

Tainting in Wine from Lubricants and Grease

Summary- Using the zNose® tainted wine samples were easily distinguished from control sample using ultra-fast chromatography. Quality control testing of wine headspace vapors at rates as high as 40 samples per hour was demonstrated. Variations in the concentration of the principal wine components (e. caproate and e. caprylate) and detection of trace impurities was used to characterize and quantify tainting.

Supplied Samples

Six 200 mL bottles labeled pure grease, lubricant oil, control wine, light swirl wine, medium swirl wine, and heavy droplets wine.

n-Alkane Reference (C6-C22) (10ps2a1b method)

Retention time was referenced to the retention time of headspace vapors from a 40 mL vial containing 10 mL methanol spiked with 125 ppm n-alkanes. Kovats indices for compounds are denoted by alkane number X 100 e.g. C12 = 1200.

The method used (10ps2a1b) enables a complete analysis to be performed in 20 seconds. The dB5 column temperature is ramped from 40°C to 190°C at 10°C per second. A new analysis can be done every 90 seconds using a 10 second sample time or 5 mL vapor sample.

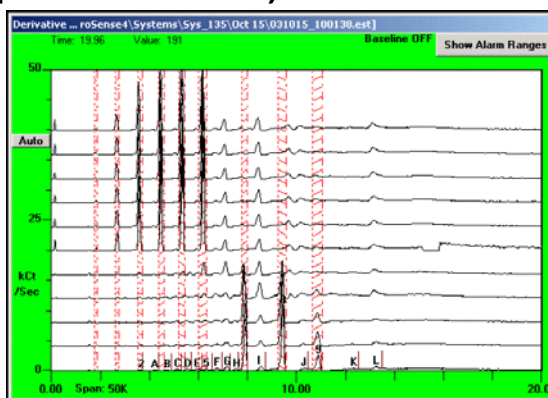


Figure 1- Replicate responses to n-alkane vapors. Reference times shown by red bands

Headspace Vapors from Grease (10ps2a1b)

Vapors within bottle containing grease were sampled for 10 seconds (5 mL vapor) with a 60°C detector. The response indicated approximately 10 major peaks spanning C9 to C13. Small trace amounts of heavier compounds out to C19 were also noted.

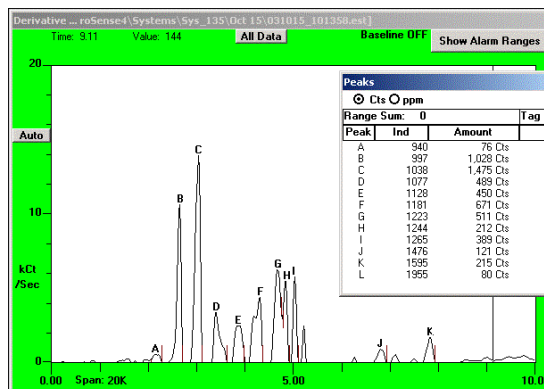


Figure 2- Compounds in grease headspace span indices 900 to approximately 1300 although a small trace appears as high as 1900.

Headspace Vapors from Lubricant (10ps2a1b)

Vapors within bottle containing lubricant oil were sampled for 10 seconds (5 mL vapor) with a 60°C detector. The response indicated one major peak at 887 and three minor peaks 1000 to 1075. Only minor trace amounts of heavier compounds were detected.

