Kjeldahl Distillation Unit *ATN-100*

User Manual

DRAWELL

Artist of Science

Please read the manual before installation and operation.

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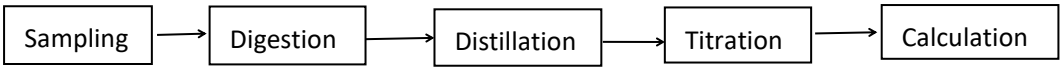
I. Summary

Currently, protein determination is still on foundation of the Kjeldahl nitrogen determination invented in 1883-based on the fact that all proteins have fixed ratios of nitrogen, by assaying nitrogen can calculate protein content. Nitrogen content determined by this method includes not only the nitrogen as ingredient of protein, but also other organic and inorganic nitrogen in non-protein materials, therefore, the converted protein content is named as crude protein content.Usually, nitrogen occupies 18% of protein, that is, every 6.25g protein contains 1g nitrogen, so 6.25 becomes the conversion factor K, but the actual nitrogen contents in different agricultural products and their finished products are different, so different agricultural products must use relevant conversion factors, for instance, the nitrogen content of peanuts, soybeans, broad beans, barleys, wheats, oats, and ryes is 17.6%. so K=5.7; the conversion factor for buckwheats, sunflower seeds, flax seeds, hemp seeds, cottonseeds, and castor beans is 5.50, corn, rapeseeds 6.00, rice 5.95, 6.25 is feasible to all other agricultural products. In spite of this, there is no feasible way to separate protein from others to measure protein content in the world, thus, at home and abroad the Kjeldahl method is still a classical one, results obtained are accurate and stable.
Absorbing new domestic and internal determination technology, chemical and physical, based on the Kjeldahl method, while taking our national situation into consideration, our ATN-100 Kjeldahl distillation unit(protein analyzer) is an ideal fast protein content testing unit for cereal, food, feed, agriculture, commodity inspection and other testing organizations.

II. Working Principle

Working procedure of the ATN-100 Kjeldahl distillation unit is as follows:



The whole device consists of two parts:electric-heated digester and distiller. To increase detection speed, the key is to speeding up the digestion of sample. The digestion unit of KDN series adopts infrared-heating quartz wick, providing even and quicker heating to digestive tubes, together with injected 10ml H_2SO_4 and sample placed inside the digestive tube which is inserted into the holder for it. Selenium tablets are added as catalyst before digestion, shortening digestion time greatly. SO_2 and other poisonous gas escaping from digestive tube will be brought into sewer together with water by suction tube through blow-off tube, effectively restraining harmful gases from escaping, so labs are saved for installing fume cupboard; after about 60-120 min of digestion, sample can be completely digested.

Main reactions in the digestion process are as follows:

$$R{\cdot}C{\cdot}H{\cdot}NH2{\cdot}COOH + H2SO4 \rightarrow CO2 + SO2 + NH3 + H2O$$

$$2NH3 + H2SO4 \rightarrow (NH4)2SO4$$

Main reactions in the distillation and absorption processes are as follows:

$$(NH4)2SO4 + 2NaOH \rightarrow Na2SO4 + 2NH3 + 2H2O$$

$$NH3 + 4H3BO3 \rightarrow NH4HB_4O7 + 5H2O$$

Then, after titration (a process hydrochloric acid and ammonium biborate reacting), protein content

can be calculated.

$NH4HB_4O7 + HCl + 5H2O \rightarrow NH4Cl + 4H3BO3$

III.Technical Parameters

a. determined materials:cereals, food, feed, brewed prodcucts, soil, sugar, dairy products, medicine, etc.

b. determination range: 0.05%~90% (0.1~200mg) nitrogen content

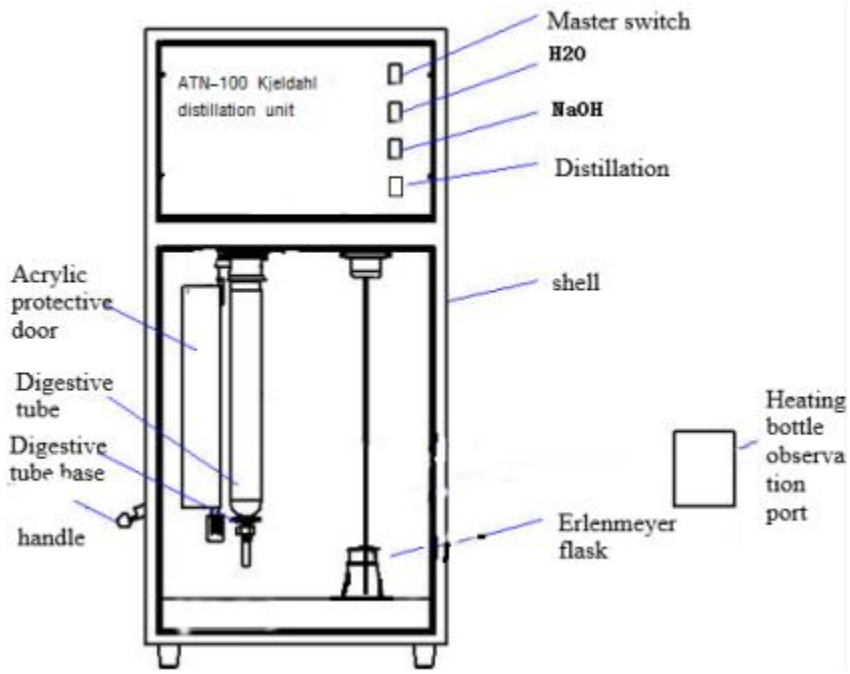
c. determination quantity:digestion quantity of a batch depends on the number of furnace holes; single distillation

