

Powder Moisture Accurate Standard Method

GEA Niro analytical method A 1 a

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1. Definition

The moisture content of a powder is the loss in weight (%) after oven drying at 102°C until constant weight is obtained.

2. Scope

This is an accurate standard method which may be used for milk powder and all other powdered dairy products which do not contain crystallized lactose (α -lactose-monohydrate).

3. Principle

The sample is dried by oven drying to constant weight at 102°C \pm 2°C for 2 hours. The oven drying is repeated until the two successive weighings do not differ more than 0.5 mg.

4. Apparatus

4.1 Drying oven, with thermostat and without forced air circulation.

4.2 Analytical balance, sensibility \pm 0.1 mg.

4.3 Desiccator with colour-indicating desiccant (e.g. silica gel).

4.4 Weighing dishes with lid.

5. Reagents

None

6. Procedure

6.1 Dry weighing dish with open lid in the oven, and cool it in desiccator.

6.2 Weigh the empty dish (a), add approx. 3 g of powder and weigh again (b)

6.3 Place the loaded dish with open lid in the oven at 102°C \pm 2°C for 2 hours.

6.4 Cool closed dish to room temperature in desiccator, and weigh (c).

6.5 Continue drying the loaded dish with open lid in the oven at 102°C \pm 2°C for 1 hour.

6.6 Repeat the cooling 6.4 and weigh again (c).

6.7 Repeat 6.5 until weight (c) is constant (i.e. until two successive weighings differ less than 0.5 mg)

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7. Calculation

$$\text{Moisture} = \frac{b-c}{b-a} \times 100\%$$

a = weight of empty dish
b = weight of dish + powder
c = weight of dish + dried powder

8. Reproducibility

± 0.1 %

9. Remarks

A sample for moisture determination has to be handled carefully in order to avoid evaporation or prevent adsorption.

10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard Nº 26:2004 / ISO Standard Nº 5537:2004](#)
- De Knecht, R.J. and Brink, H.v.d.: Improvement of the drying oven method for the Determination of the Moisture Content of Milk Powder. *Int. Dairy Journal*, 8, 1998, pp. 733-738.

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Powder Moisture Routine Method GEA Niro analytical method A 1 b

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1. Definition

The moisture content of a powder is the loss in weight (%) after oven drying at 102°C for 3 hours.

2. Scope

This is a routine method which may be used for milk powder and all other powdered dairy products which do not contain crystallized lactose (α -lactose-monohydrate).

3. Principle

Sample is dried by oven drying at 102°C \pm 2°C for 3 hours.

4. Apparatus

4.1 Drying oven, with thermostat and without forced air circulation.

4.2 Analytical balance, sensibility \pm 0.1 mg.

4.3 Desiccator with colour-indicating desiccant (e.g. silica gel).

4.4 Weighing dishes with lid.

5. Reagents

None.

6. Procedure

6.1 Dry weighing dish with open lid in the oven, and cool it in desiccator.

6.2 Weigh the empty dish (a), add approx. 3 g of powder and weigh again (b).

6.3 Place the loaded dish with open lid in the oven at 102°C \pm 2°C for 3 hours.

6.4 Cool closed dish to room temperature in desiccator, and weigh (c).

7. Calculation

$$\text{Moisture} = \frac{b-c}{b-a} \times 100\%$$

a = weight of empty dish

b = weight of dish + powder

c = weight of dish + dried powder

8. Reproducibility

\pm 0.1 %

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Powder Moisture Routine Method
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9. Remarks

A sample for moisture determination has to be handled carefully in order to avoid evaporation or prevent adsorption.

Moisture content of a powder sample depends on the drying time, our experience has shown that 3 hours is sufficient.

10. Literature

- [Niro Research Laboratory](#)
- [IDF Standard № 26:2004 / ISO Standard № 5537:2004](#)
- De Knecht, R.J. and Brink, H.v.d.: Improvement of the drying oven method for the Determination of the Moisture Content of Milk Powder. *Int. Dairy Journal*, 8, 1998, pp. 733-738.

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Free Moisture

GEA Niro analytical method A 1 c

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1. Definition

The free moisture content of a powder is the loss in weight (%) after oven drying at 87°C for 6 hours.

2. Scope

This is a routine method which may be used for any kind of dried milk product containing crystallized lactose (α -lactose-monohydrate), e.g. whey powder.

3. Principle

Sample is dried by oven drying at 87°C \pm 2°C for 6 hours.

4. Apparatus

4.1 Drying oven, with thermostat and without forced air circulation.

4.2 Analytical balance, sensibility 0.1 mg.

4.3 Desiccator with colour-indicating desiccant (e.g. silica gel).

4.4 Weighing dishes with lid.

5. Reagents

None.

6. Procedure

6.1 Dry weighing dish with open lid in the oven, and cool it in desiccator.

6.2 Weigh the empty dish (a), add approx. 3 g of powder and weigh again (b).

6.3 Place the loaded dish with open lid in the oven at 87°C \pm 2°C for 6 hours.

6.4 Cool closed dish to room temperature in desiccator, and weigh (c).

7. Calculation

$$\text{Moisture} = \frac{b - c}{b - a} \times 100\%$$

a = weight of empty dish

b = weight of dish + powder

c = weight of dish + dried powder

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Free Moisture
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8. Reproducibility

± 0.1 %

9. Remarks

A sample for moisture determination has to be handled carefully in order to avoid evaporation or prevent adsorption.

10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard N° 26:2004 / ISO Standard N° 5537:2004](#)

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Total Moisture (KF Titration) GEA Niro analytical method A 1 d

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1. Definition

The total moisture of a powder consists of free moisture content as well as crystal bounded water.

2. Scope

This method may be used for any kind of dried milk products, especially those containing crystallized lactose (α -lactose-monohydrate), e.g. whey powder.

3. Principle

The determination of total moisture by Karl Fisher titration is a calculation based on the concentration of iodine in the KF titrating reagent (i.e. titer) and the amount of KF reagent consumed in the titration. The end-point of the titration is determined by the dead-stop end-point method.

4. Apparatus

- 4.1 Karl Fisher titrator.
- 4.2 Analytical balance, sensibility 0.1 mg.
- 4.3 Closed glass weighing spoon.

5. Reagents

- 5.1 Karl Fisher reagent.
- 5.2 Methanol, moisture free
- 5.3 Sodium sulphate (Na_2SO_4), moisture free
- 5.4 Sodium tartrate dihydrate ($\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2 \text{H}_2\text{O}$)

6. Procedure

Standardization:

- 6.1 New bottles containing 'Composite 5' or 'Titrant 5' must be standardized against sodium tartrate dihydrate. 230.10 g sodium tartrate dihydrate corresponds to 36.4 g H_2O .
- 6.2 Use procedure 6.6 to 6.13. using approx. 0.1 g sodium tartrate dihydrate as sample.
- 6.3 Fresh solvent is used between each standardization.
- 6.4 The standardization is accepted when two determinations agree within 0.5% relative.

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Total Moisture (KF Titration) GEA Niro analytical method A 1 d

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6.5 The factor F (mg H₂O/ml KF reagent¹) is calculated as:

$$\frac{a \times 36.04 \times 1000}{ml \times 230.10} = \frac{a \times 156.8}{ml}$$

a = g sodium tartrate dihydrate

ml = ml KF reagent.

- 6.6 Choose titrant and solvent based on the standardization 6. Check standardization each day by doing step 6.6 to 6.13, using two drops of water as sample. Results must be between 99.0 and 101.0 % water. If that is not obtained, re-standardize the titrant using the procedure in 6.1 to 6.4.
- 6.7 Add fresh solvent into the titration vessel.
- 6.8 The solvent is titrated till dryness (drift < 20 µml/min. is used as stop criteria).
- 6.9 The sample is transferred to a closed glass weighing spoon. The amount of sample depends on the water content; an expected amount of 10-50 mg water is suitable.
- 6.10 The weighing spoon with a sample is placed on the balance. Zero the balance.
- 6.11 Dose the sample into the titration vessel. Keep the time the titration vessel is open as short as possible.
- 6.12 The weighing spoon with remaining powder is placed on the balance. Read the sample weight (w).
- 6.13 Execute the titration. When the end-point is reached (drift < 20 µml/min.) the amount of titrant is read (ml). If the amount of KF reagent added is less than 0.5 ml, increase the amount of sample to be analysed.
- 6.14 All measurements are to be made in duplicate.

7. Result

$$\% H_2O = \frac{b \times F \times 100}{1000 \times W}$$

b = ml KF reagent used for sample

F = factor mg H₂O/ml KF reagent

w = weight in g

8. Reproducibility

Two determinations must not differ more than 1% relative.

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Total Moisture (KF Titration)
GEA Niro analytical method A 1 d

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9. Remarks

Choosing the working media:

- a) Methanol is the preferred choice.
- b) Mixtures of methanol and chloroform are suitable for products containing fat. Methanol content should not be less than 25%.
- c) Mixtures of methanol and formamide improve the solubility of polar substances. The methanol content should not be less than 50%.
- d) KF titration has an optimum pH range of 5-7. At higher pH a side reaction occurs, which consumes iodine slowly. In a strongly acid solution, the reaction decreases proportionally to the pH value. Strong acid or bases have to be neutralized before titration.

If the drift in ml/min is not stable or the titration is very slow (more than 2-4 min.) it is an indication of troubles with side reactions or very slow liberation of the water.

Specify reagent, solvent and any special treatment together with the results.

10. Literature

[GEA Niro Research Laboratory.](#)

Hydranal-Praktikum, Eugen Scholtz. Riedel de Haen, Seelze, August 1987.

Radiometer Analytical.

Water of Crystallization GEA Niro analytical method A 1 e

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1. Definition

The water of crystallization (%) of a powder is the difference between total moisture and free moisture.

2. Scope

This method may be used for any kind of dried milk products containing crystallized lactose (α -lactose-monohydrate), e.g. whey powder.

3. Principle

Sample total moisture (determined by Karl Fisher titration) and free moisture (determined by oven drying 87°C/6h) is measured. The water of crystallization is calculated.

4. Apparatus

4.1 As given in GEA Niro Method N^o A 1 c.

4.2 As given in GEA Niro Method N^o A 1 d.

5. Reagents

As specified in GEA Niro Method N^o A 1 d.

6. Procedure

6.1 Determine the free moisture content as described in GEA Niro Method N^o A 1 c.

6.2 Determine the total moisture content as described in GEA Niro Method N^o A 1 d.

7. Calculation

Water of crystallization = % total moisture - % free moisture

8. Reproducibility

± 0.2%

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Water of Crystallization
GEA Niro analytical method A 1 e

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9. Remarks

The water of crystallization of whey powders provides a good indication of the 'Degree of Crystallization' of the lactose content.

Degree of Crystallization

$$= \frac{\% \text{ water of crystallization} \times 19}{\%L} \times 100$$

%L = the content of lactose in whey powder (%), expressed as anhydride. For rapid routine test of sweet whey, the %L \approx 74%.

10. Literature

[GEA Niro Research Laboratory](#)

Powder Bulk Density

GEA Niro analytical method A 2 a

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1. Definition

The bulk density of a powder is the weight of the powder divided by the volume it occupies, normally expressed as g/ml or kg/l.

2. Scope

The method is to be used for milk powders and all other dried milk products.

3. Principle

Samples are filled into a stainless steel cylinder, weighed and tapped in a Stampf-volumeter. The results of bulk density must be identified as loose, tapped 100 times or tapped 1250 times.

4. Apparatus

4.1 Balance - sensitivity 0.1 mg.

4.2 Stainless steel cylinder with detachable top, as shown in Fig. 1. The volume of the lower cylinder is exactly 100 cm³.

4.3 Stampf-volumeter, e.g. made by [Engelsmann](#), Germany (Fig.2).

4.4 Brush

5. Reagents

None.

6. Procedure

6.1 Weigh the cylinder without the top cylindrical part.

6.2 Put the top on the cylinder and carefully fill up to the rim with powder using a spoon. Avoid shaking or tapping the cylinder.

6.3 Remove the top and scrape off powder until it is flush with the rim of the cylinder. Care should be taken not to compress or vibrate the cylinder. Brush off excess powder from the outside edge of the cylinder.

6.4 Weigh the full cylinder (w_1). The weight of the powder indicates "*loose/poured bulk density*" ($0x$).

6.5 Repeat point 6.2 and tap the cylinder 100 times in the Stampf-volumeter. If necessary fill up with more powder.

6.6 Repeat point 6.3 and weigh (w_2). The weight of the powder indicates "*tapped powder bulk density*" ($100x$).

6.7 Repeat point 6.2 and tap further 1150 times in the Stampf-volumeter.

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Powder Bulk Density

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6.8 Repeat point 6.3 and weigh (w_3). The weight of the powder indicates "tapped to the extreme powder bulk density" (1250x).

7. Calculation

The results are expressed as:

- Loose/poured bulk density - tapped 0 times.
- Tapped bulk density - tapped 100 times.
- Tapped to the extreme bulk density - tapped 1250 times.

$$\text{Bulk density} = \frac{w_x}{100}$$

w_x = weight of powder in g

100 = volume of cylinder in cm³

Calculate the result to 2 decimal places.

8. Reproducibility

± 0.03 g/ml for loose bulk density.

± 0.01 g/ml for tapped 100 and 1250 times.

Unless other is stated, bulk density is made as single determination.

9. Remarks

1. Bulk density depends on water content and particle size. Avoid adsorption or desorption of water before determination.
3. To obtain reliable results, make sure the powder is at room temperature when analysing.

10. Further literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard 134A:1995](#) - Dried milk and dried milk products - Determination of bulk density.
- Svarovsky L., Powder Testing Guide: Methods of measuring the physical properties of bulk powders. ISBN 1851661379, Elsevier Science (1987).

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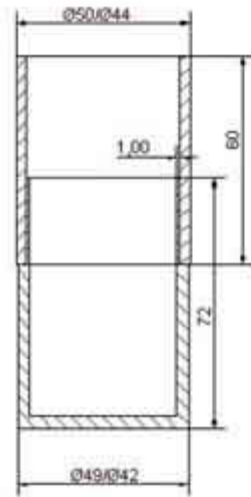


Fig. 1 Stainless steel cylinder



Fig. 2 Stampf-volumeter

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Powder Bulk Volume GEA Niro analytical method A 2 b

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1. Definition

The bulk volume of a powder is the volume of the powder divided by the weight, normally expressed in ml/100g powder.

2. Scope

The method is to be used for milk powders and all other dried milk products.

3. Principle

100 g sample is filled into a glass cylinder and tapped in a Stampf-volumeter. The results of bulk density must be identified as loose, tapped 100 times or tapped 1250 times.

4. Apparatus

4.1 Balance - sensitivity 0.1 mg.

4.2 200 ml measuring glass cylinder.

4.3 Stampf-volumeter, e.g. made by [Engelsmann](#), Germany (Fig. 1).

4.4 Brush

5. Reagents

None.

6. Procedure

6.1 Weigh out exactly 100 g of powder, and transfer it to the measuring cylinder. Avoid shaking or tapping the cylinder.

6.2 Level off the surface of the powder with the spatula.

6.3 Record the volume (v_1). The volume of the powder indicates "*loose/poured bulk volume*".

6.4 Tap the cylinder 100 times in the Stampf-volumeter.

6.5 Record the volume (v_2). The volume of the powder indicates "*tapped powder bulk volume*".

6.7 Continue tapping the sample further 1150 times in the Stampf-volumeter.

6.8 Record the volume (v_3). The volume of the powder indicates "*tapped to the extreme powder bulk volume*".

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Powder Bulk Volume

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7. Result

The results are expressed as:

- Loose/poured bulk volume - tapped 0 times.
- Tapped bulk volume - tapped 100 times.
- Tapped to the extreme bulk volume - tapped 1250 times.

$$\text{Bulk Volume (BV)} = v_x$$

8. Reproducibility

- ± 5 ml/100 g for loose bulk volume.
- ± 2 ml/100 g for tapped 100 and 1250 times.

Unless other is stated, bulk density is made as single determination.

9. Remarks

- 9.1 Bulk density depends on water content and particle size. Avoid adsorption or desorption of water before determination.
- 9.2 To obtain reliable results, make sure the powder has room temperature when analysing.
- 9.3 Powder bulk volume can easily be converted into powder bulk density by use of the formula:

$$\text{Bulk density (BD)} = \frac{100}{\text{BV}} \text{ [g/ml]}$$

BV = bulk volume of 100 g powder in ml/100g

100 = weight of powder sample in g

Calculate the result to 2 decimal places.

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Powder Bulk Volume
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10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard 134A:1995](#) - Dried milk and dried milk products - Determination of bulk density.
- Svarovsky L., Powder Testing Guide: Methods of measuring the physical properties of bulk powders. ISBN 1851661379, Elsevier Science (1987).



