Analytical Method #005
Determination of Scorched Particles

#005a: Water Disc Method
#005b: Sodium Citrate Method
#005c: Calgon Method
#005d: EDTA Method

1.0 Purpose

This Analytical Method defines the ADPI standard operating procedures for rating the level of scorched particles in dry milk products.

2.0 Scope

This SOP is applicable to the determination of scorched particles in spray-dried and roller-dried dry milks:

2.1 Dry whole milk;
2.2 Nonfat dry milk;
2.3 Dry buttermilk;
2.4 Dry buttermilk product.

Analogous products defined in standards outside the U.S. (e.g. whole milk powder, skimmed milk powder) may also be tested according to this procedure.

The validity of this method for testing any other products has not been established. For casein and caseinates, ISO 5739 / IDF 107 is the method currently referenced and endorsed by ADPI. The validity of either method (ADPI or ISO 5739) for testing any other products should be confirmed and documented by the user before application.

3.0 Definitions

3.1 **Scorched particles** (or simply "scorch") are aggregates of visibly distinct, colored Maillard reaction products that may arise when dairy powders containing suitable source(s) of nitrogen (e.g., proteins) and reducing sugars (in dairy, lactose is this sugar) are subjected to sufficient heat over time, such as can occur in the drying process. Scorched particles range in color from tan to brown to black, depending on the degree of overheating.

3.2 **Spray drying** (or the "spray process") is the method for transforming a liquid stream of feedstock into a powder by atomizing the liquid under very high pressure into a flow of hot, dry air. Water evaporates from the very fine droplets, leaving behind small particles of almost completely dry solids which settle in the dryer via gravity and are collected at the dryer outlet. Evaporative cooling experienced during spray drying subjects the dairy solids to less thermal stress than most other commercially practical methods of drying and typically causes less formation of scorched particles than roller drying.

3.3 **Roller drying** (or the "roller process") is the method for transforming a liquid stream of feedstock into a powder by coating a steam-heated, rotating drum with a thin film of the liquid; direct contact with the heated drum causes the water in the film to evaporate rapidly, leaving the almost completely dry solids to be removed and recovered from the drum via a scraper or knife. Direct contact with the highly heated surface subjects the dairy solids to more thermal stress than spray drying and typically causes more formation of scorched particles than spray drying.

3.4 **Foreign material** ("FM") in dairy powders may also be detected during evaluation for scorched particles and may have substantial food safety implications, as opposed to scorched particles which are strictly a quality concern. Foreign materials of many types may be found in a typical dairy process, for example:

a. "Soft" plastics e.g., from seals, conveyor paddles, cleaning or sampling utensils;
b. "Brittle" plastics or glass e.g., from inspection windows, sight glasses, sampling utensils, or from damaged external sources such as lighting, windows;
c. Metals e.g., from wear of equipment parts, from loose fasteners;
d. Oils and greases e.g., from failed seals or over-lubrication.

Some types of FM may lend themselves to ready distinction from scorched particles, having a markedly different color or other sufficiently distinguishing physical properties, while others may be more subtle. Whenever FM is confirmed or suspected, it should be immediately elevated to the appropriate level of concern with facility authorities for investigation, in accordance with established internal policies and protocols.

4.0 Principle

Scorched particles are relatively less soluble than the unscorched powder and can be readily separated from the reconstituted sample via simple filtration with a standardized test disc. The recovered scorched particles on the dried test disc are visually evaluated against a standard comparator and a letter rating is assigned to the powder based on that comparison.

The liquid used for reconstitution of the powder sample is specific to the type of product being evaluated, according to their differences in rehydration properties. Four (4) methods are provided here, identified by their respective solutions for reconstitution:

4.1 The **water disc method** utilizes water for reconstitution as the name suggests and is typically suitable for spray dried and instantized / agglomerated dry milks.

4.2 The **sodium citrate method** utilizes this particular salt for reconstitution and is typically suitable for roller dried nonfat dry milk and dry whole milk.

4.3 The **Calgon method** utilizes the complexation agent sodium hexametaphosphate for reconstitution and is typically suitable for roller dried nonfat dry milk and dry whole milk.

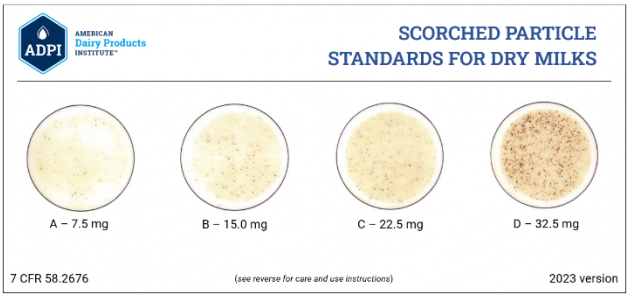
4.4 The **EDTA method** utilizes the chelating agent ethylenediamine tetraacetic acid for reconstitution and is specific to roller dried dry buttermilk and dry buttermilk product.

