

# DYNAMIC HEADSPACE SAMPLING IN THE ANALYSIS OF FOOD SOLID MATRICES OF VEGETABLE ORIGIN

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## Introduction

Dynamic headspace sampling (D-HS) also known as *purge and-trap* was first introduced by Wahlroos in 1963 [1]. D-HS is a non-equilibrium *continuous gas extraction* technique providing high and flexible concentration factors, [2] that mainly consists of three steps: i) a continuous removal of the volatiles released from a matrix by an inert gas flowing through or over it; ii) their concentration from the above flow stream on a solid adsorbent, a sorbent, or through cryo-trapping, and iii) their recovery from the trap by thermal desorption or solvent elution, either on-line or off-line to a GC or GC-MS system for analysis.

Recent technological advances in dynamic headspace sampling (D-HS) and the possibility to automate this sampling method have led to a marked improvement in its performance, a strong renewal of interest in it, and have extended its fields of application. The introduction of in-parallel and in-series automatic multi-sampling and of new trapping materials, plus the possibility to design an effective sampling process by correctly applying the breakthrough volume theory, have made profiling and fingerprinting in the food field more representative, and have enhanced selectivity, and flexibility, also offering the possibility of fractionated enrichment in particular for high-volatility compounds. This study deals with fractionated-D-HS (FD-HS) ability to produce a sample representative of the volatile fraction of solid or liquid matrices [3].

## Experimental

Experiments were carried out on a model equimolar (0.5 mM) EtOH/water solution, comprising 16 compounds with different polarities and volatilities, structures ranging from C5 to C15 and vapor pressures from 4.15 KPa (2,3-pentandione) to 0.004 KPa (*t*-β-caryophyllene), and on an Arabica roasted coffee powder. Three trapping materials were considered: Tenax TA<sup>TM</sup> (TX), Polydimethylsiloxane foam (PDMS), and a three-carbon cartridge Carbopack B/Carbopack C/Carbosieve S-III<sup>TM</sup> (CBS).

## Results

The influence of several parameters on the design of successful FD-HS sampling, including the physical and chemical characteristics of analytes and matrix, trapping material, analyte breakthrough, purge gas volumes, and sampling temperature, were investigated. The results show that, by appropriately choosing sampling conditions, FD-HS sampling, based on component volatility, can produce a fast and representative profile of the matrix volatile fraction, with total recoveries comparable to those obtained by full evaporation D-HS for liquid samples, and very high concentration factors for solid samples.

## References

- [1] Wahlroos O. *Ann. Acad. Sci. Fenn. Ser. A. II, Chemica* 122 (1963) 1
- [2] Kolb B, Ettre LS. 1997. *Static Headspace-Gas Chromatography, Theory and Practice*. Wiley-VCH: New York.
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