



What's in Your Beer?

Volatile Component Profiling by
Headspace GC and GC/MS

Ken Lynam Sept 19, 2017

Topics for this presentation

What can GC and GC/MS do for you? Think aroma and it's impact on flavor

Pertinent examples GC and GC/MS analyses

Broad range of tools used to study beer and the brewing process

Opinions and discussions

It's all about the beer...

Favorite styles?

Historical prospective?

Where are your challenges?

What can GC and GC/MS Do for You?

Excellent tools for probing flavor active volatiles

Finding the fragrance note for an exceptional beer

Raw material quality control

Raw material profiling

Process control of fermentation products, good and not so good

Finished product characterization

GC/MS identification of unknowns

EPA Method 552.3

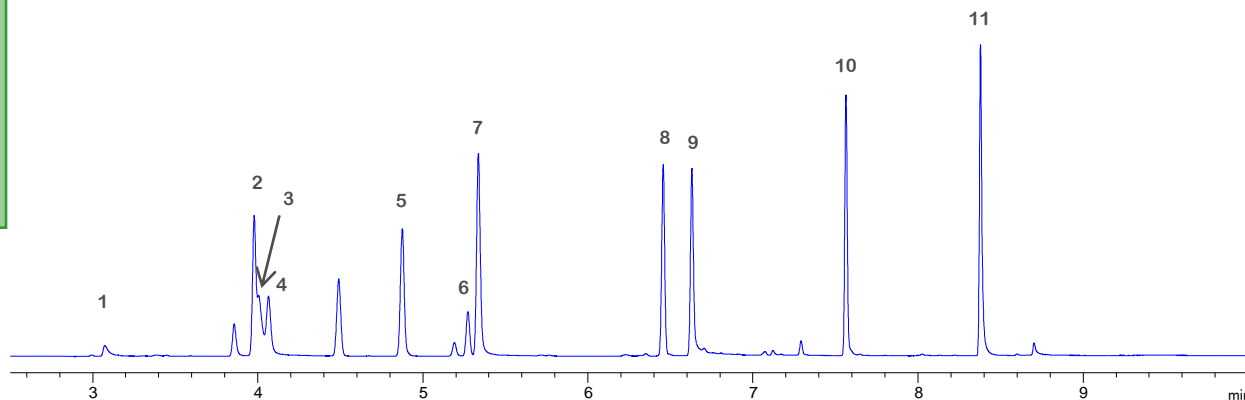
Haloacetic Acids and Dalapon

DB-CLP1 30m 0.32mm ID 0.25µm (cat.# 123-8232)
DB-CLP2 30m 0.32mm ID 0.5µm (cat.# 123-8336)
5m x 0.32mm ID deact. guard column
Inert Tee CFT device (cat.# G3184-60065) 1:1 Split

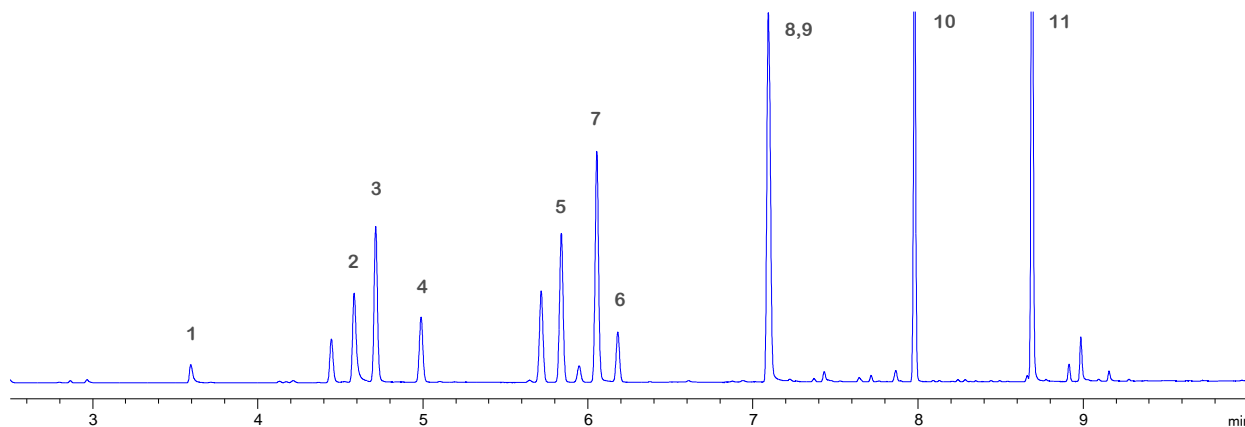
Instrument: Agilent 7890 GC with dual µECD
Sampler: Agilent 7693
Sample: 10-100 ng/mL Haloacetic acids and Dalapon (methyl esters)
Inj. Vol.: 1 µL splitless
Liner: Ultra Inert liner, single taper splitless (cat.#5190-2292)
Inj. Temp.: 180°C
Oven Temp: 40°C (hold 0.5 min) to 95°C at 10°C/min, 30°C/min to 200°C, hold 1 min
Carrier Gas: Helium at 54.79 cm/sec (constant flow)
Detector: µECD @ 340 °C

- | | |
|--|---|
| 1. Methyl chloroacetate (30ng/mL) | 7. Methyl bromochloroacetate (20ng/mL) |
| 2. Methyl bromoacetate (20ng/mL) | 8. Methyl bromodichloroacetate (20ng/mL) |
| 3. Methyl dichloroacetate (30ng/mL) | 9. Methyl dibromoacetate (10ng/mL) |
| 4. Dalapon methyl ester (20ng/mL) | 10. Methyl dibromochloroacetate (50ng/mL) |
| 5. Methyl trichloroacetate (10ng/mL) | 11. Methyl tribromoacetate (100ng/mL) |
| 6. 1,2,3-Trichloropropane (IS) (50ng/mL) | |

