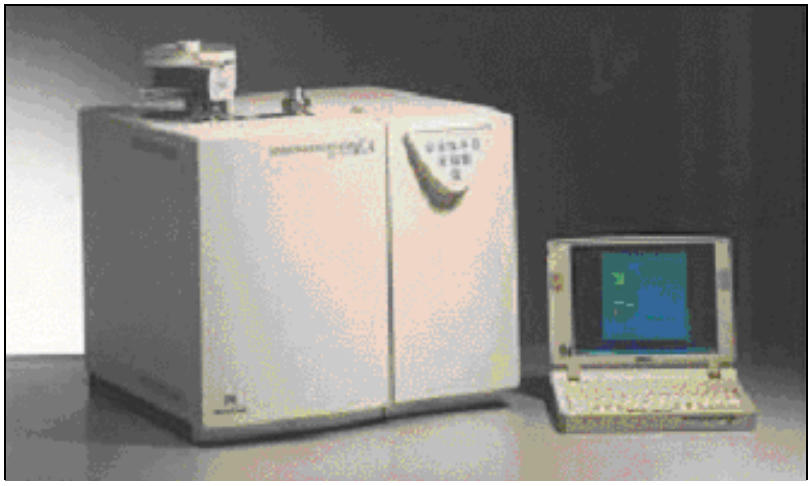
AN 642

Oxygen analysis by Flash 2000 Elemental Analyzer

OEA Team

- π Automated, unattended analysis
- π No matrix effect
- π Reliable results
- π Fast analysis



Introduction

The need of an accurate, fast and automatic method to measure the total Oxygen content in different materials has become of increasing importance in the last years.

The analytical method able to satisfy this need must be such that the experimental data has not to take into account the type of Oxygen bonding in the samples. So, it is necessary to transform in a single chemical species all the Oxygen present in the sample. This species must be able to be detected at high sensitivity due to some of its own physical or chemical characteristics.

In this light the Flash 2000 Analyzer is the most reliable instrument for Oxygen analysis and copes with all requirements of modern laboratories such as accuracy, reproducibility and low cost per analysis.

Description of the analytical method

The determination of the total Oxygen is based on the Schutz-Unterzaucher technique.

The system operates in pyrolysis mode. Samples placed in silver containers are dropped into the pyrolysis chamber which is maintained at 1060°C and contains nickel coated carbon. The oxygen in the sample combined with carbon forms CO which is then chromatographically separated from other combustion products and sensed by a thermal conductivity detector. Total run time less than 6 minutes.

A complete report is automatically generated by the Eager Xperience data handling software package and displayed at the end of the analytical routine.

Analytical Conditions

T tube: 1060°C

T oven: 65°C

Helium: Carrier Flow: 130 ml/min
Reference Flow: 100 ml/min
Sample Delay Time: 12 sec
Run time: 300 sec

Standard: CEDFNI, benzoic acid, acetanilide, hexane-ethanol solution (4.5 O%, for gasoline analysis)

Sample weight:
Depends of sample nature

Sample pretreatment:
Depends of sample nature

Results

Schema 1 shows the layout of Oxygen configuration.

Table 1 shows a typical analytical test with pure organic compounds. CEDFNI (cyclohexanone-2, 4-dinitrophenylhydrazone, 23 %O) was used as standard, K factor as calibration method and Acetanilide (11.84 %O) was analysed as unknown to check the calibration. The weight of sample was 1-2 mg. The data demonstrate an excellent reproducibility and accuracy.

Table 2 shows Oxygen data of different type of samples. Coal, coke, lignite and compost samples were homogenize using a ball mill before the analysis; while the other samples were analysed without any pre-treatment. Gasoline samples were analyzed by direct liquid injection using a chromatographic syringe. All data were obtained with a good reproducibility and no matrix effect was observed when changing type of sample.

Analytical layout of Oxygen determination

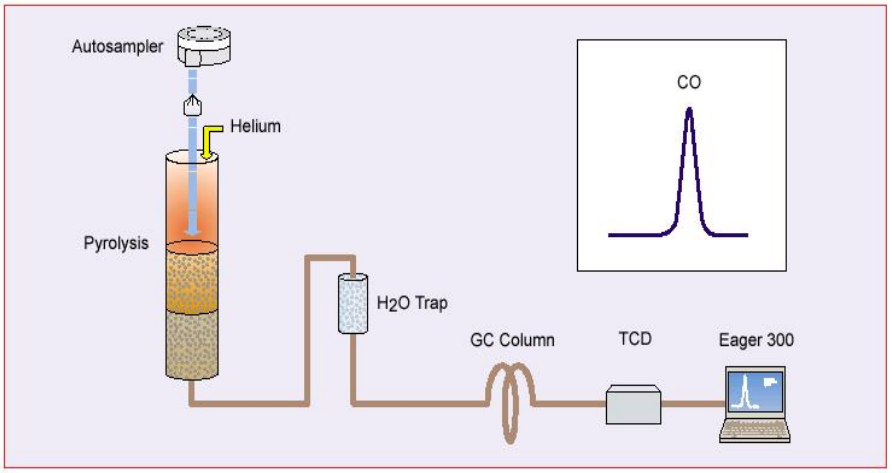


Table 1 – Typical analytical test

Sample Name	Run	Sample type	Theoretical O%	Experimental O%	Average O%	RSD %
CEDFNI	1	Standard	23.00			
	2	Standard	23.00			
	3	Standard	23.00			
Acetanilide	4	Unknown	11.84	11.85	11.86	0.4695
	5	Unknown	11.84	11.92		
	6	Unknown	11.84	11.81		

Table 2 – Oxygen determination of different sample nature

Sample	Weight (mg)	O %	RSD%
Coal 1	2-3	10.822 10.701	0.7951
Coal 2	2-3	3.730 3.723 3.690	0.5751
Coke 1	1-2	1.044 1.087 1.020	3.2319
Coke 2	1-2	0.502 0.514 0.510	1.2012
Lignite 1	2-3	25.202 25.540	0.9418
Lignite 2	2-3	21.418 21.200	0.7227
Diesel 1	2-3	0.3002 0.2711 0.2862	5.0916
Diesel 2	2-3	0.2259 0.2560 0.2431	6.2487
Gasoline 1	2-3 ul	4.0144 4.0477	0.5865
Gasoline 2	2-3 ul	0.8497 0.8653	1.2868
Gasoline 3	2-3 ul	2.6400 2.6456	0.1508

Sample	Weight (mg)	O %	RSD%
Rubber 1	2-3	2.023 1.999	0.823
Tyre 1	2-3	4.345 4.325 4.327	0.254
Wood	3-4	40.223 40.659 40.857	0.7993
Paper	2-3	42.585 42.918 42.969	0.4870
Ash	3-4	0.327 0.332	1.0730
Carbon fiber 1	2-3	0.2654 0.2679 0.2624	1.0382
Carbon fiber 2	2-3	7.3561 7.3995 7.3536	0.3502
Compost	3-4	26.360 26.022 26.550	1.0165
Stainless steel	20-30	0.0126 0.0124 0.0122	1.6129
Polymer 1	3-3.5	0.411 0.404 0.418	1.7032
Polymer 2	3-3.5	0.1595 0.1598	0.2216

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