5.0 Reagents and Materials

While the principle of this testing method is simple, it is nevertheless dependent on careful and consistent selection of certain reagents, materials, and apparatus. Adhere to the following requirements carefully for consistent and accurate results.

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

5.1 The ADPI Scorched Particle Standards for Dry Milks photoprint comparator, or its USDA equivalent, stored and maintained appropriately and without evidence of fading or other visible deterioration; follow the "Caring for your standard" instructions on the back of the comparator (2023 version or newer) and replace as often as necessary to maintain in like-new condition;



5.2 Laboratory balance, with capacity of approximately 500 grams and with sensitivity of ± 0.1 gram or better;

5.3 Mixer, Waring blender 7-speed model 7012, or equivalent type, capable of providing mixing in the range of 18,700 – 20,400 revolutions per minute (Waring blender setting #6), with jars; free from wear or observable bend to the mixer shaft (which could cause production of metal particles that will interfere with visual interpretation of scorched particles); malted milk-type mixers and solubility index mixers do not always give equivalent results and should only be used if evaluated for suitability;

5.4 Scorched particle tester, aspirator or pressure-type filtration apparatus, with 1 1/8" filtering area diameter, compatible with the scorched particle discs or test cards identified below;

5.5 Scorched particle filtering discs, cotton, 1 ¼" diameter, loose discs or optionally pre-mounted on test cards, compatible in either case with the tester identified above (Nelson-Jameson; Visible Sediment Test Card Company; Sediment Testing Supply Company; or equivalent);

5.6 Scorched particle disc test cards, compatible with the loose discs described above (only necessary when the discs are purchased separately);

5.7 Defoaming agent, "Antifoam B" emulsion, item #A 5757 (Nelson-Jameson; Ecolab, Inc.; or equivalent);

5.8 Water, sediment-free, distilled or efficiently filtered, preferably at a temperature between 65 and 80°C;

5.9 Pipettes, disposable, or equivalent, capable of measuring approximately 0.5 mL, for use in adding the defoaming agent;

5.10 Graduated cylinders, or equivalent, with adequate capacity for the 250 mL and 300 mL measurements required as specified in the procedures below;

5.11 Lab timer, or equivalent, capable of measuring in 1 second intervals up to 60 seconds or more;

5.12 Lab oven, capable of holding a consistent temperature in the range of approximately 30-40°C and protecting the test discs from exposure to particulates during drying.

The sodium citrate, Calgon, and EDTA methods also require:

5.13 Volumetric flask or graduated cylinder, "to contain", or other equivalent labware with an accurate capacity of 1 liter, suitable for preparation of the applicable reconstitution solution chosen below;

5.14 Water bath, or equivalent, capable of holding a consistent temperature of approximately 80-90°C and accommodating the vessel containing the various heated solutions prepared below.

The sodium citrate method also requires:

5.15 Sodium citrate, reagent grade or better;

5.16 Sodium citrate, 10% solution:

a. dissolve 100 grams of sodium citrate in sediment-free water and dilute to a final volume of 1 liter;
b. filter the sodium citrate 10% solution through a cotton disc;
c. hold or pre-heat the sodium citrate 10% solution to approximately 80-90°C for use in testing.

5.17 Similarly, heat sediment-free water to approximately 80-90°C for use in testing.

The Calgon method also requires:

5.18 Calgon powder (sodium hexametaphosphate, also known as sodium polyphosphate, Calgon S, "glassy sodium"), unadjusted;

5.19 Calgon, 2% solution:

a. dissolve 20 grams of Calgon powder in sediment-free water and dilute to a final volume of 1 liter;
b. filter the Calgon 2% solution through a cotton disc;
c. hold or pre-heat the Calgon 2% solution to approximately 80°C for use in testing.

The EDTA method also requires:

5.20 Ethylenediamine tetraacetic acid (EDTA), tetrasodium salt, reagent grade or better;

5.21 EDTA, 10% solution:

a. dissolve 100 grams of EDTA in sediment-free water and dilute to a final volume of 1 liter;
b. filter the EDTA 10% solution through a cotton disc;
c. hold or pre-heat the EDTA 10% solution to approximately 80-85°C for use in testing.

5.22 Similarly, heat sediment-free water to approximately 80-85°C for use in testing.

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 various reagents' Safety Data Sheets (SDSs), 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 The blender required for this testing develops substantial rotational energy. Read and understand the manufacturer's warnings and instructions for safe use.

6.6 Exercise care when using lab ovens, water baths, and other apparatus which generate heat. Read and understand the manufacturers' warnings and instructions for safe use.

