Nitrogen/Protein in Milk and Liquid Milk Products

LECO Corporation; Saint Joseph, Michigan USA

Instrument: FP-528

Accessories

502-186 Tin Foil Cups, 501-614 Spatula, disposable eyedroppers

Sample Weight

0.2 to 0.25 g

Calibration Standard

502-092 EDTA, Glycine solution (refer to reverse side for detailed instructions), or other suitable standard

Furnace Temperature

950°C

Flow Profile

All High

A. Blank (N)

0.00 (liquids), 0.04 (solids)

Protein Factor

6.38

Procedure

- Prepare the instrument by following the procedure as outlined in the operator's instruction manual (i.e. check gas supplies, perform any required maintenance, perform leak checks, etc.).
- Analyze blanks (gas) until a plateau is reached. Analyze three to five additional blanks and set blank using these data.
- Analyze five EDTA standards (using the 502-186
 Tin Foil Cups) at 0.2 g and drift correct (if using the
 PC option). NOTE: Each method on PC requires
 prior calibration with multiple weights of EDTA
 (0.035 to 0.4 g). If PC is not installed, analyze five
 EDTA standards and calibrate using the DSP screen
 menu.
- Weigh ~0.25 g glycine solution (~0.1 % N) into a 502-040 Tin Capsule (the capsule is not sealed). Analyze to verify instrument working range.
- 5. After mixing the sample well, weigh ~0.25 g milk into a 502-040 Tin Capsule (the capsule is not sealed), and analyze. NOTE: Make sure if autoloader is used, that it is adjusted for slow, smooth movement so sample is not splashed from the capsule.
- Analyze a standard at end of set to verify calibration.





Typical Results

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Sample	Weight (g)	% Nitrogen	% Protein
Milk (raw)	0.2461	0.597	3.81
	0.2000	0.600	3.83
	0.2125	0.602	3.84
	X =	0.600	3.83
	s =	0.003	0.015
Whey (raw)	0.2372	0.122	0.78
, , ,	0.2306	0.127	0.81
	0.2414	0.128	0.82
	X =	0.126	0.80
	s =	0.003	0.02
Blend	0.2473	1.745	11.13
	0.2342	1.752	11.18
	0.2244	1.751	11.17
	X =	1.729	11.16
	s =	0.004	0.026

GLYCINE SOLUTION PREPARATION

1. The following formula can be used to make a specific concentration:

$$C$$
 $G = (0.99^{\dagger} \cdot 0.18658)$

where: $C = desired nitrogen concentration as percent$
 $G = grams of glycine powder$

Example for 1% solution:

$$G = (0.99^{\dagger} \cdot 0.18658) = 5.414$$

NOTE: A quick reference chart, shown below, shows the grams of glycine powder needed to reach given concentrations.

- 2. Place a flask on the balance and tare. The flask should be large enough to hold 100 ml (where 100 g = 100 ml).
- 3. Add the amount of glycine calculated in step 1 and record the mass.
- 4. Add distilled water until the total mass equals 100 g, then record the mass (W).
- 5. Seal the flask and mix the contents.
- 6. To figure the exact concentration:

where: G = mass in grams of glycine recorded in step 3
W = mass in grams of water and glycine powder recorded in step 4

- 7. If the distilled water is not pure, determining the nitrogen concentration may be necessary.
 - a. Analyze five samples of distilled water.
 - b. Average the nitrogen content of the five samples (A).
 - c. Add this average to % nitrogen calculated for the calibration solution.

Example: To make a calibration solution of approximately 0.3% nitrogen where:
$$G = 1.672$$
 g $W = 99.824$ g $A = 0.004\%$
$$\frac{1.672 \left(18.654\right)}{\left(99.824\right)} + 0.004 = 0.316\% \text{ N}$$

QUICK REFERENCE CONCENTRATION TABLE

Nitrogen Concentration	Grams of Glycine
0.10%	0.541
0.30%	1.624
0.50%	2.707
0.75%	4.060
1.00%	5.414

[†]Assuming 99.0% purity of glycine powder.

LECO Corporation

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