Fig. 1 Stampf-volumeter

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Scorched Particles

GEA Niro analytical method A 4 a

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1. Definition

The amount of scorched particles in a powder is determined by comparison with the ADMI chart: 'Scorched Particle Standards for Dry Milk'.

2. Scope

This method is used for milk powder and all other dried dairy products.

3. Principle

4. Apparatus

- 4.1 Original ADMI chart 'Scorched Particle Standard for Dry Milk' (see Fig. 1).
- 4.2 Balance, sensitivity 0.1 g.
- 4.3 Cenco Mixer, Cenco Instrumenten B.V, Breda, The Netherlands.
- 4.4 Standard filter pads - diameter 32 mm (1¼"), Lintine brand, milk-cream sediment testers, Johnson & Johnson.
- 4.5 Scorched particles tester - aspirator or pressure type, filtering diameter 28.5 mm (1⅛"), Nagashima S.S. Ltd.
- 4.6 Water jet pump.

5. Reagents

Defoaming agent – Octylalcohol or diglycol laurate S.

6. Procedure

- 6.1 Weigh out the correct amount of powder ± 0.1 g:

Non-fat milk:	25.0 g
Whey:	15.0 g
Whole milk:	32.5 g
- 6.2 Pour the powder into the blender glass with 250 ml of 18-27°C water.
- 6.3 Add 2 or 3 drops of defoaming agent.
- 6.4 Mix for 60 seconds in the Cenco Mixer.
- 6.5 Using a vacuum to filter the solution immediately through the filter in the tester. Rinse the blender glass with about 50 ml water and filter it through the same filter.
- 6.6 Let the filters dry for 2 hours at approx. 35°C.
- 6.7 Measurements are carried out as single determinations.

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Scorched Particles**GEA Niro analytical method A 4 a**(Page 2 of 3)

7. Results

Compare the results with the original ADMI standard chart. The comparison is visual. The standard chart is divided into a scale from A-D, where

A = 7.5 mg

B = 15.0 mg

C = 22.5 mg

D = 32.5 mg

If a sample is classified as being between two standards it is always set at the highest value. A sketch of the ADMI chart is shown on Fig. 1.

8. Reproducibility

Single determination.

9. Remarks

9.1 The filtration (E4) can also be carried out with compressed air.

9.2. The procedure for caseinate and casein:

9.2.1 Weigh out 25 g powder \pm 0.1 g.

9.2.2 Pour the powder into 200 ml sodium carbonate 9.09% V/W.

9.2.3 Add 300 ml of 18-27°C water.

9.2.4 Add 2 or 3 drops of defoaming agent.

9.2.5 Mix for 60 seconds in the Cenco Mixer.

9.2.6 Use a vacuum to filter the solution immediately through the filter in the tester. Rinse the blender glass with about 50 ml water and filter it through the same filter.

9.2.7 Let the filter dry for 2 hours at approx. 35°C.

9.2.8 Measurements are carried out as single determinations.

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Scorched Particles

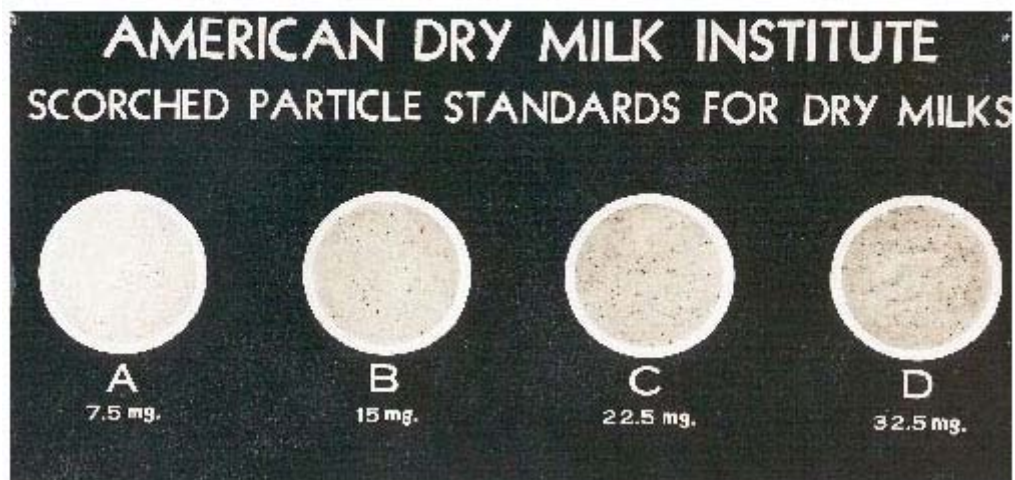
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10. Reference

- [GEA Niro Research Laboratory](#)
- [ADPI](#), Bulletin 916. (previously called ADMI, American Dry Milk Institute)
- Modification of the Harland-Ashworth method, published by Kuramoto, Jeness, Coulter and Choi. Journal of Dairy Science 42:28, 1959.

Fig.1 ADMI chart 'Scorched Particles Standards for Dry Milk'



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Wettability**GEA Niro analytical method A 5 a**(Page 1 of 3)

1. Definition

The wettability of a powder is the time necessary to achieve complete wetting of a specified amount of powder, when it is dropped into water at a specified temperature.

2. Scope

This method is used for milk powder and all other dried dairy products.

3. Principle**4. Apparatus**

- 4.1 Balance - sensitivity 0.1 g.
- 4.2 Beaker - 400 ml, diameter 70 mm, height 135 mm.
- 4.3 Funnel of anti static material - height 100 mm, lower diameter 40 mm, upper diameter 90 mm (see 9.3 and Fig. 1).
- 4.4 Porcelain pestle - length approx. 130 mm.
- 4.5 Stop watch.

5. Reagents

None.

6. Procedure

6.1 Weigh out the correct amount of powder ± 0.1 g:

Baby food:	13 g
Skim milk:	10 g
Whole milk:	13 g
Whey:	6 g

6.2 Choose water temperature depending on the powder type:

20°C $\pm 0.2^\circ$ C:	skim milk, instant whole milk and whey.
40°C $\pm 0.5^\circ$ C:	whole milk and baby food.

6.3 Pour 100 ml of deionised water with the correct temperature into the beaker. Check the temperature, and place the funnel so it rest on the upper edge of the beaker. Place the pestle inside the funnel so it blocks the lower opening.

6.4 Place the powder around the pestle.

6.5 Lift the pestle and start the stop watch.

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6.6 Stop the watch when all powder has been wetted.

6.7 Measurements are always made in duplicate.

7. Result

Wettability = the time in seconds necessary to achieve complete wetting.

The amount of powder and the water temperature have to be stated with the results. If the powder is not wetted after 5 minutes the analysis is stopped, and the result is given as >300 sec.

8. Reproducibility

Two determinations must not differ more than 20% relative.

9. Remarks

9.1 The water temperature of 20°C is the standard temperature used by GEA Niro for testing of instant properties.

9.2 For other products, the amount of sample for analysis should correspond to the powder-in-water concentration at which the given product is intended to be used.

9.3 The funnel is made by making a full-scale drawing of the figure shown in Fig. 1 and using it as a template. The funnel must be made of anti-static material, e.g anti-static plastic and stapled, glued or welded together. The inner surface of the funnel must be smooth to allow the powder to fall quickly down onto the water

10. Literature

- [GEA Niro Research Laboratory](#)
- Mohr's Standprobe, Milchwissenschaft 1960

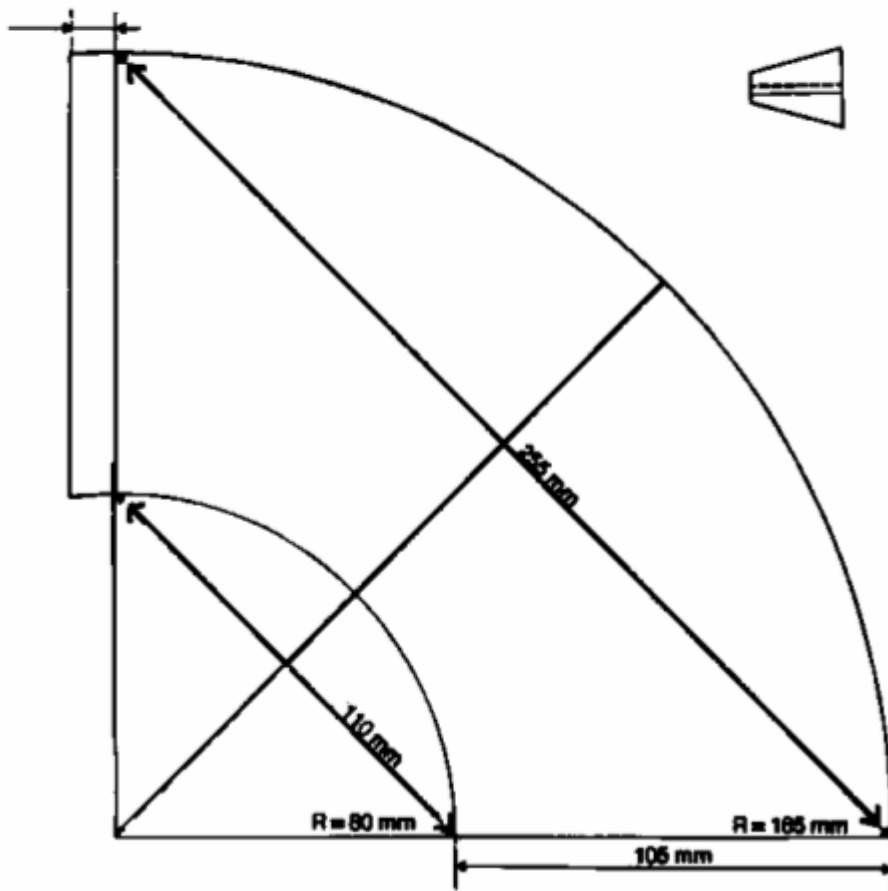
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Wettability

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Fig. 1 Template for the funnel.



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Wettability IDF Method
GEA Niro analytical method A 5 b

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1. Definition

The wettability is defined as the time in seconds required for all the particles of an instant dry milk sample to become wetted (to sink below the water surface or assume a 'typical' wet appearance) when placed on the surface of water.

2. Scope

This method may be used routinely to determine the wetting time in water of instant dried dairy products.

3. Principle

4. Apparatus

- 4.1 Balance (sensitivity 0.01 g).
- 4.2 Weighing dish.
- 4.3 600 ml beaker, internal diameter 90 mm \pm 2 mm and height 120 mm \pm 3 mm, glass plate and glass or stainless steel tube (see Fig. 1).
- 4.4 250 ml beaker.
- 4.5 Small brush.
- 4.6 Stop watch.
- 4.7 Thermometer, 0-100°C (calibrated to within \pm 0.5°C).

5. Reagents

Deionised water.

6. Procedure

- 6.1 Weigh a 10 g \pm 0.05 g well mixed instant dried milk into a weighing dish.
- 6.2 Measure 250 ml \pm 1 ml of deionised water adjusted to 25°C \pm 0.5°C into a dry 600 ml glass beaker ensuring that the inside of the beaker above the final water level remains dry.
- 6.3 Place the steel plate on top of the beaker, with one edge of the plate close to the rim of the beaker. Place the glass tube on top of the plate as shown in Fig. 1.
- 6.4 Transfer the test portion from the weighing dish to the glass tube, and spread the sample evenly over the glass plate.

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- 6.5 Start the stop watch. After 10 seconds, withdraw the glass plate with one hand (holding the steel tube with the other hand) allowing the powder sample to fall progressively, over a period of 2.5 seconds, onto the surface of the water.
- 6.6 Record the time in seconds from the beginning of withdrawal of the glass plate until all the particles have become wetted.
- 6.7 Measurements are to be carried out in duplicate.

7. Result

The wetting time = $T-10$

where:

- T = time recorded (in 6.6) in seconds.
- 10 = time elapsed before withdrawal of the glass plate.

Samples with wetting times in excess of 60 seconds are considered non-instant and the results may be given as >60 seconds.

8. Reproducibility

If two determinations do not agree within 20% relative, make two new determinations and report single determinations together with the average.

9. Remarks

- 9.1 To maintain a reasonably consistent water surface area, it is mandatory that the 600 ml beaker used is with the correct dimensions as indicated in 4.3.
- 9.2 Since particle size influences the wettability of dried milk, care should be taken to minimize particle breakdown. This may be achieved by careful sampling and subsequent handling. Sample containers should be completely filled, and bags should be packed carefully to avoid agitation of the powder.

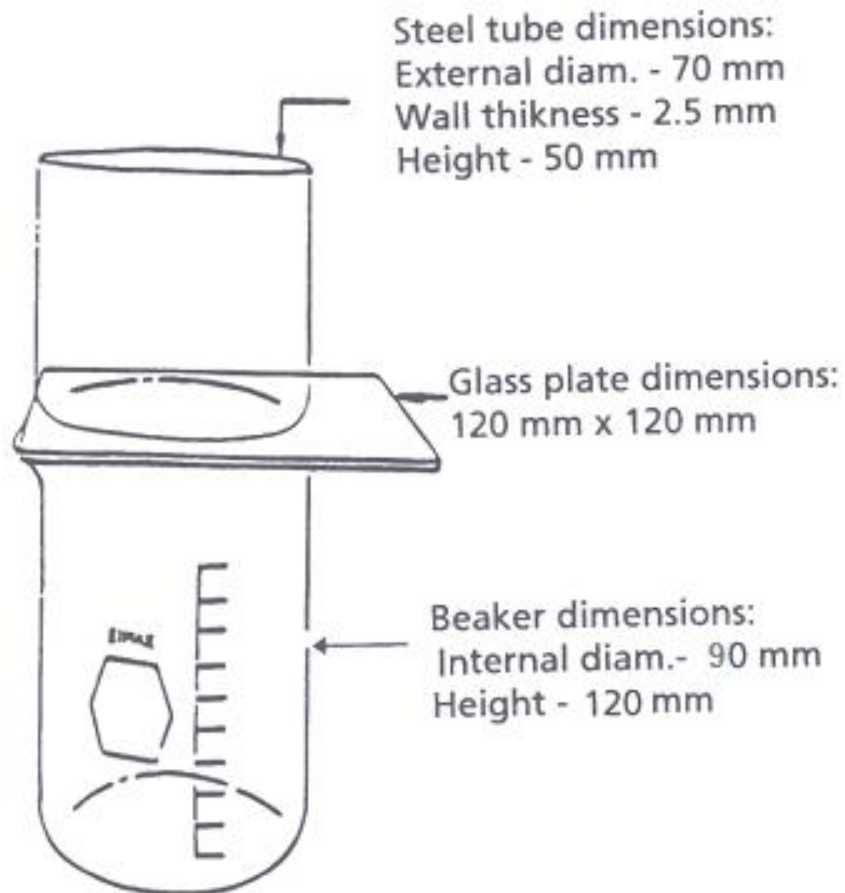
10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard 87:1979.](#)

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Wettability IDF Method
GEA Niro analytical method A 5 b

(Page 3 of 3)



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Powder Dispersibility IDF Method GEA Niro analytical method A 6 a

(Page 1 of 5)

1. Definition

The dispersibility of a powder in water is its ability to break down into particles passing through a 150 µm sieve.

2. Scope

This method is used for any kind of dried dairy product, but is especially applicable to skim milk and whole milk.

3. Principle

A powder sample of known water content is evenly spread on the surface of 25°C water. The mixture is stirred manually for a short time and part of the mixture is filtered through a sieve. The total solids content of the collected liquid is determined. Dispersibility is calculated from the mass of the test portion and the values for water content and total solids.

4. Apparatus

- 4.1 Balance, sensitivity - 0.1 g.
- 4.2 Analytical balance - sensitivity 0.1 mg.
- 4.3 Drying oven without forced air circulation, with a thermostatic control capable of maintaining the temperature at 102°C ± 2°C.
- 4.3 Desiccator.
- 4.4 Beaker - 600 ml, inside diameter 88 mm, height 123 mm, with graduation mark at 150 ml.
- 4.5 Glass plate - 120 x 120 mm, thickness 2.5 mm.
- 4.6 Metal tubing - inside diameter 73 mm, height 50 mm.
- 4.7 Stand and clamp for holding the glass tubing.
- 4.8 Spatula - stainless steel, thickness 1 mm, overall length 250 mm, length of blade 135 mm, width of blade 25 mm (see Fig. 1).
- 4.9 Stop watch.
- 4.10 Test sieve - diameter 200 mm, 150µ woven metal wire cloth complying with ISO 565.
- 4.11 Erlenmeyer flask - 250 ml with stopper.
- 4.12 Glass funnel (see Fig. 1).
- 4.13 Pipette.

