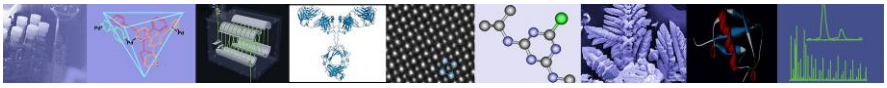


Applications Note

AccuTOF-GCv Series

High-speed 50 Hz Data Acquisition Capability for Comprehensive 2-dimensional GC Measurements

Introduction

The comprehensive 2-dimensional GC (GC x GC) technique provides higher-separation capabilities for complex mixtures than the typical 1-dimensional GC measurements. However, the GC x GC technique requires high speed data acquisition, e. g. > 20 Hz, for the GC detectors due to the shorter 2nd GC column which elutes samples within just a few seconds (comparable to those used for the ultra-fast GC measurements).

Recently, JEOL has developed a new generation GC-HRTOFMS system called the "AccuTOF GCv 4G". The AccuTOF GCv 4G has high sensitivity, high resolution, high mass accuracy and high speed data acquisition, all simultaneously. In fact, this instrument can measure data using up to a 50 Hz data acquisition speed which is more than sufficient to do not only fast GC measurements but also GC x GC measurements.

In this work, we measured diesel fuel and crude oil using the GC x GC technique with the 50 Hz data acquisition speed available on the AccuTOF GCv 4G.

Experimental

Sample information and measurement condition are shown in Table 1.

Results

To start, the sensitivity for the GCxGC/HR-TOFMS system was tested by measuring 1pg of octafluoronaphthalene (OFN). The signal to noise ratio (S/N) for this sample was checked with both the modulator OFF and then ON. These results confirmed that the GCxGC column condition and modulator were working well. The OFN mass chromatograms for each scenario are shown in Figure 1.

The S/N values for the 1pg OFN sample were over 100 for both modulator statuses. These results clearly showed high sensitivity even when the high speed 50 Hz acquisition capability is used. Additionally, 2-dimensional and 3-dimensional mass chromatograms were constructed for the OFN sample, and then the data was analyzed by using both a NIST library search and the accurate mass measurements for the sample (Figure 2).

Condition	System check	Oil application
Sample	OFN	Diesel Fuel, Crude Oil
Concentration	1 pg/uL (Hexane)	1/100 (Hexane)
GCxGC system	ZX2 thermal modulator (ZOEX)	
1st column	Rxi-5SilMS, 30 m x 0.25 mm, 0.25 um	
2nd column	Rxi-17SilMS, 2 m x 0.15 mm, 0.15 um	
Modulator	Deactivated fused silica, 1.5 m x 0.15 mm	
Modulator period	6 sec	8 sec
Modulator duration	400 msec	
Hot jet temp.	270 C	
Hot jet gas pressure	40 psi	
Cold jet flow	18 L/min	
Inlet pressure	200 kPa (Out flow: 2mL/min)	
Inlet mode	Splitless	Split 10:1
Oven temp.	50 C(1min) -> 3 C/min -> 300 C(6min)	
GC-HRTOFMS system	AccuTOF GCv 4G (JEOL)	
Ion source	EI standard (High sensitivity)	EI/FI/FD combination
Ionization mode	EI+ (70 eV, 300 uA)	
m/z range	*m/z* 35-500	
Acquisition speed	50 Hz	

Table 1. Measurement Condition.