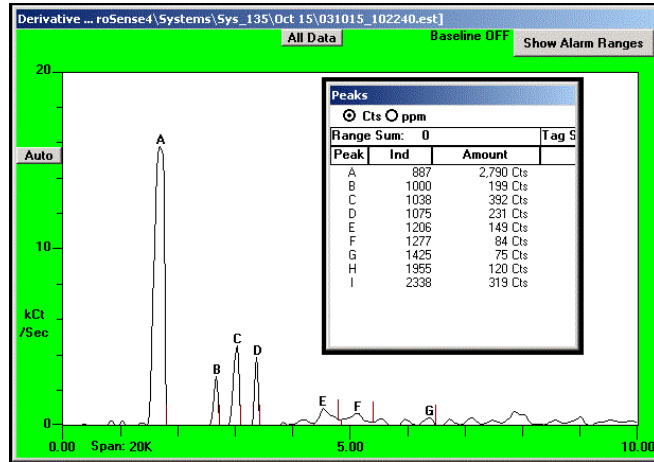


Figure 3- One major compound (887) and three minor compounds (1000, 1038, and 1075) were detected in headspace vapors from lubricant.

Creating Alarms for Grease & Lubricant Compounds

Alarm band for four major compounds which were present in both grease and lubricant oil was done using graphical interface. Alarm bands allow quantification of peak areas (in counts) and bands overlaid in red make it easier to locate these compounds in complex chromatograms.

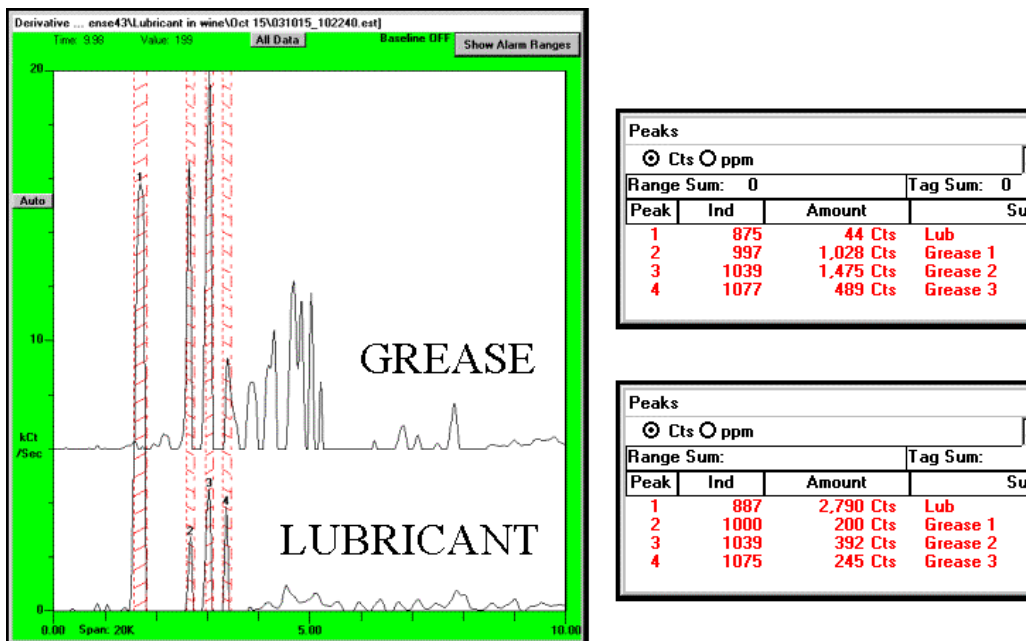
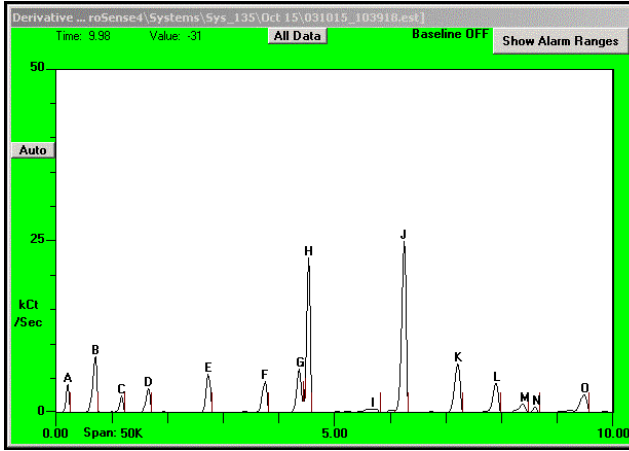


