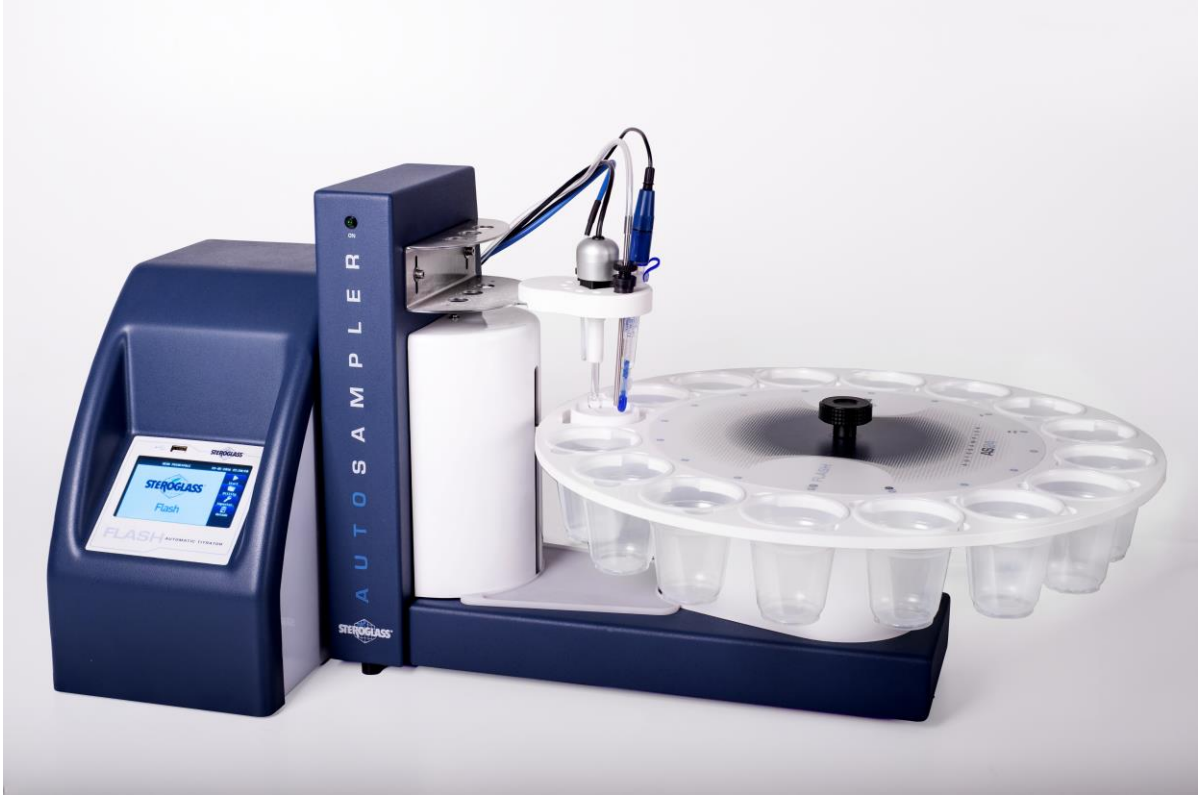
FLASH TITRATOR
Kjeldahl Nitrogen in cereals, feed and fodder



PRINCIPLE

Total Kjeldahl nitrogen (TKN) is defined as the sum of the ammoniacal nitrogen and organic nitrogen that are transformed into ammonium sulfate after mineralization.

The method (reference ICC 105/1) is applied to the determination of **nitrogenous substances in cereals and products derived from them**.

"Nitrogenous substances" are understood as the content of nitrogenous compounds in the product analyzed, calculated by multiplying the corresponding nitrogen content, determined by the method described, by a conventional factor.

The organic substances in the sample are oxidized with concentrated sulfuric acid in the presence of a catalyst; the ammonium sulfate that forms in the reaction is treated with alkalis, and the ammonia that is released is distilled and then titrated with the Flash titrator.

INSTRUMENT AND ACCESSORIES

- Flash Titrator with at least one burette;
- Single-position stand (the AS24 Automatic Sampler cannot be used);
- Combined pH electrode (with PTFE collar);
- Temperature sensor.

REAGENTS

- Titrant: Hydrochloric Acid solution HCl 0.1 N

SAMPLE PREPARATION

Weigh exactly 1-2 g of sample (depending on the presumed nitrogen content) and put it into the Kjeldahl flask. Add 0.1 g of selenium dioxide, 0.5 g of powdered copper sulfate, 5 g of potassium sulfate and 20 ml of sulfuric acid as catalyst. For weights significantly greater than 1 g, increase the quality of sulfuric acid by 10 ml per gram, and the other reagents in the same proportion. If other catalysts are used (titanium dioxide, chloric acid, etc.), it must be verified that there are no differences in the results.
If it is necessary to check for any possible influence of the reagents on the result, prepare a BLANK by replacing the sample with 1 g of sucrose, then proceed in an identical manner.
Place the flask in an inclined position, and heat slightly until foam is no longer produced, adding a few drops of paraffin as an antifoaming agent if necessary. Then boil briskly until a clear solution is obtained, and continue boiling for 30 minutes more.
Cool, add about 200 ml of water, cool again, add some grains of pumice, a small amount of paraffin, and 50 ml of sodium hydroxide solution without stirring.
Immediately connect the flask to the coolant, the end of which ends in the collection flask, into which 25 ml of boric acid solution have been introduced, ensuring that the pickup tube is immersed deeply in the solution, in order to avoid ammonia leaks.
Agitate the flask lightly to mix the contents, then heat to boiling, collecting at least 150 ml of the distillate, in order to ensure the complete passage of the ammonia into the collection flask. Remove the flask, wash the pickup tube, making the washings fall into the flask, add a few drops of the indicator and titrate the ammonia collected.

