TECHNICAL NOTE

elementar
EXCELLENCE IN ELEMENTS

Advanced Purge and Trap (APT) Technology

Introduction

The determination of the carbon, hydrogen, nitrogen and sulfur content in organic matrices is an important parameter required in many applications. The most common analysis method for monitoring these four elements simultaneously is through the use of high temperature combustion (HTC), at a furnace temperature exceeding 1000 °C, in the presence of oxygen.

During the combustion process, carbon is oxidized to carbon dioxide, hydrogen to water, and sulfur to sulfur dioxide. The nitrogen oxides formed during the combustion process are subsequently reduced to elemental nitrogen gas in a downstream reduction furnace.

Historically, helium has been the inert carrier gas of choice, but more recently argon has become more popular due to helium shortages (discussed further in a separate Technical Note). The detection of the combustion gases can be carried out in two different ways; either by quantitative separation of the combustion gases followed by quantification with a thermal conductivity detector (the most common approach), or by a series of separate infrared detectors for a gas specific quantification.

The high temperature combustion analysis method requires minimal sample preparation efforts and offers outstanding flexibility for types of matrices that can be analyzed: solid, viscous, liquid, volatile, or gaseous. As an additional benefit and unlike other methodologies a high degree of automation is possible in HTC analysis, allowing unattended 24/7 operation and high sample throughput.

APT TECHNOLOGY

vario EL cube
vario MACRO cube
vario MAX cube

Advanced gas separation

Elementar's proprietary APT technology is an industry leading and unique chromatographic technique for the quantitative separation of combustion gases. A schematic setup of an elemental analyzer equipped with the APT technology is shown in Figure 1.

The APT technology uses up to three gas-selective trapping columns that selectively adsorb CO_2, H_2O and SO_2. N_2 is not adsorbed and reaches the thermal conductivity detector first (Figure 2a). After detection and software recognition of the completely resolved N_2 peak, the CO_2-selective column is heated up, carbon dioxide is released and detected (Figure 2b). Likewise, the H_2O-selective column and the SO_2-selective column consecutively release their adsorbed gases (Figure 2c and 2d).

The three gas-selective columns are highly optimized to quantitatively adsorb the target combustion gases and are available in different sizes, allowing for capacities up to 250 times higher than ordinary gas chromatography (GC) columns. With the purge and trap approach the analysis of samples with an absolute carbon content of up to 500 mg is possible. The fast heating rate for the gas desorption assures sharp peaks and outstanding signal-to-noise ratios resulting in industry-leading accuracy and sensitivity.

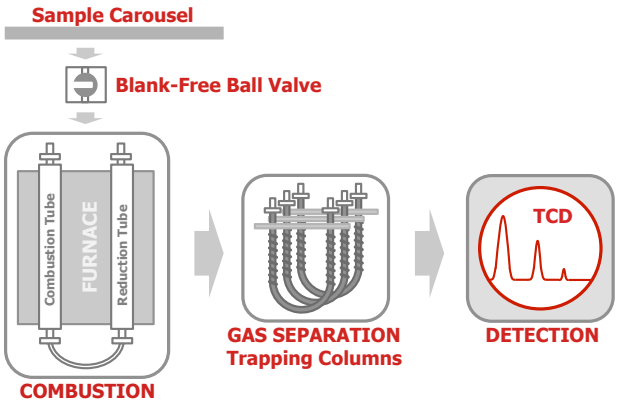


Figure 1. Functional principle of a high temperature combustion analyzer with APT technology.