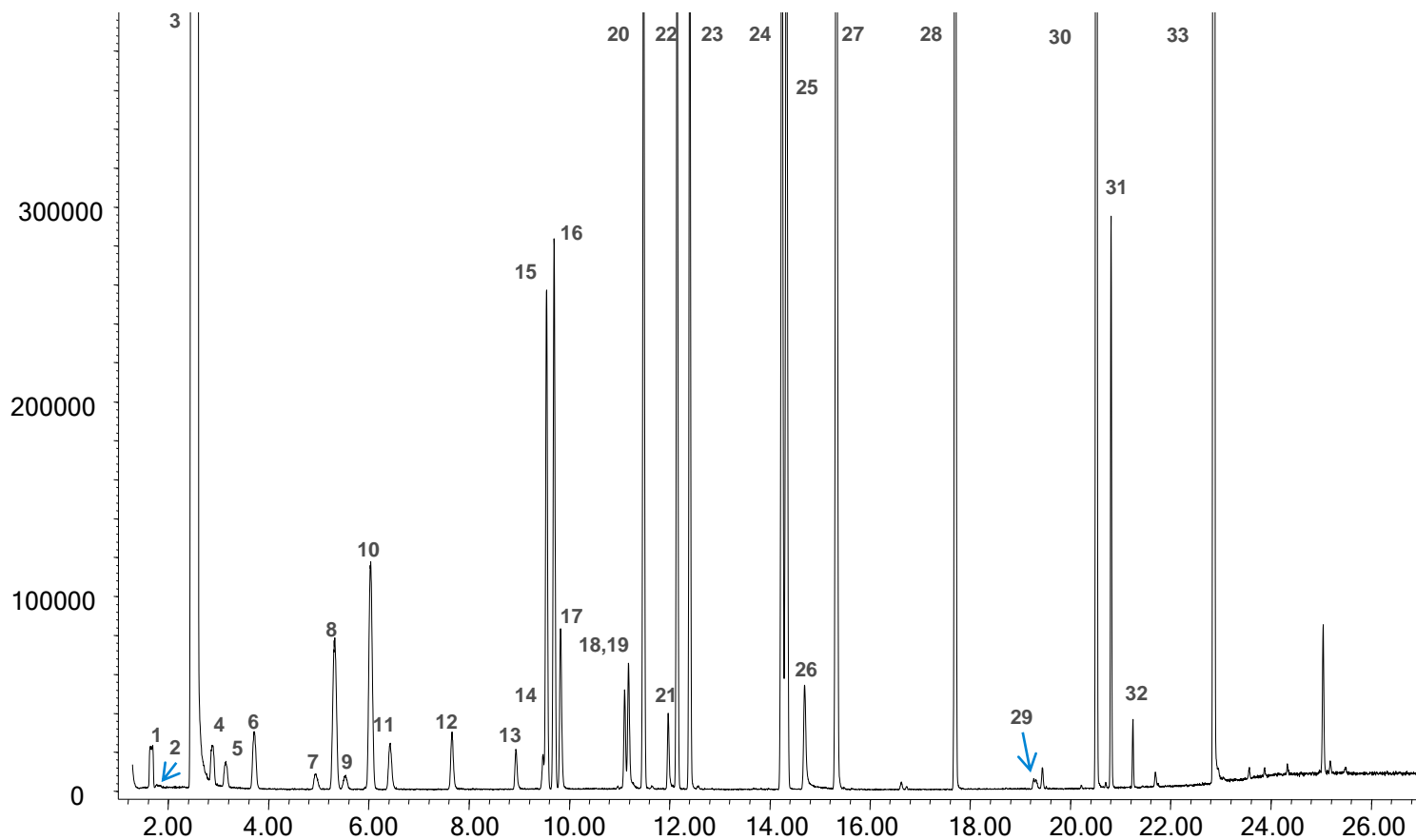
Agilent DB-CLP1



Agilent DB-CLP2



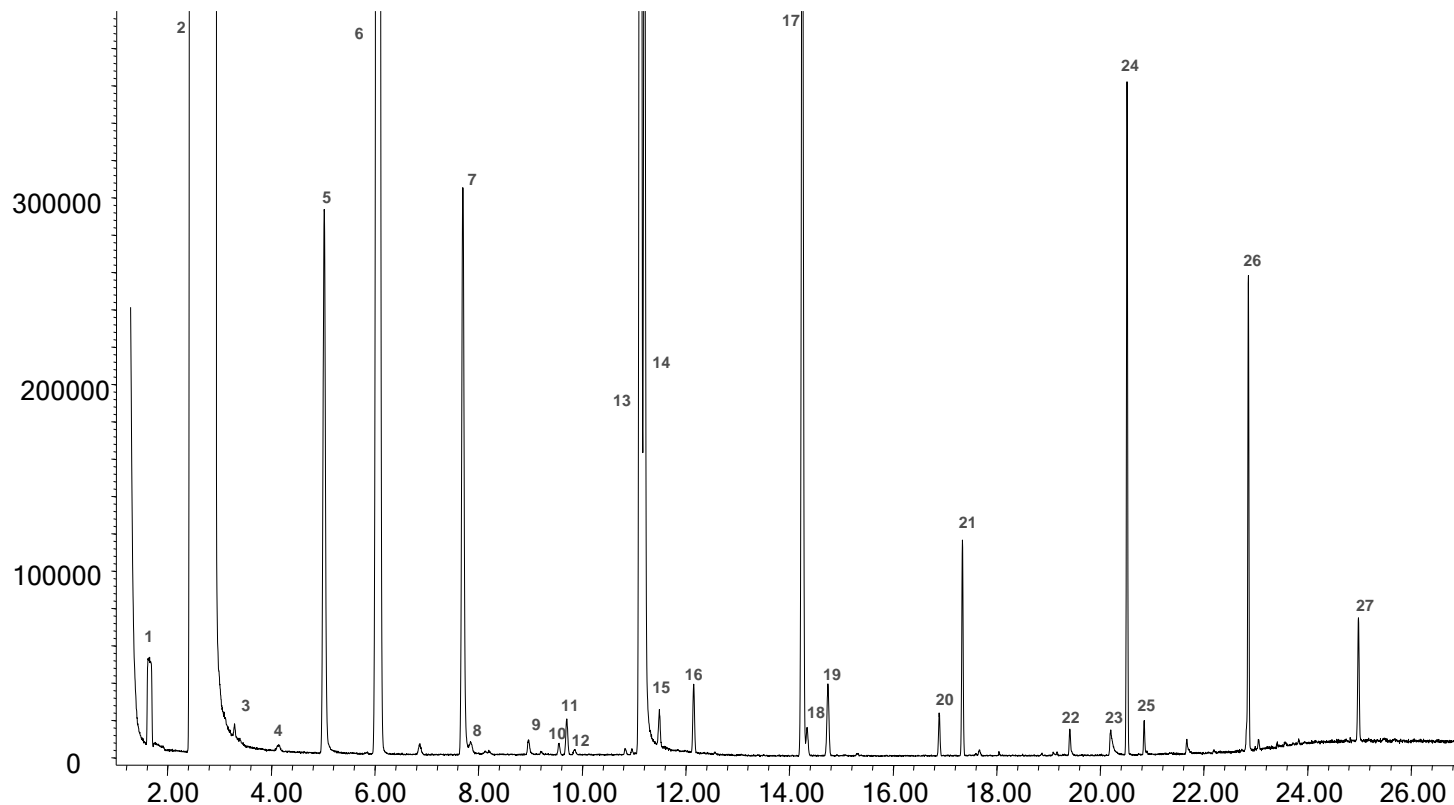
2 PPM aldehydes, alcohols and esters



- 1 acetyl aldehyde
- 2 methanol
- 3 ethanol
- 4 acetone
- 5 isopropanol
- 6 tert butanol
- 7 1-propanol
- 8 butyraldehyde
- 9 2,3 butanedione
- 10 ethyl acetate
- 11 2-butanol
- 12 isobutanol
- 13 1-butanol
- 14 2,3 pentanedione
- 15 ethyl propionate
- 16 propyl acetate
- 17 3-pentanol
- 18 isoamyl alcohol
- 19 active amyl alcohol
- 20 isobutyl acetate
- 21 1-pentanol
- 22 ethyl butanoate
- 23 hexanal
- 24 Isoamyl acetate
- 25 active amly acetate
- 26 1-hexanol
- 27 heptaldehyde
- 28 octanal
- 29 1-phenyl ethanol
- 30 ethyl octanoate
- 31 α -methylbenzyl acetate
- 32 m-anisaldehyde
- 33 ethyl decanoate

Column: Agilent J&W DB-624UI 30 m x 0.25 mm x 1.4 μ m (p/n 122-1334UI)
 Carrier : Helium 48.9 cm/s (approx. 1.8 mL/min) set at 35°C, EPC-Constant Flow
 Sampler: 7697A Headspace. temps: 85 °C oven, 1 ml loop 85°C, transfer line 100°C, 20 min vial eq. time, 0.20 min injection time, 15 psi loop fill pressure, 20 psi loop ramp rate, loop final pressure 10 psi, 0.05 min loop eq. time
 Inlet: Split, 5:1 at 220°C (total flow approximately 14 mL/min, and 14.9 psi)
 Inlet liner: Ultra Inert straight single taper, 1 mm (p/n 5190-4047)
 Oven: 35°C 5.66 min hold, 8.8°/min to 100°C (1.70 min hold), 13.3 °/min to 220°C (3.39 min hold), 22.1 °/min (3.43 min hold)
 Detector: MSD Scan range 30-400 AMU; MSD temps, source 230°C, quad 150 °C, transfer line 110 °C

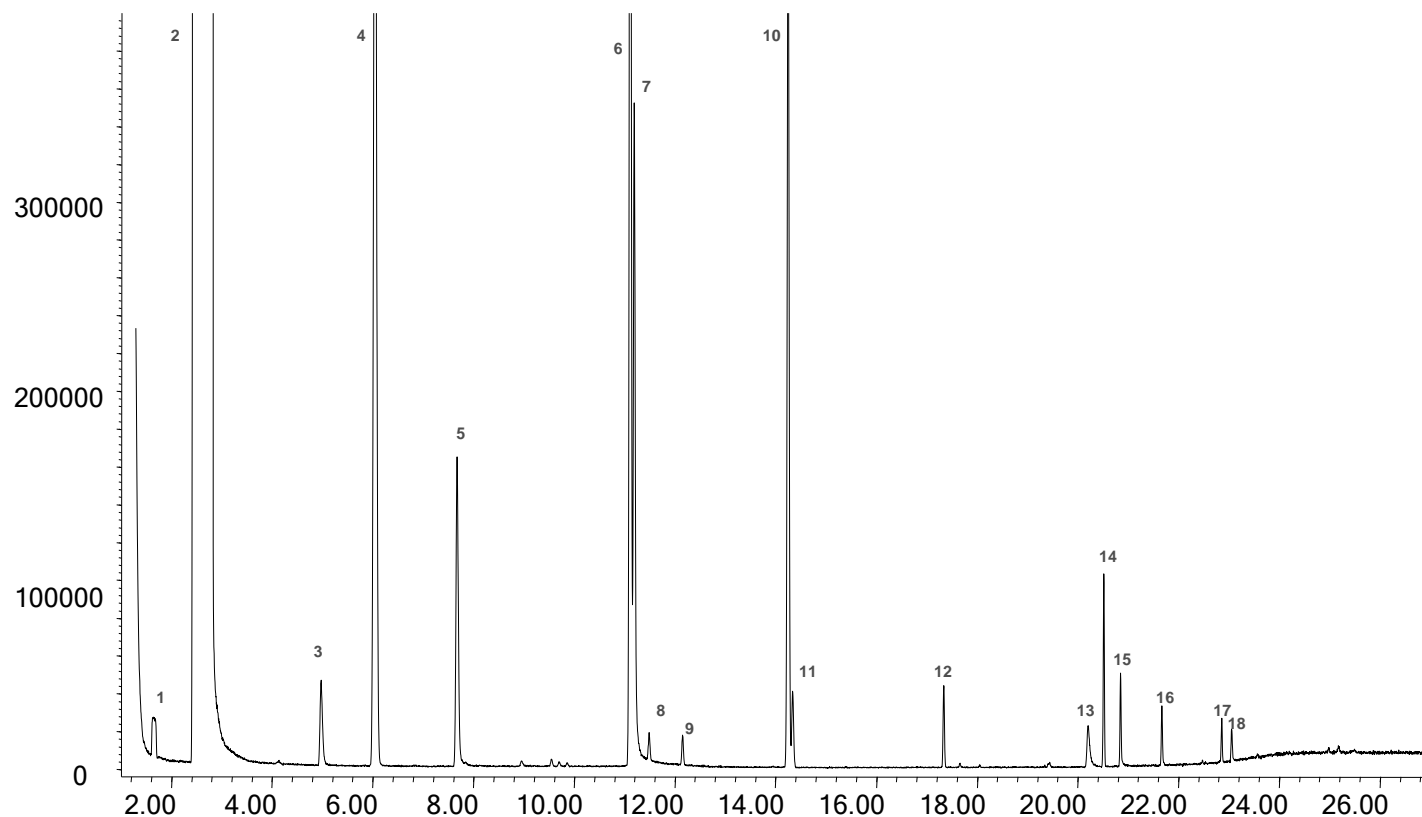
Pale Ale GC/MS Profile



- 1 acetyl aldehyde
- 2 ethanol
- 3 isopropanol
- 4 isobutyl aldehyde
- 5 1-propanol
- 6 ethyl acetate
- 7 isobutyl alcohol
- 8 3 methyl-1-butanal
- 9 1 butanol
- 10 ethyl propanoate
- 11 propyl acetate
- 12 acetal
- 13 Isoamyl alcohol
- 14 active amyl alcohol
- 15 isobutyl acetate
- 16 ethyl butanoate
- 17 Isoamyl acetate
- 18 Active amyl acetate
- 19 styrene (cinnamene)
- 20 β -myrcene
- 21 ethyl hexanoate
- 22 β -linolool
- 23 1-phenyl ethanol
- 24 ethyl octanoate
- 25 octanoic acid
- 26 ethyl decanoate
- 27 ethyl dodecanoate

Column: Agilent J&W DB-624UI 30 m x 0.25 mm x 1.4 μ m (p/n 122-1334UI)
 Carrier : Helium 48.9 cm/s (approx. 1.8 mL/min) set at 35°C, EPC-Constant Flow
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 Detector: MSD Scan range 30-400 AMU; MSD temps, source 230°C, quad 150 °C, transfer line