7.0 General Considerations

Scorched particles may be sticky in nature and as such may adhere to and accumulate on equipment surfaces during operation. These accumulations may then slough off such surfaces unpredictably, contributing to brief periods of markedly increased levels. Additional care is necessary to understand if a specific process is susceptible to such accumulations and subsequently to ensure that samples for scorched particles testing are taken in such a way that any such sloughing episodes are detected.

Under-mixing may result in reconstituted preparations that are difficult to filter, while over-mixing may cause destruction of scorched particles which will result in artificial improvement of the assigned scorched particle rating. Adhere to the mixing time and RPM instructions in the testing procedures below.

It is common throughout industry for scorched particles testing to be performed off-scope, i.e., for dairy powders other than the four specific dairy ingredients specified at the beginning of this document. For

caseins and caseinates, ADPI Ingredient Standards currently defer to ISO 5739 / IDF 107 for scorched particles testing specifically for this scope limitation. For lactose, dairy permeates, and other high-lactose dairy ingredients, it is known that the Maillard products arising from their drying may be friable and also somewhat more soluble than scorched particles for the in-scope ingredients; as such, they may readily break up and/or dissolve completely during the reconstitution process and may be undetected by this test method. At present it is unclear if these tendencies for high-lactose ingredients are irrelevant (e.g., that they have no true bearing on the functionality and/or use of the ingredients) or if a different method or a method variant would be warranted to support the manufacturing, trade, and end use of these products. Understand all these limitations if deliberately employing these methods off-scope, and consider establishing method validation, quality control, and commercial suitability documentation wherever practical.

The potential for formation of scorched particles is intrinsic to the processes of drying dairy powders, and while these particles are undesirable from a quality perspective, they do not constitute foreign material (FM) and also are not typically considered to be a food safety hazard in and of themselves. Care should be taken to recognize and distinguish foreign material from scorched particles and to raise all FM detection to the appropriate facility authorities for investigation, in accordance with established internal policies and protocols.

8.0 Method #005a: Water Disc Method

For spray dried or instantized / agglomerated milk powders, proceed as follows:

8.1 Measure 250 mL of sediment-free water and transfer into a Waring blender jar.

8.2 Install the jar and start the blender.

8.3 Weigh the required quantity of milk powder as shown in the following table and transfer the powder to the jar:

Milk powder type	required quantity
Nonfat dry milk Dry buttermilk Dry buttermilk product	25.0 g
Dry whole milk	32.5 g

8.4 Add approximately 0.5 mL of defoaming agent to the jar.

8.5 Mix for 60 seconds at blender setting #6 (Waring).

8.6 If the blended preparation is allowed to stand before proceeding to the filtration step below, cover the preparation appropriately to prevent contamination with other particles, and then stir vigorously before proceeding.

8.7 Transfer the blended preparation to the scorched particle tester equipped with the standard cotton disc or pre-mounted test disc card and filter at a rate which does not rupture the disc.

8.8 Rinse the blender jar with approximately 50 mL of sediment-free water, transfer this rinse water to the scorched particle tester and filter through the disc.

8.9 Remove the cotton disc (installing into a blank disc card) or the pre-mounted test disc card from the tester and dry completely at approximately 30-40°C.

8.10 Proceed to section 12.0 for interpretation of results.

9.0 Method #005b: Sodium Citrate Method

For roller dried nonfat dry milk or dry whole milk powders, proceed as follows:

9.1 Measure 200 mL of heated sodium citrate 10% solution and transfer into a Waring blender jar.

9.2 Install the jar and start the blender.

9.3 Weigh the required quantity of milk powder as shown in the following table and transfer the powder to the jar.

Milk powder type	required quantity
Nonfat dry milk	17.0 g
Dry whole milk	22.0 g

9.4 Add approximately 0.5 mL of defoaming agent to the jar.

9.5 Mix for 30 seconds at blender setting #6 (Waring).

9.6 If the blended preparation is allowed to stand before proceeding to the filtration step below, cover the preparation appropriately to prevent contamination with other particles, and then stir vigorously before proceeding.

9.7 Transfer the blended preparation to the scorched particle tester equipped with the standard cotton disc or pre-mounted test disc card and filter at a rate which does not rupture the disc.

9.8 Rinse the blender jar with sediment-free heated water (from 5.17), transfer this rinse water to the scorched particle tester and filter through the disc.

9.9 Remove the cotton disc or the pre-mounted test disc card from the tester and dry completely at approximately 30-40°C.

9.10 Proceed to section 12.0 for interpretation of results.

10.0 Method #005c: Calgon Method

For roller dried nonfat dry milk or dry whole milk powders, proceed as follows:

10.1 Measure 250 mL of heated Calgon 2% solution and transfer into a Waring blender jar.

10.2 Install the jar and start the blender.

10.3 Weigh the required quantity of milk powder as shown in the following table and transfer the powder to the jar.

Milk powder type	required quantity
Nonfat dry milk	17.0 g
Dry whole milk	22.0 g

10.4 Add approximately 0.5 mL of defoaming agent to the jar.

10.5 Mix for 30 seconds at blender setting #6 (Waring).

10.6 Do not allow the blended preparation to stand: transfer immediately to the scorched particle tester equipped with the standard cotton test disc or pre-mounted test disc card and filter at a rate which does not rupture the disc.

