Determining Flavor and Fragrance Formulation by Gas Chromatography

Essential oil analysis

Introduction: Essential oils have a long history as flavor additives and herbal remedies. Today, the flavoring of foods and oral care products with mint is economically of growing importance and the rising demand for all-natural products drives a need for more rigorous testing. This work demonstrates a robust GC method determining the oil formulation. The reliable methods of analyzing specific components with high resolution can be used to monitor product quality.

GC instrument: Agilent 7890 w/ FID
Column: *GsBP-Inowax 30m x 0.25mm x 0.5um*
Oven: 75 °C (hold 8 min) to 250 °C at 4 °C/min Carrier: Hydrogen, column flow 1.5ml/min
Inlet: Split, 240 °C, split flow 30ml/min Detector: FID 260 °C
Sample: Peppermint oil standard (SPECTRUM, CAS:8006-90-4)

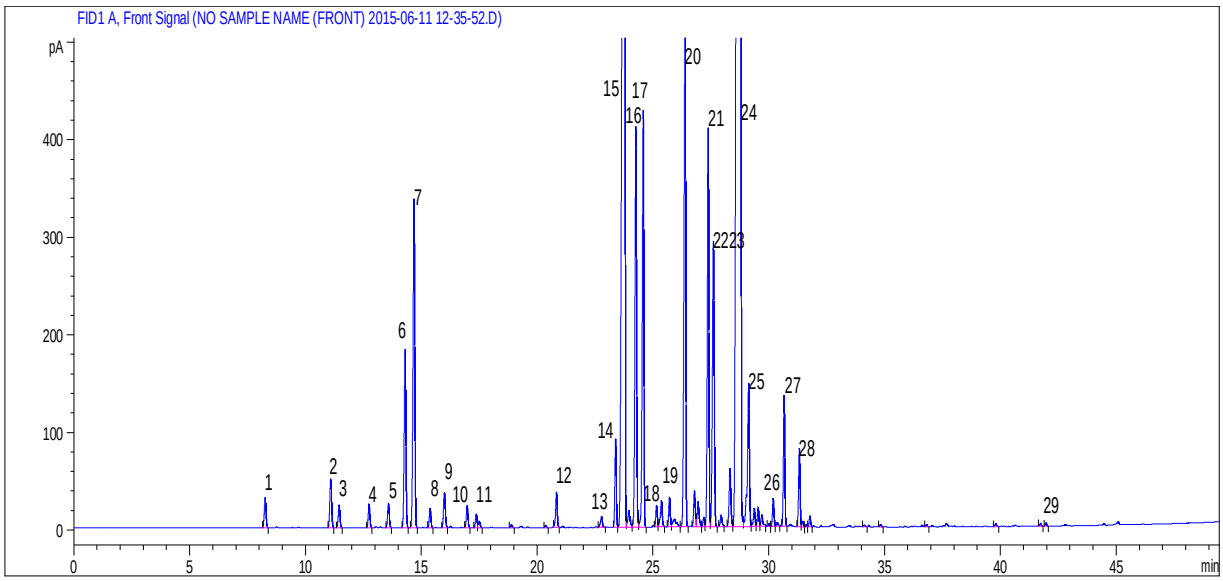


Figure 1. Gas Chromatogram of peppermint oil standard using GsBP-Ionwax column.