Lager Style GC/MS Profile

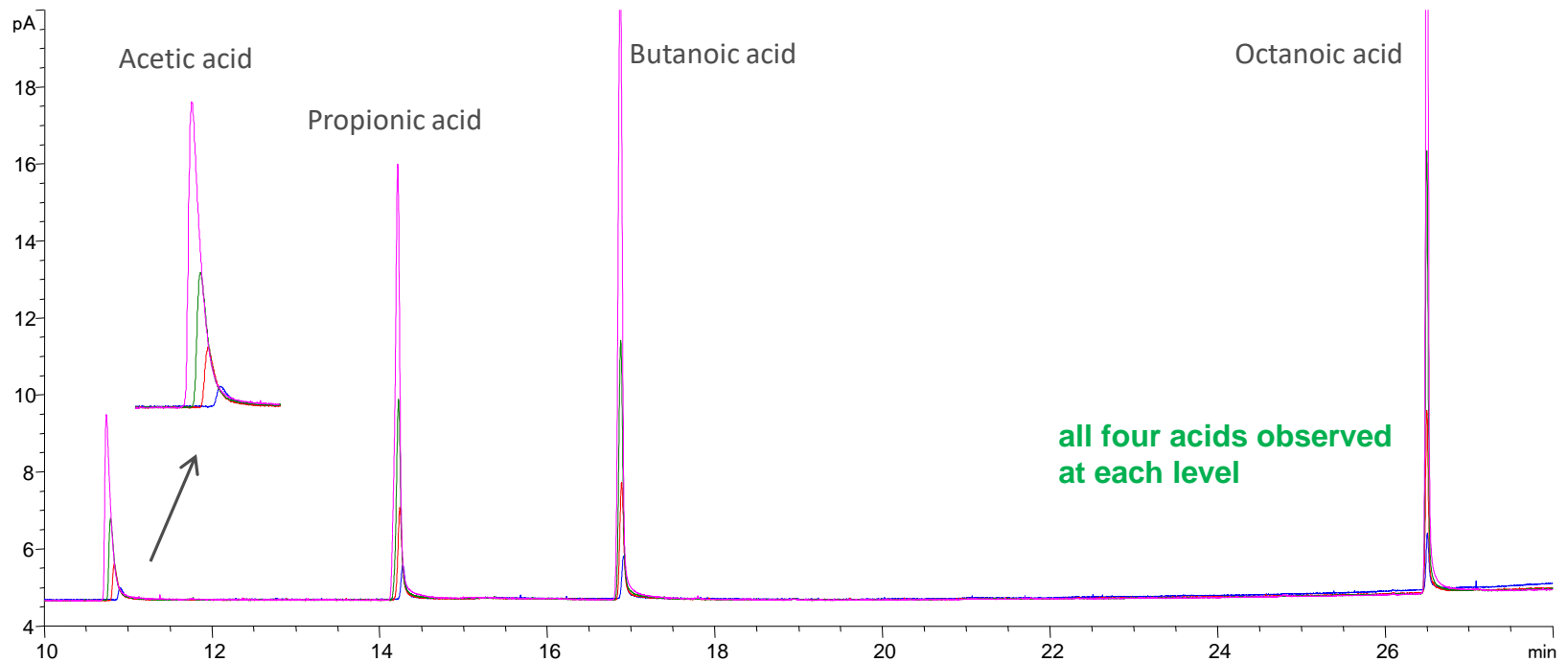


- 1 acetyl aldehyde
- 2 ethanol
- 3 1-propanol
- 4 ethyl acetate
- 5 isobutanol
- 6 isoamyl alcohol
- 7 active amyl alcohol
- 8 isobutyl acetate
- 9 ethyl butanoate
- 10 Isoamyl acetate
- 11 active amly acetate
- 12 ethyl hexanoate
- 13 1-phenyl ethanol
- 14 ethyl octanoate
- 15 octanoic acid
- 16 phenyl ethyl acetate
- 17 ethyl decanoate
- 18 decanoic acid

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Carrier : Helium 48.9 cm/s (approx. 1.8 mL/min) set at 35°C, EPC-Constant Flow
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Detector: MSD Scan range 30-400 AMU; MSD temps, source 230°C, quad 150 °C, transfer line

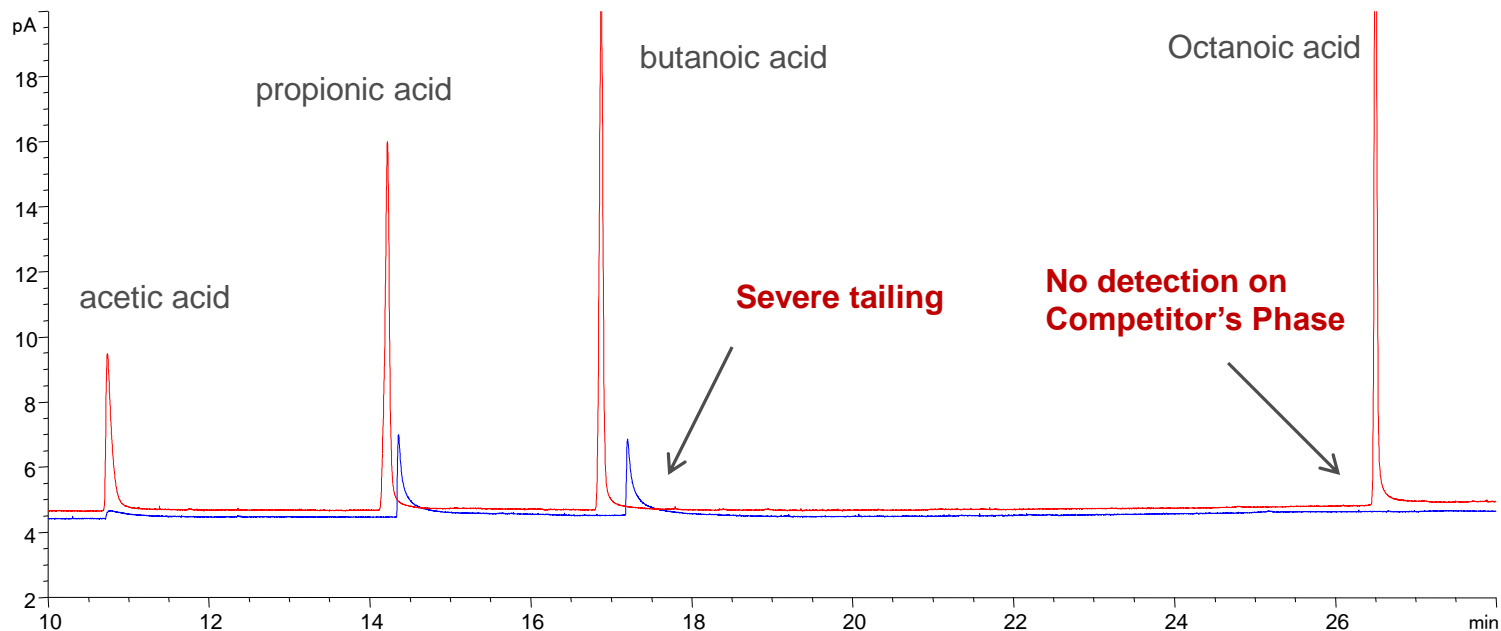
Agilent J&W DB-624UI-Organic Acid Performance

0.125 to 1 ng on column loading



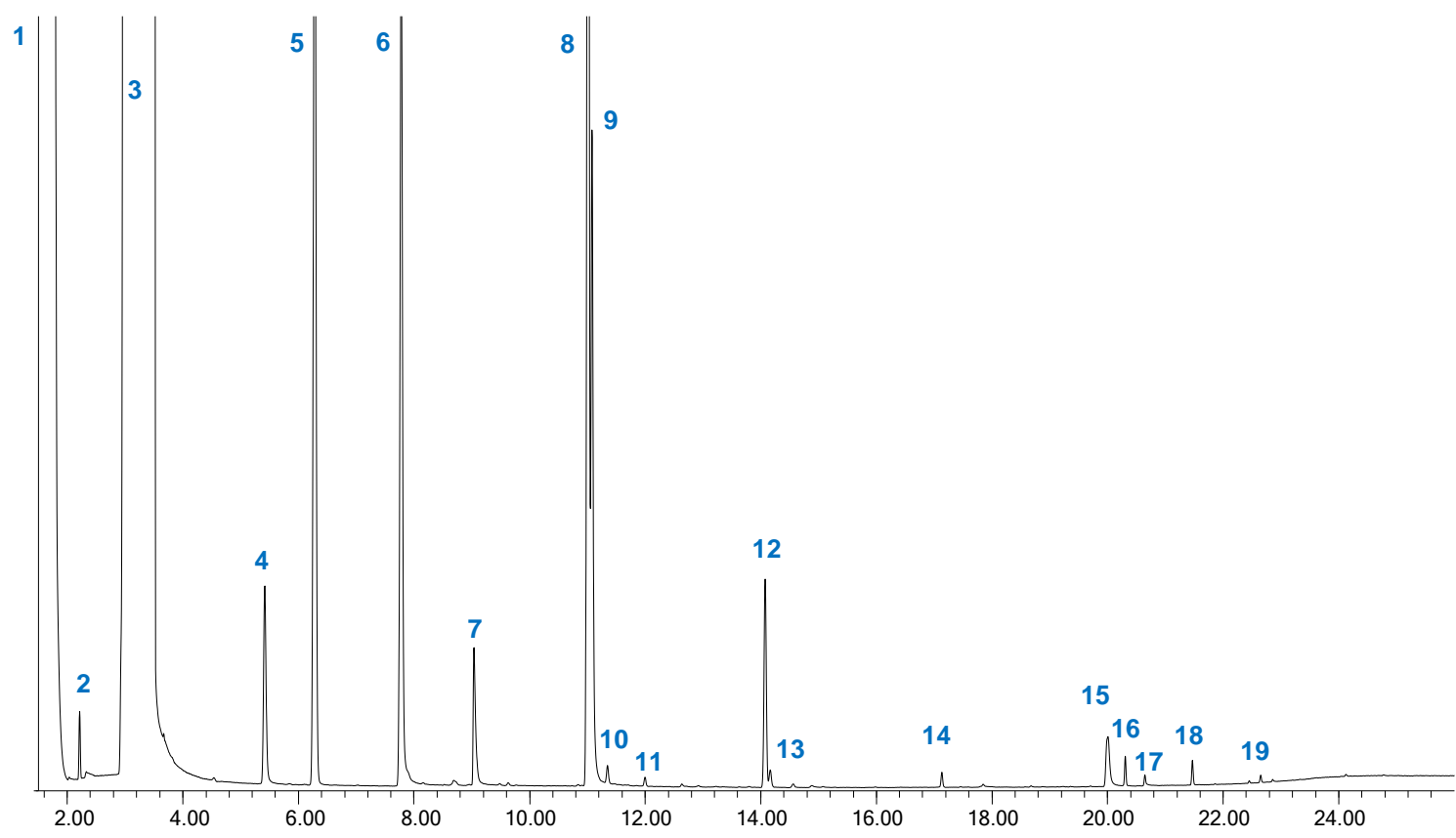
Column: Agilent J&W DB-624UI 30 m x 0.32 mm x 1.8 μ m (p/n 123-1334UI)
Oven: 35°C 7.45 min hold, 6.72°/min to 100°C(2.23min hold), 10.08 °/min to 220°C(4.47 min hold), 16.79 °/min (4.17 min hold)
Carrier : Helium 39.6 cm/s (approx. 2.6 mL/min) set at 35°C, EPC-Constant Flow
Inlet: Split, 20:1 at 250°C (total flow approx 51 mL/min, and 11.2 psi)
Inlet liner: Ultra Inert with wool
Detector: FID at 280°C, H2 @ 40 mL/min, Air @ 400 mL/min, N2 makeup @ 30 mL/min