MS-070113

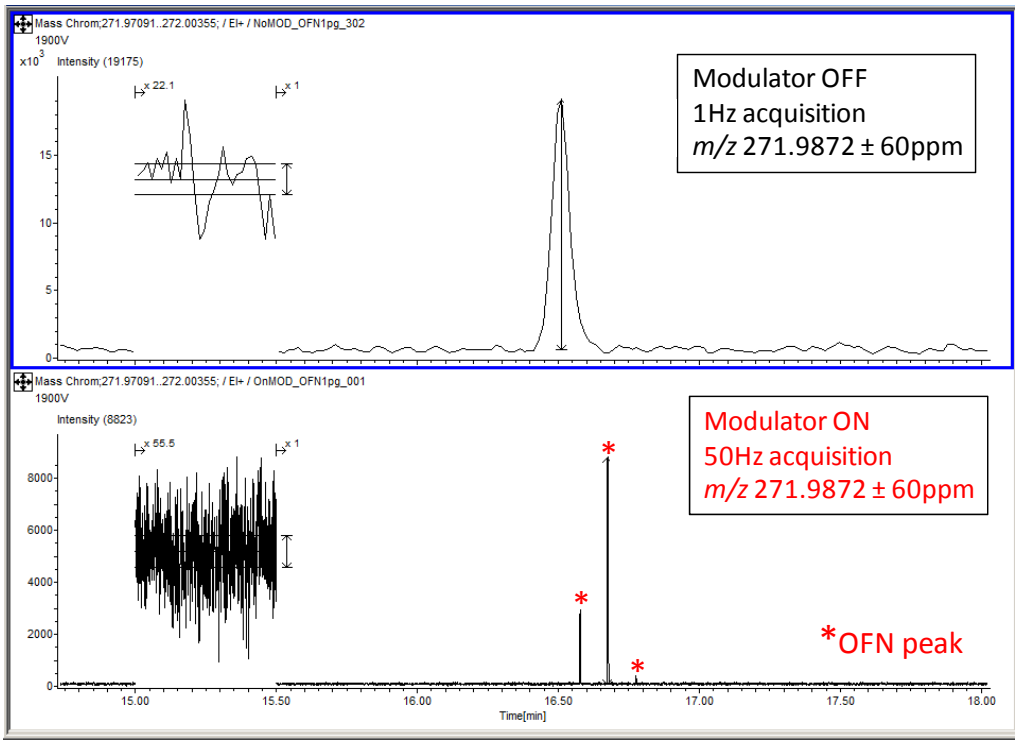


Figure 1. Mass chromatograms of OFN 1pg, Raw data, Mass windows: *m/z* 271.9872 ± 60ppm
Upper: modulator OFF, 1 Hz acquisition,
Lower: modulator ON, 50 Hz acquisition