10.7 Rinse the blender jar with approximately 25 mL of sediment-free water, transfer this rinse water to the scorched particle tester and filter through the disc.

10.8 Remove the cotton disc or the pre-mounted test disc card from the tester and dry completely at approximately 30-40°C.

10.9 Proceed to section 12.0 for interpretation of results.

11.0 Method #005d: EDTA Method

For roller dried dry buttermilk or dry buttermilk product powders, proceed as follows:

11.1 Measure 300 mL of heated EDTA 10% solution and transfer into a Waring blender jar.

11.2 Install the jar and start the blender.

11.3 Weigh 17.0 grams of dry buttermilk or dry buttermilk product and transfer the powder to the jar.

11.4 Add a few drops of defoaming agent to the jar.

11.5 Mix for 8-10 seconds.

11.6 Add additional volume of heated EDTA 10% solution to the jar until the total volume is approximately 500 mL.

11.7 Mix for an additional 45 seconds at blender setting #6 (Waring).

11.8 Do not allow the blended preparation to stand: transfer immediately to the scorched particle tester equipped with the standard cotton test disc or pre-mounted test disc card and filter at a rate which does not rupture the disc.

11.9 Rinse the blender jar with sediment-free heated water (from 5.22), transfer this rinse water to the scorched particle tester and filter through the disc.

11.10 Remove the cotton disc or the pre-mounted test disc card from the tester and dry completely at approximately 30-40°C.

11.11 Proceed to section 11.0 for interpretation of results.

12.0 Interpretation of Results

Results from all four methods described above are interpreted in the same manner. Samples are assigned an A, B, C, or D letter rating, each of which also corresponds to an equivalent mass quantity of scorched particles in 100 grams of powder as shown in the table below.

Dairy industry vernacular for these letter ratings is often “A pad”, “B pad” and so on.

12.1 Place the dry test disc on a flat, horizontal surface alongside the Standard photoprint and view directly from above under uniform, indirect light.

12.2 Assign the powder sample with the letter rating corresponding to the closest comparison on the Standard, where any disc falling between two Standard letters is assigned the higher disc’s letter. For example, a sample showing more scorched particles than Standard A but less than Standard B shall be assigned a B rating, and so on.

12.3 The visually determined, qualitative letter ratings assigned by this method also correspond to the following maximum quantities of scorched particles.

Users may elect to express their comparative results via these quantitative limits, instead of or in addition to using the letter ratings.

Rating	equivalent weight of scorched particles per 100 g of sample
A	7.5 mg maximum
B	15.0 mg maximum
C	22.5 mg maximum
D	32.5 mg maximum

13.0 External References

13.1 See USDA AMS publication *United States Scorched Particle Standards for Dry Milk* (reprinted December 2018) for details about the method of preparation of reference discs at standardized levels of scorched particles, which is the primary source of instruction from which both the ADPI and USDA comparators were created.

13.2 See USDA AMS publication *DA Instructions 918-RL Laboratory Methods and Procedures* (revised January 2001) for details about scorched particles testing, most especially the blender type and settings which are viewed to be integral to achieving accurate and repeatable results.

13.3 Note that the above information in the specified USDA AMS publication formerly resided within Title 21 Code of Federal Regulations, beginning at Section 58.2676. The USDA AMS publication retains these CFR section references as a way to provide continuity with past codification, and this reference (7 CFR 58.2676) is cited on the ADPI Standard comparator for the same reason.

13.4 See also ISO 5739 / IDF 107 *Caseins and caseinates – Determination of contents of scorched particles and of extraneous matter* for a validated procedure suitable for these products.

14.0 ADPI Document Linkages

Analytical Method #001: *Sampling Dry Powders.*

15.0 Revision History

Version	Effective Date	Notes
1.0	indeterminate	First officially approved version of this Standard Operating Procedure.
2.0	08/16/2023	Migrated this analytical method to the new modernized Standard Operating Procedure format as established by the ADPI Vice President of Technical Services.
2.1	10/30/2023	Updated apparatus and test method procedural details to incorporate the Waring blender setting recommended in USDA 918-RL. Incorporated an image of the current version of the *ADPI Scorched Particle Standards for Dry Milks* comparator. Added information in the General Considerations section regarding off-scope application of this method. Incorporated reference into USDA 918-RL into External References.
2.2	01/21/2025	Corrected multiple temperature units typographical errors where Fahrenheit was incorrectly cited, rather than the correct Celsius (centigrade) units.