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Powder Dispersibility IDF Method
GEA Niro analytical method A 6 a

(Page 2 of 5)

4.14 Glass weighing dish.

4.15 Pumice.

4.16 Brush.

5 Reagents

N/A

6. Procedure

6.1 Determine the moisture content of the powder as described in method No. A 1 b.

6.2 Weigh out 26 ± 0.1 g of skim milk or 34 ± 0.1 g of whole milk. Transfer the powder to the glass plate within the metal tubing and spread evenly with the spatula.

6.3 Weigh out 250 ± 0.1 g of deionised water at $25^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$ in a dry glass beaker, taking care that the inside of the beaker above the final water level remains dry.

6.4 Place the beaker on the stand, place the glass plate on top of the beaker and mount the metal tubing over the glass plate, clamping it so that it is centrally located above the beaker and leaving the glass plate free enough to be withdrawn.

6.5 Start the stop watch.

6.6 At the same time, start the withdrawal of the glass plate. This should be performed with a gentle and continuous movement and should be accomplished in 2.5 sec.

6.7 Immediately remove the beaker from the stand and insert the spatula down the side of the beaker until it touch the bottom, this should take exactly 5 sec. During the next 5 sec. stir the content with the spatula making one complete stirring movement per sec., i.e. a smooth and continuous movement of the spatula back and forth across the diameter of the beaker and occupying 1 sec., with the end of the spatula in continuous contact with the bottom of the beaker and slightly tilted away from the side of the beaker at the end of each half stirring movement. This will minimize the accumulation of unwetted dried milk on the side of the beaker. Without interruption, continue stirring in the same manner for 15 sec., but hold the spatula in a vertical position throughout. While making the 20 complete stirring movements in 20 sec., continuously rotate the beaker on its base so that approx. one complete turn (360°) is achieved during the stirring.

6.8 Allow the beaker to stand for 30 sec. (i.e. until the stop watch shows 55 sec.) and then, without disturbing any sediment, quickly pour some of the liquid (down to the 150 ml graduation mark) into the test sieve. Do not tilt or move the sieve

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Powder Dispersibility IDF Method

GEA Niro analytical method A 6 a

(Page 3 of 5)

during filtration. Use a wet sieve which has been wiped with a towel to remove excess water. During filtration, the sieve should be placed on a dry and clean receiver.

- 6.9 30 sec. after start of the filtration (i.e. at 1 min. 25 sec. on the stop watch), transfer the contents of the receiver to the conical flask and insert a stopper.
- 6.10 Mix the content thoroughly. Determine the dry matter in duplicate, as described in step 11-13.
- 6.11 Pipette approx. 8-12 g of the milk into a dry, glass weighing dish containing dried pumice or sea sand.
- 6.12 Dry to constant weight at 102°C in an oven. Cool to room temperature in a desiccator and weigh. The difference in weight indicates amount of milk dry matter in the reconstituted milk.
- 6.13 Measurements are carried out in duplicate.

7. Result

$$\text{Instant skim milk, } D = \frac{T \times 962}{100 - (W + T)}$$

$$\text{Instant whole milk, } D = \frac{T \times 735}{100 - (W + T)}$$

- D = Dispersibility in %.
 T = Total solids in % of the liquid (4.10).
 W = Moisture in % (4.1).

The mean value of two tests should be calculated to the nearest 1%.
 If the difference between two tests exceeds 4%, the determination is not valid and another set of duplicate values should be obtained as described in 4.

The formulas are derived as follows:

If p grams (dry matter and water) of the test portion (P grams) are dispersed in the 250 g of water, then

$$T = \frac{p \times \left(\frac{100-W}{100}\right) \times 100}{250+p} \quad \text{and therefore} \quad p = \frac{250 \times T}{100 - (W+T)}$$

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Powder Dispersibility IDF Method
GEA Niro analytical method A 6 a

(Page 4 of 5)

$$D = \frac{p \times \left(\frac{100 - W}{100}\right) \times 100}{p \times \left(\frac{100 - W}{100}\right)} = \frac{p \times 100}{P}$$

Then
$$D = \frac{250 \times T}{100 - (W - T)} \times \frac{100}{P}$$

This can be simplified as follows:

With instant dried milk where P is 26,

$$D = \frac{T \times 962}{100 - (W + T)}$$

With instant dried whole milk where P is 34,

$$D = \frac{T \times 735}{100 - (W + T)}$$

8. Reproducibility

N/A

9. Remarks

N/A

10. Reference

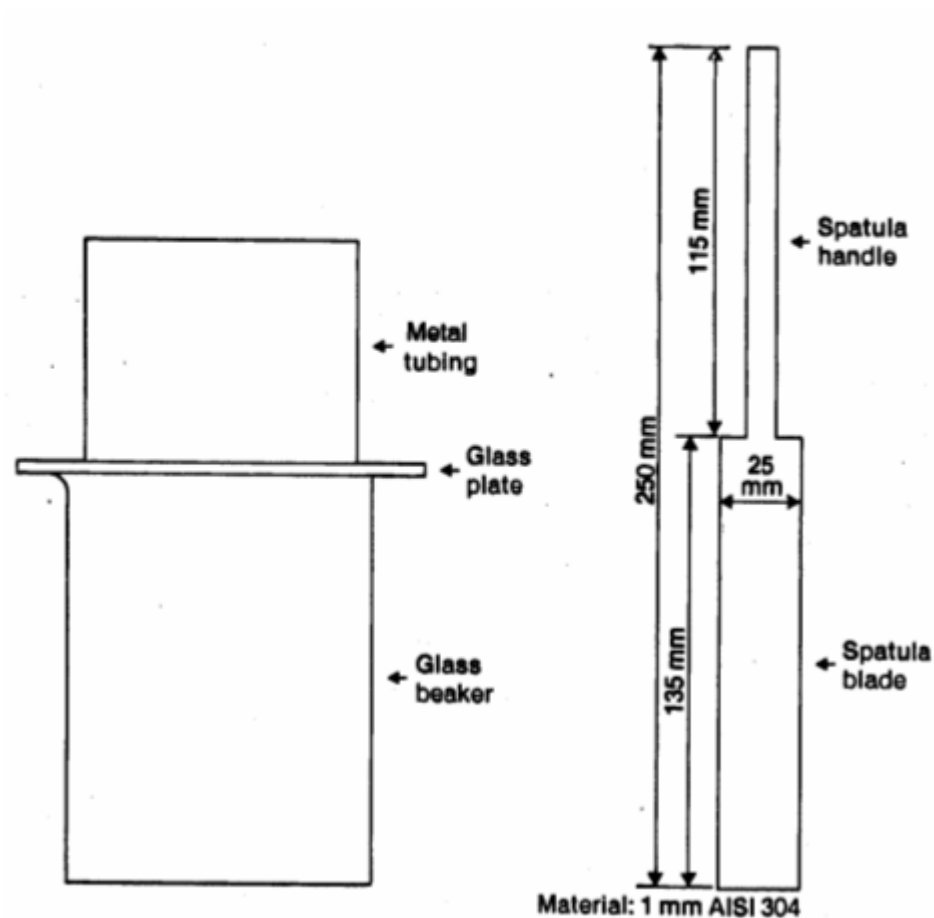
- [GEA Niro Research Laboratory](#)
- [IDF Standard 87:1979](#).

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Powder Dispersibility IDF Method
GEA Niro analytical method A 6 a

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Fig. 1. Apparatus for determination of dispersibility.



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Slowly Dispersible Particles in Agglomerated Milk Powder GEA Niro analytical method A 7 a

(Page 1 of 3)

1. Definition

The content of slowly dispersible particles in milk powders is the number of particles still not dispersed after this procedure.

2. Scope

The method is to be used for agglomerated milk powder and for all other agglomerated dairy products.

3. Principle

The sample is evenly spread on the surface of water adjusted to a certain temperature depending on the product. The mixture is stirred manually for a short time and then filtered through a filter paper.

4. Apparatus

1. Balance, sensitivity ± 0.01 g.
2. Beaker - 250 ml.
3. Teaspoon, 40 mm high, 29 mm wide.
4. Filter paper, coarse - diameter 90 mm.
5. Büchner funnel, filter flask and water air pump.
6. Standard scale 0-5 (see Fig. 1).

5. Reagents

None.

6. Procedure

1. Weigh out the correct amount of powder ± 0.1 g:

Skimmed milk:	10 g
Whole milk:	13 g
2. Pour 100 ml of tap water, at different temperatures, into the beaker and add the powder:

Skimmed milk:	20° C \pm 2° C
Whole milk:	40° C \pm 2° C
Lecithinated whole milk:	20° C \pm 2° C
3. Using 30 complete circular stirring movements, stir with the teaspoon for 20 seconds.

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Slowly Dispersible Particles in Agglomerated Milk Powder
GEA Niro analytical method A 7 a

(Page 2 of 3)

4. Allow to stand for 2 minutes.
5. Using 5 complete circular stirring movements, stir for 3 seconds.
6. Filter through a Büchner funnel.

7. Result

Compare with standards (0-5) using Fig. 1 immediately after filtering.

8. Reproducibility

N/A

9. Remarks

A sample classified as being between two standard discs is always set at the highest value. The sample is classified as '0' if no white particles are found on the filter paper.

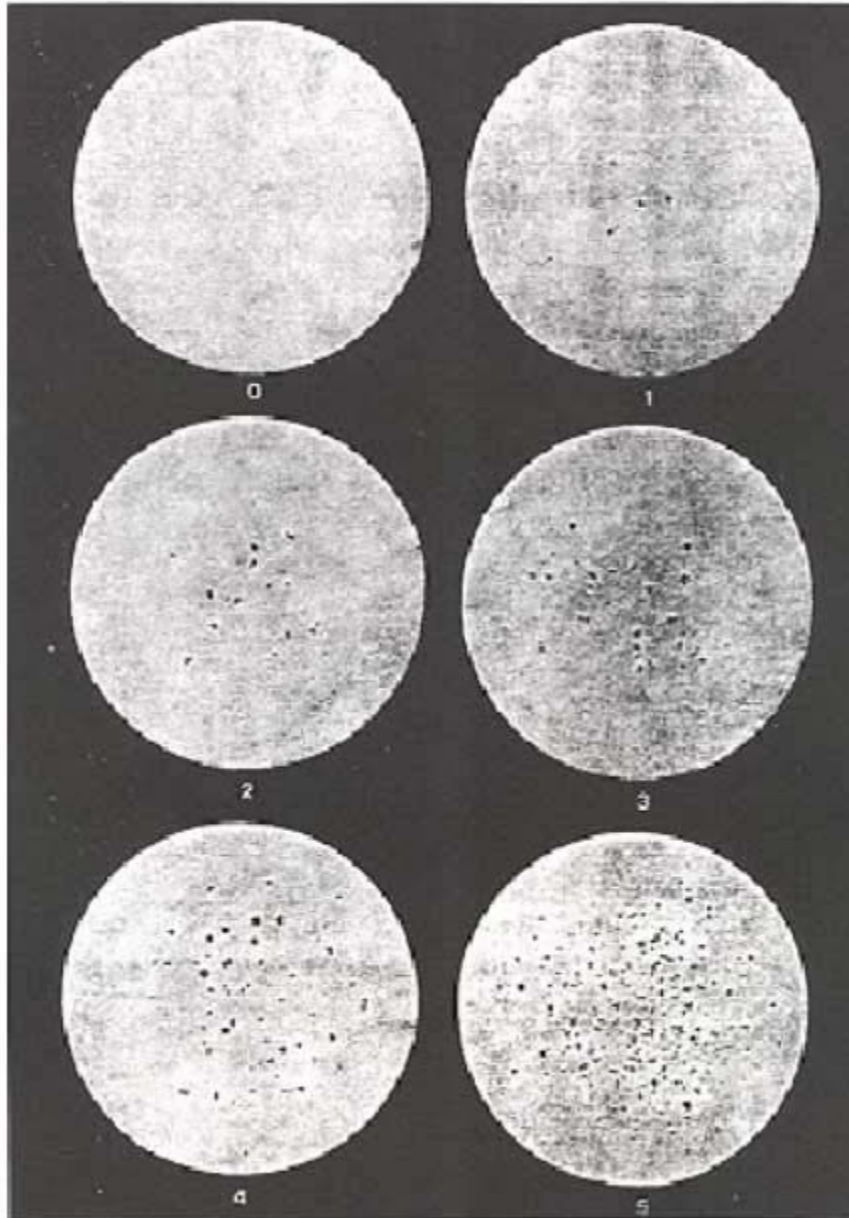
10. Literature

- [GEA Niro Research Laboratory](http://www.niro.com)

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Slowly Dispersible Particles in Agglomerated Milk Powder
GEA Niro analytical method A 7 a
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Fig. 1 Standard scale 0-5.



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Particle Size Distribution by Sieving GEA Niro analytical method A 8 a

(Page 1 of 3)

1. Definition

The powder sample is divided into fractions with different particle sizes by sieving.

2. Scope

The method is to be used for agglomerated milk powders, rewet agglomerated milk powders and other free-flowing powders. When modified, the method can also be used for fatty and sticky products – see Remarks 9.1.

3. Principle

Powder samples are sieved through a number of sieves with different mesh sizes using a horizontally oscillating movement.

4. Apparatus

1. Balance - sensitivity ± 0.1 mg.
2. Shaker for sieves, e.g. as supplied by Engelsmann, Germany (Fig. 1).
3. Brush.
4. Sieves with different mesh sizes, lid and base.

5. Reagents

None.

6. Procedure

1. Select the sieves, weigh them and the base, and place them on the base in decreasing order.

Agglomerated milk powder		Rewet agglomerated milk powder	
μm	U.S. Mesh	μm	U.S. Mesh
500	35	2000	10
355	45	1000	18
250	60	710	25
212	70	500	35
180	80	355	45
150	100	250	60
125	120	150	100

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Particle Size Distribution by Sieving
GEA Niro analytical method A 8 a

(Page 2 of 3)

2. Weigh out 50.0 g of powder and transfer it to the upper sieve.
3. Set the lid on the upper sieve and place the stack on the base of the shaker. Secure the stack and shake for 5 minutes.
4. Weigh each sieve and the base with the powder. Make sure that no powder sticking to the bottom of the sieve.
5. If >20% powder is found on the upper sieve or on the base, an additional sieve with a larger or smaller mesh size is added for a new sieve analysis.

7. Result

The result can be found in two different ways:

1. Each fraction is indicated as a percentage of the total weight.

a = weight of powder on the sieve
 w = total weight of powder

Results are reported with 1 decimal.

Example:

% powder	sieve size, m
0.1	>500
5.2	>355 - ≤500
0.4	>250 - ≤355
23.1	>212 - ≤250
30.4	>180 - ≤212
16.4	>150 - ≤180
10.2	>125 - ≤150
3.2	>90 - ≤125
1.0	≤ 90

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Particle Size Distribution by Sieving GEA Niro analytical method A 8 a

(Page 3 of 3)

2. Accumulation of the numbers found on individual sieves:

% powder	sieve size, m	% powder	sieve size, m
0.1	> 500	1.0	< 90
5.3	> 355	4.2	< 125
15.7	> 250	14.2	< 150
38.8	> 212	30.8	< 180
69.2	> 180	61.2	< 212
85.6	> 150	84.3	< 250
95.8	> 125	94.7	< 355
99.0	> 90	99.9	< 500

8. Reproducibility

N/A

9. Remarks

1. Fatty and sticky products will lump together and not pass through the sieves, especially for mesh sizes <150 μ . To prevent this, a free-flowing agent (e.g sodium aluminium silicate - Tix-O-Sil or Cal-Flo) can be used. Normally, 1-2% of free-flowing agent is suitable to prevent lumping on the sieves. Before sieving, gently mix the free-flowing agent with the powder in a beaker. When the correct amount of free-flowing agent is used, it will disperse evenly, so that no correction is needed when calculating the particle size distribution.

The use of free-flowing agent must be noted together with the results.

2. The particle size distribution of agglomerated and brittle powder will depend on the sieving time. If any deviation from this procedure is decided, specify it together with the results.

10. Literature

- [GEA Niro Research Laboratory](#)

- Allen Terence, Particle Size Measurement, 2. edition 1975 by Chapman and Hall Ltd.

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Particle Size Distribution by Sieving with Rubber Blocks

GEA Niro analytical method A 8 b

(Page 1 of 3)

1. Definition

The powder sample is divided into fractions with different particle sizes by sieving with rubber blocks.

2. Scope

The method is, to a certain extent, suitable for non-agglomerated products.

3. Scope

Powder samples are sieved together with 3 rubber blocks through a number of sieves.