Agilent J&W DB-624UI vs. Competitor's Traditional 624 column, 1 ng on column loading



Column: Agilent J&W DB-624UI 30 m x 0.32 mm x 1.8 μ m vs Rxi-624ms
Oven: 35°C 7.45 min hold, 6.72°/min to 100°C (2.23 min hold), 10.08 °/min to 220°C (4.47 min hold), 16.79 °/min (4.17 min hold)
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Detector: FID at 280°C, H₂ @ 40 mL/min, Air @ 400 mL/min, N₂ makeup @ 30 mL/min

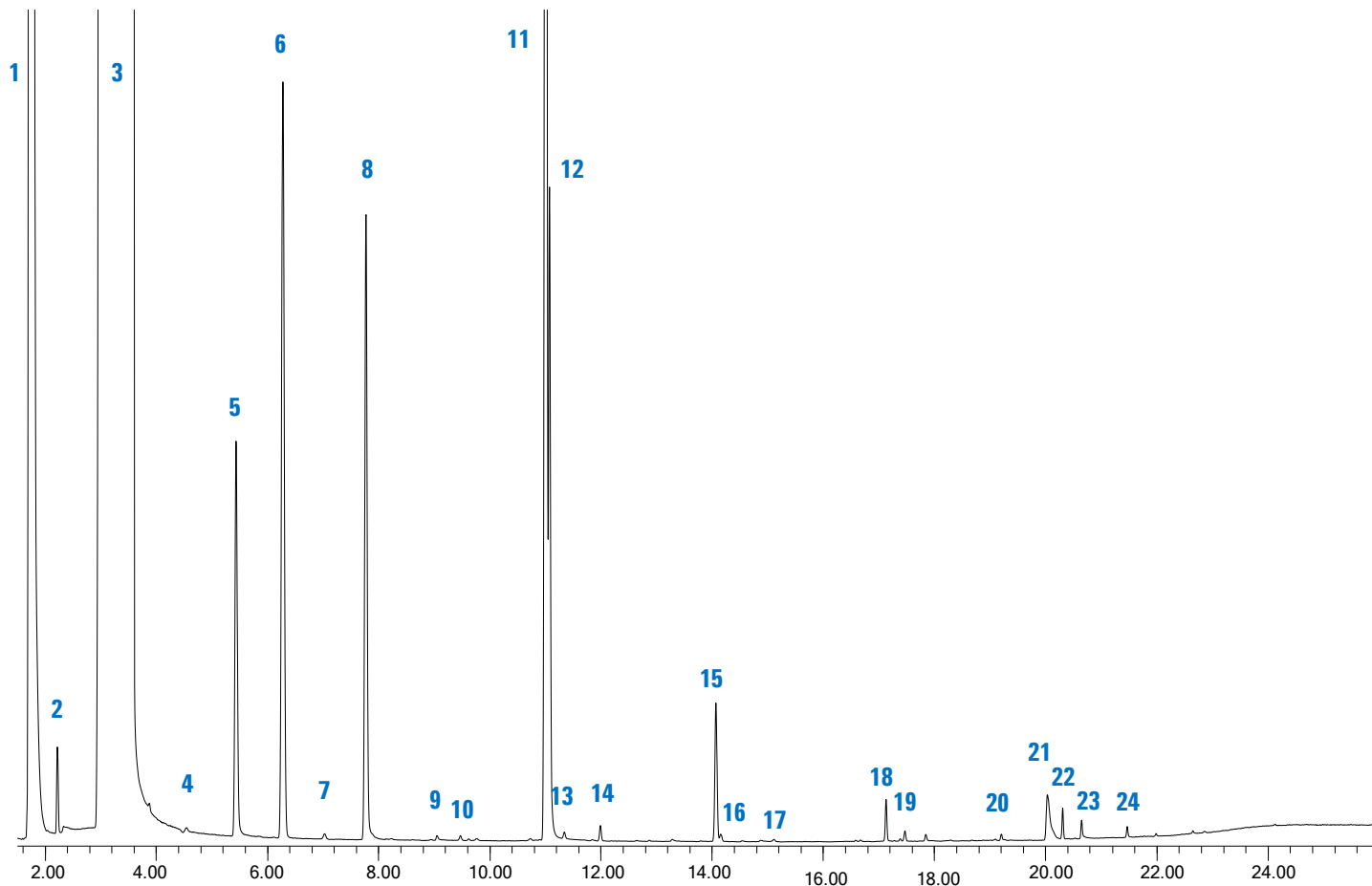
Hefe-Wizen



1	Carbon dioxide
2	Acetyl aldehyde
3	Ethanol
4	1-propanol
5	Ethyl acetate
6	Isobutyl alcohol
7	Acetic acid
8	Isoamyl alcohol (3-methyl 1-butanol)
9	Active amyl alcohol (2-methyl 1-butanol)
10	Isobutyl acetate
11	Butanoic acid ethyl ester
12	Isoamyl acetate (banana oil)
13	Active amyl acetate
14	Hexanoic acid ethyl ester
15	Phenyl ethyl alcohol
16	Octanoic acid ethyl ester
17	Octanoic acid
18	Acetic acid, phenyl ethyl ester
19	Decanoic acid ethyl ester

Column: Agilent J&W DB-624UI 30 m x 0.25 mm x 1.4 um (p/n 122-1334UI)
 Carrier : Helium 48.9 cm/s (approx. 1.8 mL/min) set at 37°C, EPC-Constant Flow
 Sampler: 7697A Headspace. temps: 85 °C oven, 1 ml loop 90°C, transfer line 105°C, 20 min vial eq. time, 0.20 min injection time, 15 psi loop fill pressure, 20 psi loop ramp rate, loop final pressure 10 psi, 0.05 min loop eq. time
 Inlet: Split, 5:1 at 220°C (total flow approximately 14 mL/min, and 14.9 psi)
 Inlet liner: Ultra Inert straight single taper, 1 mm (p/n 5190-4047)
 Oven: 37°C 5.66 min hold, 8.8°/min to 100°C (1.70 min hold), 13.3 °/min to 220°C (3.39 min hold), 22.1 °/min (3.43 min hold)
 Detector: MSD Scan range 30-400 AMU; MSD temps, source 230°C, quad 150 °C, transfer line 250 °C

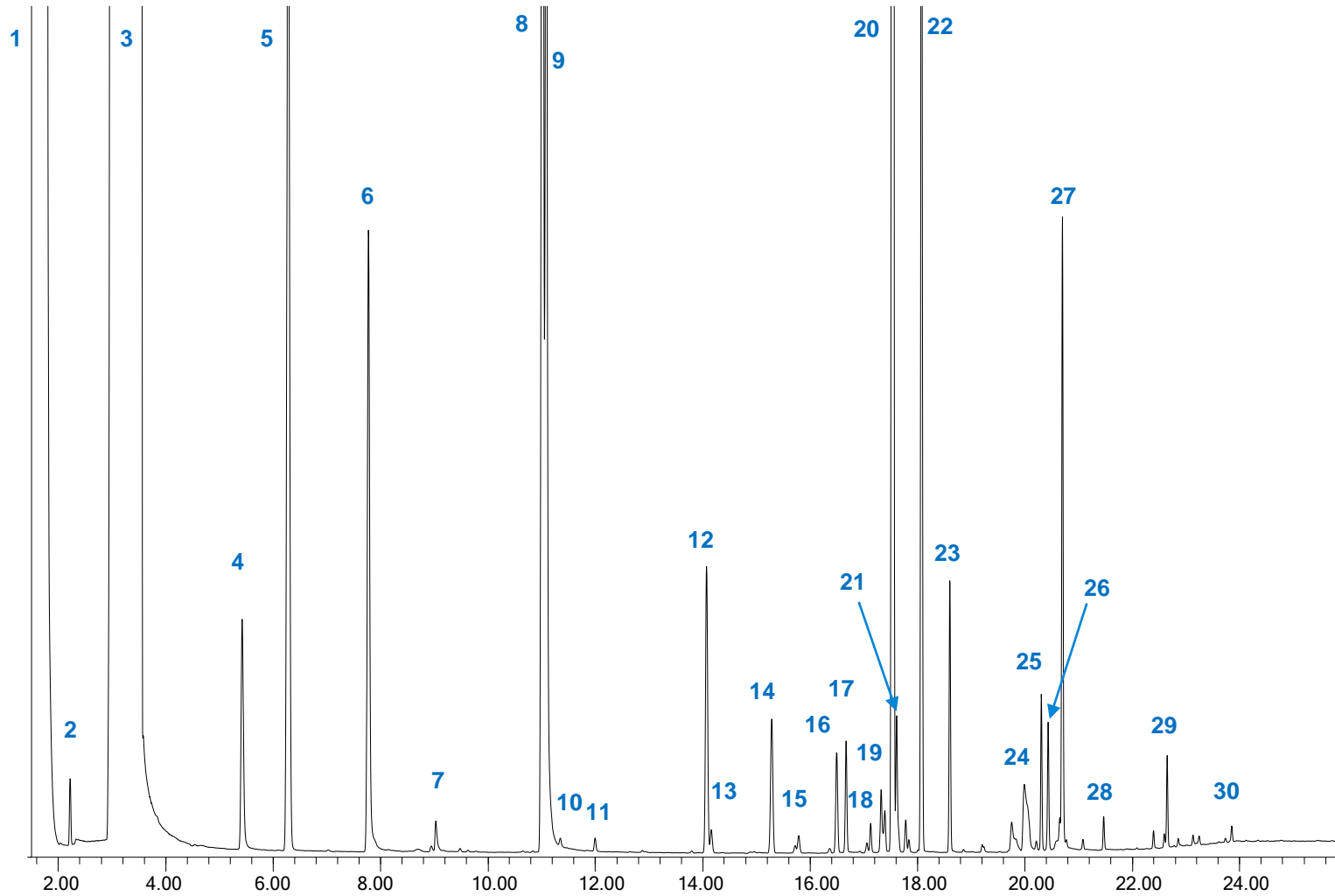
Kolsch



1	Carbon dioxide
2	Acetyl aldehyde
3	Ethanol
4	Isobutyl aldehyde
5	1-propanol
6	Ethyl acetate
7	Diethyl allyl alcohol
8	Isobutyl alcohol
9	Acetic acid
10	Propionic acid ethyl ester
11	Isoamyl alcohol (3-methyl 1-butanol)
12	Active amyl alcohol (2-methyl 1-butanol)
13	Isobutyl acetate
14	Butanoic acid ethyl ester
15	Isoamyl acetate (banana oil)
16	Active amyl acetate
17	Isobutyric acid isobutyl ester
18	Hexanoic acid ethyl ester
19	Isoamyl butanoate (check name)
20	Linolool
21	Phenyl ethyl alcohol
22	Octanoic acid ethyl ester
23	Octanoic acid
24	Acetic acid, phenyl ethyl ester