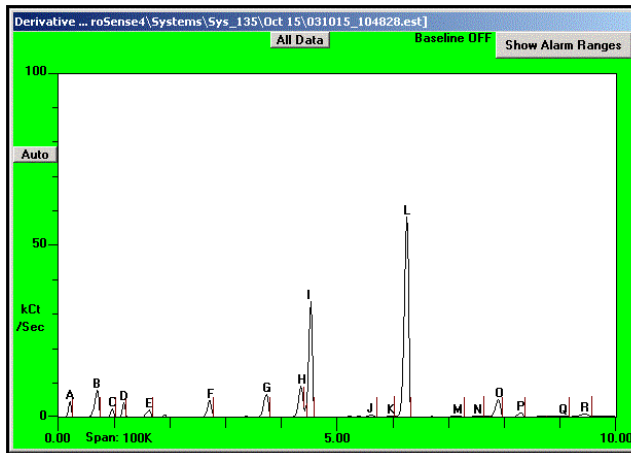
Figure 4- Vertically offset chromatograms from grease and lubricant oil. The area of the four major peaks are quantified in for each in the tables on the right of figure.

Headspace of Control Wine (10ps2a1b)



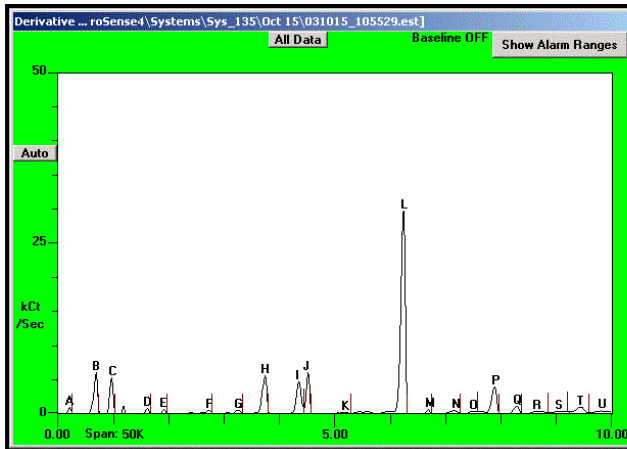
Peaks			
Cts ppm			
Range Sum: 0			
Peak	Ind	Amount	Tag S
A	716	203 Cts	
B	774	562 Cts	
C	827	140 Cts	
D	882	235 Cts	
E	1006	423 Cts	
F	1117	352 Cts	
G	1187	445 Cts	
H	1206	1,288 Cts	
I	1349	123 Cts	
J	1411	2,141 Cts	
K	1523	796 Cts	
L	1605	473 Cts	
M	1667	162 Cts	
N	1696	45 Cts	
O	1811	386 Cts	
P	1921	56 Cts	
Q	2011	338 Cts	
R	2206	43 Cts	
S	2346	159 Cts	
T	2631	33 Cts	

Headspace of Heavy Droplets in Wine (10ps2a1b)