4. Apparatus

1. Balance - sensitivity ± 0.1 mg.
2. Shaker for sieves, e.g. as supplied by [Engelsmann](#), Germany (Fig.1).
3. Brush.
4. Sieves with different mesh sizes, lid and base.
5. Rubber blocks, 3 pieces for each sieve.

5. Reagents

None.

6. Procedure

1. Select the sieves, place 3 rubber blocks on each, weigh them and the base, and place them on the base in decreasing order.
2. Weigh out 50.0 g of powder and transfer it to the upper sieve.
3. Set the lid on the upper sieve and place the stack on the base of the shaker. Secure the stack and shake for 5 minutes.
4. Weigh each sieve with rubber blocks and the base with the powder. Make sure that no powder is sticking to the bottom of the sieve – brush off to lower sieve size.
5. If >20% powder is found on the upper sieve or on the base, an additional sieve with a larger or smaller mesh size is added for a new sieve analysis.

7. Result

The result can be found in two different ways:

1. Each fraction is indicated as a percentage of the total weight.

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Particle Size Distribution by Sieving with Rubber Blocks

GEA Niro analytical method A 8 b

(Page 2 of 3)

$$\% \text{ powder on the sieve} = \frac{a \times 100}{w}$$

Where:

a = weight of powder on the sieve

w = total weight of powder

Results are reported with 1 decimal.

Example:

% powder	sieve size, μm
0.4	>250
23.1	>212 - \leq 250
30.4	>180 - \leq 212
16.4	>150 - \leq 180
10.2	>125 - \leq 150
3.2	>90 - \leq 125
1.0	\leq 90

2. Accumulation of the numbers found on individual sieves:

% powder	sieve size, μ	% powder	sieve size, μ
15.7	> 250	1.0	< 90
38.8	> 212	4.2	< 125
69.2	> 180	14.2	< 150
85.6	> 150	30.8	< 180
95.8	> 125	61.2	< 212
99.0	> 90	84.3	< 250

8. Reproducibility

N/A

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Particle Size Distribution by Sieving with Rubber Blocks

GEA Niro analytical method A 8 b

(Page 3 of 3)

9. Remarks

1. Fatty and sticky products will lump together and not pass through the sieves, especially for mesh sizes $< 150 \mu$. To prevent this, a free-flowing agent (e.g. sodium aluminium silicate - Tix-O-Sil or Cal-Flo) can be used. Normally, 1-2% of free-flowing agent is suitable to prevent lumping on the sieves. Before sieving, gently mix the free-flowing agent with the powder in a beaker. When the correct amount of free-flowing agent is used, it will disperse evenly, so that no correction is needed when calculating the particle size distribution.

The use of free-flowing agent must be noted together with the results.

2. The particle size distribution of brittle powder will depend on the sieving time and the type of rubber blocks. If any deviation from this procedure is decided, specify it together with the results.

10. Literature

- [GEA Niro Research Laboratory](#)
- Allen Terence, Particle Size Measurement, 2. edition 1975 by Chapman and Hall Ltd.



Fig. 1 Shaker for sieves.

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Particle Size Distribution by Laser (Malvern)

GEA Niro analytical method A 8 c

(Page 1 of 3)

1. Definition

A suspension of powder in isopropanol is measured with a low angle laser beam, and the particle size distribution is calculated.

2. Scope

This is a fast method for measuring particle size distribution of powders.

3. Principle

The method can be used on all powders containing less than 10% fat.

4. Apparatus

1. Malvern Instrument, Mastersizer Basic, equipped with software version B.0 or similar equipment.
2. Malvern QS Small Volume Sample Dispersion Unit.
3. Malvern in/out measuring cell, beam length 2.0 mm.
4. Dispenser 0-50 ml with container.
5. Filling knife.
6. Waste container.

5. Reagents

1. Isopropanol, IPA (technical quality).

6. Procedure

1. Look at the particle size in a microscope and choose a lens capable of measuring the largest particles, see Remarks 7.1.
2. Prepare the instrument for measuring in wet mode using IPA as the liquid, as described in the user manual.
The stirrer regulator should be set at 2000 rpm on the Malvern unit.
3. Measure the background for IPA.
4. Quickly add a sufficient amount of milk powder and measure as soon as the powder is dispersed and not later than 20 seconds after addition of the powder. For detailed instructions about measuring, see the Malvern user manual.
5. Rinse twice with IPA.

All measurements are made in duplicate

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Particle Size Distribution by Laser (Malvern)**GEA Niro analytical method A 8 c**(Page 2 of 3)

7. Result

The following calculations are done automatically:

1. The volume median diameter $D(v,0.5)$ is the diameter where 50% of the distribution is above and 50% is below.
2. Two determinations of mean particle size should not differ by more than 5% relative. The shape of the curves in the two determinations should be the same.
3. $D(v,0.9)$, 90% of the volume distribution is below this value.
4. $D(v,0.1)$, 10% of the volume distribution is below this value.
5. The span is the width of the distribution based on the 10%, 50% and 90% quantile.

$$Span = \frac{D [v, 0.9] - D [v, 0.1]}{D [v, 0.5]}$$

8. Reproducibility

N/A

9. Remarks

1. The lens should be chosen according to the actual particle sizes. All lenses are capable of measuring a specific area of microns; therefore, ensure that the lens measures 100% of the largest particle. The software estimates the particles below the lens capacity, but cannot account for larger particles. The most precise results are obtained when the lens cuts off just above the largest particles because the estimate of the particles below is more precise.
2. The obscuration is the amount of sample added to the system or more correctly the light intensity absorbed by the sample. The optimal obscuration is 0.1-0.3. The results are unreliable outside this interval.
3. The powder particles should be insoluble in the dispersing agent (IPA).
4. Fat filled products, containing more than 10% fat, and milk powder containing sugar cannot be measured by this method.
5. $D(4.3)$ is the equivalent volume mean diameter or the De Broncker mean diameter.
6. $D(3.2)$ is the equivalent surface area mean diameter or the Sauter mean diameter.
7. Non-agglomerated milk powders will give the same mean diameter measured in wet or dry mode; whereas agglomerated powders will give considerably finer mean particle sizes when measured in dry mode due to wear of the particles.

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Particle Size Distribution by Laser (Malvern)

GEA Niro analytical method A 8 c

(Page 3 of 3)

8. It is possible to use ultrasound in-line. The use of ultrasound affects the particles. Agglomerates and primary particles are separated and it is possible to measure the primary particle size.

10. Literature

- [GEA Niro Research Laboratory](#)
- The [Malvern](#) Mastersizer Basic user manual.
QS Small Volume Sample Dispersion Unit user manual.

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Total Fat (Röse-Gottlieb Method)
GEA Niro analytical method A 9 a

(Page 1 of 3)

1. Definition

The content of total fat in milk powder is defined as the mass fraction of evaporation residue obtained by the method described below.

2. Scope

This is a standard method which may be used for all milk powders and other dried dairy products.

3. Principle

The fat of the powder is extracted, followed by a removal of the solvents by evaporation and drying.

4. Apparatus

1. Analytical balance, sensitivity ± 0.01 g
2. Graduated shaking cylinder (Gottlieb) with well-fitting stopper
3. Glass siphon
4. Erlenmeyer flask - 150-200 ml
5. Fat-free granulated pumice
6. Electric hot plate (with safety device)
7. Drying oven with thermostat
8. Desiccator with water-absorbing material, e.g. silicagel

5. Reagents

1. NH_3 -solution 25% (specific gravity 0.91 at 15°C, clear, colourless)
2. 96% ethyl alcohol
3. Ethyl ether, peroxide-free, boiling point 34-35°C
4. Petroleum ether, boiling point 40-60°C

The reagents used must not leave any evaporation residue. Their purity must be checked by blind analyses and the results corrected.

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Total Fat (Röse-Gottlieb Method)
GEA Niro analytical method A 9 a(Page 2 of 3)

6. Procedure

1. Avoid water absorption by the powder during the analysis by keeping the powder container tightly closed.
2. Weigh out exactly 1 g of whole milk powder or 1.5 g of skim milk powder into the shaking cylinder.
3. Dissolve the powder into 10 ml of water. Heat if necessary.
4. Add 1.5 ml of NH₃-solution and heat in a water bath for 15 minutes to 60-70°C. Shake the mixture occasionally.
5. Cool, add 10 ml of ethyl alcohol and mix.
6. Add 25 ml of ethyl ether. Close the cylinder tightly and mix the contents by turning the cylinder up and down for 1 minute.
7. Add 25 ml of petroleum ether and repeat the mixing as under 6.
8. Allow to stand for at least 2 hours. The ether phase must be quite clear and completely separated from the water phase.
9. Transfer the ether phase to an Erlenmeyer flask by means of the siphon, which is then cleaned with a little amount of ether. This ether is also transferred to the flask. Take care that none of the water phase is introduced into the flask.
10. Repeat steps 6 to 9 a further two times, each time adding 25 ml of ether and petroleum ether. Use the same flask each time.
11. After the final siphoning, evaporate the ether on a hot plate or similar and dry the flask for about one hour in an oven at 102°C ± 2°C.
12. Cool to room temperature in a desiccator and weigh.
13. Repeat oven drying and cooling until a constant weight is reached (i.e. the loss in weight is less than or equal to 0.5 mg or the sample has increased its weight).

7. Result

$$\text{Total fat \%} = \frac{W_1 \times 100}{W_2}$$

where:

W₁ = weight in g of the evaporation residueW₂ = weight in g of the powder used

Calculate the result to 1 decimal place

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Total Fat (Röse-Gottlieb Method)
GEA Niro analytical method A 9 a

(Page 3 of 3)

8. Reproducibility

± 0.15%

9. Remarks

N/A

10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF](#) Standard 123A:1988.

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Total Fat (Gerber/Teichert Method)
GEA Niro analytical method A 9 b

(Page 1 of 2)

1. Definition

The fat can be separated from fat-containing milk/milk powder through the addition of sulphuric acid. The fat content is read directly on a special calibrated butyrometer after centrifugation.

2. Scope

The method is to be used for fat-containing milk powders.

3. Principle

The fat can be separated from fat-containing milk/milk powder through the addition of sulphuric acid. The separation is made by using amyl alcohol and centrifugation. The fat content is read directly on a special calibrated butyrometer.

4. Apparatus

1. Balance - sensitivity ± 0.01 g
2. Special butyrometer-scale 0-35% or 0-70%, Teichert, '2.5 g lait en poudre' 65°C
3. Caoutchouc stoppers
4. Pipettes - 1 and 10 ml
5. Centrifuge, Funke Gerber - 1200 rpm, equipped with heating element

5. Reagents

1. Sulphuric acid, H_2SO_4 - density 1.816 ± 0.003 g/ml 90-91%, clear, colourless.
2. Amyl alcohol, $C_5H_{12}O$ - density 0.811 ± 0.002 g/ml

6. Procedure

1. Pour successively into the butyrometer:
10 ml sulphuric acid.
8 ml distilled water (must not be mixed with the acid).
Exactly 2.5 g powder.
1 ml amylalcohol.
2. Close the butyrometer with the caoutchouc stopper and shake until the powder is dissolved. Turn the butyrometer upside-down 5 times.
3. Spin in the centrifuge for 15 minutes at 65°C.

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Total Fat (Gerber/Teichert Method)
GEA Niro analytical method A 9 b

(Page 2 of 2)

4. Shake for further 5 minutes. Spin in the centrifuge for 15 minutes at 65°C. Adjust the fat column by using the stopper, so that it will be in the graduated part of the

butyrometer. The fat percentage can then be read directly after spinning again for further 5 minutes.

5. Measurements are carried out in duplicate.

7. Result

The fat content is read directly on the butyrometer and an average value of the two determinations is calculated. Calculate the result to 1 decimal place.

8. Reproducibility

± 0.3 % for the 0-35 % scale

± 0.5 % for the 0-70 % scale

9. Remarks

1. It is important to wear acid resistant gloves, protection glasses or a protection shield.
2. Samples containing sugar must not be analyzed by this method. Sugar can react very violently with concentrated sulphuric acid and cause an explosion.
3. If the powder is not dissolved after step 5.2, place it in a water bath at $\leq 65^{\circ}\text{C}$ until it is dissolved.
4. If the fat content is $>70\%$, the procedure is repeated using only 1.25 g of powder instead of 2.5 g, and the result is multiplied by 2.
5. For baby food and whole milk, a butyrometer with the scale from 0-35% is used. For powders with a higher fat content, a butyrometer with a scale from 0-70% is used.
6. If the expected fat content is close to 1%, or if a higher precision of the fat content is required, use the Röse Gottlieb Method A 9 a.
7. The sample must be 65°C when reading the fat percentage. If the centrifuge is not adjusted to the right temperature, the butyrometer must be heated in a water bath.

10. Literature

- [GEA Niro Research Laboratory](#)
- [IDF Standard 1C:1987](#).

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Surface Free Fat of Powder
GEA Niro analytical method A 10 a

(Page 1 of 3)

1. Definition

The content of free fat on the surface of milk powder particles is defined as the evaporation residue remaining, after the sample has been gently mixed with petroleum ether, filtered and dried.

2. Scope

The method is to be used for whole milk powder and all other dried dairy products containing fat.

3. Principle

The determination of free fat on the surface of milk powder particles is based on extraction of the fat on the surface of the particles.

4. Apparatus

1. Analytical balance, sensitivity ± 0.1 mg.
2. Balance - sensitivity 10 mg.
3. Funnel.
4. Erlenmeyer flask - 250 ml with ground glass stopper.
5. Pipettes - 25 ml.
6. Measuring cylinder - 50 ml.
7. Shaking apparatus (Stuart flask shakers).
8. Filter paper - Selecta 572 $\frac{1}{2}$ or similar.
9. Erlenmeyer flask, 100 ml.
10. Aluminium weighing dish.
11. Drying oven without forced air circulation with a thermostatic control capable of maintaining the temperature at $105^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
12. Desiccator with water-absorbing material, e.g. silica gel.
13. Stop watch.
14. 50 ml dispenser.

5. Reagents

Petroleum ether, boiling point $<50^{\circ}\text{C}$, density 0.645 - 0.665 g/ml

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Surface Free Fat of Powder
GEA Niro analytical method A 10 a(Page 2 of 3)

6. Procedure

1. Weigh out $10\text{ g} \pm 0.01\text{ g}$ of the sample into a 250 ml Erlenmeyer flask.
2. Start the stop watch and add 50.0 ml of petroleum ether to the flask. Petroleum ether is added using a 50 ml dispenser. Check twice that the dispenser measures the correct volume by dispensing 50 ml into a measuring cylinder.
3. Close the flask and agitate in the shaking device. The degree of shaking must be regulated so the powder is moving and the contents are not splashed up on the sides of the upper half of the flask.
4. After exactly 15 minutes, stop the shaking and filter the solution. Two samples are filtered at a time. Collect the filtrate in a 100 ml Erlenmeyer flask. Record the extraction time from the moment the solvent comes in contact with the powder and until filtration begins.
5. As soon as the filtration is finished, pipette 25 ml of the filtrate into the pre-weighed aluminium dish.
6. Allow most of the petroleum ether to evaporate in the fume hood.
7. Dry in the drying oven for one hour at $105^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
8. The sample is cooled in a desiccator until room temperature and weighed.
9. Measurements are carried out in duplicate.
10. Make a blind analysis for each batch of petroleum ether and correct if necessary.

7. Result

The content of surface free fat may be expressed as a percentage of the powder (or as a percentage of the amount of total fat).

$$\% \text{ free fat} = \frac{a \times 50 \times 100}{\left(ml - \frac{a}{0.94} \right) \times b}$$

ml = ml filtrate taken out with pipette.

a = evaporation residue in g.

b = g of powder used.

0.94 = estimated value of the density of free fat.

Calculate the result to 2 decimal places.

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Surface Free Fat of Powder
GEA Niro analytical method A 10 a

(Page 3 of 3)

8. Reproducibility

Two determinations must not differ more than 5% relative.

9. Remarks

N/A

10. Literature

- [GEA Niro Research Laboratory](#)

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Particle Density Occluded Air and Interstitial Air (Air Pycnometer Method)

GEA Niro analytical method A 11 a

(Page 1 of 7)

1. Definition

Particle density g/ml is defined as the mass of particles having a total volume of 1 ml.
Occluded air ml/100g is defined as the difference between the volume of a given mass of particles and the volume of the same mass of air-free solids.
Interstitial air ml/100g is defined as the difference between the volume of a given mass of particles and the volume of the same mass of 100x tapped powder.

2. Scope

This method may to be used for all powders.

3. Principle

The true volume of a sample (the volume in g/cm³ enclosed by its outer surface an excluding its open pores) is determined by measuring the pressure change of helium in a calibrated volume.

