

A Comparative Analysis of Hop Essential Oil in North Carolina and the Pacific Northwest by Headspace GC-MS

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Abstract

As there are currently 84 breweries in North Carolina, there is an increasing demand for locally produced hops.^{1, 2} Currently, hops are produced in the Pacific Northwest due to appropriate soil composition and climate. For this study, the essential oils are to be compared in the hop cultivars Cascade, Zeus, Magnum, and Chinook produced in North Carolina and the Pacific Northwest. This method of essential oil analysis is fast, reproducible and requires little sample quantity or preparation. This comparative analysis of essential oil of hops produced in North Carolina and the Pacific Northwest provides information for sourcing hop aroma.

Table 1. Description of Aroma Compounds

Analytes	Compound Class	Flavor Description	Smells like...
α -Caryophyllene	terpene	Spicy	Cloves, black pepper
Caryophyllene oxide	terpene oxide	Spicy, herbal	Basil
Farnesene (all isomers)	terpene	Woody, green, citrus	Gardenia, apple peel
Geraniol	terpene alcohol	Floral	Geranium
α -Humulene	terpene	Woody, earthy	Hops, coriander
Linalool	terpene alcohol	Sweet, floral, woody	Coriander, orange, lavender
Myrcene	terpene	Citrus, earthy	Bay leaves, thyme
β -Pinene	terpene	Green, piney	Pine, rosemary

The aroma compounds in hops derive from the volatile lipophilic fraction referred to as the essential oil which amounts to 0.1-2.0% (v/w) of hops.⁶ Although the aroma compounds are minimally soluble in the matrix of beer, odor thresholds are large. This allows the aroma compounds to be detectable at concentrations below 10 ppm in beer. α -humulene was anticipated to degrade rapidly and farnesene was anticipated to be difficult to quantify due to isomerization.

Calibration Methods

- Five standard solutions of aroma compounds were prepared at the concentrations of 50, 100, 300, 700, 1000 parts-per-million Standard solutions prepared in ethyl acetate
- Internal standard - n-nonane used at the concentration of 1 part-per-thousand¹



Figure 1. Cascade hops in Boone, NC Summer 2013

Headspace Sampler:

Agilent Technologies 7697A

Gas Chromatograph (GC):

Agilent Technologies 6890N

Injection Mode: Splitless

Injection volume: 1 μ L

Column: J&W Scientific DB-1701

30 meter, 0.25 mm ID, 0.25 μ m

Mass Spectrometer (MS):

Agilent Technologies 5973 *inert*

Table 2: Temperature Program

Stage	Rate (°C/s)	Temp (°C)
Initial		40
Ramp 1	20	60
Ramp 2	7	115
Ramp 3	10	210

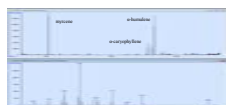


Figure 2. Chromatogram and mass spectrum of Magnum hops essential oil



Figure 3. Chromatogram and mass spectrum of Zeus hops essential oil

Sample Methods

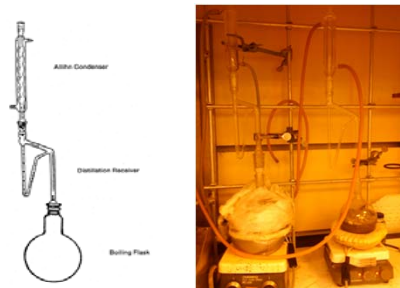


Figure 4: Extraction of Oil by Steam Distillation⁷

Of each hop sample, 25 g ground to a fine powder then added to a round bottom flask with 1 L deionized water

- The mixture boiled for 4-7 hours and the distillate was collected in the distillation receiver
- The essential oil was transferred to 20 mL headspace vials with 10 mL deionized water for analysis and the internal standard, n-nonane was added at 1 part-per-thousand



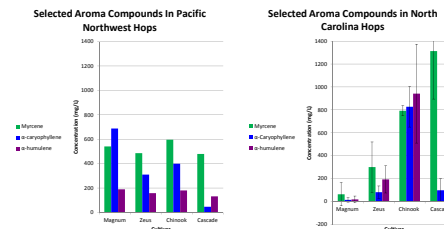
Figure 5: Headspace-GC-MS used for this study



Figure 6. Zeus and Cascade ready to harvest in NC Piedmont Summer 2011.*

The hops used for analysis of aroma compounds were produced by the NCSU Hops Project.

Results



Conclusions

Several varieties such as Chinook and Cascade produce larger concentrations of predominant aroma compounds in NC compared to PNW. Perhaps short growing season causes rapid development of essential oil resulting in high concentrations of some aroma compounds and little presence of others.

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