ADPI Analytical Method #003
Determination of Moisture

#003a: Vacuum Oven Method
#003b: Mechanical Convection Oven Method

1.0 Purpose

This Analytical Method defines the standard operating procedures for determination of moisture content in dairy powders.

2.0 Scope

This SOP is applicable to determination of moisture for dry powder dairy products of all kinds.

3.0 Definitions

3.1 **Moisture** is the loss on drying for a sample, determined under controlled conditions. For dairy powders, this loss is entirely (or practically entirely) equivalent to the free water content of the sample. This method does not account for water of crystallization, such as in hydrated types of lactose, where such water remains bound in the crystal lattice and cannot be liberated under the drying conditions of the method.

3.2 **Solids** (or "dry solids") is the fraction of a sample which is not moisture, simply determined by subtracting the moisture result from 100%.

4.0 Principle

Systematic drying of dairy samples achieves the elimination of water from those samples by heating (and in the vacuum oven method, also by the reduction of the partial pressure of water in the drying environment by application of vacuum). Comparing the mass of the sample before drying to the mass after drying allows for determining this loss on drying, which is equivalent to the moisture content of the sample.

5.0 Reagents and Materials

While the principle of this testing method is simple, it is nevertheless dependent on careful selection of apparatus, particularly the ovens which must be capable of very consistent performance in terms of temperature accuracy and uniformity throughout the drying chamber.

In the vacuum oven method, the accuracy and consistency of the associated vacuum pump is similarly important.

Adhere to the following requirements carefully for consistent and accurate results.

The vacuum oven method (#004a) requires the following:

5.1 Drying oven, vacuum type, capable of maintaining a temperature of 100 ± 2°C throughout the drying chamber, equipped with a thermometer or equivalent for accurate indication of oven temperature, equipped with a moisture trap or equivalent on the vacuum relief vent, and with a rotameter or other flow measurement device on the pressure relief vent to meter the ingress of dry air consistent with the rate defined in the procedure below;

5.2 Vacuum pump, compatible with the drying oven, capable of maintaining a minimum vacuum of -86 kPa (about -26" of mercury absolute), preferably -96 kPa (about -28" of mercury absolute) in the oven.

The convection oven method (#004b) requires the following:

5.3 Drying oven, mechanical convection type, capable of maintaining 100-103°C, equipped with a thermometer or equivalent for accurate indication of oven temperature.

Both methods described in this standard operating procedure require all of the following:

5.4 Laboratory balance, with capacity of approximately 200 grams and with sensitivity of ± 0.0001 grams or better;

5.5 Spatula, or equivalent, suitable for weighing the samples;

5.6 Weighing vessels, approximately 5-6 cm in diameter, with close-fitting lids e.g., 5 cm aluminum weighing pans, 60 x 30 mm glass weighing bottles with ground-glass joint closures, or equivalent;

5.7 Tongs or similar utensil, suitable for handling the weighing vessels;

5.8 Dessicator, suitable for holding the weighing vessels, with color-changing calcium sulfate dessicant or equivalent.

6.0 Personal Safety Precautions

In all cases, the practitioner's company's internal policies and procedures regarding personal safety supersede the following ADPI recommendations:

6.1 Milk (dairy) is globally classified as an allergen and should be properly handled with personal safety needs in mind.

6.2 Read and understand all precautions for safe handling and disposal shown in the dessicant Safety Data Sheet (SDS), if applicable, including use of any prescribed Personal Protective Equipment (PPE).

6.3 Dairy ingredients are foods and as such are exempt from U.S. requirements regarding Safety Data Sheets (SDSs), where ingredient-specific safe handling instructions would be provided. Despite this exemption, many dairy ingredients are manufactured and marketed in powder form, and powders should be recognized as potential physical irritants, such as to the eyes, nose, and if inhaled.

6.4 Some testing apparatus described above may be susceptible to breakage, therefore be aware of associated personal risks. Inspect apparatus before use and replace any items which are compromised.

6.5 Vacuum apparatus develops stored energy in use. Read and understand the manufacturer's warnings and instructions for safe use.

6.6 Exercise care when using lab ovens and heated testing materials. Read and understand the oven manufacturer's warnings and instructions for safe use and handle any heated materials with tongs or other suitable utensil.

7.0 General Considerations

Rapid methods, such as quartz-halogen moisture balances, mid-infrared (MIR) or other methods, may be used for routine determination of moisture, such as for process control. Where possible, rapid methods should be calibrated against the vacuum oven method, especially if rapid methods are utilized for official purposes such as release testing of finished products. Daily accuracy checks against the vacuum oven method are recommended as an additional safeguard.

The vacuum oven method is the referee method for determination of moisture. In the event of a dispute or conflict where other moisture methods have been used, investigate and resolve on the basis of the vacuum oven method.

General procedural considerations:

7.1 Confirm that any dessicant media are not exhausted prior to engaging in this procedure, in all locations where used, if applicable.

7.2 It is advisable that the means for oven temperature indication (e.g., separate thermometer, electronic thermocouple, built-in temperature controller) be traceable to a recognized third-party standard (e.g., NIST), maintained according to a calibration policy/procedure, and that oven operating temperatures be documented during testing.

7.3 Avoid transferring any measurable quantities of residues onto the weighing vessels that could result from direct contact with the hands. Use utensils or equivalent suitable handling technique.

