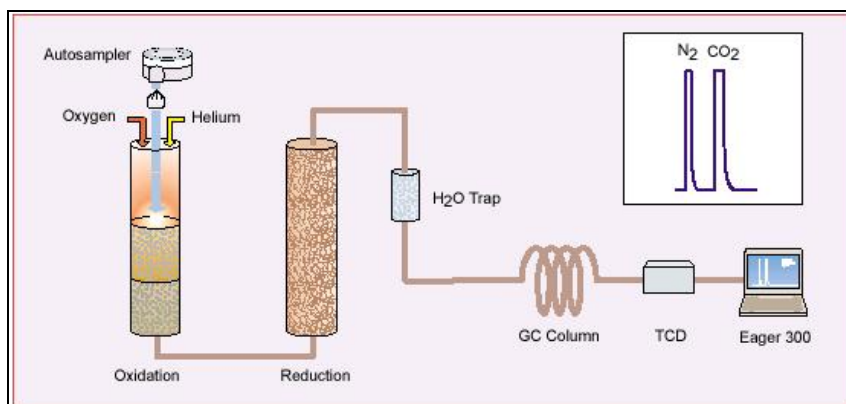


AN 637

Nitrogen and Carbon determination in particulate matter in water (filters)

OEA Team

- π Easier sample handling
- π Automated, unattended analysis
- π Fast analysis
- π No matrix effect



Introduction

Nitrogen and Carbon determination in particulate matter in waters provides useful information to biologists, ecologists and chemists involved in the study of the importance and the role of those elements, their chemical forms and their biological and physio-chemical transformations.

The importance of this analysis has grown dramatically in the last years and many of the classical methods are now no longer suitable for routine analysis due to time consuming and environmentally hazardous reagents employed.

The Flash 2000 NC Filters Analyzer provides rapid and automatic Nitrogen and Carbon determination as well as advantages in terms of accuracy, reproducibility and low cost per analysis.

Description of the analytical method

Particulate matter in water is normally analysed after filtration on glass filters (typically 24 mm diameter or less) and dried; the dried filter is analysed as such.

The analyzer is based on the dynamic combustion of the sample. The sample is contained in a tin capsule and introduced into the combustion reactor via the MAS 200R Autosampler together with a proper amount of Oxygen. Weighing the sample is not required because it is the total content of the elements which need determinations.

After combustion, the produced gases are carried by a helium flow to a second reactor filled with copper, then swept through H₂O trap, a GC column and finally detected by a thermal conductivity detector. A NC report is automatically generated by the Eager Xperience dedicated software and displayed at the end of the analysis.

Analytical conditions

T left tube: 900°C
 T right tube: 840°C
 T oven: 50°C

Helium Carrier Flow: 130 ml/min
 Helium Reference Flow: 100 ml/min

Oxygen Flow: 250 ml/min
 Oxygen Injection Time: 8 sec
 Sample Delay Time: 12 sec

Total Run time: 300 sec
 Standard: Atropine 1 – 1.5 mg

Note: 1) For filters 12 mm diameter use tin capsule P/N 24006400 while for filters 24 mm diameter use tin capsule P/N 25208000.

2) For a correct total determination it is important to evaluate the blank level by analysing a clean filter, the resulting blank value is then automatically subtracted by the software.

Calculation and results

1) Determination of the Area of 1 µg Nitrogen and the Area of 1 µg Carbon through the analysis of Atropine as standard:

a) Analysis of blank (only tin capsule): the analysis was repeated three times to check the reproducibility of the blank, the Nitrogen Area was 0 and the average of the Carbon Area was 8351.

b) Analysis of Atropine (4.84 %N, 70.56 %C) in duplicate weighing 1 – 1.5 mg. See Table 1.

c) Values of N Area/µg N and C Area/ug C to use for sample calculation: it was used the average of the two runs of Atropine: 1658 N Area/ug N and 5014 C Area/µg C.

2) Analysis of filters containing the samples:

a) Analysis of blank (tin capsule + filter) analyzed in triplicate to check the reproducibility of the blank, see Table 2.

b) NC results of filters containing the samples analyzed in triplicate: see Table 3.

Figure 1 show an overlay between the chromatogram of a sample (chromatogram black) and the chromatogram of the blank (only tin capsule plus clean filter) (chromatogram red) indicating no interference with the analysis.

Conclusion

The Flash 2000 NC Filters Analyzer has provided a good reproducibility for the extremely low content of Nitrogen and Carbon in this type of application.

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Table 1

Weight (mg)	N amount (ug)	C amount (ug)	N Area	C Area	C Area corrected by blank	N Area / ug N	C Area / ug C
1.384	66.98	976.55	110903	4897750	4889399	1656	5007
1.305	63.16	920.81	104818	4632224	4623873	1660	5021

Table 2

N°. Run	N Area	C Area	Average C Area
1	--	17016	14938
2	--	13349	
3	--	14449	

Table 3

Sample	N Area	C Area	C Area corrected	ug N	ug C
1	541	41475	26537	0.33	5.29
	585	37275	22337	0.35	4.45
	618	41079	26141	0.37	5.21
2	3857	78837	63899	2.33	12.74
	3878	78166	63228	2.34	12.61
	3820	77138	62200	2.30	12.40
3	10367	41666	26728	6.25	5.33
	10990	46245	31307	6.63	6.24
	10274	43952	29014	6.20	5.78

Figure 1

