

## A 18 a - $\alpha$ -Lactose and Total Lactose

### GEA Niro Method No. A 18 a

Revised: September 2005

#### 1. Definition

The content of  $\alpha$ -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  $\alpha$ -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  $2.5 \pm 0.01$ g tannic acid powder ( $C_76H_{52}O_{46}$ ) into a 100ml volumetric flask and dilute with deionized water.

2. 10% lead acetate solution.

Add  $10.0 \pm 0.01\text{g}$  lead acetate trihydrate ( $\text{C}_4\text{H}_6\text{O}_4 \text{ Pb}, 3\text{H}_2\text{O}$ ) 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  $\alpha$ -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  $5 \pm 0.1$  ml of tannin solution and  $10 \pm 0.1$  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  $80^\circ\text{C} \pm 1^\circ \text{C}$  for 30 minutes.
8. Cool the filtrate to  $5^\circ\text{C}$ . 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  $\alpha$ -lactose in % of total lactose (TL) content is expressed as follows:

First polarimetric reading	$P_1$
Second polarimetric reading	$P_2$
$\alpha$ -lactose content of total lactose	% $\alpha_{\text{TL}}$

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

It is possible to calculate other values as follows:

% total lactose (as anhydride)	% TL
% amorphous lactose	% AL
% $\alpha$ -lactose-monohydrate (as anhydride)	% $\alpha \text{ L}$
% water of crystallisation	% $\text{H}_2\text{O}$ cryst.
% $\alpha$ -lactose-monohydrate (as monohydrate)	% $\alpha \text{ L}_{\text{H}_2\text{O}}$
Degree of crystallisation of the powder (crystallised lactose of total lactose)	% cryst.

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

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

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

$$\% \text{H}_2\text{O cryst.} = \frac{\% TL - \% AL}{19}$$

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

$$\% \alpha_{\text{LH}_2\text{O}} = \% \alpha L + \% \text{H}_2\text{O cryst.}$$

where 'x' expresses the proportion of  $\beta$ -lactose to  $\alpha$ -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 = ( $\beta$ : $\alpha$ )	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

## 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).