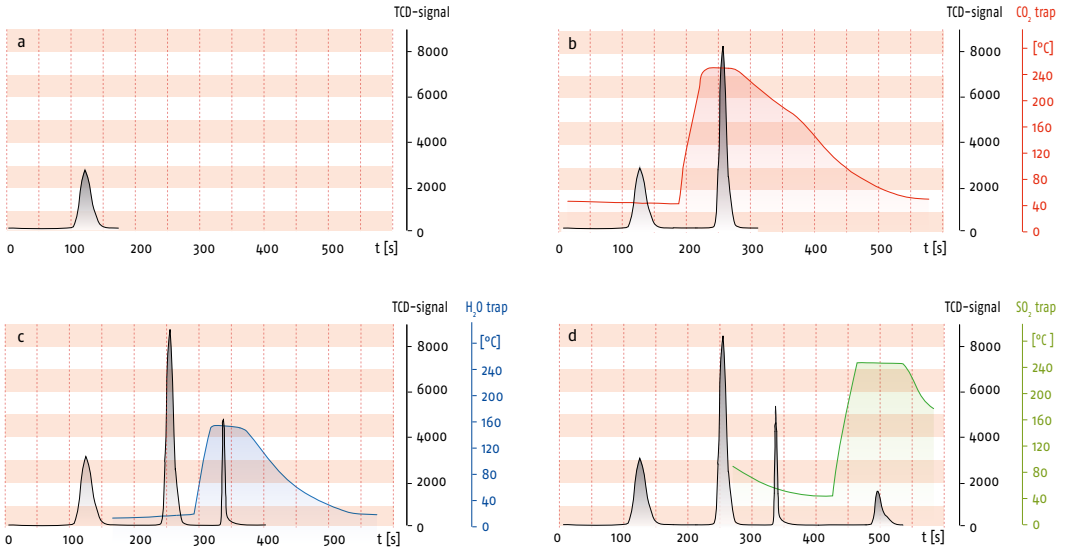
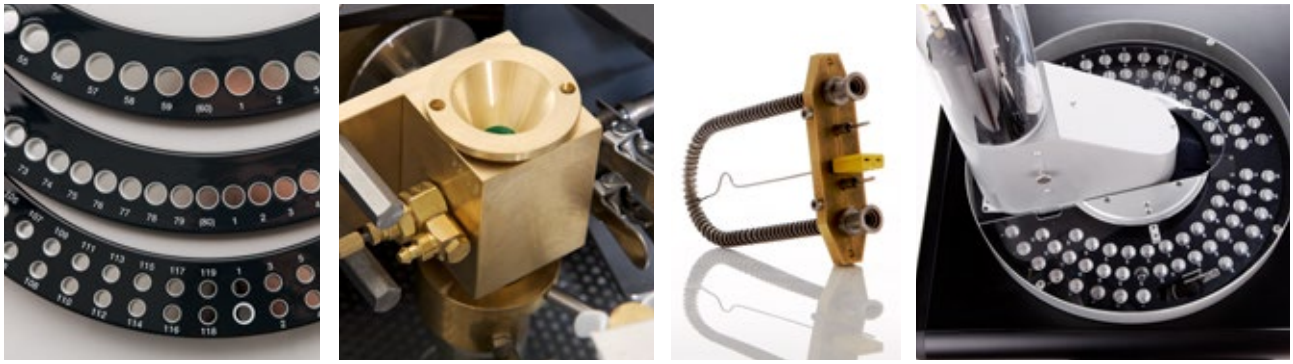


Figure 2. Peak graphics of a CHNS analysis of sulfanilamide, visualizing the advanced gas separation using the three gas-selective trapping columns for CO_2, H_2O and SO_2.



Always baseline separated

One of the main advantages of the APT technology is that it allows full control of the individual desorption of each combustion gas. Each heating step commences only after completion of the previous peak detection by the thermal conductivity detector, so that complete peak separation is always achieved – independent from the elemental content and concentration ratio. Even challenging C:N and C:S elemental ratios of up to 12000:1 can, therefore, be easily quantified (Figure 3).

The distinct peak separation assures absolutely reliable and trouble-free data acquisition. The data analysis can therefore be easily automated for larger sample amounts while maintaining highest possible data quality and accuracy. This results in significantly higher laboratory efficiency.

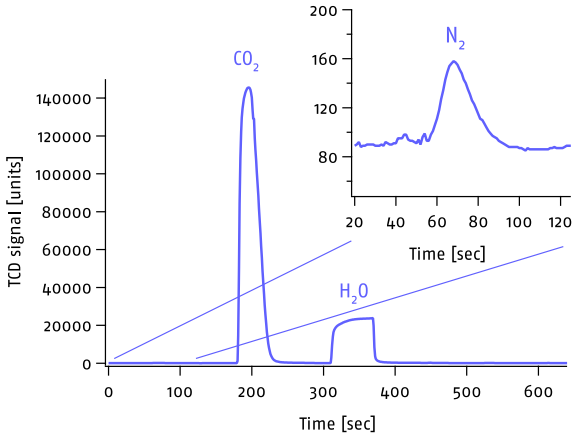


Figure 3. Experimentally determined C:N elemental ratio of 12000:1 found in a toluene sample measured with the vario EL cube in CHN mode.

Outstanding sulfur sensitivity

One of the most notable advantages of the APT technology is the fact that peak broadening is not proportional to retention time. In ordinary gas chromatographic analyses peak broadening is generally observed because of increased longitudinal diffusion inside the GC column. Gas molecules are not only traveling forward but also in radial direction and backwards. This affects most prominently the determination of SO_2 with its prolonged retention time, resulting in a broad peak, which significantly deteriorates the detection limit for sulfur (Figure 4). With APT technology, SO_2 is quantitatively adsorbed using the gas-selective column. The fast heating rate for the gas desorption assures a sharp peak and best possible detection limit for sulfur. Using an infrared detector, an outstanding limit of detection (LOD) can be achieved, as shown in Figure 5. Even lower sulfur concentrations, down to <10 ppb can be determined with our trace analyzer trace SN cube.

