Test methods used for the analysis of whey powder

by

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General information about whey powder

Dry matter (102°C): 98 – 99%
Ash: 7 – 8%
Fat: around 1%
Protein: 13 – 14%
Lactose (monohydrate): 72 – 75%
Nitrate (FIA): 140 – 180 mg/kg
Determination of Water Content, IDF 26A:1993

1. Heat uncovered dish and its lid in the oven at 102 ± 2 °C for 1 h
2. Cool down in desiccator and weigh to the nearest of 0.1 mg
3. Weigh 1 – 3 g whey powder into the dish to the nearest of 0.1 mg
4. Place it in the oven for 2 h at 102 ± 2 °C
5. Cool down the dish and weigh to the nearest of 0.1 mg
7. Repeat heating until constant weight (< 0.5 mg to previous weight)

Repeatability: Absolute difference between the results of two single determinations shall not exceed 0.2 g of water per 100 g of product.

Reproducibility: The absolute difference between the results of two single determinations, found out by different operators shall not exceed 0.4 g per 100 g product
1. Heat the platinum or silica dish in the furnace at 500°C for 30 min
2. Cool down in desiccator and weigh to the nearest of 0.1 mg
3. Weigh 2 – 2.5 g whey powder into the dish to the nearest of 0.1 mg
4. Cover with ashless filter and start ashing using bunsen burner
5. Ashing of content in the dish at 500°C (max. 550°C)
6. Cool down the dish and weigh to the nearest of 0.1 mg
7. Repeat heating in the furnace for 15 min until constant weight

Repeatability: Absolute difference between the results of two single determinations shall not exceed 0.15 % of ash.
1. After homogenization about 1.5 g of dried whey (nearest to 1 mg) transferred to fat-extraction flask (Mojonnier type)

2. Prepare a blank test, instead of dried whey take 10 ml water

3. Prepare fat-collecting vessels: drying of vessels, add a few boiling aids, heating at 102 ± 2°C for 1 h, cooling down and weigh to the nearest of 0.1 mg.

4. Add 10 ml water at 65 ± 5°C to wash the test portion into the small bulb of the flasks until the product is completely dispersed.

5. Add 2 ml 25% ammonia, mixing

6. Heat the flask at 65 ± 5°C in the water bath, occasionally shaking.

7. Cooling down to room temperature
8. Add 10 ml EtOH, at least 94% (v/v), mix gently
9. Add two drops of Congo red solution (1 g, dilution to 100 ml)
10. Add 25 ml diethyl ether, shaking vigorously avoiding emulsions
11. Transfer organic layer into fat-collecting vessel by decantation
12. Add 25 ml light petroleum (boiling point 30-60°C)
13. Shaking gently and then centrifuge the flask (500–600 rpm)
14. Allow the closed flask to stand for at least 30 min (separation of
the organic supernatant layer has to be clear and separated
from the aqueous layer (if necessary cool the flask
15. Rinsing of organic layer to fat-collecting vessel, adding gently
some water to raise the aqueous layer and transfer again
organic layer into fat-collecting vessel by decantation
16. Rinse the outside of the extraction flask with some mixed solvent, collecting the rinsings in the fat-collecting vessel
17. If desired, solvent parts may be removed from the vessel by evaporation etc.
18. Add 5 ml ethanol to the rests in extraction flask
19. Carry out a second extraction by repeating steps 10 to 16
20. Carry out a third extraction (without addition of ethanol) by repeating steps 10 to 16
21. Remove solvents from fat-collecting vessel as completely possible (evaporation, destillation)
22. Heat fat-collecting vessel for 1 hour at 102 ± 2°C, allow to cool
23. Weigh to the nearest of 0.1 mg.
24. Repeat the heating for checking removal of solvents
Determination of nitrogen using Kjeldahl method

Calculation of protein, please use

Protein = Nitrogen $\times$ 6.38

Repeatability: 0.51 %

Reproducibility: 1.6 %
Both determination are based on enzymatic reactions:

Measurement of NADH spectrophotometrically.
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ISO 5765-1 IDF 79-1: Utilizes the glucose moiety of the lactose

ISO 5765-2 IDF 79-2: Utilizes the galactose moiety of the lactose

Please look at GLP requirements in the Annex A
Both determination are based on enzymatic reactions:

ISO 5765-1 IDF 79-1: Utilizes the glucose moiety of the lactose

ISO 5765-2 IDF 79-2: Utilizes the galactose moiety of the lactose

Please look at GLP requirements in the Annex A
Determinations are based on spectrophotometric determinations of nitrite:

ISO 14673-1 IDF 189-1:2005 “Method using cadmium reduction and spectrometry”

ISO 14673-2 IDF 189-2:2005 “Method using segmented flow analysis (Routine method)

ISO 14673-3 IDF 189-3:2005 “Method using cadmium reduction and flow injection analysis with in-line dialysis (Routine method)

Please take care about repeatability and reproducibility requirements in standards.