

Nitrogen/Protein in Milk and Juice

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruSpec® N

Sampling and Sample Preparation

It is essential that a representative and uniform sample be analyzed.

Accessories

502-040 Tin Capsule

Sample Mass

~0.30 g

Calibration Standards

LECO 502-092 EDTA, 502-211 Glycine Solution (see reverse side for glycine solution preparation), or other suitable standard.

Analysis Parameters

Combustion Furnace Temperature	950°C
Afterburner Temperature	850°C

Element Parameters

Analyze	Nitrogen
Minimum Analysis Time	Yes
Comparator Level	30 seconds
Endline Time	1.00
Conversion Factor	1 second
Significant Digits	1.00
TC Baseline Delay Time	5
TC Baseline Time	seconds

Burn Profile

Burn Steps	Time	Furnace Flow
1	60 seconds	High

Macro Ballast Parameters

Ballast	
Equilibrate Time	30 seconds
Not Filled Timeout	600 seconds

Aliquot Loop

Fill Time	20 seconds
Equilibrate Pressure Time	4 seconds



Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine and calibrate systems blank as outlined in the operator's instruction manual.
3. Instrument must be calibrated as outlined in the operator's instruction manual.
4. Perform Drift Correction as outlined in the operator's instruction manual.
Note: Drift should be performed at the start of every day or when the check standard doesn't return the correct result(s).
5. Weigh ~0.3 g sample into a 502-040 Tin Capsule. With the capsule left open, enter sample identification and mass into Sample Login (F3), place into the appropriate position of the sample carousel, and proceed with analysis.

Typical Results

Sample	Mass	% Nitrogen	% Protein*
2% Milk	0.3233	0.503	3.21
	0.3360	0.495	3.16
	0.3263	0.493	3.14
	0.3155	0.508	3.24
	X = 0.500	3.19	
	s = 0.0070	0.046	
Orange	0.3110	0.107	0.669
Juice	0.3128	0.105	0.655
	0.3252	0.107	0.667
	0.3342	0.106	0.665
	X = 0.106	0.664	
	s = 0.0010	0.0062	

*Protein factor for milk: 6.38, protein factor for juice: 6.25.

GLYCINE SOLUTION PREPARATION

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

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

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

Example for 1% solution:

$$G = \frac{1}{(0.99^{\dagger} \cdot 0.18658)} = 5.414$$

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:

$$\% \text{ Nitrogen} = \frac{G (18.658 \cdot 0.99^{\dagger})}{W}$$

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%

$$\frac{1.672(18.654)}{(99.824)} + 0.004 = 0.316\% \text{ N}$$

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.

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