



How can manufacturers be assured that their highly complex raw ingredients or finished products are superior in taste and aroma? Consistency is one of the top reasons consumers will continue to buy a specific food product. With over 300 companies world-wide providing manufactured flavors to the food industry, how can a company be assured that their raw products are consistent in flavor and fragrance every time?

To make qualified purchasing decisions manufacturers must be aware that raw materials with the same name, such as oils that come from different geographical areas, are not necessarily the same. While taste testers would not be able to tell subtle differences in raw materials, there are certain instruments available that can. With regular GC/MS systems analysts can spend hours, days, and even weeks manually searching for unknown sample contaminants. Advancements in computerization and software are making that tedious searching obsolete. Today's instruments have the ability to resolve flavor/fragrance inconsistencies in your manufactured product by automatically analyzing all raw materials, searching for unknown contaminants in raw and finished product, and providing comparison testing of finished product lots.

While there are many instruments on the market, the Pegasus®II Fast Gas Chromatography Time-of-Flight Mass Spectrometer (GC/TOFMS) from LECO Corporation has many strengths that other systems do not. It can perform qualitative and quantitative analysis of complex mixtures containing 1000 components or more. The built-in software easily identifies crucial differences, even troublesome contaminants buried below the total ion chromatogram (TIC) baseline. But, absolutely best of all is that this instrument does all of it automatically in just minutes. The Pegasus II can take hours or days off your regular analysis times with automated peak find, deconvolution, and sample comparison algorithms. This system is the world's fastest and a lab technician can easily run it.

For an example of how the Pegasus works, let's go back to the oils from different regions mentioned earlier. In a recent application Scotch and Midwest Spearmint Oils were compared using the GC/TOFMS. The two samples were processed and compared with algorithms unique to the Pegasus II.

The automated data processing algorithms include peak finding and spectral deconvolution. Peak finding effectively locates the positions of all peaks in a sample including multiple components in complex coelutions. Deconvolution then resolves the mixed mass spectra of the coelution into accurate individual mass spectra for each analyte, including the accurate distribution of signal from masses shared by several components in the coelution. Once the peaks are located and the spectra determined for each sample, a comparison is performed based on peak positions and spectral similarities. Components present in one sample and not in another are identified, as well as components present in both samples, but at widely varying concentrations (based on a user-defined threshold).

The Scotch and Midwest Spearmint Oil samples were analyzed using the following instrument conditions.

**Analysis Time:** 2 minutes

**Transfer Line:** 300°C

**Source:** 300°C

**Acquisition Rate:** 30 spectra/sec

**Stored Mass Range:** 35 to 400 u

**GC:** Hewlett Packard@6890\*

**Column:** DB-5 4mx0.1mm ID, 0.1 µm phase film

**Oven:** 40°C for 0.5 min., then to 280°C at 75°C/min., hold for 1 min.

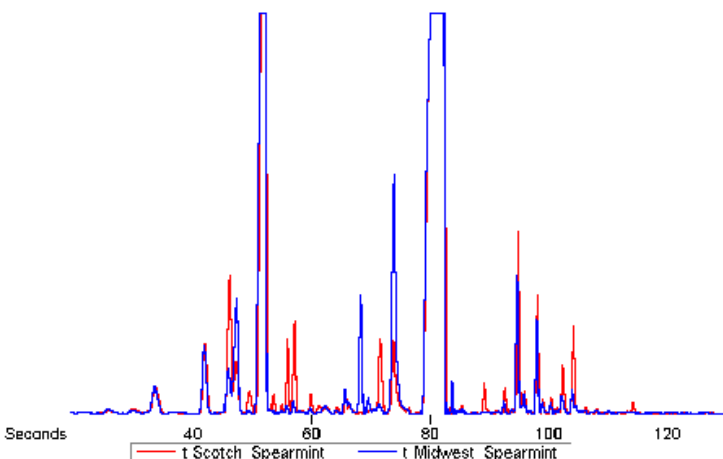
**Injector:** Split/Splitless at 290°C

**Carrier Gas:** Helium, 2.0 ml/min. constant flow

**Sample:** No preparation required. 0.1 µm split (150:1) injection

\*HP6890 GC is equipped with fast oven temperature ramp capabilities and a high pressure EPC module.

Automated data processing was performed and the resulting mass spectra were searched against both the National Institute for Standard and Technology (NIST) 1998 Mass Spectral Database and Terpene Essential Oil Library. (The Terpene Library contains mass spectra of essential oil components and DB-5 retention indices compiled by Robert P. Adams, Baylor University Plant Biotechnology Center.)



**Figure 1:** Total Ion Chromatogram (TIC) Overlay of Scotch and Midwest Spearmint Oils.

Based upon the Total Ion Chromatogram overlay (Figure 1), the samples appear quite similar in composition. The comparison algorithm detects and identifies components present in one oil and not in the other, and analytes present in both oils, but outside of a user-defined tolerance level for concentration variation. The results from these analyses are in Tables 1, 2 and 3. An extracted ion profile of  $m/z$  83 is plotted for both oils in Figure 2. This profile highlights two analytes present in the Midwest Spearmint and not the Scotch Spearmint as well as analytes present in both, but outside of the concentration tolerance set for the analysis.