d. determination time:for digestion, 45 min./batch; distillation, 4-7 min./one

e. reproducibility: parallel experimental results in line with GB/T 6432-94

f. power supply: 220V, 50Hz

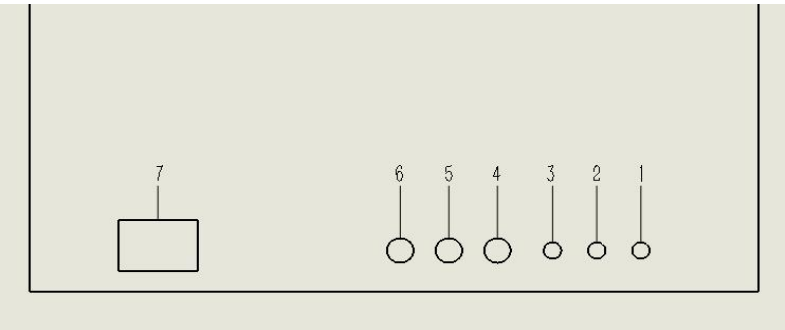
IV. Configuration Drawing



Remark: the right drawing is a side profile of the main body.

the way adjusting the base:

digestive tube base: lift the lifting handle, the digestive tube can be placed inside or taken out.



Remark:the above is a sketch map of the back side.

1.distilled water inlet 2. distilled water inlet 3. alkali liquid inlet
4.cooling water inlet 5. cooling water outlet 6.waste liquid outlet
7.electric outlet

Remark:waste liquid outlet is connected with one valve, its working states are as shown below.

Pic.1:in close state

Pic.2: in open state

V. Operation Instructions

1. Other devices and Tools

a. analytical balance: sensitivity 0.0001g
b. lab microniser or mortars
c. acid buret: 25ml or 10ml
e. Erlenmeyer flask: volume 250ml
f. sample screen: aperture 0.45mm (40 meshes)

2. Reagents

2.1 Hydrochloric acid (HCl): AR (GB622), 0.05mol/L standard solution, (4.2ml hydrochloric acid, injected with 1000ml of distilled water) take the sodium carbonate method to calibrate hydrochloric acid.
2.2 Sodium hydroxide (NaOH):CP or TP (GB629), 40g, dissolved in distilled water into 100ml 40% solution (m/v).
2.3 Boric acid (H_3BO_3): AR (GB628), 2g, dissolved in distilled water to make 100ml 2% solution (m/v).
2.4 Mixed indicator: methyl red (G5H15N3O2) (HG3-958), dissolved in ethanol into 0.1% ethanol solution, bromcresol green (HG3-1220) dissolved in ethanol into 0.5% ethanol solution, mix the two

solutions with same volume, keep the indicator in shaded area, no more than three months.
2.5 Catalyst:copper sulfate ($CuSO_4 \cdot 5H_2O$), AR(GB655), 10g; potassium sulphate(K_2SO_4) (HG3-920), AR, 150g, grind them in a mortar, carefully mix them then pass sieve of 40 meshes (catalyst).
2.6 Concentrated sulfuric acid (H_2SO_4) , CP (GB625)(98%, no nitrogen).

3. Operating Regulations

a. Sample digestion
To weigh 0.1-0.5g sample(depending on the content) that is already ground and has passed sieve of 40-60 meshes, place full of it into a already cleaned and dried test tube, and add 5g catalyst,10ml H_2SO_4.

Plug digestive tubes into each hole of the rack for them, then place the rack on digester, and open the water tap connected with gas exhaustion tee, to make the tee in a suction state. Connect power supply, in the initial stage of heating, observe carefully to prevent the sample from splashing because of quick boiling. (initially, set the temperature at about 200℃, when the sample is fully boiling, increase temperature linearly until the liquid is transparent.)
After digestion(the digestive juice is clear and transparent), move the rack for digestive tubes to cooling rack, cool it to 40-50℃, the next step will be distillation.

b. Distillation
For ATN-100 Kjeldahl distillation unit, its distiller is added alkali liquor and distilled water by a built-in electromagnetic pump, NaOH and H_2O inlets should be rang with rubber tubes then insert the tubes into NaOH container (self-prepared) and H_2O container separately; when adding liquid, press corresponding buttons on the panel, liquid will automatically flows into digestive tube through electromagnetic pump, volumes of liquid added are depended by the amount of sample (the yellow ruler is for volume reference, usually, alkali liquor added is 5 times the amount of sulfuric acid).