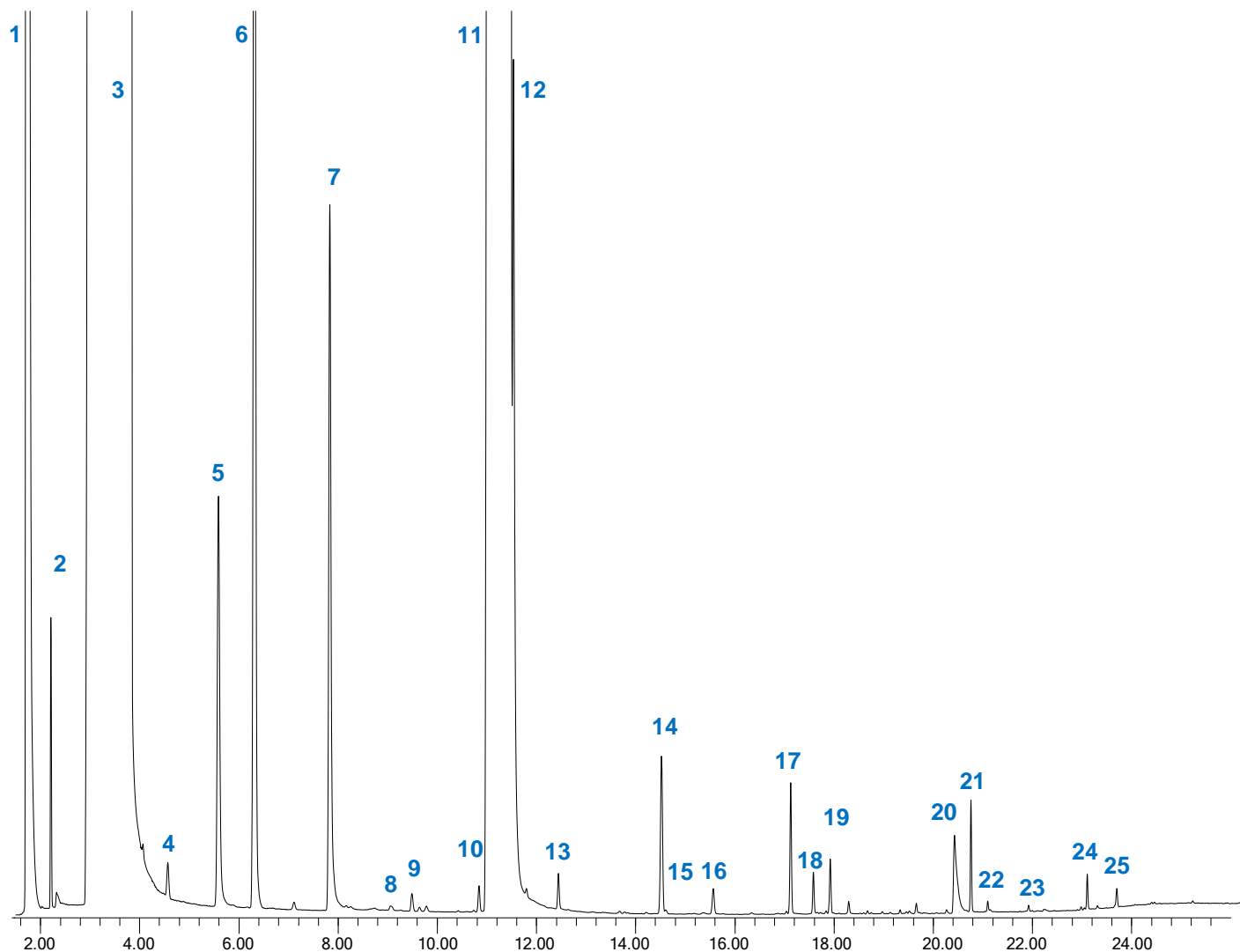
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 Detector: MSD Scan range 30-400 AMU; MSD temps, source 230°C, quad 150 °C, transfer line 250 °C

Lemon Shandy



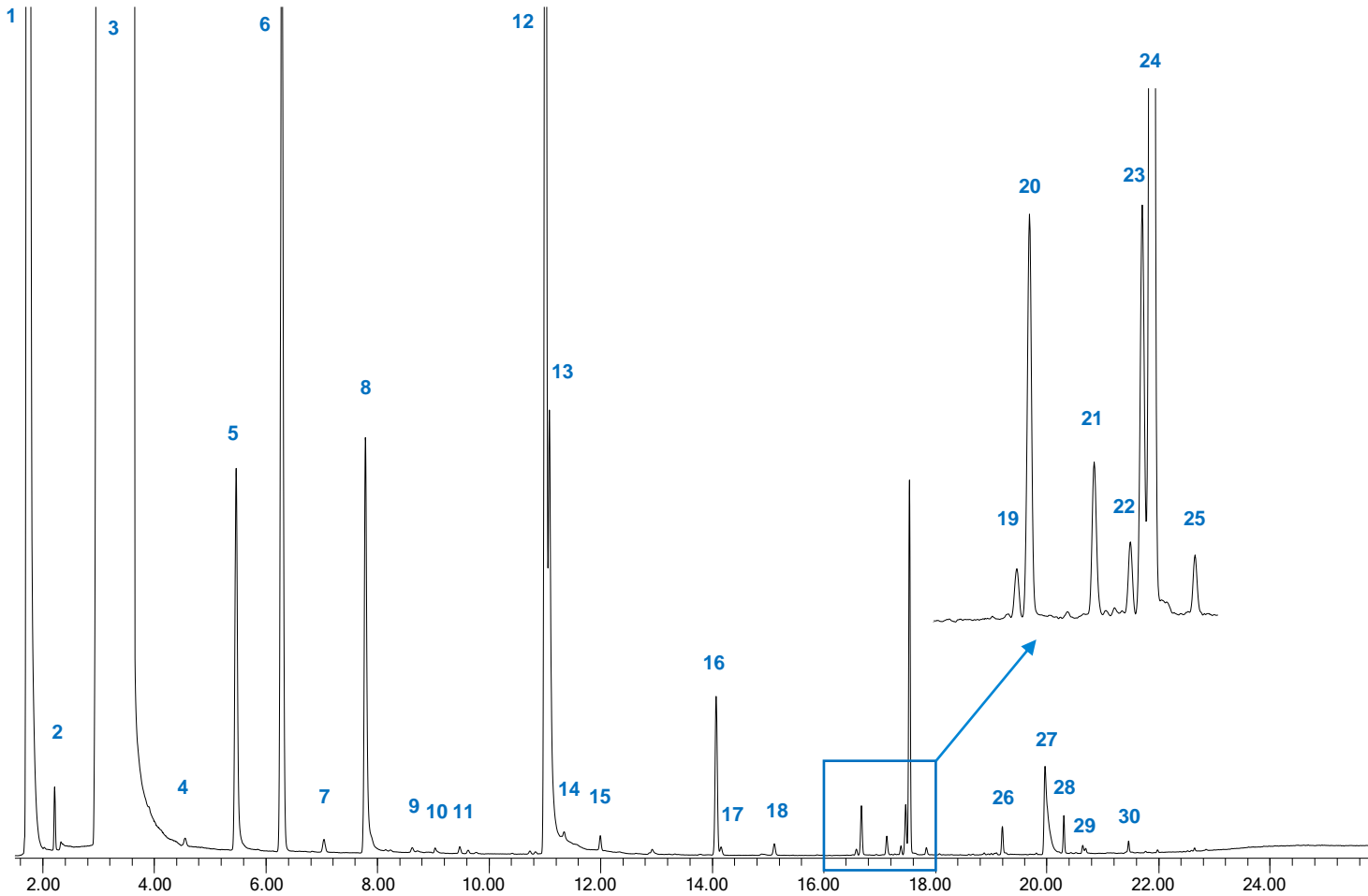
1	Carbon dioxide
2	Acetyl aldehyde
3	Ethanol
4	1-propanol
5	Ethyl acetate
6	Isobutyl alcohol
7	Acetic acid
8	Isoamyl alcohol (3-methyl 1-butanol)
9	Active amyl alcohol (2-methyl 1-butanol)
10	Isobutyl acetate
11	Butanoic acid ethyl ester
12	Isoamyl acetate (banana oil)
13	Active amyl acetate
14	α -pinene
15	camphene
16	β -pinene
17	b-Myrcene
18	α -phelladrene
19	Hexanoic acid ethyl ester
20	d-limonene
21	o-Cymene
22	α -Terpinene
23	α -Terpinolene
24	Phenyl ethyl alcohol
25	Octanoic acid ethyl ester
26	Terpinen-4-ol
27	α -Terpineol
28	Acetic acid, phenyl ethyl ester
29	Decanoic acid ethyl ester
30	β -Bisabolene

Citrus cocoanut ale



1	Carbon dioxide
2	Acetyl aldehyde
3	Ethanol
4	Isobutyl aldehyde
5	1-propanol
6	Ethyl acetate
7	Isobutyl alcohol
8	Acetic acid
9	Propionic acid ethyl ester
10	Isobutyric acid ethyl ester
11	Isoamyl alcohol (3-methyl 1-butanol)
12	Active amyl alcohol (2-methyl 1-butanol)
13	Isobutyl acetate
14	Butanoic acid ethyl ester
15	Isoamyl acetate (banana oil)
16	Active amyl acetate
17	Isobutyric acid isobutyl ester
18	b-Myrcene
19	Hexanoic acid ethyl ester
20	Phenyl ethyl alcohol
21	Octanoic acid ethyl ester
22	Octanoic acid
23	Acetic acid, phenyl ethyl ester
24	Decanoic acid ethyl ester
25	Carophyllene