**Table 1:** Spearmint Oil Difference Peak Table. Analytes found in Scotch Spearmint and not in Midwest Spearmint.

Name	R.T.
7-Octen-4-ol	44.18
trans-Piperitol	78.13
2-Cyclohexen-1-one, 3-methyl-6-(1-methylethenyl)-	83.38
Globulol	114.33

**Table 2:** Spearmint Oil Difference Peak Table. Analytes found in Midwest Spearmint and not in Scotch Spearmint.

Name	R.T.
2-Methoxyethyl benzene	58.87
Benzene, 4-ethenyl-1,2-dimethyl-	60.15
Isomenthone	69.54
Bicyclo[3.1.1]heptan-3-one, 2,6,6-trimethyl-, (1a,2a,5a)-	70.45
8-a-Acetoxyelemol	110.19
cis-3-Hexenyl Phenyl Acetate	118.07

**Table 3:** Spearmint 'Out of Tolerance' Peak Table. Analytes in both spearmint oils, but at concentrations outside of a user-defined tolerance (150% used here). The Match Number indicates the spectral similarity between the spectra in the two spearmint oils. The Relative Concentration is the concentration of analytes in the Scotch sample relative to those in the Midwest sample. Concentrations in the Midwest sample are set to 100% by default. RT is the Retention Time.

Name	R.T.	Match	Relative Concentration
Butanal, 3-methyl-	5.78	773	157.2 %
Butanal, 2-methyl-	5.93	851	19.52 %
Furan, 2-ethyl-	7.08	783	150.02 %
1-Pentanol	8.58	673	236.9 %
Thuja-2,4(10)-diene	37.78	720	40.7 %
Benzaldehyde	38.33	686	39.9 %
3-Octanone	44.98	666	13.09 %
b-Myrcene	46.13	961	304.99 %
3-Octanol	47.23	918	42.19 %

a-Terpinene	49.48	917	810.4 %
p-Cymene	50.48	712	273.9 %
cis-b-Ocimene	53.53	920	828.3 %
trans-b-Ocimene	54.93	925	263.5 %
g-Terpinene	55.93	954	1089.8 %
2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-, cis-	57.13	967	797.6 %
1-Octanol	58.58	726	206.4 %
Terpinolene	59.98	924	313.4 %
trans-Sabinene hydrate	61.28	849	500.54 %
3-Nonanol	61.88	633	49.5 %
Nonanal	62.48	825	44.4 %
7-Hydroxy-6-methyl-oct-3-enoic acid	64.13	585	459.0 %
Cyclohexanone, 5-methyl-2-(1-methylethyl)-	68.38	848	1.7 %
Terpinen-4-ol	71.58	918	867.1 %
cis-Dihydro carvone	73.88	937	25.4 %
neo-iso-Dihydro Carveol	75.93	778	166.6 %
Carvone	82.43	956	4.1 %
cis-Carvone oxide	82.93	828	257.2 %
Edulan I, dihydro-	85.43	870	201.0 %
Dihydrocarvyl acetate	89.18	866	2607.0 %
Piperitenone	89.88	781	158.0 %
10,13-Octadecadiynoic acid, methyl ester	91.28	432	665.2 %
Piperitone oxide	92.58	398	14.4 %
cis-Carvyl Acetate	92.73	937	281.3 %
b-Elemene	95.63	850	173.1 %

1,6,10-Dodecatriene, 7,11-dimethyl-3-methylene-, (Z)-	99.78	715	158.5 %
$\alpha$ -Humulene	101.38	905	164.2 %
1,6,10-Dodecatriene, 7,11-dimethyl-3-methylene-, (E)-	102.38	961	268.7 %
$\gamma$ -Muurolene	102.63	932	188.6 %
Germacrene D	104.23	972	364.0 %
Phenethyl isovalerate	104.83	876	22.8 %
Germacrene A	106.43	915	174.1 %

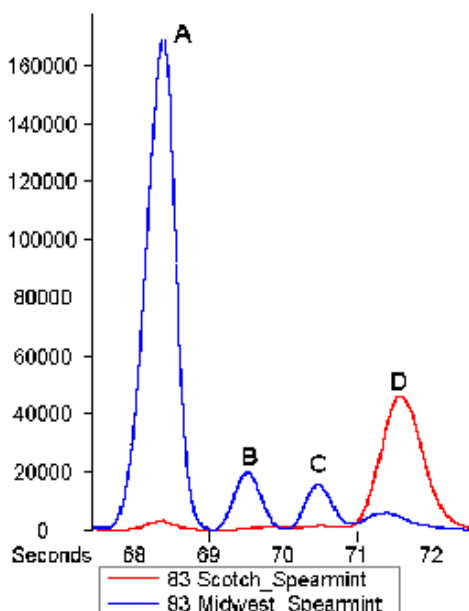
**Figure 2:** Extracted Ion Profile Chromatogram Overlay of m/z 83 in Scotch and Midwest Spearmint Oils.

**Peak A:** Cyclohexanone, 5-methyl-2-(1-methylethyl)-, Scotch oil contains 1.7% of that in Midwest oil.

**Peak B:** Isomenthone, Not Found in Scotch oil.

**Peak C:** Bicyclo[3.1.1]heptan-3-one, 2,6,6-trimethyl-, (1a,2a,5a), Not Found in Scotch oil.

**Peak D:** Terpinen-4-ol, present in Scotch oil at 8.7 times greater than that in Midwest oil.



The combination of unique and automated peak find, spectral deconvolution, and comparison algorithms from the Pegasus II allow for the rapid identification of even minor differences between two spearmint oils taken from different geographical regions. Both oils provide good flavor in a variety of applications, but each spearmint oil has chemical properties not found in the other.

While the subtle differences in raw material may not seem to be important to the final product, slight chemical differences may drastically affect the taste and aroma qualities through the cooking, heating, and cooling processes involved in the manufacture of the product. Other contaminants, such as pesticides, herbicides, and insecticides may even pose health concerns if they are undetected in the final product.

Don't take chances with your food quality. Gain complete control over your raw food ingredients, your final manufactured product, and the analysis of competitive products. Troubleshoot production problems, develop rapid, reliable quality control protocols, and solve your customer complaints with the Fast GC/MS from LECO Corporation.