1	α-Pinene	11	Terpinolene	21	Neomenthol
2	β-Pinene	12	3-Octanol	22	Terpinen-4-ol
3	Sabinene	13	l-Octen-3-ol	23	β-Caryophyllene
4	Myrcene	14	trans-Sabinene hydrate	24	l-Menthol
5	α-Terpinene	15	l-Menthone	25	Pulegone
6	l-Limonene	16	Menthofuran	26	α-Terpineol
7	1,8-Cineol	17	d-Isomenthone	27	Germacrene-D
8	cis-Ocimene	18	β-Bourbonene	28	Piperitone
9	γ-Terpinene	19	Linalool	29	Viridiflorol
10	p-Cymene	20	Menthyl acetate		

GC: Agilent 7890 w/ FID Column: GsBP-Inowax 30m x 0.25mm x 0.5um

Oven: 75 °C (hold 8 min) to 250 °C at 4 °C/min

Carrier: Hydrogen, column flow 1.5ml/min

Inlet: Split, 240 °C, split flow 30ml/min Detector: FID 260 °C

Sample: customer's mentha oil

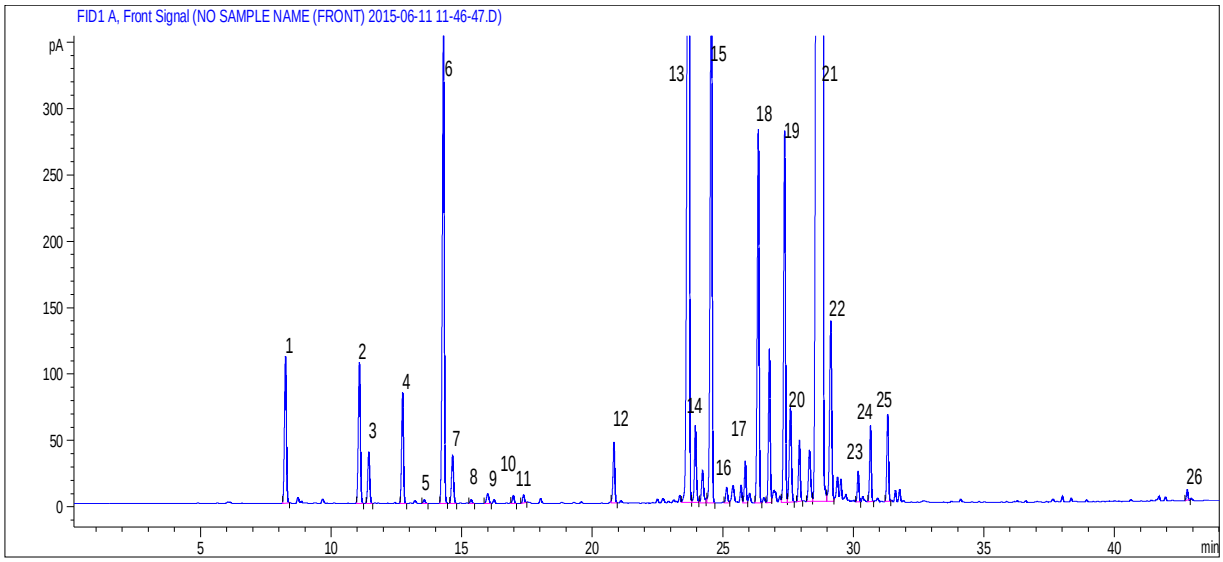


Figure 2. Gas Chromatogram of customer's mentha oil using GsBP-Inowax column.

1	α-Pinene	10	p-Cymene	19	Neomenthol
2	β-Pinene	11	Terpinolene	20	Terpinen-4-ol
3	Sabinene	12	3-Octanol	21	l-Menthol
4	Myrcene	13	l-Menthone	22	Pulegone
5	α-Terpinene	14	Menthofuran	23	α-Terpineol
6	l-Limonene	15	d-Isomenthone	24	Germacrene-D
7	1,8-Cineol	16	β-Bourbonene	25	Piperitone
8	cis-Ocimene	17	Linalool	26	Viridiflorol
9	γ-Terpinene	18	Menthyl acetate		

Lavender essential oil is one of the most versatile essential oils. It is an often used in hair and skin care products and also as a component in the bouquet of fragrances found in perfumes. We also provide the analysis results for lavender oil as follows.

