A 1 d - Total Moisture (KF Titration)

GEA NIRO® Method No. 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 re-agent 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 ($Na_2C_4H_4O_6 \bullet 2\ H_2O$)

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 dehydrate 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.

6.5 The factor F (mg H_2O/ml KF reagent´) is calculated as:

$$F = \frac{a \cdot 36 \times 04 \cdot 100}{ml \times 230.10} = \frac{a \times 156.8}{ml}$$

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

$$\% H_2O = \frac{b \cdot F \cdot 100}{1000 \cdot w}$$

b = ml KF reagent used for sample

F = factor mg H_2O/ml KF reagent

w = weight in g

8. Reproducibility

Two determinations must not differ more than 1% relative.

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 sub- stances. 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.