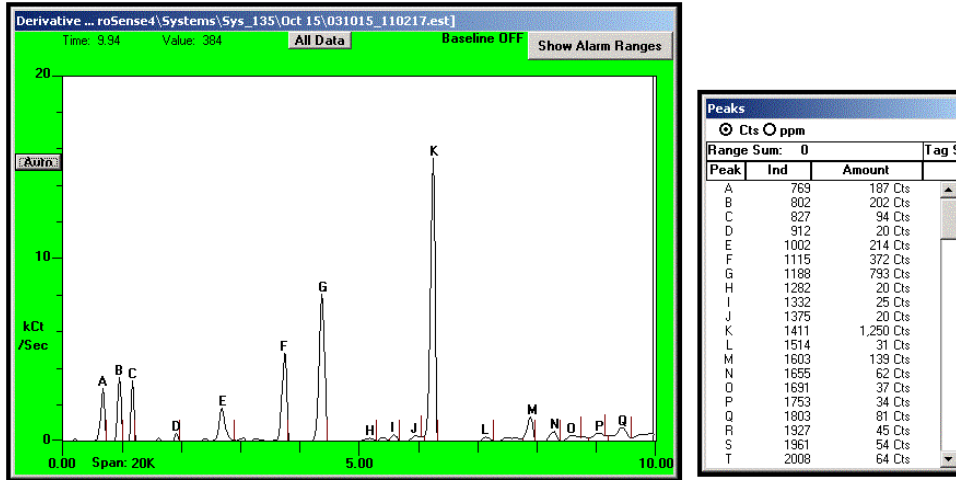
Peaks			
Cts ppm			
Range Sum: 0			
Peak	Ind	Amount	Tag S
A	716	223 Cts	
B	772	658 Cts	
C	804	129 Cts	
D	827	134 Cts	
E	880	138 Cts	
F	1006	323 Cts	
G	1114	507 Cts	
H	1185	540 Cts	
I	1206	1,898 Cts	
J	1335	55 Cts	
K	1377	20 Cts	
L	1411	4,905 Cts	
M	1516	58 Cts	
N	1558	38 Cts	
O	1602	530 Cts	
P	1654	123 Cts	
Q	1753	38 Cts	
R	1803	119 Cts	
S	1921	40 Cts	
T	1960	49 Cts	

Headspace of Medium Swirl Wine (10ps2a1b)



Peaks			
Cts ppm			
Range Sum: 0			
Peak	Ind	Amount	Tag S
A	716	35 Cts	
B	772	378 Cts	
C	804	354 Cts	
D	878	45 Cts	
E	912	28 Cts	
F	1006	47 Cts	
G	1062	40 Cts	
H	1115	435 Cts	
I	1185	405 Cts	
J	1204	331 Cts	
K	1285	28 Cts	
L	1411	2,748 Cts	
M	1462	28 Cts	
N	1518	64 Cts	
O	1556	21 Cts	
P	1603	392 Cts	
Q	1655	110 Cts	
R	1699	48 Cts	
S	1753	21 Cts	
T	1803	110 Cts	

Headspace of Lite Swirl Wine (10ps2a1b)