4. Apparatus

1. Analytical balance, capable of weighing to 0.1 mg.
2. AccuPyc 1330 Pycnometer, Micromeritics (Fig. 1 and 2).
3. LC-20 Dot matrix printer, Star.

5. Reagents

Helium (g).

6. Procedure

1. Adjust the pressure of the helium to 2 bar on the gas flask.
2. Check the parameters by pressing the white button and button No. 2 on the keypad. Press 'Enter'.
The parameters must be as follows:
Number of purges: 3.
Purge fill pressure: 19.5 psig.
Number of runs: 3
Run fill pressure: 19.5 psig
Equilibration rate: 0.050 psig/min.
Use run precision: No.
3. Press 'Save' to store the information. The display should show 'Reload'.
4. Weigh out an amount of powder into the sample cup and remove excess powder on the sides of the cup.
5. Remove the chamber cap by turning it counter clockwise, then lifting up. Insert the sample

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Particle Density Occluded Air and Interstitial Air (Air Pycnometer Method)

GEA Niro analytical method A 11 a

(Page 2 of 7)

cup in the cell chamber and put on the chamber cap again.

6. Press the white button and button No. 4.
7. Type sample identification followed by 'Enter' and the sample weight followed by 'Enter'.
8. To start the analysis press 'Enter'.
9. When the analysis stops (after approx. 10-12 min.) the results are printed.
10. Determine the moisture content (Method A 1 a), the fat content (Method A 9 a) and the 100x tapped powder bulk density (Method A 2 a).

7. Result

1. The particle density $D_{particle}$ is calculated as:

$$D_{particle} = \frac{w_{sample}}{V_{sample}} \quad [\text{g/ml}]$$

w_{sample} = Weight of the sample in g.

V_{sample} = Volume of the sample in ml.

An example of the print of the results is seen on Fig. 3.

2. The theoretical density of powder solids D_{solids} in milk powder is calculated as:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF/1.52 + \%W} \quad [\text{g/ml}]$$

%F = fat content

%SNF = solid non-fat content

%W = moisture content

For whey powder of normal composition, the following formula can be used:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF(whey)/1.58 + \%W} \quad [\text{g/ml}]$$

%F = fat content

%SNF = solid non-fat content in whey powder

%W = moisture content

3. Occluded air content V_{oa} is calculated as:

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Particle Density Occluded Air and Interstitial Air (Air Pycnometer Method)

GEA Niro analytical method A 11 a

(Page 3 of 7)

$$V_{oa} = \frac{100}{D_{particle}} - \frac{100}{D_{solids}} \quad [\text{ml}/100\text{g}]$$

$D_{particle}$ = particle density (from 7.1)

D_{solids} = density of powder solids (from 7.2)

4. Interstitial air content is calculated as:

$$V_{ia} = \frac{100}{D_{powder}} - \frac{100}{D_{particle}} \quad [\text{ml}/100\text{g}]$$

D_{powder} = Powder bulk density, tapped 100x (from 6.10)

$D_{particle}$ = particle density (from 7.1)

8. Reproducibility

Particle density \pm 0.02 g/ml

9. Remarks

1. Particle densities measured on powders produced by injection of inert gas(es), e.g. CO₂ and N₂, into the feed will depend on the pressure used for the determination. The results will often not be the same as those measured with the Beckman Air Pycnometer. For baby food, produced with CO₂ injection a pressure change to 10 psig on the AccuPyc will result in the same values as previously measured by the Beckman Air Pycnometer.
2. If there is any excess powder on the outside of the sample cup or around the O-ring, the analysis stops.
3. The analysis time is approx. 10-12 min.
4. If the analysis takes more than 1000 secs the analysis stops automatically.
5. If the deviation between determinations is >0.01 the pressure must be changed.
6. The temperature of the helium used must be the same as that of the instrument, in order to get correct results.
7. When calculating powder of different compositions, the density and the amounts of the constituents must be taken in consideration. For this purpose, the following values may be used:

Powder material, moisture free	Density at 20°C
Whole milk powder (28% fat)	1.28
Non-fat milk solids	1.52

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**Particle Density Occluded Air and Interstitial Air
(Air Pycnometer Method)**

GEA Niro analytical method A 11 a

(Page 4 of 7)

Milk fat in powder	0.94
Ca-caseinate phosphate complex	1.39
Amorphus lactose	1.52
Beta-lactose	1.59
Alpha-lactose monohydrate	1.545
Anhydrous alpha-lactose	1.545
Spray dried whey powder	1.58
Residual whey components	1.8
Demineralized whey powder	1.525

10. Literature

1. [GEA Niro Research Laboratory](#)
2. [Micromeritics](#): 'AccuPyc 1330 Pycnometer' operator's manual.
3. Buma T.J.: 'The true density of spray milk powder and of certain constituents' (Netherlands Milk and Dairy Journal, 1965, 19, pp. 249-265)

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**Particle Density Occluded Air and Interstitial Air
(Air Pycnometer Method)**

GEA Niro analytical method A 11 a

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Fig. 1 AccuPyc 1330 Pycnometer

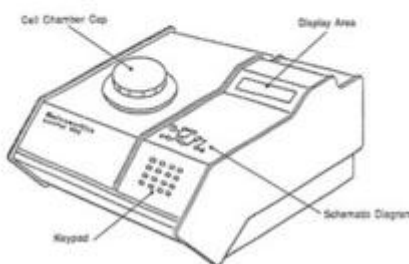


Fig. 2 Pycnometer Keys

Function	Keys	Used for
ZERO	0	Zero the pressure transducer
CALIBRATE	•	Calibrate the pycnometer
MANUAL	1	Manually control the valves. After pressing the MANUAL key, you may use the FILL EXPAND and VENT keys to open and close the valves
SET UP	2	Display or edit analysis parameters, report options, calibration volumes, data transmission parameters, unit types and operation language
TRANSMIT	3	Transmit analysis or calibration data over the serial line. If an automatic operation is in progress, transmit a partial report

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**Particle Density Occluded Air and Interstitial Air
(Air Pycnometer Method)**

GEA Niro analytical method A 11 a

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ESCAPE	Clear	Delete all data entered in the current mode and return to display mode. If an automatic operation is in progress, cancel it
ANALYZE	4	Perform analysis
REVIEW	5	Review or edit completed analysis or calibration data
PRINT	6	Print analysis or calibration report. If an automatic operation is in progress, print partial report
FILL	7 (manual mode)	Open and close the fill valve. The indicator above the FILL key is on when the valve is open and off when the valve is closed
EXPAND	8 (manual mode)	Open and close the expansion valve. The indicator above the EXPAND key is on when the valve is open and off when the valve is closed
VENT	9 (manual mode)	Open and close the vent valve. The indicator above the VENT key is on when the valve is open and off when the valve is closed

Fig. 3 Example - print of results

ACCUPYC 1330 VI.03

Serial number: 529
 Density and volume report
 Sample ID: 920268
 Sample weight: 1.8702 g
 Number of purges: 3
 Equilibration rate: 0.0500 psig/min.
 Cell volume: 12.1664 cm³
 Expansion volume: 8.7231

	Volume	Deviation	Density	Deviation
Run	cm ³	cm ³	g/cm ³	g/cm ³
1	1.4992	0.0001	1.2475	0.0001

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**Particle Density Occluded Air and Interstitial Air
(Air Pycnometer Method)**

GEA Niro analytical method A 11 a

(Page 7 of 7)

2	1.5014	0.0023	1.2457	0.0019
3	1.4967	0.0024	1.2495	0.0020

Average volume: 1.4991 cm³
Standard deviation: 0.0023 cm³

Average density: 1.2475 g/ cm³
Standard deviation: 0.0019 g/ cm³

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Particle Density, Occluded Air and Interstitial Air (Petroleum Ether Method)

GEA Niro analytical method A 11 b

(Page 1 of 3)

1. Definition

Particle density (g/ml) is defined as the mass of particles having a total volume of 1 ml.

Occluded air (ml/100g) is defined as the difference between the volume of a given mass of particles and the volume of the same mass of air-free solids.

Interstitial air (ml/100g) is defined as the difference between the volume of a given mass of particles and the volume of the same mass of powder tapped 100 times (100x tapped powder).

2. Scope

This method may to be used for all powders.

3. Principle

The weighted amount of powder is added to petroleum ether in a measuring cylinder. The weight of the powder divided by the volume increase of the petroleum ether gives the particle density.

4. Apparatus

1. Analytical balance, capable of weighing to 0.1 mg.
2. Calibrated 100 ml measuring cylinder with glass stopper.
3. Rubber spatula.
4. Pipettes – 10 ml and 50 ml

5. Reagents

Petroleum ether.

6. Procedure

1. Weigh out 25 g powder into the measuring cylinder.
2. Add 50 ml petroleum ether with a pipette and shake the measuring cylinder gently until all the powder is suspended.
3. Using the rubber spatula, scrape down all the powder particles on the wall of the measuring cylinder. Rinse with a further 10 ml of petroleum ether from a pipette.
4. Read the total volume of petroleum ether with suspended powder.
5. Determine the moisture content (Method A1a), the fat content (Method A9a) and the 100x tapped powder bulk density (Method A2a).

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Particle Density, Occluded Air and Interstitial Air (Petroleum Ether Method)

GEA Niro analytical method A 11 b

(Page 2 of 3)

7. Result

1. The particle density $D_{particle}$ is calculated as:

$$D_{particle} = \frac{W_{sample}}{V_{ether-60}} \text{ [g/ml]}$$

W_{sample} = Weight of the sample in g

V_{ether} = Volume of petroleum ether with suspended powder in ml

2. The theoretical density of powder solids D_{solids} in milk powder is calculated as:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF/1.52 + \%W} \text{ [g/ml]}$$

%F = fat content

%SNF = solid non-fat content

%W = moisture content

For whey powder of normal composition the following formula can be used:

$$D_{solids} = \frac{100}{\%F/0.94 + \%SNF(whey)/1.58 + \%W} \text{ [g/ml]}$$

%F = fat content

%SNF = solid non-fat content in whey powder

%W = moisture content

3. Occluded air content V_{oa} is calculated as:

$$V_{oa} = \frac{100}{D_{particle}} - \frac{100}{D_{solids}} \text{ [ml/100g]}$$

$D_{particle}$ = particle density (from 7.1)

D_{solids} = density of powder solids (from 7.2)

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Particle Density, Occluded Air and Interstitial Air (Petroleum Ether Method)

GEA Niro analytical method A 11 b

(Page 3 of 3)

4. Interstitial air content is calculated as:

$$V_{ia} = \frac{100}{D_{powder}} - \frac{100}{D_{particle}} \quad [\text{ml}/100\text{g}]$$

D_{powder} = Powder bulk density, tapped 100x (from 6.10)

$D_{particle}$ = Particle density (from 7.1)

8. Reproducibility

Particle density \pm 0.03 g/ml

9. Remarks

- When calculating powder of different compositions, the density and the amounts of the constituents must be taken in consideration. For this purpose, the following values may be used.

Powder material, moisture free	Density at 20° C
Whole milk powder (28% fat)	1.280
Non-fat milk solids	1.520
Milk fat in powder	0.940
Ca-caseinate phosphate complex	1.390
Amorphous lactose	1.520
Beta-lactose	1.590
Alpha-lactose monohydrate	1.545
Anhydrous alpha-lactose	1.545
Spray dried whey powder	1.580
Residual whey components	1.800
Demineralized whey powder	1.525

10. Literature

- [GEA Niro Research Laboratory](#)
- Buma T.J.: 'The true density of spray milk powder and of certain constituents' (Netherlands Milk and Dairy Journal, 1965, 19, pp.

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Mechanical Stability of Agglomerated Milk Powder

GEA Niro analytical method A 13 a

(Page 1 of 2)

1. Definition

The mechanical stability is expressed as percentage of broken down particles during mechanical treatment by sieving.

2. Scope

The method is to be used for agglomerated powder.

3. Principle

The fraction of particles smaller than the size of the sieve is removed by gentle hand sifting. The remaining part of the original powder is thereafter mechanically treated by sieving for 10 minutes, and the amount of created smaller particles is determined.

4. Apparatus

1. Balance - sensitivity ± 0.01 g
2. Shaker for sieves, [Engelsmann](#), Germany (Fig. 1)
3. Sieves – 150 μ or 250 μ (incl. lid and base)
4. Brush.

5. Reagents

None.

6. Procedure

1. Depending on the type of powder choose an appropriate sieve:
Spray agglomerated instant powder: 150 μ
Rewet agglomerated instant powder: 250 μ
2. Manually sieve 60-80 g of powder to separate the fines from the agglomerates.
3. Weigh out 50 g of the powder remaining on the sieve.
4. Clean the sieve carefully. Place 50 g of powder on the sieve, and sieve for 10 minutes on the shaker.
5. Weigh the powder remaining on the sieve.

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Mechanical Stability of Agglomerated Milk Powder GEA Niro analytical method A 13 a

(Page 2 of 2)

7. Result

$$\text{Mechanical stability, \%} = \frac{a \times 100}{b}$$

a = g powder left on the sieve

b = g powder weighed on the sieve

8. Reproducibility

± 1 %

9. Remarks

N/A

10. Literature

[GEA Niro Research Laboratory](#)

Fig. 1 Shaker for sieves.



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Hygroscopicity

GEA Niro analytical method A 14 a

(Page 1 of 4)

1. Definition

The hygroscopicity of a powder is its equilibrium moisture content after being exposed to air humidity under given conditions.

2. Scope

This method is particularly suitable for whey powder, but may be applied for other dried products.

3. Principle

Air with a 79.5% relative humidity (RH) is sucked through the powder sample until a constant increase in weight has been reached.

4. Apparatus

1. Analytical balance, sensitivity ± 0.1 mg
2. Vacuum flask, 500 ml.
3. Gooch filter with ground cone 34/35, porosity 1.
4. Gooch filter adapter with ground glass socket 34/35, see Fig. 1.
5. Glass covering tube assembled by means of 2 rubber stoppers to the vacuum flask, see Fig. 1.
6. Water vacuum pump.
7. Flow meter, 0-400 ml/min.
8. Washing bottle, 250 ml.
9. Three way glass cock.
10. 500 μ sieve.

5. Reagents

Ammonium chloride, saturated solution at 20°C.

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Hygroscopicity

GEA Niro analytical method A 14 a

(Page 2 of 4)

6. Procedure

1. Assemble the apparatus with rubber tubes as follows (see Fig. 1):

- 1.1 vacuum pump
- 1.2 three-way cock
- 1.3 vacuum flask with filter and filter adapter
- 1.4 washing bottle
- 1.5 flow meter

One position of the three-way cock should allow passage of air through the apparatus. The other passage of air directly from the atmosphere to the pump and at the same time keeping the apparatus tightly closed from the atmosphere.

2. Fill the washing bottle with a saturated solution of ammonium chloride and add a surplus of ammonium chloride crystals.
3. Place a clean and dry Gooch filter in the adapter and assemble as shown in Fig. 1. Turn the cock to position 1 (pump atmosphere). Start the vacuum pump. Turn the cock to position 2 (pump washing bottle), set the flow rate to 3-400 ml/min and pump for 5 minutes.
4. Turn the cock to position 1 and wait until the circulation through the washing bottle has ceased. Open the apparatus and weigh the Gooch filter.
5. Weigh out approx. 0.5 g of the sample on the filter with an accuracy of 0.1 mg of powder, which has been forced through a 500 μ sieve. Spread the powder evenly over the filter bottom by tapping gently.
6. Place the Gooch filter in the apparatus and start as described in 6.3.
7. Check the flow rate from time to time and clean the washing bottle if necessary (the tube can be blocked by crystals).
8. Check the increase in weight every 10 min. during the first 30-40 min. and then every 20 min. Stop the air flow as described in 6.4.

The increase in weight normally shows a maximum and then slightly decreases and reaches a steady level. When the maximum has been reached the analysis can be stopped. Normally this does not take more than 4 hours. If degree of caking (Method A 15 a) has to be determined, keep the sample in the apparatus until it can be transferred to the oven.

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Hygroscopicity

GEA Niro analytical method A 14 a

(Page 3 of 4)

7. Result

$$\% \text{ Hygroscopicity} = \frac{(\%WI + \%FW) \times 100}{100 + \%WI}$$

%FW = % free water (determined according to Niro Method A1c for whey powders and according to Niro Method A1a for other dried products)

$$\%WI = \frac{c-b}{b-a} \times 100$$

a = weight of Gooch filter in g

b = weight of Gooch filter + powder in g

c = weight of Gooch filter + powder in equilibrium in g

Calculate the result to 1 decimal

8. Reproducibility

± 1% relative

9. Remarks

1. All whey powders are more or less hygroscopic, and it is therefore important to determine free water content at the same time as the hygroscopicity.