GC: Agilent 7890 w/ FID Column: GsBP-Inowax 30m x 0.25mm x 0.5um

Oven: 50 °C (hold 2 min) to 5°C /min to 80°C (hold 4min), 4°C /min to 250 °C(hold 1min)

Carrier: Hydrogen, column flow 1.5ml/min

Inlet: Split, 240 °C, split flow 30ml/min Detector: FID 260 °C

Sample: Lavender essential oil

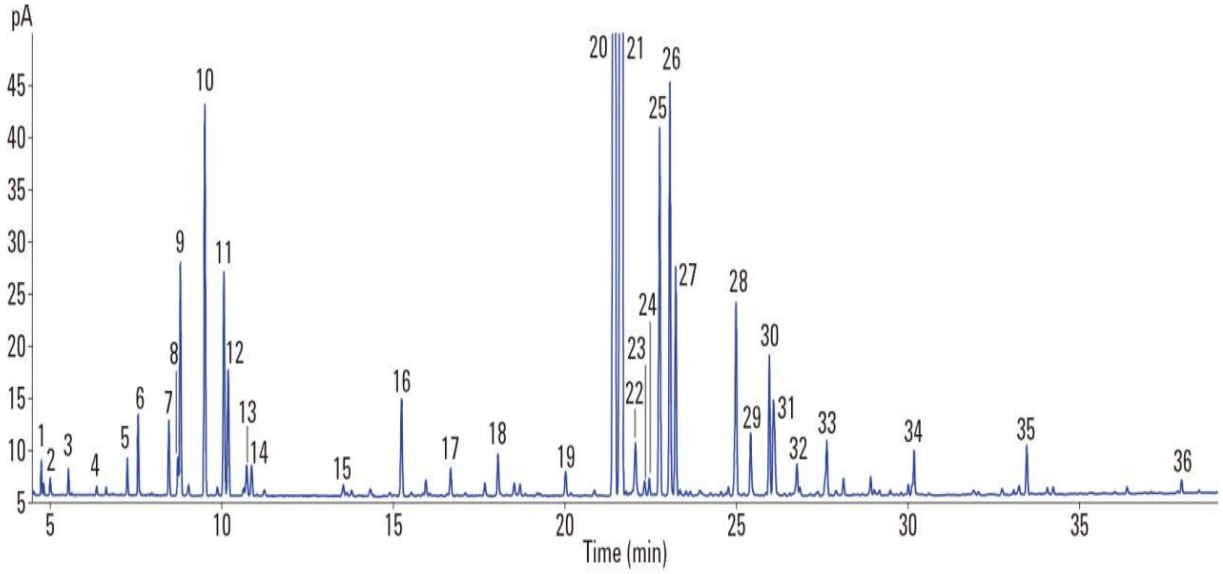


Figure 3. Gas Chromatogram of Lavender essential oil using GsBP-Inowax column.

1	α-Pinene	10	*cis*-β-Ocimene	19	Camphor	28	Lavandulyl acetate
2	a-Thujene	11	*trans*-β-Ocimene	20	β-Linalool	29	β-Farnesene
3	Camphene	12	3-Octanone	21	Linalool acetate	30	Lavandulol
4	β-Pinene	13	O-cymene	22	a-Santalene	31	a-Terpineol
5	3-carene	14	Hexyl acetate	23	Bornyl acetate	32	Bornanol
6	Myrcene	15	Hexyl isobutanoate	24	a-Bergamotene	33	Nerol acetate
7	D-limonene	16	1-Octen-3-yl-acetate	25	Caryophyllene	34	Geranyl acetate
8	β-Phellandrene	17	Hexyl butyrate	26	Terpinen-4-ol	35	Geraniol
9	Eucalyptol	18	1-Octen-3-ol	27	Lavandulyl acetate	36	Caryophyllene oxide

Conclusion: Peppermint oil have a characteristic pattern and isotope ratios (not published here) that is common to most samples. This pattern is observed in customer's sample and it is an indication that the oil is peppermint oil and is not adulterated. Because the clear pattern with high resolution is required to determine the oil formulation, we switched to the polar column with the higher film thickness. For reduction of the analytical time, 30m column is recommended. After adjusting the instrumentation condition, the baseline separation of 29 components in peppermint oil could be achieved. But due to the instrumental and environmental variation, the detail parameters need to be further changed in the practical application.