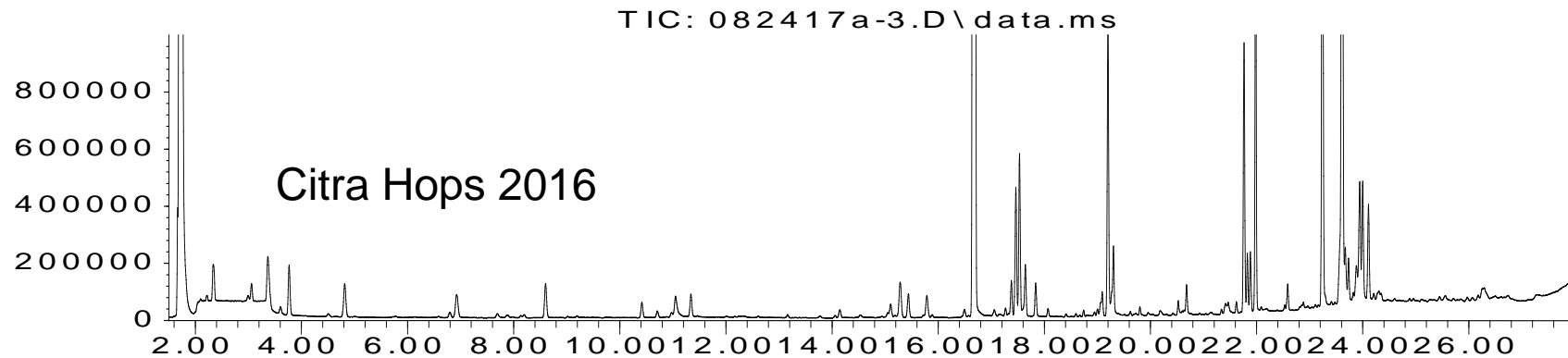
Tangerine Ale



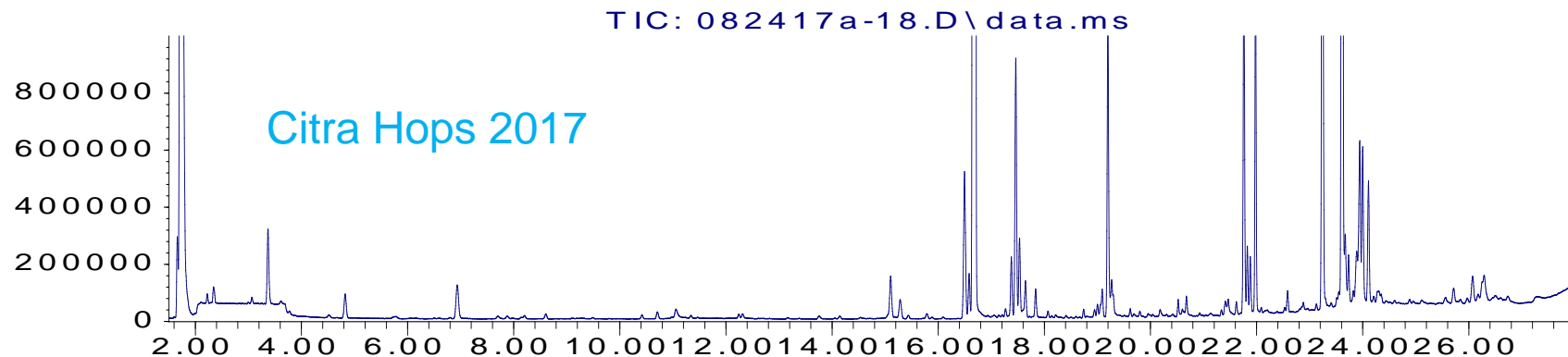
1	Carbon dioxide
2	Acetyl aldehyde
3	Ethanol
4	Isobutyl aldehyde
5	1-propanol
6	Ethyl acetate
7	Diethyl allyl alcohol
8	Isobutyl alcohol
9	Isobutyric acid methyl ester
10	Acetic acid
11	Propionic acid ethyl ester
12	Isoamyl alcohol (3-methyl 1-butanol)
13	Active amyl alcohol (2-methyl 1-butanol)
14	Isobutyl acetate
15	Butanoic acid ethyl ester
16	Isoamyl acetate (banana oil)
17	Active amyl acetate
18	Isobutyric acid isobutyl ester
19	1-butanol 2 methyl propionate
20	b-Myrcene
21	Hexanoic acid ethyl ester
22	Isoamyl butanoate
23	propionic acid 2 methyl,2 methyl butyl ester
24	Limonene
25	3-hexanoic acid 3 methyl 3 hexanoate
26	Linolool
27	Phenyl ethyl alcohol
28	Octanoic acid ethyl ester
29	Octanoic acid
30	Acetic acid, phenyl ethyl ester