PRELIMINARY OPERATIONS

Check that the burette and the fluid circuit are rinsed with and full of titrant.
Check that the pH electrode is present in the titration holder. We suggest that the pH electrode be calibrated every day before starting the analyses:
1) Insert the pH electrode into the titration holder.
2) Go to the UTILITY menu and select CALIBRATION.
3) Select the type of calibration you prefer. Perform the “classic” “AUTO 2 BUFFER” calibration (pH 7 and 4 buffers).
The instrument will ask you to insert two buffers in sequence; follow the instructions on the display.
4) The electrode is considered good if at the end of the procedure the asymmetry is within the range of +/- 20 mV and the efficiency is between 90 and 110%.
To double-check that the calibration is good, you can:
Go to UTILITY>MEASUREMENTS> measure the buffer pH.
Readings should be between pH 4.00 +/- 0.03 and pH 7.00 +/- 0.03. If the values do not correspond to this, repeat the pH calibration.

If it is already calibrated:
1) Go to START from the main menu and select the Kjeldahl NITROGEN method;
2) If connected to the autosampler, enter the number of samples present, e.g. "2", and place the beakers in the sampler;
3) If you want to give a name to the samples, enter the name in SAMPLE DESCRIPTION;
4) Press START to start the analysis.

Method Type	*End Point*
Method Name	*Kjeldahl Nitrogen*
Descript./Sample no.	*Sample*
Pump level N	*0*
Pump level sec	*0*
Degassing sec:	*0*
Stirrer speed	*6*
Pre-stirring time (s)	*10*
Measurement type	*pH*
Initial auto-stability (pH)	*0.02*
Initial auto-stability time (s)	*5*
Initial addition	*0.00*
Initial stirring	*3*
Titrant burette	*1*
Addition type	*Progressive*
Addition (ml)	*0.25*
Limit volume (ml)	*30.0*
Polarization value	*NA*
End Point value (pH)	*4.10*
Auto-stability (pH)	*0.05*
Auto-stability time (s)	*1*
Max. stability time (s)	*60*
End titration delay	*3*
Factor	*8.7550*
Concentration (mol/l)	*0.1000*
Sample volume (ml)	*0.0*
Result unit	*N% d.m.*
Number decimals	*2*
Approaching factor	*250*
Blank (ml)	*0.000*
Washing type	*Washing position*
Washing time (s)	*5*
Reagent standardization	*NO*
Equation type	*DEFAULT*

NOTES:
The official method calculates the content of nitrogenous substances per 100 g of dry matter with the formula:

$$\% \text{ d.m.} = \frac{V \cdot N \cdot F \cdot 10000}{E \cdot (100 - U)}$$

where:
V = ml of acid 0.1 N;
N = 0.0014008 (grams of nitrogen corresponding to 1 ml of acid 0.1 N);
E = sample weight in grams;
U = sample percentage humidity;
F = factor of nitrogen conversion into nitrogenous substances, equal to:
- 5.70 for wheat and rye;
- 5.95 for rice;
- 6.25 for maize and barley.

By convention, the protein content is obtained by multiplying the weight of Kjeldahl Nitrogen by the factor 6.25.

The factor given here in the method derives from the nitrogen molecular weight 14.008 x 6.25/10 (conversion from g/kg to %)

If you want to distill and titrate a 1% and 2% ammonium sulfate solution as a standard solution, replace the factor with **6.607**; in fact 132.14 (molecular weight of ammonium sulfate)/2 (being bivalent) → 66.07 factor to express the result in g/l. To express it in % just divide by 10, therefore the factor to be entered is **6.607**.

DETERMINATION OF THE BLANK:

Method Type	*End Point*
Method Name	*Nitrogen BLANK*
Descript./Sample no.	*Sample*
Pump level N	*0*
Pump level sec	*0*
Degassing sec:	*0*
Stirrer speed	*6*
Pre-stirring time (s)	*10*
Measurement type	*pH*
Initial auto-stability (pH)	*0.02*
Initial auto-stability time (s)	*5*
Initial addition	*0.00*
Initial stirring	*3*
Titrant burette	*1*
Addition type	*Progressive*
Addition (ml)	*0.25*
Limit volume (ml)	*30.0*
Polarization value	*NA*
End Point value (pH)	*4.10*
Auto-stability (pH)	*0.05*
Auto-stability time (s)	*1*
Max. stability time (s)	*60*
End titration delay	*3*
Factor	*1.000*
Concentration (mol/l)	*0.1000*
Sample volume (ml)	*0.0*
Result unit	*N% d.m.*
Number decimals	*2*
Approaching factor	*250*
Blank (ml)	*0.000*
Washing type	*Washing position*
Washing time (s)	*5*
Reagent standardization	*NO*
Equation type	*DEFAULT*

NOTES:

It is important to perform the determination of the blank before titration of the sample in order to subtract the contribution of the reagents from the total nitrogen content.
Normally a blank is considered acceptable if it is less than 0.1 ml of titrant; this value must be entered in the field “BLANK” (ml) of the Kjeldahl NITROGEN method given above.