A 14 a - Hygroscopicity

GEA Niro Method No. A 14 a

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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 milk 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. 500µ sieve.

5. Reagents

Ammonium chloride, saturated solution at 20°C.

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.

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)

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

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

10. Reference

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Fig. 1 Apparatus for determination of hygroscopicity