



Analytical Method #009

Determination of Solubility Index

1.0 Purpose

This Analytical Method defines the ADPI standard operating procedures for determining the solubility index of dry dairy products.

2.0 Scope

This SOP is applicable to the determination of solubility index of:

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|-----|--------------------------------|------|------------------------------|
| 2.1 | Nonfat dry milk; | 2.7 | Whey protein isolates; |
| 2.2 | Skimmed milk powder; | 2.8 | Whole milk powder; |
| 2.3 | Dry buttermilk; | 2.9 | Milk protein concentrates; |
| 2.4 | Dry buttermilk product; | 2.10 | Milk protein isolates; |
| 2.5 | Whey powders (sweet and acid); | 2.11 | Micellar casein concentrate. |
| 2.6 | Whey protein concentrates; | | |

3.0 Definitions

- 3.1 **Solubility index** (sometimes identified as **insolubility index**) is the volume of undissolved powder “sediment” which remains after the vigorous systematic rehydration of a dairy powder. Clear visualization of this remaining sediment layer depends in part on the centrifugation of the test preparation at an RPM setting which is appropriate to the dimensions of the centrifuge rotor.
- 3.2 **Head diameter** of a centrifuge is the distance between the inside bottom of opposite cups on the centrifuge rotor, measured through the center of rotation and with the cups in their horizontally extended positions (simulating their positions during operation).

4.0 Principle

Dairy powder is rehydrated under controlled conditions of time, temperature, and mixing. The rehydration conditions prescribed in this analytical method are vigorous, ensuring that “reasonable” conditions of end use are appropriately simulated.

Once rehydrated, what remains as “sediment” is the volume that does not readily disperse and dissolve under these reasonable test conditions. This could be the result of high levels of protein denaturation,

for example, potentially arising from excessive heat exposure or other extreme handling conditions during manufacture. The lower the amount of sediment under the prescribed test conditions, the more readily soluble the powder is; and conversely, higher levels of sediment correspond to a less readily soluble powder.

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 apparatus, especially the mixing and centrifugation equipment. Adhere to the following requirements carefully for consistent and accurate results.

- 5.1 Laboratory balance, with capacity of approximately 500 grams and with sensitivity of ± 0.1 gram or better;
- 5.2 Graduated cylinders, or equivalent, with adequate capacity for the 200 mL and 25 mL measurements required as specified in the procedure below;
- 5.3 Water bath, or equivalent, capable of holding a consistent temperature of approximately 75°F or 140°F, for heating the sediment-free water as described below;
- 5.4 Water, sediment-free, distilled or efficiently filtered, at a temperature of 75°F or 140°F as dictated by the type of powder being tested (see the table in section 8 below);
- 5.5 Pipettes, disposable, or equivalent, suitable for delivering the defoaming agent dropwise;
- 5.6 Defoaming agent, "Antifoam B" emulsion, item #A 5757 (Nelson-Jameson; Ecolab, Inc.; or equivalent);
- 5.7 Mixer, Waring blender, 7-speed model, or equivalent type, with jars (Waring model 31 BL42, catalog #7012G, or equivalent);
- 5.8 Lab timer, or equivalent, capable of measuring up to 15 minutes or more;
- 5.9 Spoon, spatula, or equivalent, suitable for mixing the sample preparation;
- 5.10 Centrifuge, capable of meeting the RPM requirements corresponding to the head diameter of its rotor, as shown in the table below; and compatible with the centrifuge tubes defined in section 5.11;

Head diameter	RPM requirement	Head diameter	RPM requirement
10 inches	1075	18 inches	800
12 inches	980	20 inches	759
14 inches	909	22 inches	724
16 inches	848	24 inches	695

(If verification of RPM accuracy is required, conduct with test samples loaded into the rotor and with the centrifuge lid closed. Consult the manufacturer's manual for other instructions.)

5.11 Centrifuge tubes, conical, graduated as follows:

from	0.0 – 1.0 mL	in	divisions of 0.1 mL
from	1.0 – 2.0 mL	in	divisions of 0.2 mL
from	2.0 – 10.0 mL	in	divisions of 0.5 mL
from	10.0 – 20.0 mL	in	divisions of 1.0 mL
and with a 50.0 mL mark at least ½" from the top of the tube			

5.12 Siphon tube, or equivalent, suitable for drawing off the supernatant layer from the sediment layer after centrifugation;

5.13 Wire, glass rod, or equivalent, suitable for dislodging sticky traces of sediment from the walls of the centrifuge tubes when required.

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 The centrifuge required for this testing also develops substantial rotational energy. Read and understand the manufacturer's warnings and instructions for safe use.
- 6.7 Exercise care when using hot water baths. Read and understand the manufacturers' warnings and instructions for safe use.

7.0 General Considerations

Because visual interpretation is the means by which the solubility index determination is ultimately made, good results depend on adequate lighting.

Avoid common mistakes such as parallax when making the volume determination for the sediment layer. Use good technique for volumetric glassware; hold up the centrifuge tube with the top of the sediment layer at eye level.

8.0 Determination of solubility index

- 8.1 Weigh the required quantity of milk powder as shown in the following table and transfer the powder into a Waring blender jar.
- 8.2 Add 200 mL of water to the jar, following the water temperature requirement shown in the table.
- 8.3 Add 2-3 drops of defoaming agent to the jar.
- 8.4 Blend according to the minimum time shown in the table, using the #1 blender setting (approximately 3,000-3,500 RPM).

powder type	powder quantity (g)	water temperature (°F)	minimum blending time (minutes)
nonfat dry milk; skimmed milk powder	20	75	1 ½
dry buttermilk; dry buttermilk product	20	75	1 ½
whey powders, WPCs, WPI	20	75	1 ½
whole milk powder	26	75	1 ½
MPCs up to and including MPC70	15	140	2
MPC greater than MPC70; MPI; micellar casein concentrate	8	140	10

- 8.5 Allow the blended preparation to stand for a period of at least 5 minutes but not more than 15 minutes.
- 8.6 After standing, stir the preparation thoroughly with a spoon or similar, and immediately fill to the 50.0 mL mark of a centrifuge tube.
- 8.7 Centrifuge for 5 minutes at the RPM setting that is appropriate for the centrifuge rotor.
- 8.8 Immediately siphon off the supernatant layer to within 5 mL of the upper surface of the sediment layer, taking care not to disturb the sediment layer in the process.
- 8.9 Add 25 mL of water at a temperature of 75°F to the tube.
- 8.10 Shake the tube gently to disperse the sediment, dislodging any sticking / pelleted material with a wire or glass rod if necessary. Do not withdraw any sediment from the tube in the process.
- 8.11 Fill the tube to the 50.0 mL mark with water at a temperature of 75°F.
- 8.12 Invert the tube several times to mix the contents thoroughly.
- 8.13 Centrifuge again for 5 minutes at the appropriate RPM setting.

9.0 Interpretation of Results

- 9.1 Hold the centrifuge tube vertically, between the eye and a strong source of illumination.
- 9.2 Read the volume of sediment corresponding to the upper level of the sediment layer, to the nearest graduation on the tube.
- 9.3 Record the result in units of mL.

10.0 External References

No external references are cited.

11.0 ADPI Document Linkages

Analytical Method #001: *Sampling Dry Powders*.

12.0 Revision History

Version	Effective Date	Notes
1.0	indeterminate	First officially approved version of this Standard Operating Procedure.
2.0	10/30/2023	Migrated this analytical method to the new modernized Standard Operating Procedure format as established by the ADPI Vice President of Technical Services.