Citra Hops comparison

Abundance

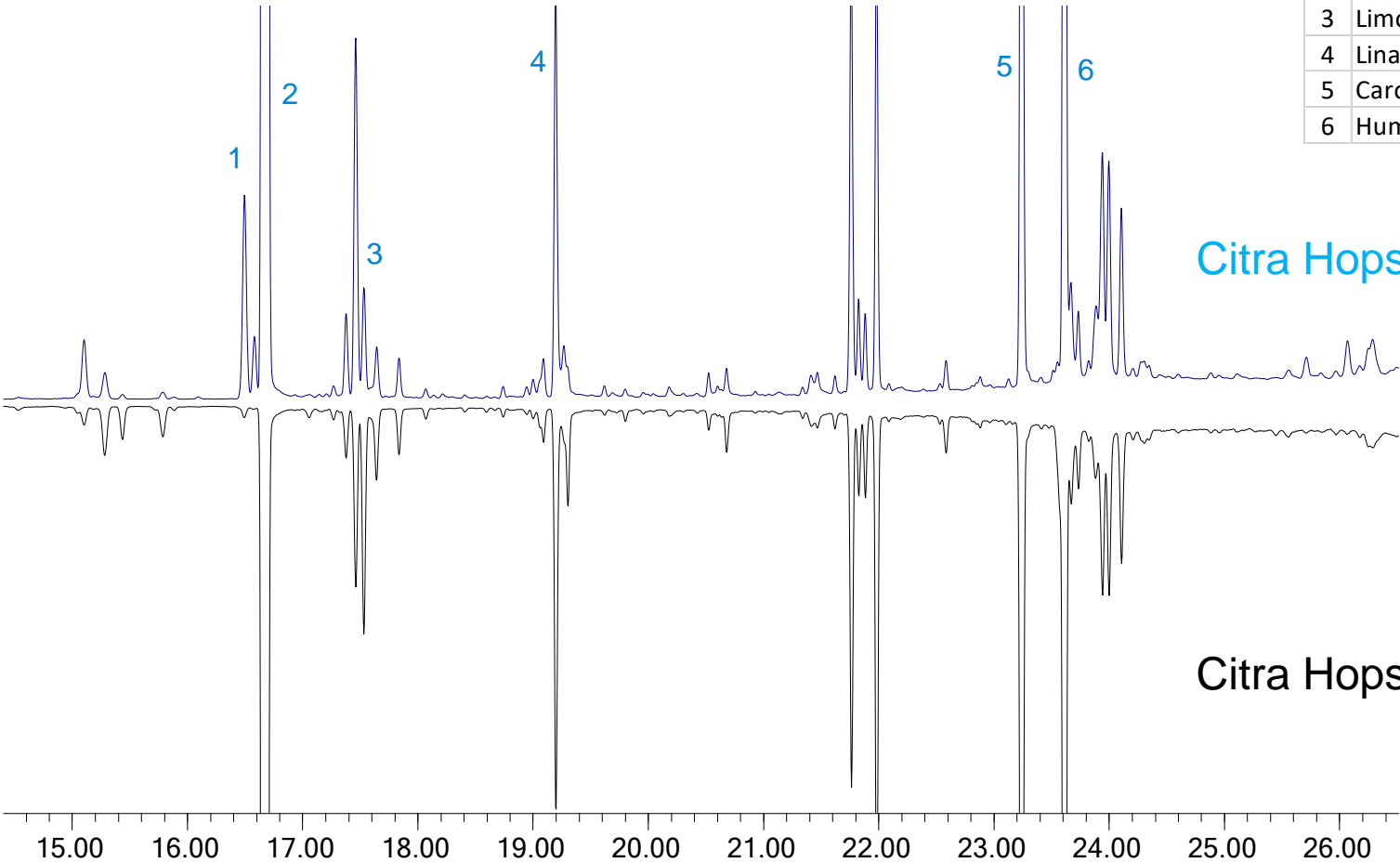


Abundance



Citra hops overlay

1	β -pinene
2	β -myrcene
3	Limonene
4	Linalool
5	Carophyllene
6	Humalene

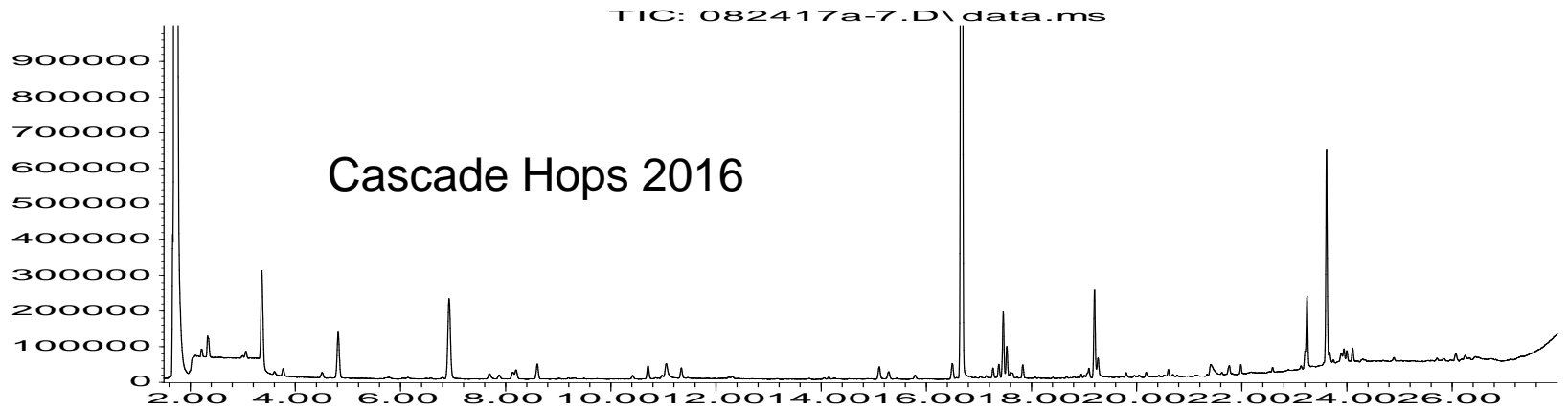


Citra Hops 2017

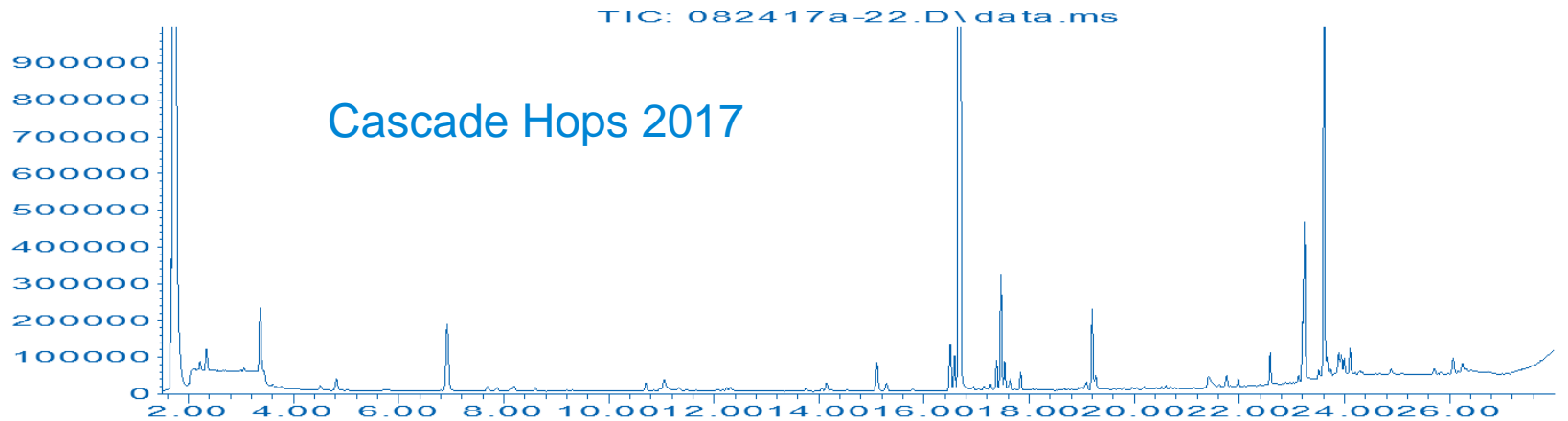
Citra Hops 2016

Cascade hops comparison

Abundance

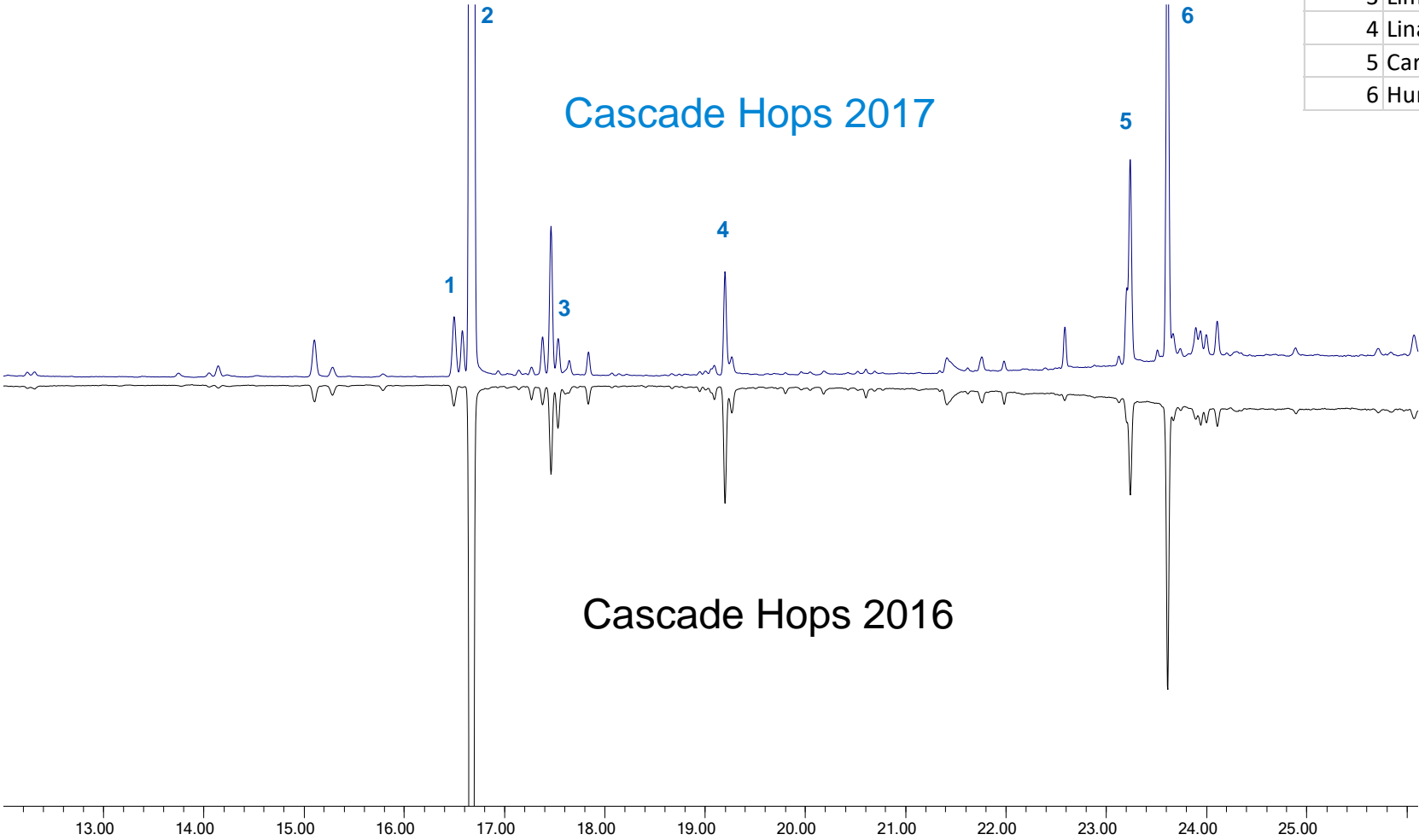


Abundance



Cascade Hops Overlay

1	β -pinene
2	β -myrcene
3	Limonene
4	Linalool
5	Carophyllene
6	Humalene



Is Beer Complex ?

Wide variety of styles

Natural product derived

Flavor impact of some minor components

Synergetic contributions of flavor elements

It's all about the beer

Broad Range of Analytical Tools used to Study Beer and the Brewing Process

GC-MS/MS

GC x GC

SPME- GC with SCD detection

Spectroscopy-

Molecular UV, IR

Atomic , ICP-OES, ICP-MS

LC-MS/MS

2 D LC



Opinions and discussions-Favorite Styles?

Ales

Lagers

Seasonal/specialty

Root beer with a kick?



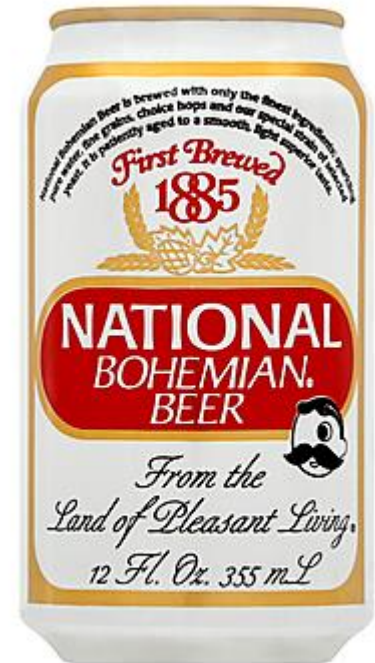
Opinions and discussions-Historical Prospective?

First beer you can remember

Cheap beer stories?

First really good beer you can remember

First time you brewed



Opinions and discussions-Where are your challenges?

Raw materials

water, barley, hops, yeast

rising costs

quality consistency

quality control

Opinions and discussions-Where are your challenges?

Processing

Malting

Milling

Mashing-lautering

Boil

Fermentation

Conditioning

Packing

Cleaning in place



Beer Advocate's top 10 for PA

The general statistical formula is:

$$\text{weighted rank (WR)} = (v \div (v+m)) \times R + (m \div (v+m)) \times C$$

R = average (mean) rating for the beer = (Rating)

v = number of ratings for the beer = (votes)

m = minimum ratings required to be considered (currently 54)
= (the average number of ratings across the dataset)

C = the mean rating across the dataset (currently 3.76)

Top Rated Beers: Pennsylvania (US)

		WR	Ratings
1	Parageusia1 Tired Hands Brewing Company American Wild Ale / 6.50% ABV	4.37	148
2	Tröegs Nugget Nectar Tröegs Brewing Company American Amber / Red Ale / 7.50% ABV	4.33	7,724
3	Le Roar Grrrz Bullfrog Brewery American Wild Ale	4.31	89
4	Sunday Morning Stout Weyerbacher Brewing Co. American Double / Imperial Stout / 11.30% ABV	4.31	855
5	Sunny Side Up (Little Amps! Double Coffee) Al's of Hampden / Pizza Boy Brewing American Double / Imperial Stout / 11.00% ABV	4.3	347
6	Cowbell Imperial Oatmeal Milk Stout Voodoo Brewing Company Milk / Sweet Stout / 8.50% ABV	4.3	231
7	Mago Tago Tired Hands Brewing Company American IPA / 7.80% ABV	4.29	175
8	DirtWolf Victory Brewing Company American Double / Imperial IPA / 8.70% ABV	4.29	5,822
9	Le Roar Grrrz Kriek Bullfrog Brewery American Wild Ale / 6.00% ABV	4.28	95
10	The Light That Spills Out Of The Hole In Your Head Tired Hands Brewing Company American Pale Ale (APA) / 6.00% ABV	4.27	200