Hygroscopicity	
<i>Non hygroscopic:</i>	<10%
<i>Slightly hygroscopic:</i>	10.1-15%
<i>Hygroscopic:</i>	15.1-20%
<i>Very hygroscopic:</i>	20.1-25%
<i>Extremely hygroscopic:</i>	>25%

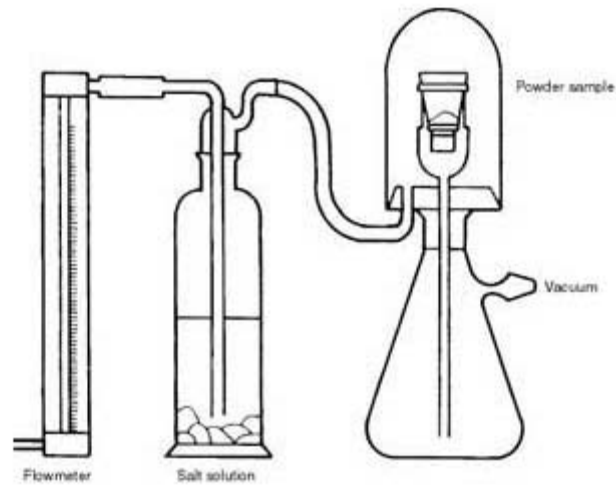
10. Reference

[GEA Niro Research Laboratory](#)

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Hygroscopicity
GEA Niro analytical method A 14 a
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Fig. 1 Apparatus for determination of hygroscopicity



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Degree of Caking

GEA Niro analytical method A 15 a

(Page 1 of 2)

1. Definition

The degree of caking is the amount of powder appearing as lumps which cannot pass through a 500 μ sieve, after allowing the powder first to absorb moisture to equilibrium and then to release moisture by drying.

2. Scope

This method may be used for dried products, especially whey powders.

3. Principle

The powder is allowed to absorb moisture from air with 79.5% relative humidity until the equilibrium is reached. The powder is then dried and sieved under standard conditions. What is left on the sieve is expressed as the degree of caking.

4. Apparatus

1. Analytical balance sensitivity ± 0.1 mg.
2. Drying oven.
3. Desiccator with silica gel or an equivalent drying agent.
4. Shaker for sieves. [Engelsmann](#) (See Method A 8 a).
5. 500 μ sieve, lid and base.
6. Spatula and brush.

5. Reagents

None.

6. Procedure

1. Determine the hygroscopicity (See Method A 14 a).
2. Place the Gooch filter with the wet material in a drying oven at 102°C \pm 2°C for one hour.
3. Cool in desiccator for 1/2 an hour.
4. Transfer the powder quantitatively to a piece of paper by means of the spatula. Weigh powder and paper and transfer the sample to the 500 μ sieve using the brush. Weigh the paper alone.
5. Sieve for 5 min. in the sieving apparatus. Transfer the powder remaining on the sieve to the paper and weigh again.

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Degree of Caking

GEA Niro analytical method A 15 a

(Page 2 of 2)

7. Result

$$\% \text{ Degree of caking} = \frac{b \times 100}{a}$$

a = g of powder used

b = g of powder left on sieve

Calculate the result with 1 decimal.

8. Reproducibility

± 15% relative

9. Remarks

1. The following table may be used for characterisation of the degree of caking:

Degree of caking	
<i>Non-caking powder:</i>	10%<
<i>Slightly caking powder:</i>	10.1-20%
<i>Very caking powder:</i>	>20.1-50%
<i>Very caking powder:</i>	>50%
<i>Extremely caking powder:</i>	100%

2. In some cases it is not possible to remove the hard, glassy cake from the filter. The powder is then said to be 100% caking.
3. It is important to keep the Gooch filter in the humid atmosphere, 79.5% relative humidity, until the drying can be started.

10. Literature

[GEA Niro Research Laboratory.](#)

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Coffee Test

GEA Niro analytical method A 16 a

(Page 1 of 2)

1. Definition

The thermostability in an acid environment is expressed by the number of white particles on the surface after reconstituting the powder in hot coffee.

2. Scope

The method can be used for coffee creamers and milk powders.

3. Principle

The protein stability of powders used for hot beverages is analyzed by adding the powder to hot coffee and determining whether there are flocculated particles on the surface.

4. Apparatus

1. Balance, sensitivity ± 0.01 g
2. pH-meter, capable of reading ± 0.2 pH
3. 250 ml beaker
4. Teaspoon
5. Weighing dish, polystyrene
6. Stop watch
7. Electrical kettle or hot plate
8. Thermometer, $0-100^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$.

5. Reagents

1. Standard instant coffee (Nescafé extra or similar).
2. Deionized water.

6. Procedure

1. Weigh 1.5 ± 0.01 g of coffee into the 250 ml beaker.
2. Weigh 5.0 ± 0.1 g of powder into the weighing dish.
3. Boil approx. 200 ml of water and measure 150 ml into the 250 ml beaker containing the coffee.
Check that the solution has a pH of 4.9-5.4 and adjust the pH with base or acid if it is out of range.

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Coffee Test

GEA Niro analytical method A 16 a

(Page 2 of 2)

4. When the temperature of the coffee has cooled to $80^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$, tip in the 5 g of powder.
When all powder is wetted, start the stop watch and stir with a teaspoon for 30 seconds.
5. The procedure should be carried out in duplicate.

7. Result

Record 0 as passed - if 2 determinations show no particles on the surface.

Record 1 as failed - if 2 determinations show particles on the surface.

8. Reproducibility

N/A

9. Remarks

The results can also be given with information expressing number of particles on the surface.

10. Reference

[GEA Niro Research Laboratory](http://www.niro.com)

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Coffee Sediment Test GEA Niro analytical method A 16 b

(Page 1 of 2)

1. Definition

The coffee sediment is equal to the sum of volumes of sediments after centrifugation of 2 g powder reconstituted in coffee under standardized conditions.

2. Scope

The method is to be used for all dried milk products whether instant or not instant.

3. Principle

A test portion of the sample is added to coffee at 80°C. The mixture is stirred manually. After a specified standing period, the mixture is centrifuged and the amount of sediment is determined.

4. Apparatus

1. Balance, sensitivity 0.01 g.
2. Weighing dish, white polystyrene.
3. 250 ml beaker - external diameter 70 ± 2 mm, height 95 ± 3 mm, calibrated with a mark at 100 ml.
4. Centrifuge - Funke-Gerber Super Quattro, or equivalent. Speed 900 rpm.
5. Centrifuge glasses, 50 ml conical.
6. Stop watch.
7. Teaspoon - height 40 mm, width 29 mm.
8. Thermometer - $0-100^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$.

5. Reagents

1. Standard instant coffee (Nescafé extra or similar).
2. Deionized water.

6. Procedure

1. Weigh 0.8 ± 0.01 g of coffee into the 250 ml beaker, calibrated as described in 4.3.
2. Weigh 2.0 ± 0.01 g of dried milk into the weighing dish.
3. Fill the 250 ml beaker containing the coffee to the 100 ml mark with boiling deionized water.
4. When the temperature of the black coffee has cooled to $80^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$, tip in the 2 g of dried milk sample and start the stop watch.

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Coffee Sediment Test
GEA Niro analytical method A 16 b

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5. After 5 seconds, stir the contents of the beaker with a teaspoon, using 6 clockwise and 6 anti-clockwise stirs over 6 seconds. Circular movements of the spoon should be used, following the side of the beaker. The spoon should touch the bottom of the beaker at all times.
6. After completion of the stirring, allow the contents of the beaker to stand for 10 minutes.
7. Give the contents of the beaker one stir and immediately after pour the contents into two 50 ml centrifuge tubes, and let it stand for 5 minutes.
8. Centrifuge the tubes for 5 minutes at 900 rpm. Record the volume of sediment in each tube to the nearest 0.05 ml if the volume is < 0.5 ml, and to the nearest 0.1 ml if the volume is > 0.5 ml.
9. The procedure should be carried out in duplicate.

7. Results

1. The sediment is equal to the sum of volumes of sediments in the two centrifuge tubes.
2. Any abnormalities such as colour or a fatty appearance on the surface of the coffee after step 6.6 should be noted.

8. Reproducibility

Two determinations on the same sample should not differ by more than 0.05 ml

9. Remarks

If another coffee type is used instead of the specified, report the type and the pH of the coffee together with the results.

10. Literature

- [GEA Niro Research Laboratory](#)
- Ministry of Agriculture and Fisheries Dairy Division (1977), Standard Chemical Methods, 16.1 ADMI Solubility Index. 2nd ed. Wellington NZ.
- NZDRI 4.10.1 Coffee sediment test. September 1990.
- IDF Provisional International Draft 2.

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Foam Test

GEA Niro analytical method A 17 a

(Page 1 of 2)

1. Definition

The foam is the height of the foam layer on top of the liquid in mm after 3 and 15 minutes

2. Scope

The method is used for foaming agent powders, e.g. cappuccino powder.

3. Principle

A foaming agent powder is mixed with coffee, sugar and water. The foam layer is evaluated after a given time.

4. Apparatus

1. Balance - ± 0.1 g
2. 250 ml beaker - external diameter 70 ± 2 mm, height 95 ± 3 mm, calibrated with a mark at 100 ml.
3. Teaspoon - (height 40 mm, width 29 mm)
4. Stop watch
5. Thermometer - $0-100^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

5. Reagents

1. Standard instant coffee (Nescafé extra or similar)
2. Sugar - granulated sugar beet ('Dansukker melis, De danske Sukkerfabrikker')

6. Procedure

1. Dry mix 6.0 ± 0.1 g of foaming agent powder, 3.6 ± 0.1 g of sugar and 2.4 ± 0.1 g of instant coffee in a 250 ml beaker.
2. Add water at $80 \pm 1^{\circ}\text{C}$ to the 150 ml mark of the beaker.
3. Immediately mix the content of the beaker with a teaspoon for 10 seconds using 2 sets of:

6 complete revolutions clockwise

6 complete revolutions anti-clockwise.
4. Read the height of the foam layer on top of the liquid in mm after 3 and 15 minutes.

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Foam Test

GEA Niro analytical method A 17 a

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7. Result

The results are reported as the height in mm after 3 and 15 min.

The presence of lumps etc. in the foam should also be reported.

8. Reproducibility

± 0.5 ml

9. Remarks

N/A

10. Reference

[GEA Niro Research Laboratory](#)

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a-Lactose and Total Lactose
GEA Niro analytical method A 18 a

(Page 1 of 4)

1. Definition

The content of α -lactose and total lactose is determined on the basis of 2 polarimetric readings.

2. Scope

The method can be used on milk and whey powder

3. Principle

The proportion of α -lactose (of total lactose) is determined on the basis of 2 polarimetric readings. The first optical rotation of the clear filtrate is taken under such conditions that the temperature of the solution is kept low during the whole procedure to prevent mutarotation. The second reading is made after the equilibrium has been reached.

The analysis must be carried out in a cold room, with a temperature not higher than 5°C.

4. Apparatus

1. Analytical balance, capable of weighing to 0.1mg.
2. Polarimeter with 200 mm polarimetric tube and a sodium lamp - sensitivity 0.01°.
3. Water bath, capable of maintaining a temperature at 80°C \pm 1°C.
4. Porcelain mortar with pestle, diam. approx. 10 cm.
5. Glass funnels diam. 7-8 cm.
6. Pleated filter paper.
7. 100 ml volumetric flask.
8. 250 ml Erlenmeyer flask.
9. 5 and 10 ml graduated cylinders.
10. 1000 ml beaker (for making an ice bath).

5. Reagents

1. 2.5% tannin solution.
Add 2.5 \pm 0.01g tannic acid powder (C₇₆H₅₂O₄₆) into a 100ml volumetric flask and dilute with deionized water.
2. 10% lead acetate solution.

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a-Lactose and Total Lactose
GEA Niro analytical method A 18 a(Page 2 of 4)

Add 10.0 ± 0.01 g lead acetate trihydrate ($C_4H_6O_4 Pb, 3H_2O$) into a 100 ml volumetric flask and dilute with deionized water.

6. Procedure

All the glass ware and reagents must be kept in a cold-storage room or in a refrigerator overnight before analyzing.

1. Weigh the amount of sample that corresponds to approx. 1-1.5 g of milk sugar (if only the proportion of α -lactose has to be determined, weighing is not necessary).
2. Transfer the sample into a mortar, and grind it to a smooth paste with approx. 10 ml of deionized water.
3. Dilute the sample with 10-15 ml of deionized water, and transfer it quantitatively by means of a glass funnel into a 100 ml volumetric flask.
4. Add 5 ± 0.1 ml of tannin solution and 10 ± 0.1 ml of lead acetate solution. Mix the contents and fill up to 100 ml.
5. Mix the solution carefully, and filter through a dry paper filter, into an Erlenmeyer flask. Re-filter the first few ml of the filtrate, as these are usually unclear.
6. Fill a pre-cooled polarimetric tube with the filtrate and close it. Insert in the polarimeter and make the reading within a couple of minutes.
7. Keep the rest of the filtrate in a water bath at $80^\circ C \pm 1^\circ C$ for 30 minutes.
8. Cool the filtrate to $5^\circ C$. Fill a polarimetric tube and make a second reading. The cooling can take place in a 1000 ml beaker containing ice water.

7. Results

The proportion of α -lactose in % of total lactose (TL) content is expressed as follows:

First polarimetric reading	P_1
Second polarimetric reading	P_2
α -lactose content of total lactose	% α_{TL}

$$\% \alpha_{TL} = \left(\frac{P_1}{P_2} \times 0.623 \right) \times 100.54$$

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a-Lactose and Total Lactose
GEA Niro analytical method A 18 a

(Page 3 of 4)

It is possible to calculate other values as follows:

% total lactose (as anhydride)	% TL
% amorphous lactose	% AL
% α-lactose-monohydrate (as anhydride)	% α L
% water of crystallisation	% H ₂ O cryst.
% α-lactose-monohydrate (as monohydrate)	% α L _{H₂O}
Degree of crystallisation of the powder (crystallised lactose of total lactose)	% cryst.

$$\% TL = 90.25 \times \frac{P_2}{W} \quad \text{where} \quad W = \text{g sample}$$

$$\% AL = \frac{(100 - \% \alpha TL) \times \% TL}{100} \times y \quad \text{where} \quad y = \frac{1+x}{x}$$

$$\% \alpha L = \% TL - \% AL \%$$

$$\% H_2O \text{ cryst.} = \frac{\% TL - \% AL}{19}$$

$$\% \text{ cryst.} = \frac{\% \alpha L}{\% TL} \times 100$$

$$\% \alpha L_{H_2O} = \% \alpha L + \% H_2O \text{ cryst.}$$

where 'x' expresses the proportion of β-lactose to α-lactose at the temperature of the concentrate prior to drying. The value of 'x' at various temperatures is as follows:

Temp.°C	0	10	20	25	30	40	50
X = (β:α)	1.65	1.62	1.59	1.58	1.57	1.54	1.51
$y = \frac{1+x}{x}$	1.61	1.62	1.63	1.63	1.64	1.65	1.66

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a-Lactose and Total Lactose
GEA Niro analytical method A 18 a

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8. Reproducibility

N/A

9. Remarks

The analysis must be carried out in a cold room, with a temperature not higher than 5°C.

10. Reference

- [GEA Niro Research Laboratory](#)
- Landbrugsministeriet (Danish Ministry of Agriculture): Arbejdsmetoder for kemiske undersøgelser af mælk og mejeriprodukter (1958).

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Total Lactose in Milk and Whey Powders (Gravimetric Method)

GEA Niro analytical method A 18 b

(Page 1 of 4)

1. Principle

The method is based on lactose reducing a cupric salt complex to Cu₂O under specified conditions. The amount of Cu₂O precipitated is proportional to the lactose content.

2. Scope

The method is used on all milk and whey powders.

