Trace Contamination Detection in Cooking Oils using a Pyroprobe

Author:

Shilei Liu
Karen Sam

Application Note
Food

Abstract
This application note investigates the trace contamination of cooking oil caused by transportation tanker trucks using evolved gas analysis coupled with thermal extraction GC-MS.

Introduction

As part of manufacturing process, the cooking oil is often transported by tanker trucks from the crushing facility to the packaging facility. In certain countries, it was an “open secret” in the transportation industry that tanker trucks used to transport both fuel and cooking oil were often violating regulated cleaning protocols between exchanges to reduce operation cost, until a recent media coverage sparked public concerns on food safety. Even though transportation units and carrier vehicles found in violation of the regulations were terminated immediately, the relevant regulatory authorities were urgently seeking an analytical measurement to quickly screen cooking oil.

In this application note, evolved gas analysis (EGA) and thermal extraction techniques with GC-MS were used to detect gasoline and mineral oil in cooking oil, which require no sample preparation through simple, time-saving direct injection to the GC-MS. These advantages make these techniques ideal for monitoring cooking oil contamination from tanker trucks. These techniques can detect 2 g of mineral oil in a typical 20 ton load.

Experiment Setup

Samples of cooking oil were spiked with gasoline or mineral oil, then loaded into DISC tubes for analysis. A 1m piece of fused silica was used to connect the GC inlet to the MS detector in a preliminary EGA run. After which, a 30 meter long 5% phenyl capillary column was used for multi-step pyrolysis. A vent-free adapter was installed to enable a fast switch between the fused silica and the column without losing vacuum in the mass spectrometer.

EGA

Pyroprobe 6150
Initial: 50°C
Final: 800°C
Ramp Rate: 100°C per min
Interface: 300°C
Transfer Line: 320°C
Valve Oven: 300°C

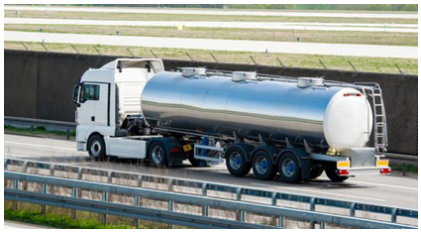
GC-MS
Column: Fused silica (1m x 0.10mm)
Carrier: He 1.00mL/min 50:1 spl
Injector: 320°C
Oven: 300°C
Ion Source: 250°C
Mass Range: 35-700amu

Thermal Extraction

Pyroprobe 6150
DISC:
100°C 60 sec (gasoline)
250°C 60 sec (mineral oil)

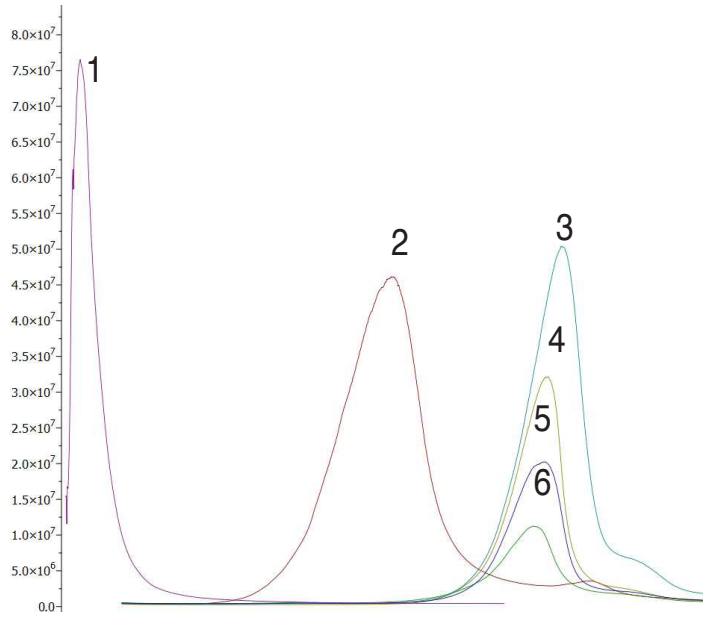
Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

GC-MS
Column:
5% phenyl (30m x 0.25mm x 0.25μm)
Carrier: He 1.00mL/min, 50:1 spl
Injector: 320°C
Oven: 40°C for 2 minutes
10°C/min to 100°C
50°C/min to 300°C (3 min)
Ion Source: 250°C
Mass Range: 35-600amu



Results and Discussion

As a fast-screening technique, evolved gas analysis was first performed; the DISC temperature was ramped up at 100 °C/min from 50°C to 800 °C and the GC oven was kept at 300°C. The results in Figure 1 show that gasoline vaporizes between room temperature and 150°C, mineral oil vaporizes between 250°C and 350°C, and four different cooking oils decompose between 400°C and 500°C. Therefore, to quantitatively analyze gasoline and mineral oil in cooking oils by GC-MS, gasoline and mineral oil can be thermally extracted at 100°C and 250°C, respectively, from cooking oil.