U.S. BEER SALES VOLUME GROWTH 2014

OVERALL BEER
0.5%
197,124,407 bbls

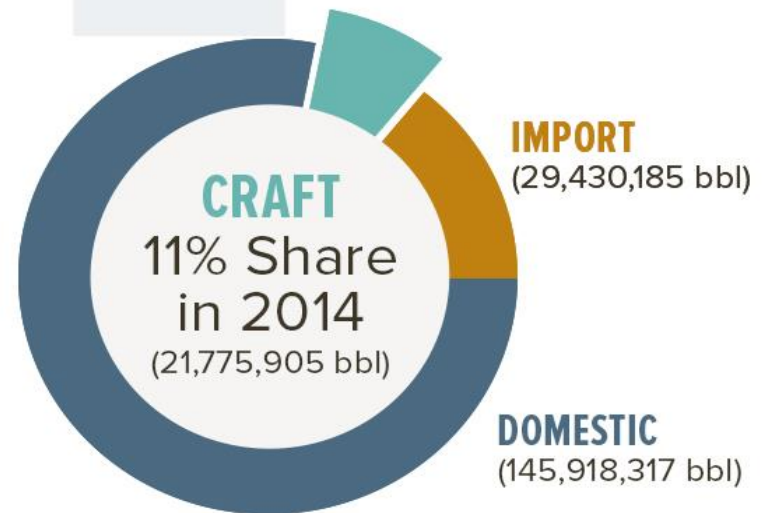
17.6%
CRAFT
21,775,905 bbls

IMPORT BEER
6.9%
29,430,185 bbls

36%
EXPORT CRAFT BEER
383,422 bbls

OVERALL BEER MARKET
\$101.5 BILLION

CRAFT BEER MARKET
\$19.6 BILLION
22% DOLLAR SALES GROWTH

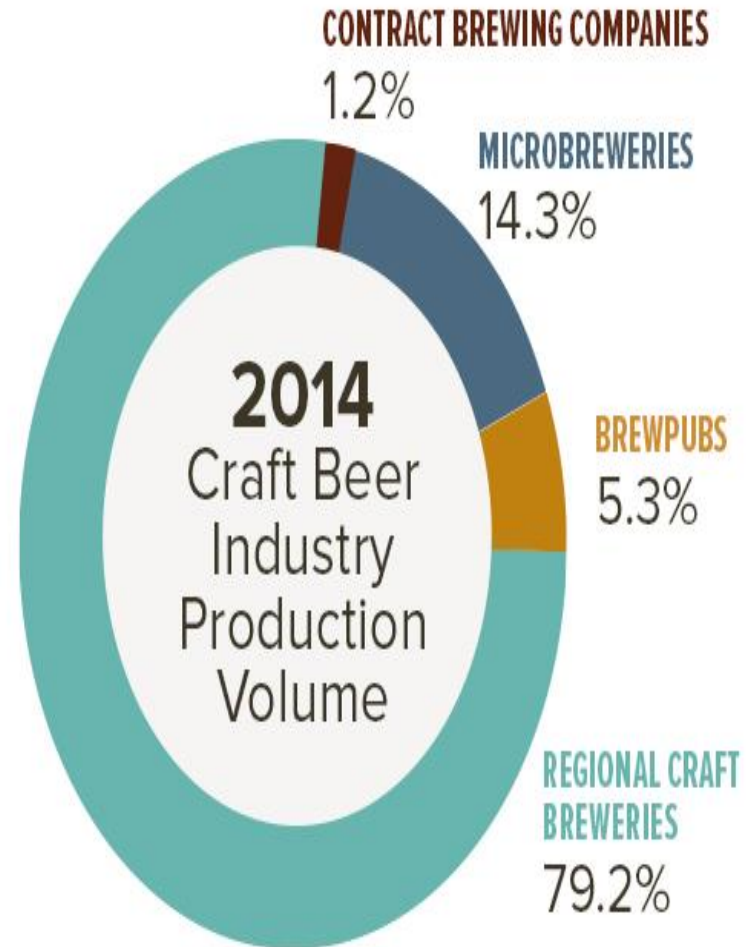


Source: Brewers Association, Boulder, CO

U.S. BEER PRODUCTION VOLUME 2014

18%
CRAFT

OVERALL
BEER
0.5%



Source: Brewers Association, Boulder, CO

U.S. BEER SALES VOLUME GROWTH 2016

OVERALL
BEER
0.0%

196,749,624 BBLS

6.2%
CRAFT

24,104,852 BBLS

IMPORT
BEER
6.8%

33,366,352 BBLS

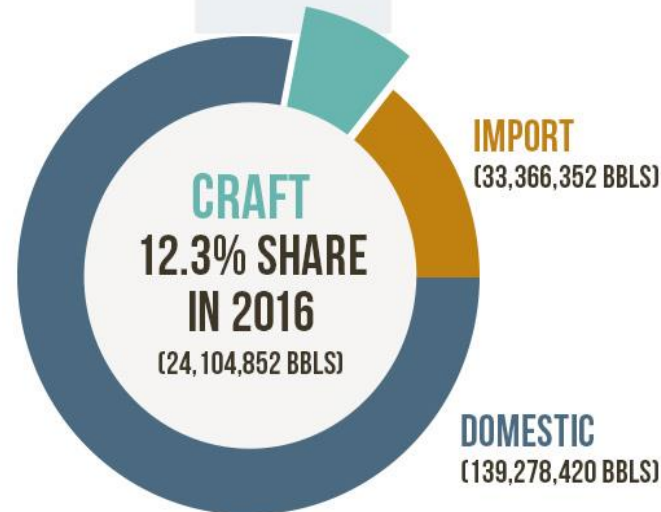
4.4%
EXPORT
CRAFT
BEER

465,617 BBLS

OVERALL BEER MARKET
\$107.6 BILLION

CRAFT BEER MARKET
\$23.5 BILLION

10% DOLLAR SALES GROWTH

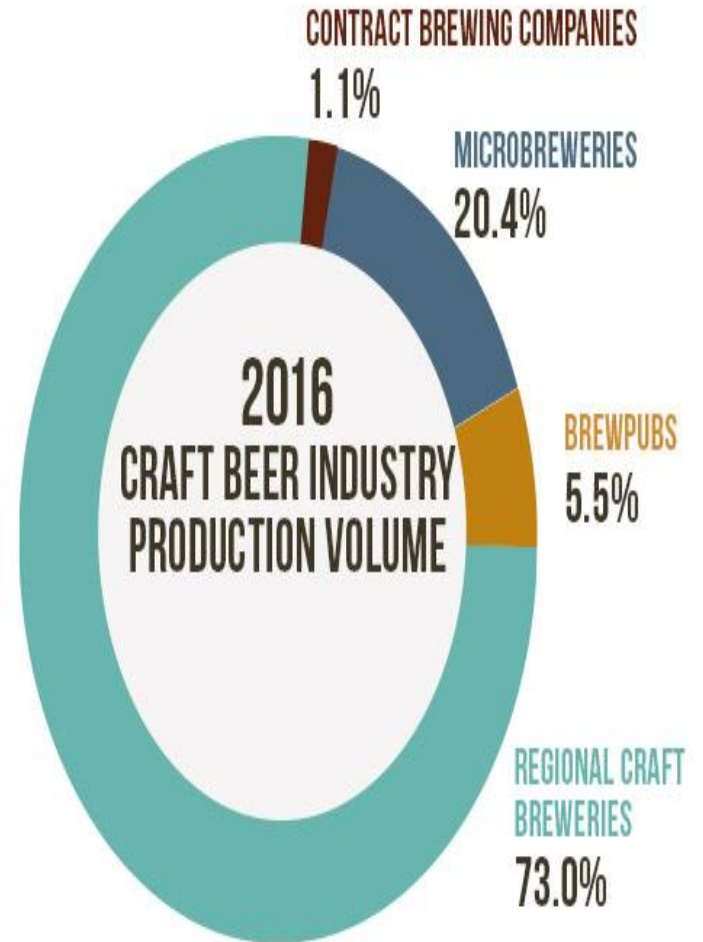


SOURCE: BREWERS ASSOCIATION, BOULDER, CO

U.S. BEER PRODUCTION VOLUME 2016

6%
CRAFT

OVERALL
BEER
0.0%



SOURCE: BREWERS ASSOCIATION, BOULDER, CO

Beer Advocate Top 10 Rated California Beers

Top Rated Beers: California (US)

		Avg	Ratings
1	Pliny The Younger Russian River Brewing Company American Double / Imperial IPA / 10.25% ABV	4.67	3,082
2	Fundamental Observation Bottle Logic Brewing American Double / Imperial Stout / 14.30% ABV	4.68	853
3	AleSmith Speedway Stout - Vietnamese Coffee (Bourbon Barrel Aged) AleSmith Brewing Company American Double / Imperial Stout / 12.00% ABV	4.67	753
4	Pliny The Elder Russian River Brewing Company American Double / Imperial IPA / 8.00% ABV	4.6	14,471
5	Citra Kern River Brewing Company American Double / Imperial IPA / 8.00% ABV	4.56	1,751
6	Parabola Firestone Walker Brewing Co. Russian Imperial Stout / 14.00% ABV	4.54	6,498
7	Duck Duck Gooze The Lost Abbey Gueuze / 7.00% ABV	4.54	1,777
8	Pugachev Royale Hangar 24 Brewery Russian Imperial Stout / 14.80% ABV	4.65	232
9	Westly Sante Adairius Rustic Ales American Wild Ale / 8.50% ABV	4.63	256
10	Parabajava Firestone Walker Brewing Co. Russian Imperial Stout / 14.00% ABV	4.55	737

Beer Advocate Top 10 Most Popular US Beers

Most Popular Beers			Avg	Ratings
1	Founders Breakfast Stout Founders Brewing Company American Double / Imperial Stout / 8.30% ABV		4.47	16,536
2	90 Minute IPA Dogfish Head Craft Brewery American Double / Imperial IPA / 9.00% ABV		4.24	15,573
3	Pliny The Elder Russian River Brewing Company American Double / Imperial IPA / 8.00% ABV		4.6	14,471
4	Two Hearted Ale Bell's Brewery, Inc. American IPA / 7.00% ABV		4.26	14,327
5	Sculpin IPA Ballast Point Brewing Company American IPA / 7.00% ABV		4.37	13,844
6	Heady Topper The Alchemist Brewery and Visitors Center American Double / Imperial IPA / 8.00% ABV		4.65	13,438
7	Hopslam Ale Bell's Brewery, Inc. American Double / Imperial IPA / 10.00% ABV		4.43	13,044
8	Founders KBS (Kentucky Breakfast Stout) Founders Brewing Company American Double / Imperial Stout / 11.80% ABV		4.56	12,610
9	Old Rasputin Russian Imperial Stout North Coast Brewing Co. Russian Imperial Stout / 9.00% ABV		4.28	12,217
10	Bourbon County Brand Stout Goose Island Beer Co. American Double / Imperial Stout / 13.80% ABV		4.5	12,20

Topics Discussed Today

What can GC and GC/MS do for? Think aroma and it's contribution to flavor

Pertinent example GC and GC/MS analyses

It's all about the beer..

Broad range of tools used to study beer and the brewing process

Opinions and discussions

Related References

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2. Ken Lynam “Screen Beer by GC/MS Static Headspace with an Agilent J&W DB-624 Ultra Inert Capillary Column” Agilent publication # 5991-1136EN
3. Yunfei Bai, Hao Wang, Shun Na “Determination of diacetyl (butanedione) and pentadione in beer by HS-GC” Agilent publication # 5991-1563EN
4. Beer Advocate “Top Beers” list on-line blog
5. Beer and Brewing Magazine “Myrcene” on-line magazine
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9. A.J. DeLange Wetnewf.org “Measuring Bitterness” on-line blog
10. R.E. Miracle, S.E. Ebeler and C.W. Bamforth “The Measurement of Sulfur-Containing Aroma Compounds in Samples form Production-Scale Brewery Operations” J. Am. Soc. Brew. Chem. 63(3):129-134,2005
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Thank You for your kind attention

