



Application Note

No. 223/2016

Nitrogen determination in Tobacco

SpeedDigester K-425, KjelMaster K-375 with Scrubber K-415:
Nitrogen Determination in Tobacco according to the Kjeldahl Method



1. Introduction

Generally it is considered that flavour and taste of smoke is correlated with the nitrogenous constituents. According to Indian Central Tobacco Research Institute (CTRI) flue-cured tobacco containing 1.6 to 2.3 % total nitrogen gives the most satisfying smoke, whereas the acceptable limit of total nitrogen is 1.0-3.0 %. Apart from curing difficulty, tobacco with a nitrogen content higher than 3.0 % would result in deep brown coloured trashy leaf which shatters readily and has flat, insipid tasting smoke [1].

High level of nitrogen in tobacco is associated with a high level of nicotine. A low nitrogen content would result in 'washed out', pale coloured leaf, lacking in rich colour characteristic of good tobacco. Therefore, the determination of total nitrogen is an important quality control measure in the tobacco industry [1].

Furthermore, the ratio of nitrogen to nicotine is assumed to give some chemical balance within the leaf. A value exceeding 1.0 has been ascribed as unbalanced. A too low value (below 0.5), on the other hand, may be considered undesirable because the tobacco is heavy bodied [1].

Here, the determination of nitrogen in tobacco is performed using the SpeedDigester K-425 and KjellMaster K-375.

2. Equipment

- KjellDigester K-425 (the parameters used are also valid for K-436)
- Scrubber K-415 TripleScrub
- KjellMaster K-375
- Analytical balance (accuracy ± 0.1 mg)

3. Chemicals and Materials

Chemicals:

- Sulfuric acid conc. 98 %, Merck (1007482500)
- Titanium, BUCHI Kjeldahl Tablet (11057980)
- Sodium hydroxide 32 %, Merck (106469)
- Boric acid 4 %, 400 g boric acid, Merck diluted to 10 L with deionized water, pH adjusted to 4.65
- Sulfuric acid 0.5 mol/L, Fluka (35357)
- Neutralization solution for the Scrubber: 600 g sodium carbonate, calcined, technical, Synopharm (0179420) about 2 mL ethanol and a spatula tip of bromthymol blue, Fluka (18460) diluted to 3 L with distilled water

For a safe handling please pay attention to all corresponding MSDS.

Samples:

- Tobacco sample purchased at a local market. The sample was dry and cut in small pieces of tobacco leaves (Figure 1).



Figure 1. Tobacco sample analysed for its nitrogen content.

4. Procedure

The determination of nitrogen in tobacco includes the following steps:

- Sample was used as received.
- Digestion of the sample, using SpeedDigester K-425.
- Distillation and titration of the sample, using KjelMaster K-375.

4.1. Digestion method

Preheat the SpeedDigester K-425 according to the parameter listed in Table 1.

1. Place approx. 0.6 g of sample directly in a 300 mL sample tube.
2. Add 2 Titanium Tablets and a tip of a spatula of stearic acid to each sample. Stearic acid acts as antifoam agent.
3. Add 20 mL of sulfuric acid (conc. 98 %).
4. Prepare additional blanks, chemicals without sample.
5. Connect the Scrubber K-415 to the K-425 for absorbing the acid fumes created during digestion.
6. Insert the rack containing the samples into the preheated unit.
7. Digest the samples according to the parameters listed in Table 1.

Table 1: Temperature profile for digestion with the K-425.

Step	Heating Level	Time [min]
Preheating	4	10
1	4	10
2	7	110
Cooling	-	30

8. Let the samples cool down to ambient temperature and start the distillation according to Table 2.

NOTE: When the samples are placed in the cooling position it takes approx. 30 min to cool them down; when they are left in the heating chamber it takes at least 60 min.

4.2. Distillation and titration

Distill the samples according to the parameters listed in Table 2.

Table 2: Parameters for distillation and titration with the KjelMaster system K-375.

Method parameters KjelMaster K-375

H ₂ O volume	50 mL	Titration solution	H ₂ SO ₄ 0.5 mol/L
NaOH volume	110 mL	Sensor type	Potentiometric
Reaction time	5 s	Titration mode	Standard
Distillation mode	Fixed time	Titration start time	0 s
Distillation time	240 s	Measuring mode	Endpoint pH
Stirrer speed distillation	5	End point pH	4.65
Steam output	100 %	Stirrer speed titration	7
Titration type	Boric acid	Titration start volume	0 mL
Receiving solution vol.	60 mL	Titration algorithm	Optimal

4.3. Calculation

The results are calculated as a percentage of nitrogen. The following equations (1) and (2) are used to calculate the results.

$$w_N = \frac{(V_{\text{Sample}} - V_{\text{Blank}}) \cdot z \cdot c \cdot f \cdot M_N}{m_{\text{Sample}} \cdot 1000} \quad (1)$$

$$\%N = w_N \cdot 100 \% \quad (2)$$

- w_N : weight fraction of nitrogen
- V_{Sample} : amount of titrant for the sample [mL]
- V_{Blank} : mean amount of titrant for the blank [mL]
- z : molar valence factor (1 for HCl, 2 for H₂SO₄)
- c : titrant concentration [mol/L]
- f : titrant factor (for commercial solutions normally 1.000)
- M_N : molecular weight of nitrogen (14.007 g/mol)
- m_{Sample} : sample weight [g]
- 1000 : conversion factor [mL/L]
- %N : percentage of weight of nitrogen

5. Results

5.1. Repeatability

The results of nitrogen determination are presented in Table 3. 2.88 % nitrogen in was measured in the tobacco analyzed. The nitrogen content was analyzed in triplicate.

Table 3: Results of the nitrogen content in tobacco.

Tobacco	mSample [g]	V _{Sample} [mL]	%N
Sample 1	0.6113	1.316	2.84
Sample 2	0.6077	1.337	2.91
Sample 3	0.6122	1.340	2.89
Average [%]	–	–	2.88
Rsd [%]	–	–	1.2

The mean blank volume (V_{Blank}) was 0.075 mL ($n = 3$).

5.2. Reproducibility

Reproducibility of the results was checked by performing same experiment with same sample on another day.

Table 4: Results of the nitrogen content in tobacco

Tobacco	m _{Sample} [g]	V _{Sample} [mL]	%N
Sample 4	0.6339	1.418	2.82
Sample 5	0.6287	1.427	2.86
Sample 6	0.6331	1.408	2.80
Average [%]	–	–	2.82
Rsd [%]	–	–	1.1

The mean blank volume (V_{Blank}) was 0.144 mL.

6. Conclusion

The determination of nitrogen in tobacco using the SpeedDigester K-425 and KjelMaster K-375 provides reliable and reproducible results with low relative standard deviations (Rsd).

These results correspond well to the values of the nitrogen in tobacco products indicating the quality of the product.

Together with the fully-automatic KjelMaster K-375, the time to result is significantly reduced.

7. References

[1] Central Tobacco Research Institute; http://www.ctri.org.in/for_quality.php.