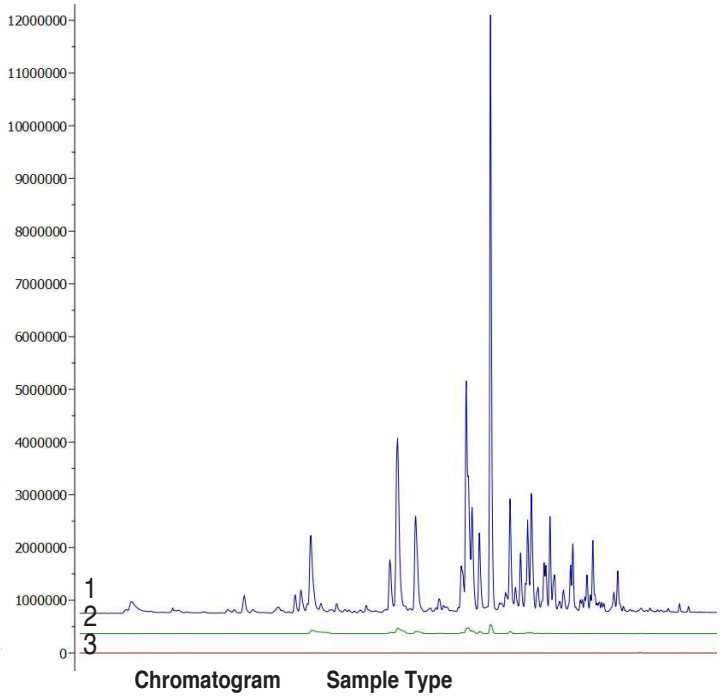
EGA	Sample Type	EGA	Sample Type
1	gasoline	4	canola oil
2	mineral oil	5	melon seed oil
3	soybean oil	6	corn oil

Figure 1. EGA Overlay of Gasoline, Mineral Oil and four different cooking oils

Once extraction temperatures were determined, canola oil was spiked with gasoline and was extracted at 100°C. Gasoline, canola oil, and canola oil spiked with gasoline are shown in Figure 2. The gasoline spiked canola oil had both xylene and trimethylbenzene, which are characteristic components in gasoline. The matrix oil did not interfere with the analysis of the gasoline spiked sample.

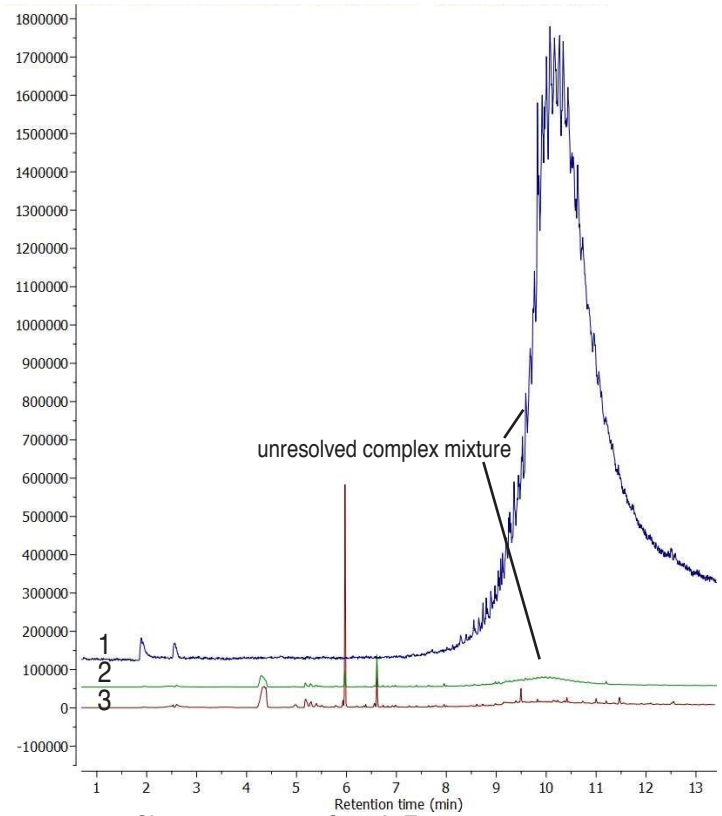
Next, canola oil was spiked with mineral oil and extracted at 250°C (Figure 3). An unresolved complex mixture of alkanes from mineral oil is present in the spiked sample between 9 and 11 minutes, with no canola oil interference.

