Headspace Analysis of Botanicals

Application Note
Botanicals

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The technique of dynamic headspace involves removing volatiles and semi-volatiles using a combination of temperature and carrier gas. The volatiles are then concentrated on an absorbent trap. The sample is simply placed inside a bulk dynamic headspace vessel and sealed. The vessel is then permitted to come to thermal equilibrium, while a controlled, constant flow of helium ensures efficiency in the sampling process. The volatiles are collected on a Tenax trap which can then be backflushed to the gas chromatograph with a mass selective detector for analysis. It should be noted that headspace analysis on natural products such as herbs and spices requires no sample preparation (other than to dry the material, if desired).

Figure 1 is a total ion chromatogram of chamomile flower volatiles collected at 85°C.The dried flowers were ground to a powder and placed into the sampling vessel. The vessel was heated and purged for 60 minutes. The trap was then desorbed into the GC/MSD, producing a chromatogram which could be considered a fingerprint for this natural product.

The ion chromatogram in Figure 2 is from a sample of white willow bark using a purge temperature of 100°C. It clearly is different from the chamomile observed in Figure 1.

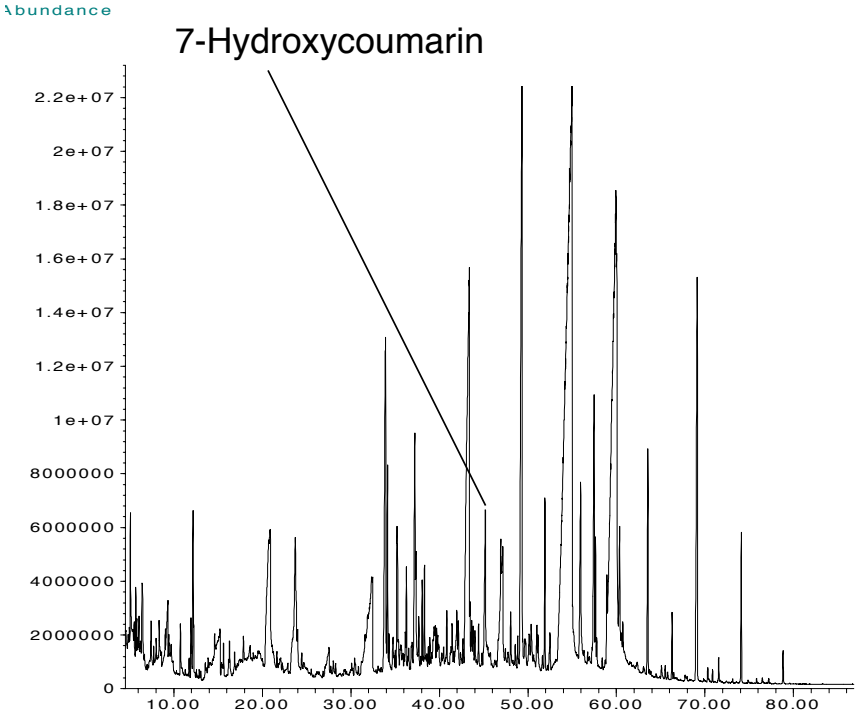

Figure 1. Chamomile Flowers at 85°C

Dynamic Headspace Instrument Conditions

Bulk Vessel Temperature: 85°C, 100°C
Sampling Time: 60min
Flow: 100mL/min
Trap: Tenax
Desorption: 300°C for 5 min.
Valve Oven: 300°C
Transfer Line: 300°

GC/MS

Column: 5% phenyl (30m x 0.25mm x .25μm)
Carrier: Helium, 25:1 split
Injector: 300°C
Oven: 40°C for 2 minutes
6°C/min to 295°C hold 10 min

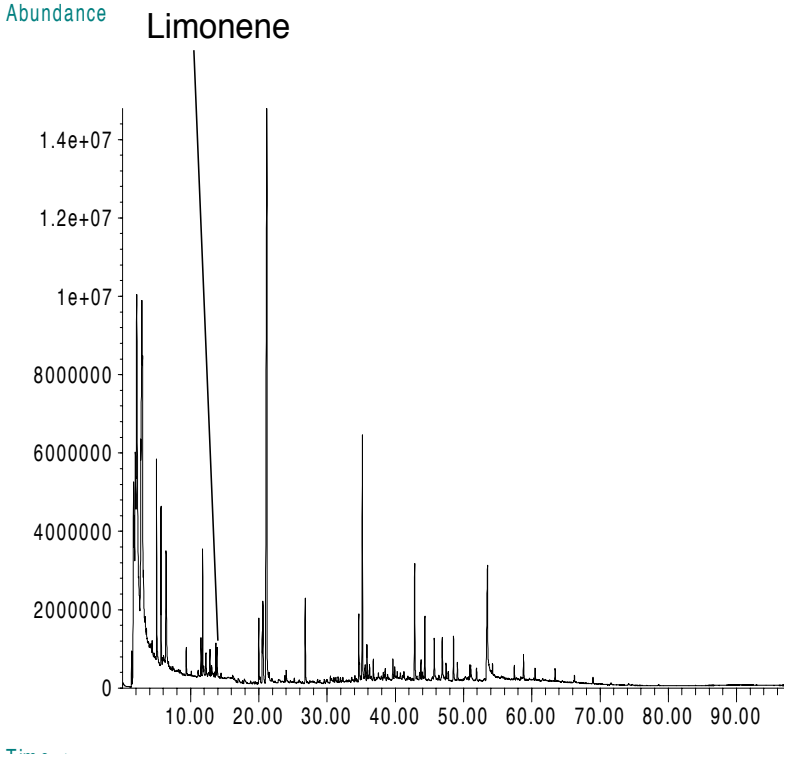

Figure 2. Willow Bark at 100°C.

FOR MORE INFORMATION CONCERNING THIS APPLICATION,
WE RECOMMEND THE FOLLOWING READING:

M. Wichtl, Herbal Drugs and Phytopharmaceuticals, Med Pharm, Boca Raton, 1994

J. Robbers, Pharmacognosy and Pharmacobiotechnology, Williams and Wilkins, Baltimore, Maryland, 1996

W. Coleman, J. Chromat. Sci. 30: 159 (1992)