

Headspace Analysis of Botanicals

The technique of dynamic headspace involves removing volatiles and semivolatiles using a combination of temperature and carrier gas. The volatiles are then concentrated on an absorbent trap. The CDS Model 6500 Bulk Dynamic Headspace Autosampler is a fully automated instrument capable of performing multiple analyses using this technique. The sample is simply placed inside one of the vessels 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 and then are 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.

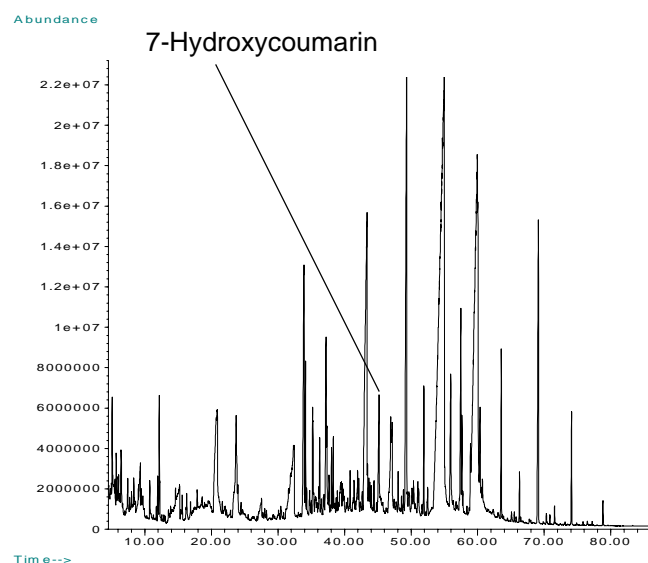


Figure 1. Chamomile Flowers at 85°C

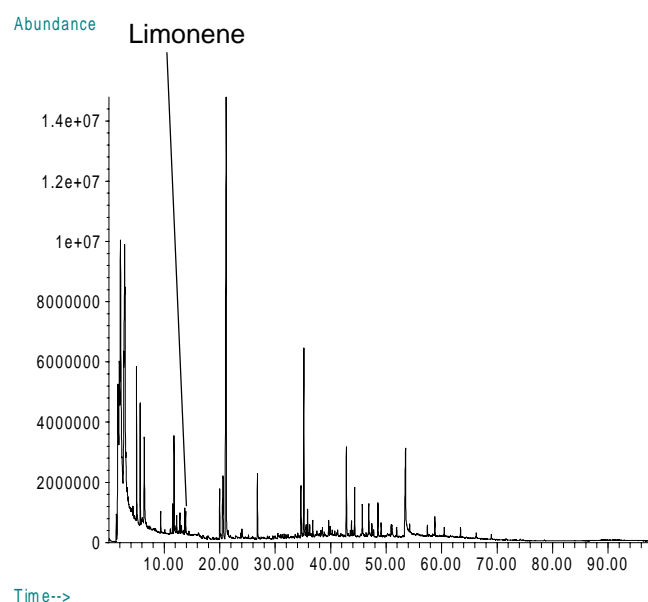


Figure 2. White Willow Bark at 100°C

Equipment

Samples were analyzed using a CDS Analytical Model 6500 Bulk Dynamic Headspace Autosampler interfaced to a Hewlett Packard 6890 gas chromatograph equipped with a Hewlett Packard 5972A mass selective detector.

Model 6500 Conditions

Valve Oven: 300°C
Transfer Line: 300°C
Sample Temperature: 85°C, 100°C
Time: 60 min.
Flow: 100ml/min.
Trap: Tenax
Desorption: 300°C for 5 min.

Gas Chromatograph Conditions

Carrier: He, 5.9psi, 25:1 Split
Column: HP-5M
(30m x 250µm x.25µm)
Detector: MSD

Initial Oven Temperature: 40°C for 2 min.
Ramp: 6°C/min.
Final Temperature: 295°C for 10min.

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 Pharmaco-biotechnology*, Williams and Wilkins, Baltimore, Maryland, 1996

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

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Headquarters

JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

Zoex Europe
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 257 39 72
F +31 (0) 40 251 47 58

Sales and Service

Netherlands
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 320 87 00 18
F +31 (0) 320 87 00 19

Belgium
Grensstraat 7
Box 3 1831 Diegem
T +32 (0) 2 721 92 11
F +32 (0) 2 720 76 22

Germany
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

UK & Ireland
Cedar Court,
Grove Park Business Est.
White Waltham, Maidenhead
Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

info@go-jsb.com
www.go-jsb.com