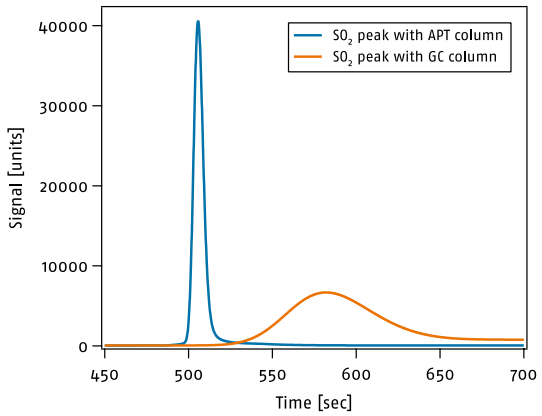


Figure 4. Example of peak focusing effect of the APT technology for sulfur analysis of a barium sulfate sample.

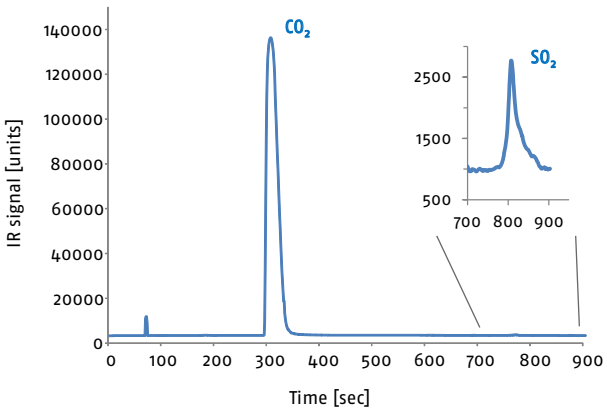


Figure 5. Even absolute S concentrations of only 2 µg can be determined using an optional infrared detector for SO_2. This translates into a relative LOD of 2 ppm.

Greater sample weight range

Starting from low mg sample weights for high purity samples with limited availability up to 5 grams of inhomogeneous sample, almost anything is possible thanks to the APT technology. Our one-of-a-kind APT technology is utilized in the vario EL cube, vario MACRO cube and vario MAX cube. Each elemental analyzer is optimized for different sample weights, sample homogeneity and maximum carbon content, thus providing versatility and performance. Even for the largest sample weights it is possible to analyze the complete combustion gas without the need for gas splitting or dilution, which would result in a degradation of sensitivity and maximization of contaminant effects.

The vario EL cube is the instrument of choice for low sample weights and highly homogeneous samples, such as chemicals, pharmaceuticals or polymers. For samples with medium homogeneity, which require larger sample weights for representative results, such as coal, biomass or waste, the vario MACRO cube is the ideal instrument. Finally, for inhomogeneous samples or samples with higher ash content, such as soil, plants or fertilizer, the vario MAX cube is the perfect option. This instrument offers automatic sample introduction and ash removal via a robust, maintenance-free robotic arm.

	vario EL cube	vario MACRO cube	vario MAX cube
elements	C, H, N, S and O or Cl with multiple modes		N, CN, CNS
maximum C content	40 mg	150 mg	500 mg
sample weight (for solids with 1% C)	< 1 g	< 1.5 g	< 5 g
sample homogeneity (recommended)	high	medium	low
ash removal	manual		automatic

Table 1. A multitude of options for optimal elemental analysis with the vario EL cube, vario MACRO cube and vario MAX cube.

Summary

The experimental data demonstrates that the APT technology clearly outperforms, in virtually any aspect, other technical solutions for gas separation, such as ordinary gas chromatography. APT technology offers the highest sample weight flexibility, peak resolution, and therefore unrivaled sensitivity.

Furthermore, the gas-selective columns are optimized to provide unmatched robustness and longevity compared to GC columns. In combination with 10 years warranty on furnace and thermal conductivity detector cell, Elementar's elemental analyzers offer an unmatched future-proof investment.

Elementar – your partner for elemental analysis

Elementar is the world leader in high performance analysis of organic elements. Continuous innovation, creative solutions and comprehensive support form the foundation of the Elementar brand, ensuring our products continue to advance science across agriculture, chemical, environmental, energy, materials and forensics markets in more than 80 countries.

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