3. Apparatus

1. 500 ml volumetric flask.
2. 2 x 500 ml brown glass bottles.
3. Pipettes - 25 and 50 ml.
4. 400 ml graduated beaker.
5. Bunsen burner or hot plate.
6. Wire gauze with a ceramic centre.
7. Filter paper.
8. Glass funnel.
9. Stop watch.
10. Watch glass.
11. Glass spatula.
12. Glass filter crucible, porosity 4.
13. Disposable weighing dish
14. 10 and 25 ml graduated cylinders.
15. Vacuum pump.
16. Analytical balance, capable of weighing 0.1 mg.
17. Desiccator.
18. Drying oven without forced air circulation, and with a thermostatic control capable of maintaining the $C \pm 1^\circ C$ temperature at 100

4. Chemicals

1. CuSO₄, 5H₂O, p.a., Merck.
Copper(II)sulfate: R 22 and S 24.
2. C₄H₄Na₂O₆, H₂O, p.a., Merck.
Potassium sodium tartrate.
2. NaOH, p.a., Merck.
Sodium hydroxide pellets: R 35 and S 2-26-37/39.
3. KOH, p.a., Merck.
Potassium hydroxide, pellets: R 35 and S 2-26-37/39.
4. 96% Ethanol: R 11 and S 7-16.
5. Diethyl ether, technical: R 12-19 and S 9-16-29-33.
R ≈ DK risk sentences
S ≈ DK safety sentences

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Total Lactose in Milk and Whey Powders (Gravimetric Method)**GEA Niro analytical method A 18 b**

(Page 2 of 4)

5. Reagents

1. Fehling I.
Copper(II)sulphate solution.
Dissolve 34.639 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in deionized water.
Dilute to 500.0 ml in a volumetric flask, and filter through a paper filter.
Store in a 500 ml brown glass bottle.
2. Fehling II.
Alkaline tartrate solution.
Dissolve 173.0 ± 0.1 g $\text{C}_4\text{H}_4\text{Na}_2\text{O}_6$, H_2O and 50.0 ± 0.1 g NaOH pellets in deionized water and dilute to 500 ml in a volumetric flask.
Allow to stand for 2 days, before filtering through a paper filter.
Store in a 500 ml brown glass bottle.
3. Potassium hydroxide solution.
Dissolve 15.567 g KOH in deionized water. Dilute to 500.0 ml in a volumetric flask.

6. Procedure

1. Weigh out according to type of powder ± 0.05 g:
whey 1.60 g
skim milk 2.00 g
whole milk 2.80 g
2. Dissolve the powder in approx. 200 ml 60°C deionized water in a 500 ml volumetric flask. Invert the solution until all powder is dissolved, and cool to 20°C.
3. Add 10 ml of Fehling I solution and 7.5 ml (measuring pipette) KOH solution (the solution must still be acidic, check with pH paper), and dilute to 500.0 ml.
4. Mix carefully and filter through a dry filter.
5. Pipette 25.0 ml of Fehling I solution and 25.0 ml of Fehling II solution into a 400 ml beaker.
6. Add 25 ml of the filtrate (F4) and 25 ml of deionized water.
7. Cover the beaker with a watch glass and heat it over a Bunsen burner or a hot plate. The heat must be regulated so boiling begins after 4 minutes. Continue boiling for exactly 2 minutes. It is important that these regulations are strictly maintained. For this purpose it is recommended to make a preliminary test, using 50 ml deionized water and 50 ml reagent.
8. Filter the solution immediately through a dried and weighed glass filter crucible by means of suction.
9. Transfer the precipitated Cu_2O quantitatively to the glass filter crucible, and wash it carefully, first with 60°C deionized water, then with 10 ml alcohol and finally with 10 ml of ether.
10. Dry the precipitate in an oven at 100°C for 30 minutes, cool in a desiccator and weigh.
11. Carry out a blank test according to F5-F10 using deionized water instead of reducing sugar filtrate. If the weight of the Cu_2O obtained in the blank is more than 0.5 mg, correct the results of reducing sugar determination accordingly.

7. Result

Use the Hammond Table to express the weight of lactose equivalent to the weight of Cu_2O .

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Total Lactose in Milk and Whey Powders (Gravimetric Method)

GEA Niro analytical method A 18 b

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$$\& \text{ lactose in powder} = \frac{A \times 500 \times 100}{W \times ml \times 1000}$$

A = mg lactose equivalent to the weight of Cu₂O as found in the table.

W = weight of milk powder

ml = ml filtrate taken with pipette

8. Reference

- [GEA Niro Research Laboratory](#)
- Landbrugsministeriet (Danish Ministry of Agriculture): Arbejdsmetoder for kemiske undersøgelser af mælk og mejeriprodukter (1958).

Hammond table for calculating lactose values

Expressed in mg

Cu ₂ O	Lactose, H ₂ O	Cu ₂ O	Lactose, H ₂ O
20	13,6	76	51,7
30	20,2	77	52,4
40	27,2	78	53,0
50	34,0	79	53,7
51	34,7	80	54,4
52	35,4	81	55,1
53	36,0	82	55,8
54	36,7	83	56,4
55	37,4	84	57,1
56	38,1	85	57,8
57	38,8	86	58,5
58	39,4	87	59,2
59	40,1	88	59,8
60	40,8	89	60,5
61	41,5	90	61,2
62	42,2	91	61,9
63	42,9	92	62,6
64	43,5	93	63,2
65	44,2	94	63,9
66	44,9	95	64,6
67	45,6	96	65,3
68	46,2	97	66,0
69	46,9	98	66,6
70	47,6	99	67,3
71	48,3	100	68,0

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Total Lactose in Milk and Whey Powders (Gravimetric Method)

GEA Niro analytical method A 18 b

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72	49,0	150	102,3
73	49,6	200	136,6
74	50,3	250	171,1
75	51,0	300	205,7

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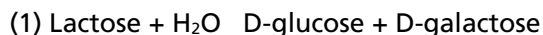
Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

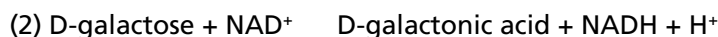
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1. Principle

Lactose is hydrolyzed to D-glucose and D-galactose at pH 6.6 in the presence of the enzyme β -galactosidase and water (1).



D-galactose is oxidized at pH 8.6 by nicotinamide-adenine dinucleotide (NAD) to D-galactonic acid in the presence of the enzyme β -galactose dehydrogenase (Gal -DH) (2).



The amount of NADH formed in reaction (2) is stoichiometric to the amount of lactose and D-galactose respectively. The increase in NADH is measured by means of its light absorbance at 340 nm.

2. Scope

The method is to be used for milk powders, liquid milk and milk concentrates.

3. Apparatus

1. Spectrophotometer Perkin Elmer, Lambda 2, UV/VIS.
2. Analytical balance - capable of weighing to 0.0001 g.
3. Water bath - thermostatically controlled to $70^\circ\text{C} \pm 1^\circ\text{C}$.
4. 100 ml volumetric flask.
5. 50 ml Erlenmeyer flask with stopper.
6. Glass funnel - short stem 65 mm, diameter 50 mm.
7. Pleated filter paper - 11 cm, S&S No. 589.
8. Micropipette (Socorex) & microtip.
9. Volumetric pipettes 5 - 7 - 10 ml.
10. Weighing dish, disposable.
11. Disposable cuvettes, Brand Cat. No. 7590 05.
12. Parafilm "M"

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Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

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4. Chemicals

1. Lactose/D-galactose UV-Test No. 176 303, Boehringer Mannheim.
 - 1.1 Bottle 1 containing 600 mg of lyophilisate, citrate buffer pH 6.6, NAD approx. 35 mg, magnesium sulphate and stabilizers.
This solution is stable for 3 months at 4°C.
 - 1.2 Bottle 2 containing 1.7 ml of a β -galactosidase suspension.
 - 1.3 Bottle 3 containing 34 ml of a solution of potassium diphosphate buffer, pH 8.6 and stabilizers.
 - 1.4 Bottle 4 containing 1.7 ml of a galactose dehydrogenase suspension.
 - 1.5 Bottle 5 containing lactose standard.
2. Zinc sulphate heptahydrate, p.a. Merck.
ZnSO₄, 7 H₂O: R 36 and S 24.
3. Potassium hexacyanoferrate(II) trihydrate, p.a.
Merck. K₄ (Fe(CN)₆), 3H₂O .
4. Sodium hydroxide Titrisol 0.1 N, p.a. No. 9956 Merck.
NaOH: R 36/37 and S 26.

R ≈ DK risk sentences.

S ≈ DK safety sentences.

5. Reagents

1. Solution 1:
Dissolve contents of bottle 1 with 7.0 ml of deionized water.
Solution 1 is stable for 3 months at 4°C.
 2. Suspension 2:
Use bottle 2.
 3. Solution 3:
Use bottle 3.
 4. Suspension 4:
Use bottle 4.
- Bring all solutions and suspensions to room temperature before use.

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Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

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5. Carrez-I solution:
Dissolve 3.60 g of potassium hexacyanoferrate-II in 100 ml of deionized water.
6. Carrez-II solution:
Dissolve 7.20 g of zinc sulphate in 100 ml of deionized water.
7. 0.1 N NaOH:
Dilute 0.1 N Titrisol to 1000 ml with deionized water.

6. Procedure

1. Weigh out an amount of sample corresponding to a final expected concentration of lactose and D-galactose between 0.2 and 1.0 g/l.
If the amount is unknown, weigh out 0.2 to 0.4 g with an accuracy of 0.1 mg in a 100 ml volumetric flask.
2. Add approx. 60 ml water and incubate for 15 min. at 70°C in a water bath; shake from time to time.
3. Add 5 ml of Carrez-I, 5 ml of Carrez-II and 10 ml of 0.1 mol/l NaOH. Shake vigorously after each addition.
4. Adjust to room temperature and fill up to the mark with water.
5. Mix the solution and filter it through a paper filter into an Erlenmeyer flask. Re-filter the first few ml of the filtrate, as these are usually unclear.
6. Carry out a blank test, proceeding as described above, using all the reagents but omitting the sample.
7. Add the amounts specified in the scheme to disposable cuvettes. Treat the blank test of the reagent as a sample. Read absorption against water using the flow-through cuvette.

Pipette into cuvettes	Blank lactose samples ml	Lactose sample ml	Blank D-galactose sample ml	D-galactose sample ml
Solution 1	0.200	0.200	0.200	0.200
Suspension 2	0.050	0.050	-	-
Sample solution	-	0.100	-	0.100
Mix and incubate for 20 minutes and add:				

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Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

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Solution 3	1.000	1.000	1.000	1.000
Deionized water	2.000	1.900	2.050	1.950
Mix and read the absorbances (A_1) for the solutions after approx. 2 minutes. Start the reaction by addition of:				
Suspension 4	0.050	0.050	0.050	0.050
Mix and wait until the reaction has stopped (approx. 15 min.) and read the absorbances (A_2) of the solutions. If the reaction has not stopped after 15 minutes, continue to read the absorbances at 5 minute intervals until the absorbance increases constantly over 5 min.				

8. Determine the absorbance differences ($A_2 - A_1$) for both blank and sample. Subtract the absorbance difference of the blank from the absorbance difference of the corresponding sample:

$$\Delta A = (A_2 - A_1)_{\text{sample}} - (A_2 - A_1)_{\text{blank}}$$

It follows for: $\Delta A_{\text{D-galactose}}$ (from 'D-galactose sample')

and $\Delta A_{\text{lactose + D-galactose}}$ (from 'lactose sample')

The difference of these values stands for $\Delta A_{\text{lactose}}$

9. The measured absorbance differences should, as a rule, be at least 0.100 absorbance units to achieve sufficiently accurate results. If that is not the case, adjust the sample volume or weigh in a larger amount of the sample. If the absorbance difference is too high, dilute the sample further.
10. If the reagent blank shows significant amounts of lactose or D-galactose, correct the sample results.

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Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

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7. Result

$$c \text{ in } \frac{g}{l} = \frac{V \times MW \times F}{\varepsilon \times d \times v \times 1000} \times \Delta A$$

c = concentration, g/l sample solution

V = final volume, ml

MW = molecular weight g/mol

F = dilution factor

v = sample volume, ml

d = light path, cm

ε = extinction coefficient of NADH at 340 nm = 6.3 ($l \times mmol^{-1} \times cm^{-1}$)

ΔA = absorbance difference of sample minus absorbance difference of blank

Lactose:

$$c \text{ in } \frac{g}{l} = \frac{3.300 \times 342.3 \times F}{6.3 \times 1.00 \times 0.100 \times 1000} \times \Delta A_{lactose}$$

$$c \text{ in } \frac{g}{l} = 1.793 \times F \times \Delta A_{lactose}$$

$$c \text{ in } \% = \frac{17.93 \times F \times ml}{w} \times \Delta A_{lactose}$$

D-galactose:

$$c \text{ in } \frac{g}{l} = \frac{3.300 \times 180.16 \times F}{6.3 \times 1.00 \times 0.100 \times 1000} \times \Delta A_{D-galactose}$$

$$c \text{ in } \frac{g}{l} = 0.9437 \times F \times \Delta A_{D-galactose}$$

$$c \text{ in } \% = \frac{9.437 \times F \times ml}{w} \times \Delta A_{D-galactose}$$

where

F = dilution factor

ml = original dilution, ml

w = weight of milk powder

?A = absorbance difference of sample minus absorbance difference of blank

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Lactose and D-Galactose (Enzymatic method)

GEA Niro analytical method A 18 c

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If the sample has been diluted during preparation, the result must be multiplied by the dilution factor F.

Detection limits of 0.2 mg/l for lactose and 1 mg/l for D-galactose are derived from the absorbance difference of 0.010 measured at 340 nm and a maximum sample volume $v = 0.5$ ml.

In a double determination using one sample solution (1 g diluted to 100 ml) a difference of 0.05 - 0.1% for D-galactose and 0.15 - 0.25% for lactose can be expected.

If the sample is diluted further during sample preparation, the expected difference has to be multiplied by the dilution factor.

8. Remarks

1. Control the method by analysing the standard samples frequently.
2. If the conversions of lactose and of D-galactose have been completed according to the time given in the scheme, it can be concluded (in general) that no interference has occurred. If interference is suspected, use the standard addition for control or consult the reference.

9. Reference

- Methods of Biochemical Analysis and Food Analysis, Boehringer Mannheim
- [GEA Niro Research Laboratory](#)

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Titrateable Acidity

GEA Niro analytical method A 19 a

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1. Principle

The titrateable acidity is expressed as % lactic acid and is determined by titration of a known amount of reconstituted milk with 0.1 N NaOH using phenolphthalein as indicator.

2. Scope

This method may be applied for all kind of dried milk products.

3. Apparatus

1. Analytical balance ± 0.1 mg
2. Methrom autoburette
3. Solubility index mixer, Snijders, The Netherlands.
Speed 3800-4000 rpm
4. 100 ml Erlenmeyer flask
5. 20 ml pipette, other sizes may be used

4. Chemicals

1. Titrisol, 0.1 N NaOH - R 35, and S 26, 27, 37/39
R \approx DK risk sentences
S \approx DK safety sentences
2. Phenolphthalein
3. 96% Ethanol

5. Reagents

1. 0.1 N NaOH.
Dilute the Titrisol solution to 1 litre. Standard Method no. R-7.1
2. 1 % Phenolphthalein solution
Dissolve 1g of phenolphthalein in 50 ml 96% ethanol and dilute to 100 ml with deionized water.

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Titrateable Acidity

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6. Procedure

1. Disperse and dissolve the following amount of powder in 100 ml of deionized water using the mixer.

Powder:

Skim or buttermilk:	10 g
Whole milk:	13 g
Whey:	6 g

2. Allow the mixture to stand for approx. 1 hour, stir gently.
3. Pipette 20 ml into a 100 ml Erlenmeyer flask.
4. Add 0.5 ml of phenolphthalein and titrate with 0.1 N NaOH until a faint pink colour persists for 30 sec.

7. Result

$$\% \text{ titrateable acidity} = \frac{ml \times N \times 90 \times 100}{V \times 1000}$$

Where:

ml = ml 0.1 NaOH used

N = Normality of 0.1 N NaOH

V = ml milk solution used

Titrateable acidity is expressed as % lactic acid, (CH₃-CHOH-COOH, MW = 90)

Reproducibility : ± 0.01% lactic acid

8. Remarks

1. Ref. 1 (ADMI) prescribes that exactly 17.6 ml of milk solution is used. If that is the case the Titrateable Acidity can be calculated by dividing ml 0.1 N NaOH by 20.
2. Adjust the amount of milk used in the titration until a reasonable amount of base is used.

9. Reference

- ADMI, Standards for grades of dry milk, bulletin 916, revised 1990

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Titrateable Acidity

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GEA Niro

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Whey Protein Nitrogen Index GEA Niro analytical method A 21 a

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1. Principle

The undenaturated Whey Protein Nitrogen Index (WPNI) is a measure of the heat treatment applied to the milk during processing to milk powder. It is the basis of the following heat classifications:

Class	WPNI
High heat powder	< 1.5
Medium heat powder	> 1.5 - < 6.0
Low heat powder	> 6.0

WPNI is expressed as milligrams (mg) undenaturated whey protein nitrogen per gram of non-fat milk powder with a moisture content of 3.16%.