Fragrance analysis

Gas chromatography (GC) are by far the most important analytical techniques in the perfume industry. Because of the complexity of perfumes, use is made of GC columns and conditions that offer maximum resolution rather than minimum analysis time.

GC: Agilent 7890 w/ FID Column: GsBP-FFAP 50m x 0.32mm x 0.33um

Oven: 100°C (hold 1 min) to 250 °C (hold 1min) at 5 °C/min

Carrier: Hydrogen, column flow 1.5ml/min

Inlet: Split, 240 °C, split flow 30ml/min Detector: FID 260 °C

Sample: Fragrance mix standard

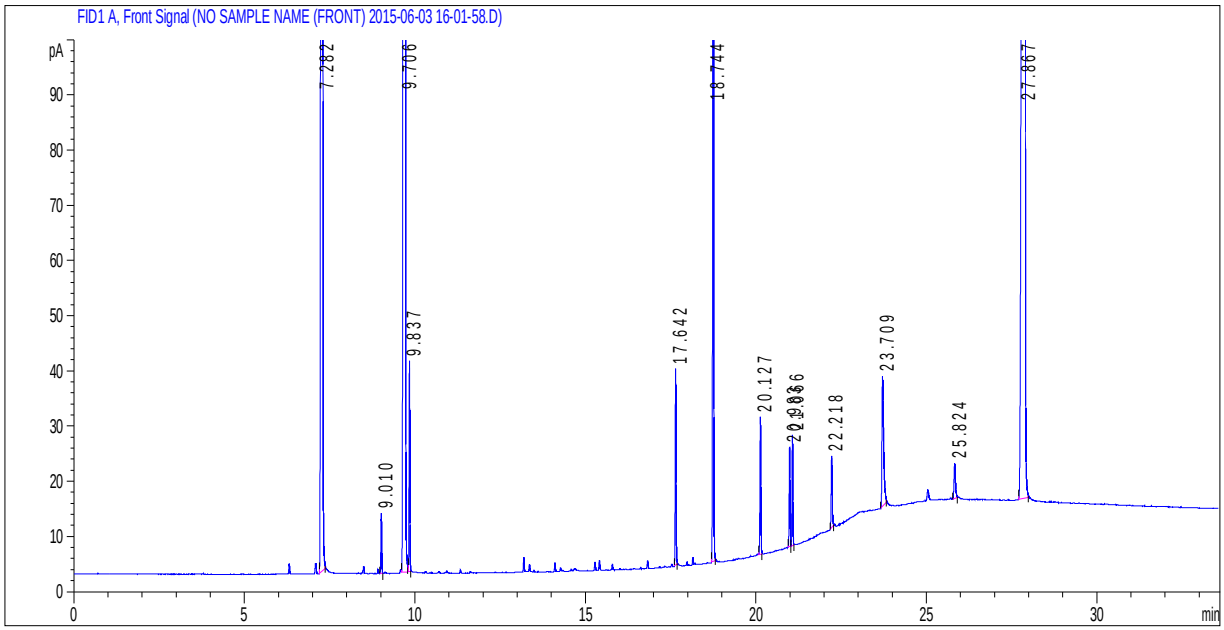
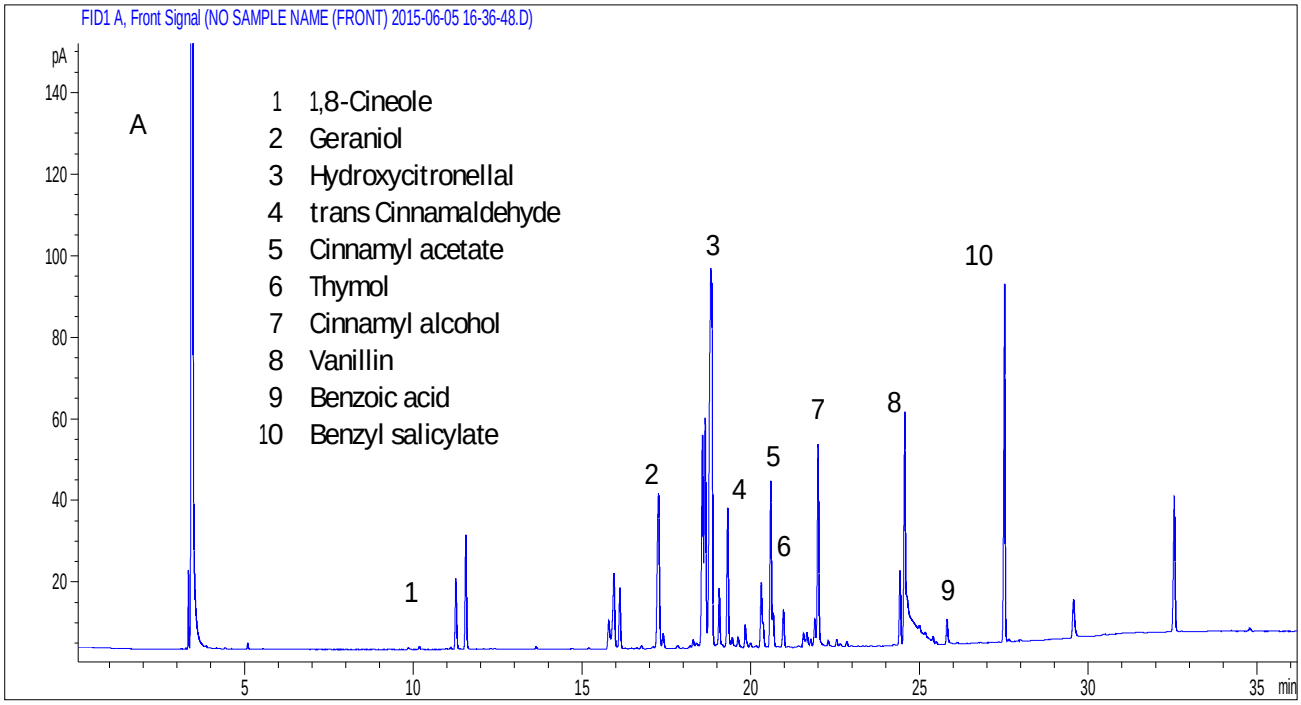