3.2.1 Switch on the power, then the water tap(the one connected with hose for “cooling water”), make sure water flows out from the hose(to sewer or recollect the water) and the valve connecting with “exhaust outlet” is closed.

3.2.2 Preheat: place empty digestive tube and conical flask of 250ml in right places, shut up the protective door for digestive tube; switch on “H_2O”, “NaOH”, make certain their connecting hoses are smooth, then switch off them; start up “Distillation”, to make 2-3 min of empty distillation, till then preheating is finished, shut down “Distillation”.

3.2.3 Distill Samples

(1) fetch a conical flask of 250ml, add 50ml 2%H_3BO_3 and 2-3 drops of mixed indicator, put the flask at the right of the apparatus, to immerse dropper, while, the cooled digestive tube with sample should be at left, then close the protective door.

(2) switch on “H_2O” to add 15-25ml distilled water to dilute sample;

(3) switch on “NaOH” to add 50ml NaOH solution(commonly 5 times of the volume of concentrated H_2SO_4)

(4) start up “Distillation” and move away conical flask when its received liquid reaches 100ml(when total volume reaches 150ml), to separate dropper from solution, rinse the dropper with washing bottle containing distilled water, at the end, take away the conical flask.

(5) when distillation is finished, turn off the “Distillations” switch;

(6) change for another digestive tube with new sample, start all steps from (1)

3.2.4 concluding work after all samples distilled

(1) switch off “Distillation” and the water tap for cooling water.、

(2) turn on the valve connecting with “exhaust outlet”, turn it off when water inside vaporizing furnace is emptied.

(3) put an empty digestive tube in place and the hose connecting alkali pump into distilled water container, draw distilled water to rinse the alkali pump.

(4) finally, cut off power, wipe the apparatus.

c. titration

use calibrated HCL to titrate solution inside conical flask, stop titrating when color of the titrated solution changes from green to pale purple, record the volume of consumed HCL, then calculate protein content according to the following formula:

$$\text{Protein content}(\%)=\frac{(V_2-V_1)\times C\times 0.014\times K}{W}$$

In the above formula:

V2— volume of consumed standard acid solution when titrating sample (ml)

V1—volume of consumed standard acid solution when making blank titration(ml)

C—molar concentration of standard acid solution

0.014—mEq of nitrogen

K—conversion factor of nitrogen to crude protein

W—sample weight (g)

(Blank determination: use 0.1g of sugar to replace sample or blank determination without sample)

The result of parallel determination is shown by arithmetic mean, and two decimal places retained.

4. Conversion Factors

The results report must indicate the conversion factor of nitrogen to crude protein, the conversion factors are shown in the table below.

Sample	Conversion Factor	Source of Data
wheat	5.70	GB2905
barley	5.70	GB2905
brewing barley	6.25	TOCT10846
flour	5.70	GB5009.5
bread	5.70	AOAC14.103
soybean	6.25	GB2905
corn	6.25	Food Data Manual
feed	6.25	GB6432
meat products	6.25	GB5009.5
dairy products	6.38	GB5009.5
milk	6.38	ACAC16.036
sesame	5.30	GB5009.5
rapeseed	5.53	Comprehensive Utilization of Rapeseed

5. Precautions

a. If there is gas overflowing when digesting, increase pressure from the water tap connected with gas extraction tee. Clean sealing ring at end of each test to extend its service life.
b. Every day after work, shift the rubber tube connecting NaOH solution to distilled water bottle, then place the digestive tube well to make several times of suction and cleaning, preventing alkali liquor remained at the alkali pump corroding the pump and non-returning valve becoming adhesive; when using next time, discharge certain NaOH before distilling sample, in case NaOH is diluted, affecting the first sample.

c. Each time after distillation, the digestive tube must be removed immediately, to prevent liquid inside being siphoned to evaporation furnace and corroding heating tube.

d. Under conditions of complying with transportation, storage, installation and usage regulations, if the apparatus malfunctions for sake of manufacturing within one year from the day of receiving, we provide three guarantees; when opening packing box, if there are cases like shortage, broken places because of bad package, accessories not in compliance with packing list, please contact with our customer service dept., let us know the product's model, serial number and production date. When using the apparatus, please follow instructions strictly, if you have any question or suggestion for optimizing structure, please inform our quality inspection department.

VI.Common Breakdowns and Treatment

No.	breakdowns	causes and remedies
1	no electricity through the apparatus	Fuse blows out, please replace the fuse; the power plug is not plugged well, please plug it into socket firmly.
2	little steam	Leakage from the steam generator reduces steam output, check if the fixing screw or adaptor at the fixing-use sealing bolt is loose, as a result steam escapes from fixing-use sealing ring; check whether the sealing ring is aging, replace it in time; or increase proper distillation time.

VII.Packing List

No.	Parts	Specifications	Quantity
1	ATN-100 distiller	main body	1
2	power cable	250V/10A	1
3	fuse tube	10A	2
4	4*6 rubber hose	meter	3
5	6*10 rubber hose	meter	3
6	gloves		1 pair
7	warranty card	piece	1
8	Certification card	piece	1
9	operating instructions	copy	1

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