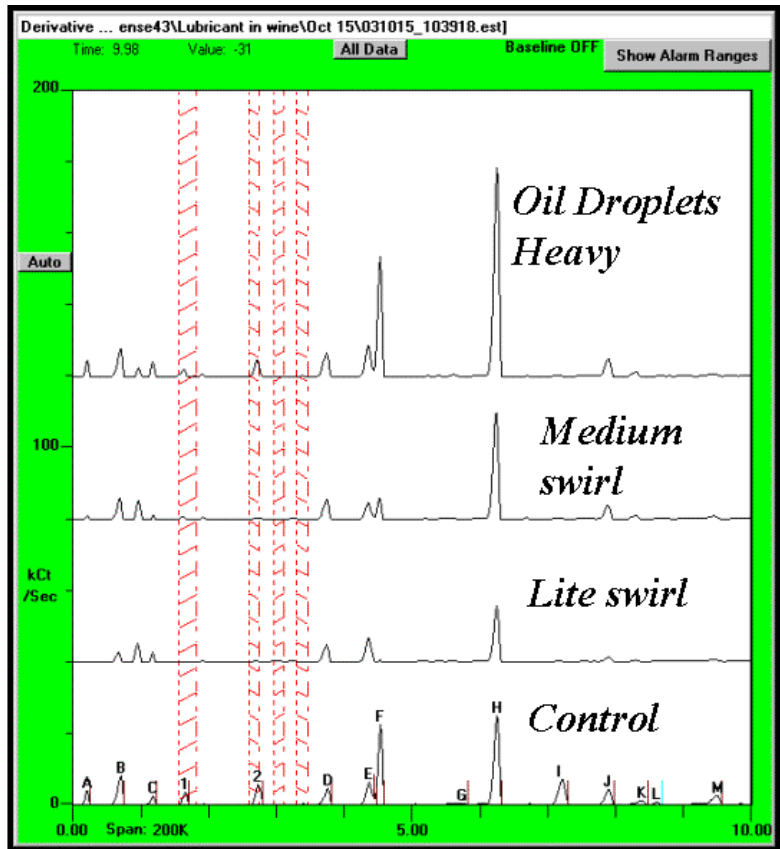


Headspace of Lite Swirl Wine (10ps2a1b)

Offset plotting of chromatograms from wine headspace in wine bottle samples did not reveal any major compound peaks from the grease or lubricant oils as seen in the alarm bands in red.

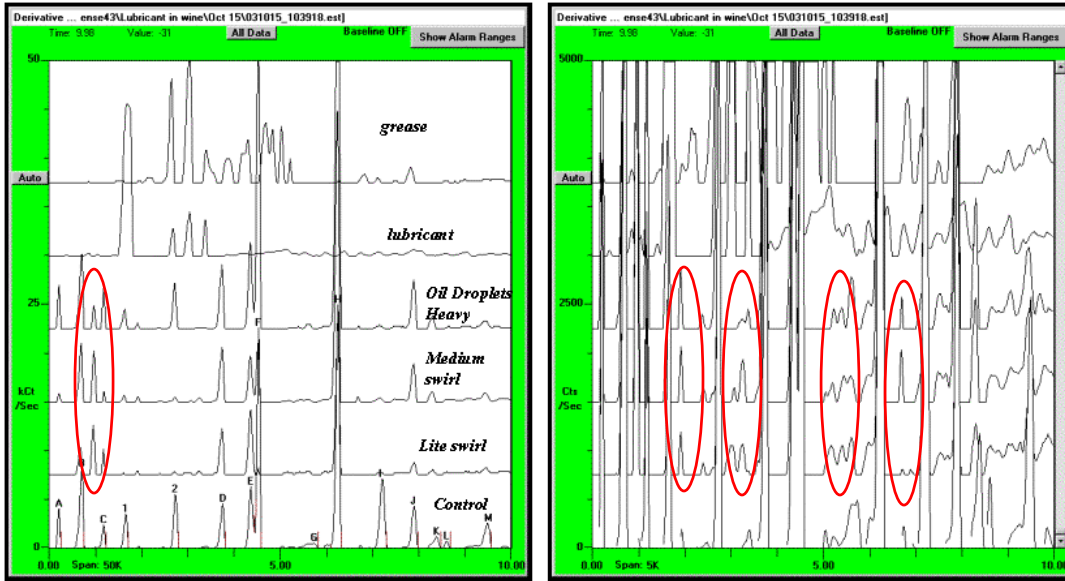
However, the principal compounds within the wine were greatly reduced in the medium and lite swirl samples indicating alteration of their solubility in the wine as well as partitioning into the headspace (Henry's constant).

Increased concentration of heavy oil droplet sample was more due to the larger amount of wine in this bottle relative to the control bottle.



Offset Comparisons of Headspace in Sample bottles

More detailed offset comparisons show clear contamination (denoted by red) of wine samples compared with control sample bottle. Expanded traces also shows contamination from heavier compounds.



VaporPrint® Comparisons of Wine Samples

High resolution olfactory images depicting logarithmic radial odor intensity vs retention time (angle, top=0, Max) provide a visual representation of the entire odor. These images are directly measured by the SAW sensor and chromatograms are derived by derivatization of the sensor signal.

Lite and medium swirl images are easily distinguished from that of the control sample. However, only the previous trace analysis using expanded chromatograms can distinguish between heavy droplets and the control sample.

