

NITROGEN ANALYSIS IN FERTILIZER WITH THE FLASH 2000 COMBUSTION NITROGEN ANALYZER

- **Large Sample Size Capability**
- **Inconel crucible for ash removal**
- **Easy and fast to maintain**
- **Reliable and reproducible results**
- **Conforms to the AOAC Method 990.13 Nitrogen in Fertilizer**



Discussion

Most fertilizers use nitrate based compounds due to their solubility in water. However, nitrates do cause some problems when trying to determine the amount of total nitrogen. The method used in many laboratories for nitrogen determination is the classical wet chemistry Kjeldahl method. Unfortunately this digestion process is dependent on the type of nitrogen bonding in the sample. The matrix dependent results obtained by the Kjeldahl method highlighted the need for a reliable and precise alternative for determining the total amount of nitrogen in fertilizers.

The Thermo Flash 2000 is the most reliable instrument for elemental analysis. This analyzer copes with all of the requirements of modern laboratories such as accuracy, reproducibility, high sample throughput, and low cost of analysis. With the addition of the MAS200R automatic sampler this instrument provides the efficiency of automated, unattended analysis of up to 125 samples.

The simple system design ensures that the Flash Combustion Elemental Analyzer is the easiest unit of its kind to use and maintain. There are no valves, no switches, and no mixing devices thus guaranteeing reliable results. The system simply consists of oxidation and reduction reactors, a GC column and a thermal conductivity detector. The quality of results is assured by the fact that all of the gases yielded by the combustion are swept onto the GC column and are measured without being split or diluted.

Description of the analytical method

The Flash 2000 Nitrogen Analyzer, which is based on the dynamic flash combustion principle, requires no sample digestion or toxic chemicals, while providing important advantages in terms of time, automation, and quantitative determination of nitrogen in all sample matrices. The instrument equipped with the new MAS200R electronic autosampler provides automated, unattended analyses of up to 125 samples. Additionally, the new electronic mass flow control of both the helium carrier gas and oxygen for combustion assures the stability of the analytical conditions and eliminates the need for frequent re-calibration.

The proper amount of oxygen, based on the weight and type of sample, is determined by OxyTune™ and introduced to insure a complete combustion of the sample.

Following combustion, the gases produced are carried by the helium flow through the reduction reactor filled with copper, the swept through CO₂ and H₂O traps, a GC separation column and finally detected by a Thermal Conductivity Detector. **The Eager Xperience dedicated software then automatically generates a complete report.**

Maintenance

With the Inconel crucible the ash removal can be performed at operating conditions (900°C), avoiding time consumed due to cooling down and heating up the reactor. We suggest removing the ash after 100 analyses. The crucible, cleaned after cooling, can be used again for further runs (4-5 cycles). The lifetime of the reactor tube is indefinite with the catalyst lasting from 1,000 to 2,000 analyses. Copper lasts 750 to 1,000 samples with a properly optimised OxyTune™. Adsorption traps for CO₂ and H₂O are replaced after 100 to 200 samples depending on the moisture and carbon content of the sample.

Analytical conditions

Combustion temperature:	900°C
Reduction Temperature:	840°C
Oven temperature:	60°C
GC Column:	0.5M Activated Carbon
Helium flow rate:	Measurement: 140 ml/min Reference: 100 ml/min
Oxygen flow rate:	300 ml/min
Analytical Time:	300 Seconds
Sample Delay Time:	10 Seconds
Oxygen Inject Time:	15 Seconds

Figure 1 – Nitrogen Configuration

Analytical Layout of FlashEA™ 1112 Protein Analyzer

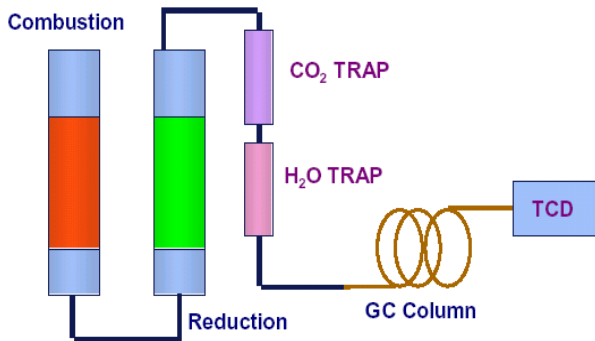


Table 1 – AOAC 992.15 Performance Requirements

(10 samples)

Standards	N%	SD %	RSD %
Nicotinic Acid	11.375	0.067	0.590
Lysine-HCl	15.217	0.054	0.361
AOAC Mix N%	3.263	0.012	0.076
AOAC Mix N/P	20.396	0.327	0.373

SOLID FERTILIZER

Number of Samples: 3 (200-400 mg)
Average: 8.84% N₂
RSD %: 0.64

LIQUID FERTILIZER

Number of Samples: 3 (100-200 mg)
Average: 9.75% N₂
RSD %: 1.15

RESULTS ON THE FOLLOWING 5 FERTILIZER SAMPLES ARE THE AVERAGE OF TEN (10) REPLICATES VARYING THE WEIGHTS FROM 100 TO 300 MG.

SAMPLE NUMBER 1

Nitrogen %: 45.90
Std. Deviation: 1.04

SAMPLE NUMBER 2

Nitrogen %: 17.44
Std. Deviation: 0.85

SAMPLE NUMBER 3

Nitrogen %: 8.23
Std. Deviation: 0.47

SAMPLE NUMBER 4

Nitrogen %: 32.98
Std. Deviation: 0.98

SAMPLE NUMBER 5

Nitrogen %: 12.74
Std. Deviation: 0.69