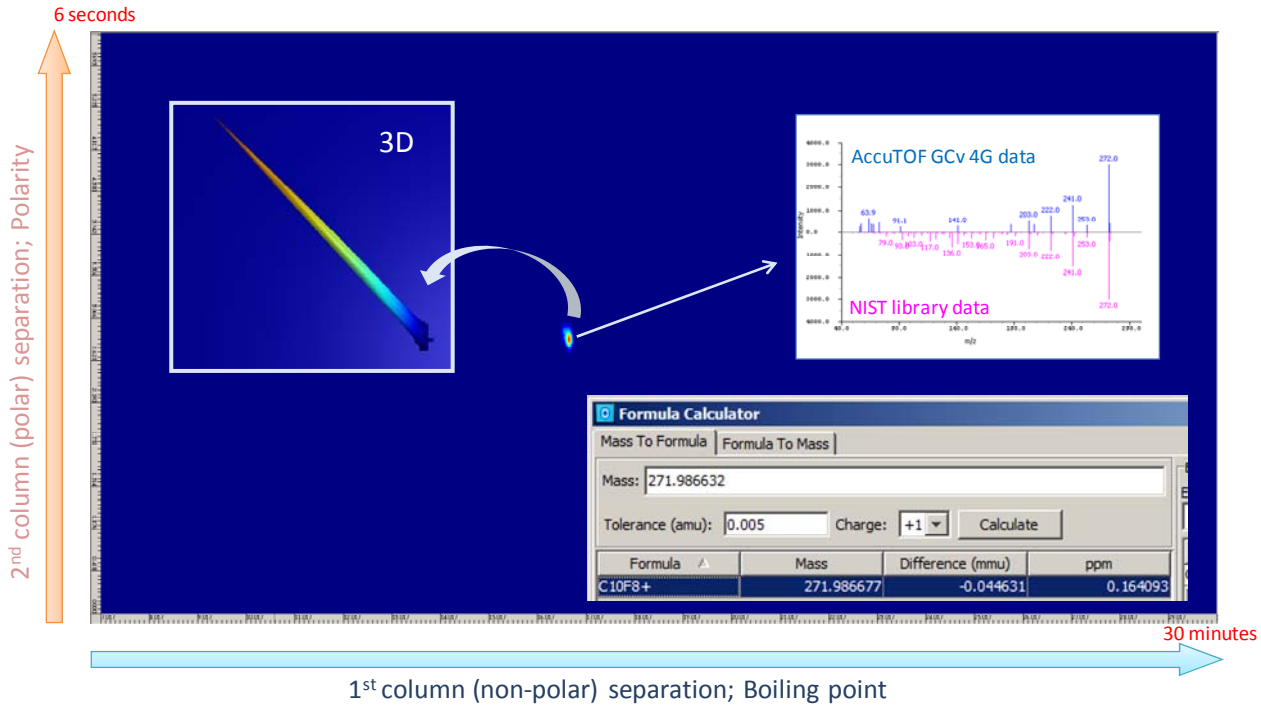


Figure 2. 2-dimensional and 3-dimensional mass chromatograms, library search result and accurate mass measurement results using the OFN 1pg data.