To investigate sensitivity, canola oil was spiked with 10 ppm of gasoline and 1 μL was added to a DISC tube for analysis. When it was extracted at 100°C, xylene and trimethyl benzene from gasoline were present at S/N>10 (Figure 4).



Chromatogram	Sample Type
1	gasoline
2	gasoline in canola oil
3	canola oil

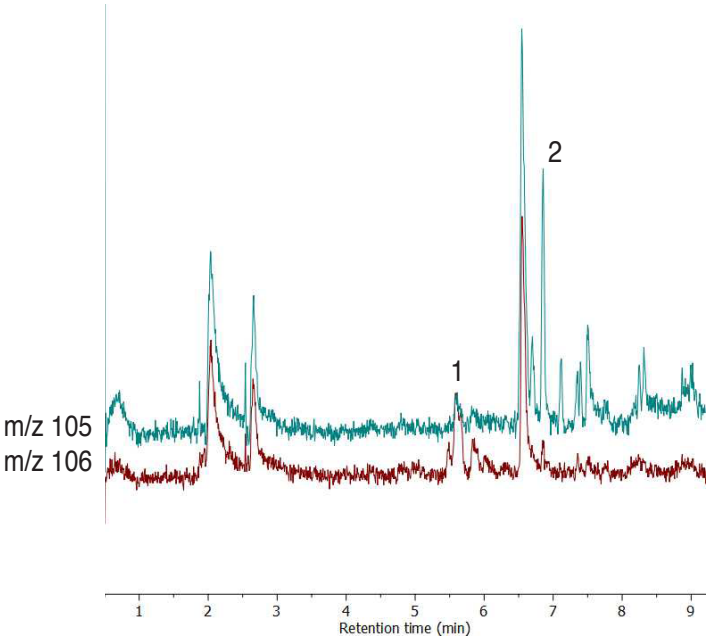
Figure 2. Thermal Extraction at 100°C of gasoline, canola oil and gasoline in canola oil.



Chromatogram	Sample Type
1	mineral oil
2	mineral oil in canola oil
3	canola oil

Figure 3. Thermal Extraction at 250°C of mineral oil, canola oil and canola oil spiked with mineral oil.

Additionally, canola oil was spiked with mineral oil at 10 ppm, and 1 μL was added to a DISC tube. When it was extracted at 250°C, the unresolved complex mixture of alkanes from the mineral oil was present at S/N>10 (Figure 5).



Peak	Identification
1	xylene
2	trimethyl benzene

Figure 4. m/z 105 and 106 to show trace gasoline in canola oil, thermally extracted at 100°C.

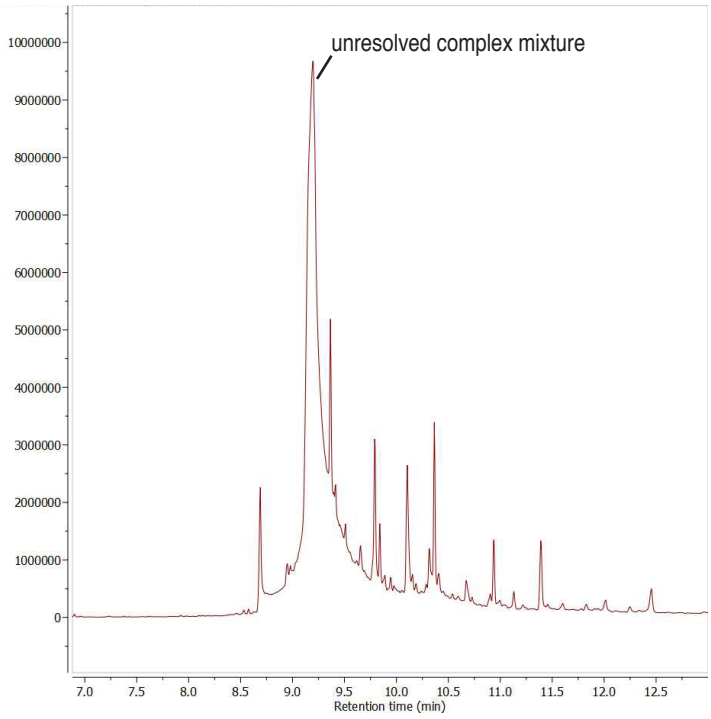


Figure 5. Thermal Extraction at 250°C of trace mineral oil in canola oil.

Conclusion

EGA combined with thermal extraction are shown to be are powerful tools for detecting adulteration of cooking oil transported by tanker trucks. The first tool was used to quickly screen cooking oils and adulteration candidates for proper thermal extraction settings, while the second tool effectively detected low levels of contamination in cooking oils after applying the correct thermal extraction temperature. Furthermore, this analysis can be performed quickly, without lengthy sample preparation steps, to ensure transportation companies follow regulated health and safetly guidelines.