Figure 4. Gas Chromatogram of fragrance mix standard using GsBP-FFAP column.

	Compound	Retention time(min)
1	Ethyl butyrate 36.2%	7.282
2	d-Limonene 20.0%	9.706
3	1,8-Cineole (Eucalyptol) 0.5%	9.837
4	Geraniol 0.6%	17.642
5	Hydroxycitronellal (3,7-Dimethyl-7-hydroxyoctanal) 5.0%	18.744
6	trans Cinnamaldehyde 0.5%	20.127
7	Cinnamyl acetate 0.3%	20.983
8	Thymol 0.3%	21.066
9	Cinnamyl alcohol 0.3%	22.218
10	Vanillin 0.1%	23.709
11	Benzoic acid 1.0%	25.824
12	Benzyl salicylate 36.2%	27.867


FID1 A, Front Signal (NO SAMPLE NAME (FRONT) 2015-06-05 16-36-48.D)
pA
A
1 1,8-Cineole
2 Geraniol
3 Hydroxycitronellal
4 trans Cinnamaldehyde
5 Cinnamyl acetate
6 Thymol
7 Cinnamyl alcohol
8 Vanillin
9 Benzoic acid
10 Benzyl salicylate
min

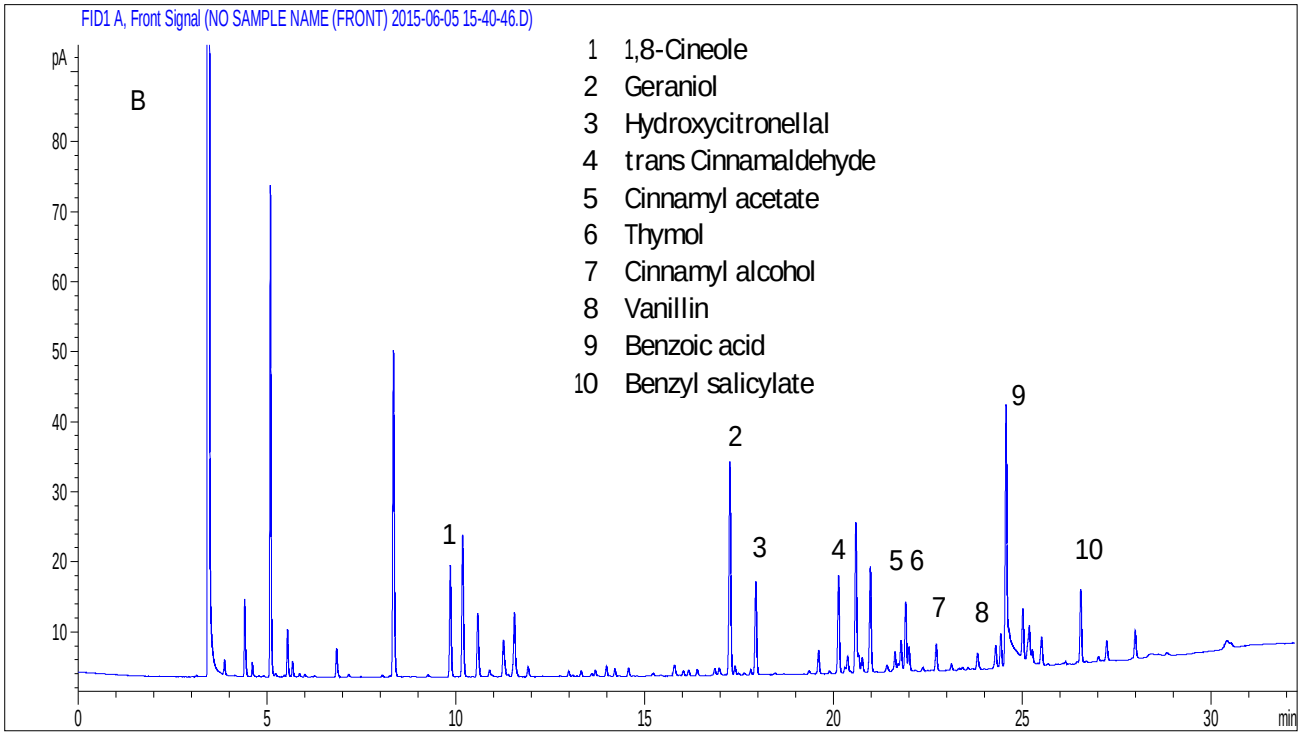

FID1 A, Front Signal (NO SAMPLE NAME (FRONT) 2015-06-05 15-40-46.D)
pA
B
1 1,8-Cineole
2 Geraniol
3 Hydroxycitronellal
4 trans Cinnamaldehyde
5 Cinnamyl acetate
6 Thymol
7 Cinnamyl alcohol
8 Vanillin
9 Benzoic acid
10 Benzyl salicylate
min

Figure 5. Gas Chromatogram of name brand perfume 1(A) and perfume 2(B) using GsBP-FFAP column.