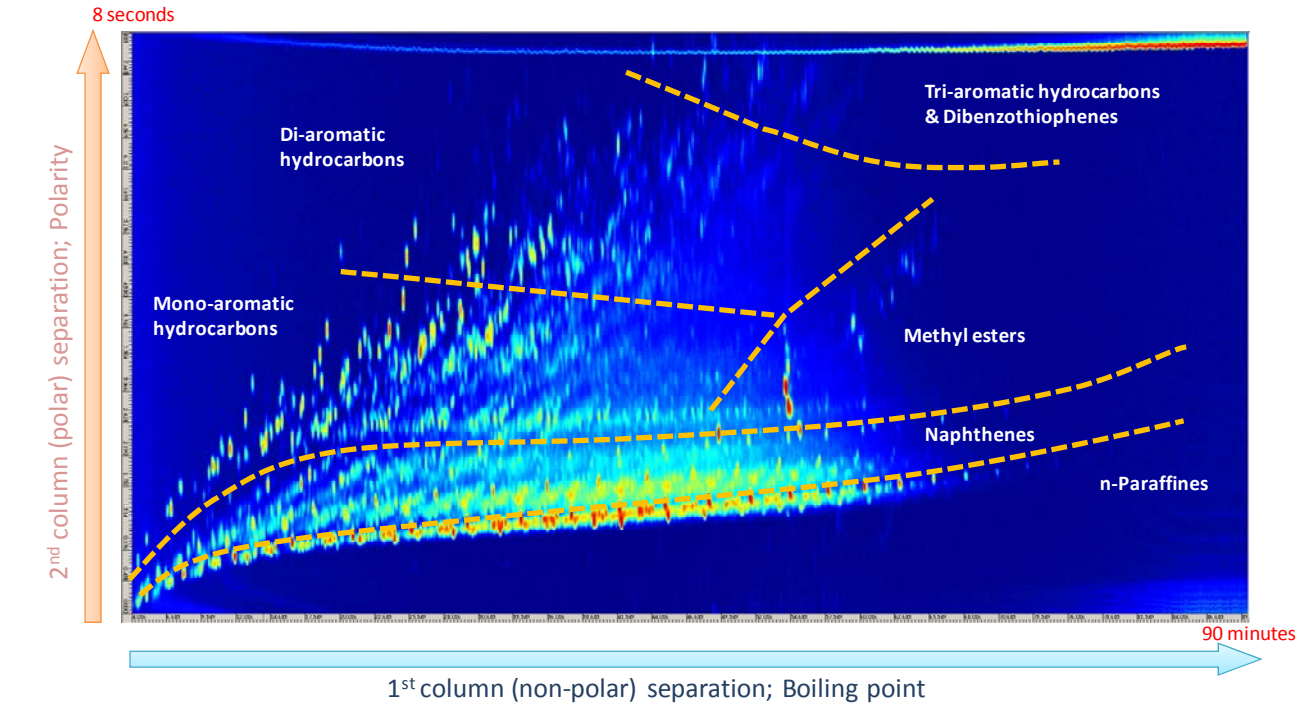


Figure 3. 2-dimensional TIC chromatogram for diesel fuel (50 Hz data acquisition).

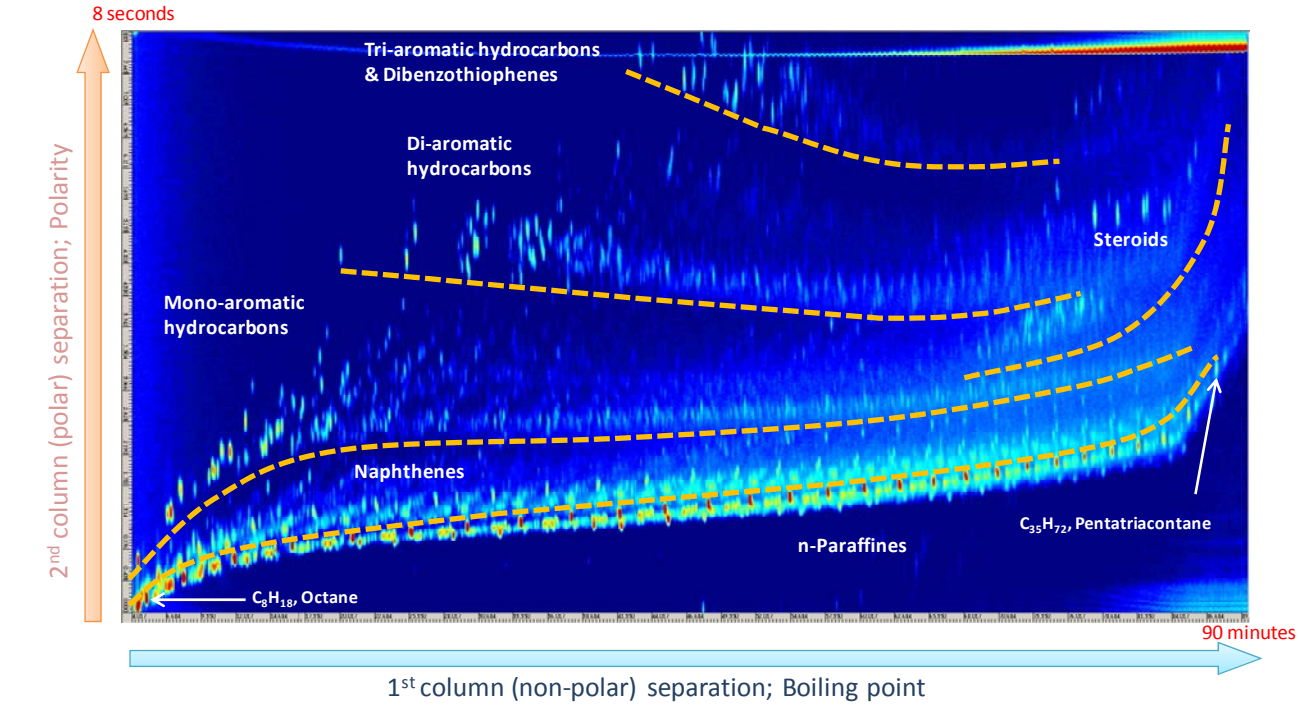
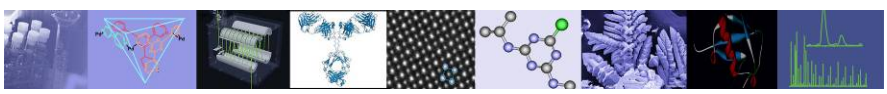


Figure 4. 2-dimensional TIC chromatogram for crude oil (50 Hz data acquisition).



