

## A 18 b - Total Lactose in Milk and Whey Powders by Gravimetric

### GEA Niro Method No. A 18 b

Revised: June 2006

#### 1. Principle

The method is based on lactose reducing a cupric salt complex to  $\text{Cu}_2\text{O}$  under specified conditions. The amount of  $\text{Cu}_2\text{O}$  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 temperature at  $100^\circ\text{C} \pm 1^\circ\text{C}$ .

#### 4. Chemicals

1.  $\text{CuSO}_4, 5\text{H}_2\text{O}$ , p.a., Merck.  
Copper(II)sulfate: R 22 and S 24.
2.  $\text{C}_4\text{H}_4\text{Na}_2\text{O}_6, \text{H}_2\text{O}$ , p.a., Merck.  
Potassium sodium tartrate.
2.  $\text{NaOH}$ , p.a., Merck.  
Sodium hydroxide pellets: R 35 and S 2-26-37/39.
3.  $\text{KOH}$ , 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  $\approx$  DK risk sentences  
S  $\approx$  DK safety sentences

## 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  $173.0 \pm 0.1$  g  $\text{C}_4\text{H}_4\text{Na}_2\text{O}_6$ ,  $\text{H}_2\text{O}$  and  $50.0 \pm 0.1$  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  $\pm 0.05$  g:  
whey 1.60 g  
skim milk 2.00 g  
whole milk 2.80 g
2. Dissolve the powder in approx. 200 ml  $60^\circ\text{C}$  deionized water in a 500 ml volumetric flask. Invert the solution until all powder is dissolved, and cool to  $20^\circ\text{C}$ .
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  $\text{Cu}_2\text{O}$  quantitatively to the glass filter crucible, and wash it carefully, first with  $60^\circ\text{C}$  deionized water, then with 10 ml alcohol and finally with 10 ml of ether.
10. Dry the precipitate in an oven at  $100^\circ\text{C}$  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  $\text{Cu}_2\text{O}$  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  $\text{Cu}_2\text{O}$ .

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

A = mg lactose equivalent to the weight of  $\text{Cu}_2\text{O}$  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**

<b>Cu<sub>2</sub>O</b>	<b>Lactose, H<sub>2</sub>O</b>	<b>Cu<sub>2</sub>O</b>	<b>Lactose, H<sub>2</sub>O</b>
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
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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