7.4 Manage the weighing vessels very carefully throughout testing, given that measurements are being made to the nearest 0.1 mg. Utilize a method for marking the weighing vessels that will not affect the validity of weight measurements obtained during testing, and ensure that covers / closures remain paired with their respective vessels whenever necessary.

7.5 Weighing vessels may be prepared in advance of testing, provided that they can be held under conditions that preserve their dry condition and that they are allowed to equilibrate to ambient temperature before use.

7.6 Samples should be homogeneous, representative, and equilibrated to ambient temperature before handling, in a manner which will not compromise their suitability for moisture testing.

7.7 Follow Good Laboratory Practices (GLPs) wherever applicable.

8.0 Method #003a: Vacuum Oven Method

8.1 Prepare weighing vessels and their closures:

a. Dry all weighing vessel components to constant weight at about 100°C (this typically takes approximately 1 hour for fiberglass vessel covers, approximately 3 hours for disposable or reusable dishes);

b. Place dried weighing vessels with their closures into a dessicator and allow them to equilibrate to ambient temperature before use.

8.2 Accurately weigh a weighing vessel and closure, recording this tare weight to the nearest 0.1 mg.

8.3 Accurately weigh 1 to 2 grams of sample powder into a weighing vessel, distributing the powder across the bottom of the vessel, and recording the sample weight to the nearest 0.1 mg.

8.4 Place the weighing vessel plus sample into the vacuum oven with the weighing vessel's closure slightly offset or completely to the side, to afford adequate exposure to the operating conditions of the oven.

8.5 Dry to constant weight at 100 ± 2°C (this typically takes approximately 5 hours), operating the vacuum oven as follows:

a. at least -86 kPa vacuum, preferably -96 kPa;

b. regulating the ingress of dry air into the oven at a consistent flow rate of about 117 mL per minute, or about 2 bubbles per second if monitoring via a glycol-type moisture trap.

8.6 Once the sample is dried to constant weight, shut off or bypass the vacuum pump and slowly equalize the oven's internal pressure by admitting a slow stream of dry air into the oven via the vacuum relief vent.

8.7 Immediately open the pressure-equalized vacuum oven and transfer the weighing vessel and its closure to a dessicator, again leaving the weighing vessel's closure slightly offset or completely to the side.

8.8 Immediately seal the dessicator and allow the materials to equilibrate to ambient temperature (typically takes approximately 30 minutes).

8.9 Once equilibrated to ambient temperature, open the dessicator and immediately place the closure onto the vessel.

8.10 Record the final weight of the vessel to the nearest 0.1 mg.

8.11 Proceed to section 10.0 for calculation of results.

9.0 Method #003b: Convection Oven Method

9.1 Prepare weighing vessels and their closures:

a. Dry all components to constant weight at about 100°C (this typically takes approximately 1 hour for fiberglass vessel covers, approximately 3 hours for disposable or reusable dishes);

b. Place dried weighing vessels with their closures into a dessicator and allow them to equilibrate to ambient temperature before use.

9.2 Accurately weigh a weighing vessel and its closure, recording this tare weight to the nearest 0.1 mg.

9.3 Accurately weigh 10 grams of sample powder into the weighing vessel, distributing the powder across the bottom of the vessel, and recording the sample weight to the nearest 0.1 mg.

9.4 Place the weighing vessel plus sample into the convection oven with the weighing vessel's closure slightly offset or completely to the side, to afford adequate exposure to the operating conditions of the oven.

9.5 Dry for 16 hours at 100-103°C.

9.6 Open the convection oven and immediately transfer the weighing vessel to a dessicator along with its closure, again leaving the weighing vessel's closure slightly offset or completely to the side.

9.7 Immediately seal the dessicator and allow the materials to equilibrate to ambient temperature (typically takes approximately 30 minutes).

9.8 Once equilibrated to ambient temperature, open the dessicator and immediately place the closure onto the vessel.

9.9 Record the final weight of the vessel to the nearest 0.1 mg.

9.10 Proceed to section 10.0 for calculation of results.

10.0 Results Calculation

$$\textbf{moisture (\%)} = \frac{\text{(initial weight of vessel \& sample, g) - (weight of dried vessel \& sample, g)}}{\text{(initial weight of vessel \& sample, g) - (tare weight of vessel, g)}} \times 100\%$$

solids (%) = 100% - moisture (%)

11.0 External References

11.1 *Standard Methods for the Examination of Dairy Products* ("SMEDP"), 17th edition, Ch. 15 – Chemical and Physical Methods, section 15.110 – Moisture and Solids Tests;

11.2 USDA Instruction No. 918-RL, *Instruction for Resident Grading Quality Control Service Programs and Laboratory Analysis*, is no longer in print. USDA documents referencing this outdated instruction also cite the *Official Methods of Analysis of the Association of Official Analytical Chemists* (AOAC) without reference to a specific method, as well as SMEDP.

12.0 ADPI Document Linkages

Analytical Method #001: *Sampling Dry Powders*

13.0 Revision History

Version	Effective Date	Notes
1.0	???	First officially approved version of this Standard Operating Procedure.
2.0	08/22/2023	Migrated this analytical method to the new modernized Standard Operating Procedure format as established by the ADPI Vice President of Technical Services.