Casein and the heat denaturated whey proteins are removed by filtration after precipitation of the reconstituted milk with NaCl. The filtrate contains all undenaturated whey proteins. By adding HCl, the proteins denature and develop a turbidity depending on the concentration of the whey proteins. The turbidity is measured as % transmittance, in a spectrophotometer at a wave length of 420 nm.

By using the standard curve, this reading can be converted directly into mg undenaturated whey protein N/g powder (WPNI).

2. Scope

The method is to be used for milk powders, liquid milk and milk concentrates.

3. Apparatus

1. Spectrophotometer - Perkin Elmer, Lambda 2, UV / VIS.
2. Analytical balance - capable of weighing to 0.01 g.
3. Water bath - thermostatically 0.5° C.±controlled to 37
4. Electrical heat plate.
5. Vibrator - Vibrofix VF 1, Electronic.
6. Test tube - 25 x 150 mm round bottom, Pyrex or Kimax, or soft glass.
Rubber stoppers.
7. Glass funnel - short stem 65 mm, diameter 50 mm.
Glass funnel - short stem 65 mm, diameter 90 mm.
8. 15 ml specimen tube with stopper - socket size 14/23.
9. Pleated filter paper - 9 cm, S & S No. 602.
Pleated filter paper - 15 cm, S & S No. 605.
10. Volumetric pipettes - 1 - 2 - 3 - 4 - 5 - 10 - 20 -100 ml.
Disposable pipette.
11. 100 ml volumetric flask.
12. 50 ml + 500 ml Erlenmeyer flasks.
13. 2000 ml graduated beaker.
14. 2 l bottle with a lid.
15. Stop watch.
16. 60 mm + 90 mm watch glasses.

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Whey Protein Nitrogen Index

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17. Weighing dish, disposable.

4. Chemicals

1. Hydrochloric acid, fuming, p.a. Merck.
Conc. HCl 37%. R 34-37 and S 2-26.
2. Sodium chloride (NaCl), p.a., Merck.
Do not use NaCl containing anti-caking agents.
3. Standard reference samples.
Low heat and high heat non-fat dry milk (NDM).
(Available from the American Dairy Products Institute)

R ≈ DK risk sentences
S ≈ DK safety sentences

5. Reagents

1. HCl solution (10 g/100 ml).
Add 23.0 ± 0.1 ml conc. HCl to 77 ml deionized water.
2. NaCl, saturated.
Add 1 kg of NaCl to 2 l of deionized water and heat the mixture to just below boiling point while stirring to ensure complete saturation. After cooling to room temperature, filter the solution through S & S No. 605 pleated filter paper.

6. Standard Curve

1. Reconstitute 20 ± 0.01 g of both standards with 200.0 ml of deionized water in 500 ml Erlenmeyer flasks.
2. Add 80 g of NaCl to saturate the reconstituted milk. Stopper the Erlenmeyer flask, shake for 1 min., and incubate for 30 min. at $37^\circ\text{C} \pm 0.5^\circ\text{C}$ in a water bath. Turn the solution upside down 8-10 times during the first 15 min. to insure complete saturation. Do not touch the solution in the last 15 min. of the incubation period.
3. Without cooling, filter the solution through S & S No. 605 pleated filter paper. If the first portion of the filtrate is cloudy, re-filter through the same filter paper. Continue filtration until approx. 100 ml of filtrate have been collected. Cover the funnel with a watch glass during filtration, to prevent evaporation.
4. Pipette proportions of low heat and high heat filtrates into 25 x 150 mm tubes as follows:

Tube No.	Low heat filtrate, ml		High heat filtrate, ml
1	10	+	0
2	8	+	2
3	6	+	4
4	4	+	6
5	2	+	8
6	0	+	10

5. Stopper the tubes with the combined filtrates, and mix carefully by inverting twice. Continue as described in 7.5 - 7.10
6. Make duplicate determinations of each point.

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Whey Protein Nitrogen Index GEA Niro analytical method A 21 a

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7. Check that each measurement is within the confidence limit of the existing standard curve. If that is the case continue to use the curve, if not, calculate the linear regression curve that fits the numbers measured with the new standard samples. Stat Graphic program or equivalent is suitable.
An example of a standard curve is shown in Fig. 1 and Fig. 2.
8. New standard samples have to be bought every 6 months and used for control of the standard curve.

7. Procedure

1. The method uses 2.0 g of non-fat dry powder.
For milk powders containing more than 1% fat, a correction in the amount of sample used for analysis is required.

$$\text{Amount of powder for analysis} = \frac{20 \times 100}{(100 - \%fat)}$$

% fat can be determined by using Gerber Method, GEA Niro Method A 9 b

2. Reconstitute the milk powder in 20.0 ml of deionized water, in a 25 x 150 mm test tube. Make duplicates from each sample. Use the Vibrofix until all milk powder is dissolved.
3. Add 8.0 ± 0.1 g of NaCl, stopper, and place the test tube in a water bath at $37 \pm 0.5^\circ\text{C}$ for 30 min. Shake the content of the tube 8-10 times during the first 15 min. to ensure complete saturation of the milk solution with NaCl.
4. Without cooling, shake the mixture to facilitate pouring, and filter through S & S No. 602 filter paper. If the first portion of the filtrate is cloudy, re-filter through the same filter paper. Collect approx. 5 ml of the filtrate in a 50 ml Erlenmeyer flask.
5. Pipette 2 x 1.0 ml of the filtrate, one for a sample and one for a blind determination, into two 15 ml tubes. Dilute the filtrates with 10 ml of saturated NaCl solution. Stopper the tube and mix by slowly inverting once.
6. Add 2 drops of HCl solution (use a disposable pipette) to the sample (not the blind sample) for developing a turbidity. Stopper the tube and mix the acid with the diluted filtrate by slowly inverting twice. Do it very carefully to prevent formation of foam.
7. Adjust the spectrophotometer to 100% transmittance by using the blind sample, with the wavelength set at 420 nm.
8. Rinse carefully with deionized water between each transmittance reading.
9. Within 5-10 min. after adding the HCl solution, invert the tube once again and measure the turbidity on the spectrophotometer with the wavelength set at 420 nm.
10. Before measuring the duplicate sample, check the 100% transmittance reading with the corresponding blind sample.

8. Result

1. Transmittance readings on the duplicates must not differ by more than 2%. If they do, another pair of filtrates must be analyzed, and the average of 4 determinations is used as the final value.
2. To obtain a value for serum protein nitrogen, use the Standard Curve. Report the result as: mg WPN/g powder NDM.
3. Whey Protein Nitrogen (WPN) values obtained by a modified Kjeldahl analysis on standard reference samples are corrected to a 3.16% moisture content. Therefore measured WPN

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values have to be corrected to the same water content.

$$WPN\text{I at } 3.16\% H_2O = \frac{(100 + (\%A - 3.16\%) \times (WPN))}{100}$$

A = % moisture in the sample (GEA Niro Method A 1 a)

9. Remarks

1. When producing skimmed milk powder with a certain heat-treatment specification, it is necessary to know the whey protein content in the raw skim milk as well as the heat stability of the whey proteins. Both are influenced by seasonal variations. Therefore the heat treatments applied to the milk prior to evaporation must differ through the year in order to meet required specifications.
2. Liquid skimmed milk, skim milk concentrates, whole milk powder etc.:
The amount of sample and water for step 7.1 must be calculated in such a way to obtain approx. the same amount of non-fat solids and water in the sample as when dissolving 2.0 g of skim milk in 20 ml of water. The result found is expressed in mg WPN/1 g of skim milk powder and can be used directly for heat classification.
3. Whey powder:
Use 1.0 g of whey powder and 20 ml of water for step 7.1 and multiply the result of step 8.3 by 2.
The result is expressed as mg WPN/1 g powder. In the case of whey powder express the degree of denaturation in %. Multiply the result by 0.638 to obtain the amount of undenaturated whey protein of the powder in %. Simultaneously determine the total protein content by using the Kjeldahl method, and calculate the % of denaturation.
4. It is recommended not to filtrate the milk for more than approx. 2 hours, as there is a risk of crystallization in the residue.

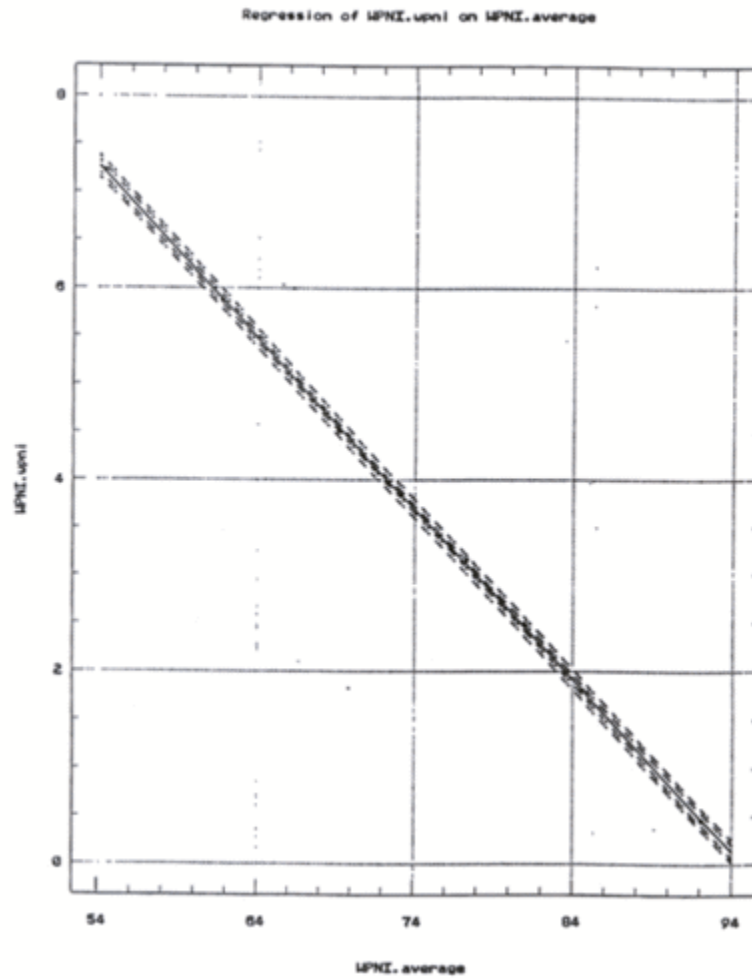
10. Reference

1. ADMI, Standards for grades of dry milk, Bulletin 916, revised 1990.
2. Modification of the Harland-Ashworth method, published by Kuramoto, Jeness, Coulter and Choi. Journal of Dairy Science. 42:28. 1959.

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Fig. 1 Example of standard curve



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Fig. 2. Calculation of Standard Curve

Regression Analysis - Linear model: $f(x) = a+bx$

Dependent variable: WPNI. wpni Independent variable: WPNI. average

Parameter	Estimate	Std. error	T-value	Probe level
Intercept	16.8753	0.0765988	220.308	0.00000
Slope	-0.178078	1.0303 SE-3	-172.827	0.00000

Analysis of variance

Source	Sum of squares	Df	Mean square	F-Ratio	Probe level
Model	32.011	1	32.011	29869.26	0.00000
Residual	0.0042868	4	0.0010717		
Total (corr.)	32.015400	5			

Correlation coefficient = - 0.999933

R-squared = 99.99%

Standard error of estimate = 0.0327369

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Ash Content**GEA Niro analytical method A 25 a**(Page 1 of 2)

1. Principle

Samples are ignited in a heating furnace at 525°C for approx. 16 hours.

2. Scope

The method is to be used for milk powders.

3. Apparatus

1. Carbolite heating furnace max. 1100°C or equivalent.
2. Analytical balance, capable of weighing to 0.1 mg.
3. Porcelain crucibles.

4. Procedure

1. Ignite a porcelain crucible.
2. Cool the crucible till room temperature and weigh.
3. Weigh out 1-5 g of sample, and transfer to the pre-ignited crucible.
4. Heat up the sample carefully with a bunsen burner until all is calcined.
Avoid that the product catches fire as some of the material may burn off.
5. Put the sample into the heating furnace overnight at 525°C, or until it is carbon-free.
6. Cool in desiccator and weigh. Do not put too many crucibles in one desiccator as it will prolong the cooling time.
7. All measurements are to be made in duplicate.

5. Result

$$\% \text{ ash} = \frac{a - b}{c - b} \times 100$$

a = weight of crucible + dry sample

b = weight of crucible

c = weight of sample and crucible

Two determinations must not differ more than 5 % relative.

Specify temperature and time with each result.

6. Remarks

1. If the sample is still black after approx. 16 hours, a few drops of nitric acid can be added after cooling. After another 2-3 hours in the heating furnace, the sample should be C-free.

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Ash Content

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7. Reference

1. Official methods of analysis of the Association of Official Analytical Chemists, Washington, DC 20044, 1970.
2. Own experience.

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Flowability GEA Niro analytical method A 23 a

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1. Principle

Flowability is the ability of a powder to flow, and it is measured as the time in seconds necessary for a given volume of powder to leave a rotary drum through a slit of a certain size.

2. Scope

This method may be used for all powders, dairy and non-dairy products.

3. Apparatus

1. Motor with gear suitable for running at 30 rpm.
2. Stainless steel drum as shown in Fig. 1.
3. Stop watch.

4. Procedure

1. Determine the bulk density tapped 100 times in g/cm^3 by using GEA Niro Analytical Method A 2 a.
2. Weigh out an amount of powder ± 0.1 g corresponding to:
 $A = B \times 25$
A = amount of powder in g
B = bulk density in g/cm^3 tapped 100 times.
3. Pour the powder into the drum and put on the lid.
4. Start the drum (set at 30 rpm) and the stop watch simultaneously.
The drum should rotate so that the powder that is not leaving the drum through the slits is broken up by the metal rod 24° later.
This is very important when measuring sticky products.
5. When the last powder has left the drum, stop the watch and record the time.
6. The determination has to be made in duplicate.

5. Result

Flowability = average time in sec.

Record the results in sec.

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Flowability
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Two determinations must not differ more than shown below, if they do, repeat the analysis.

Flowability	Reproducibility
< 30 sec.	20% relative
> 30 sec.	10% relative

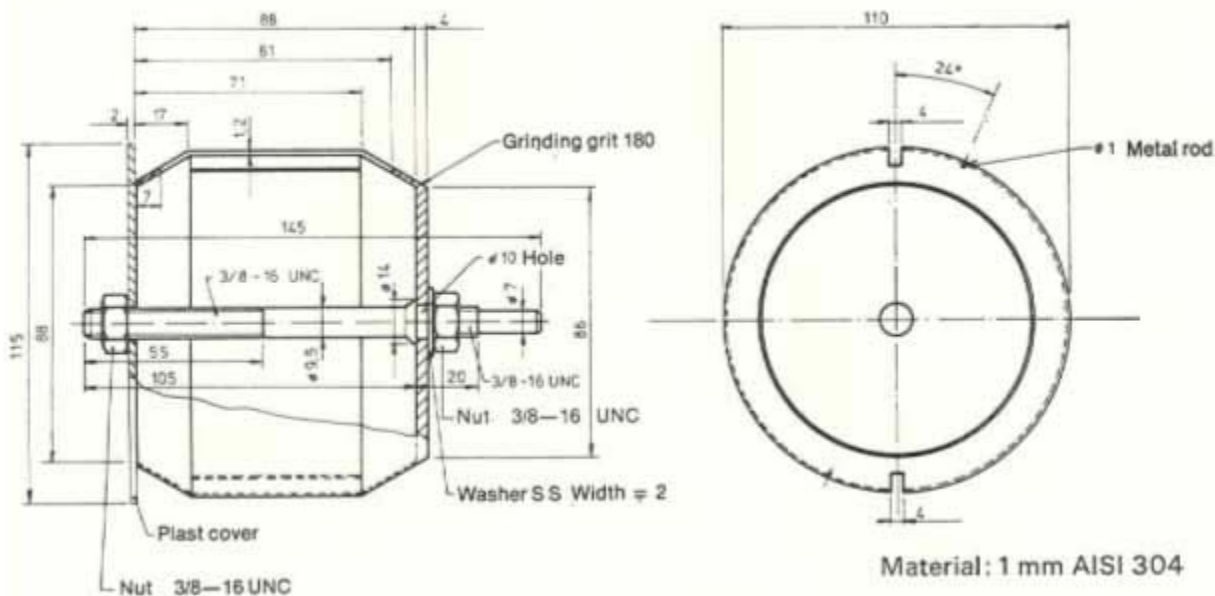
6. Remarks

The method is in principle not designed for powders containing agglomerates >4 mm. However, when testing powders containing some agglomerates >4 mm, the watch should be stopped, when powder ceases to flow out of the drum.

7. Reference

Own experience.

Fig 1. Drum for determination of flowability



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Flowability

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