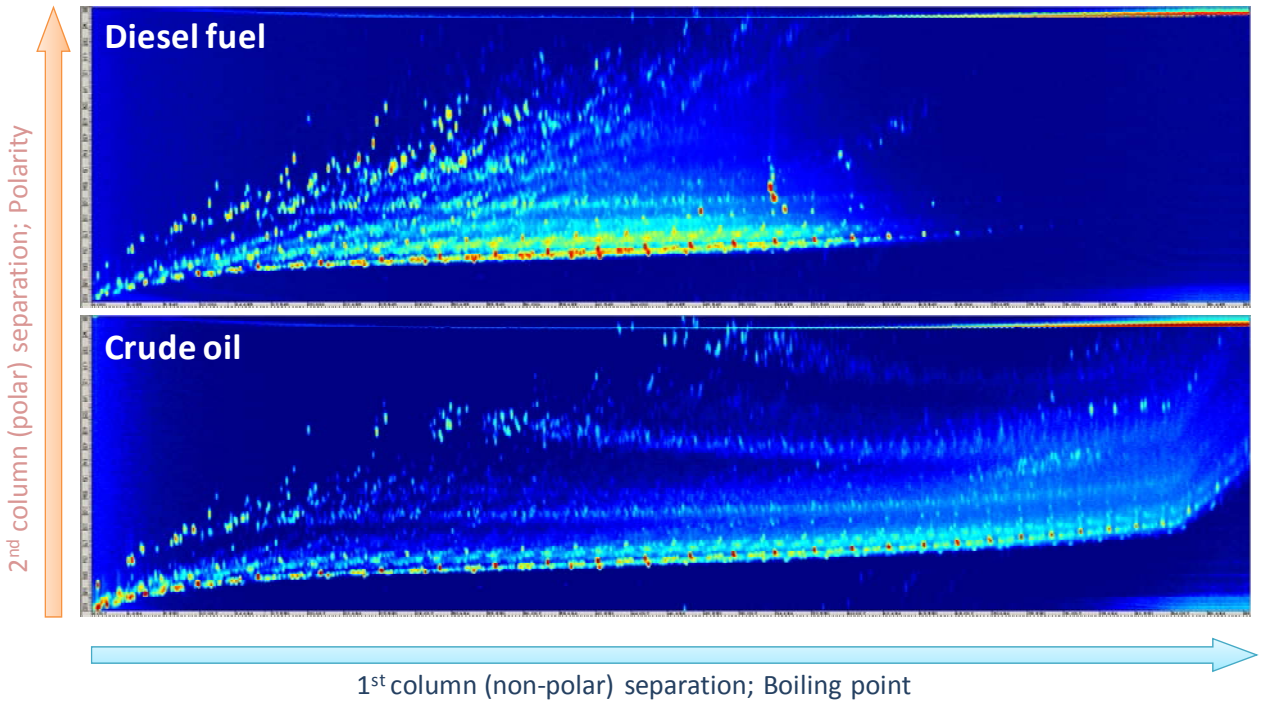


Figure 5. Comparing the 2-dimensional TIC chromatograms for diesel fuel and crude oil.

The data showed good spectral matching to the NIST data for OFN (also shown in Figure 2). Additionally, the mass accuracy obtained for the measurement was less than 1 mDa for the molecular ion ($C_{10}F_8^+$, *m/z* 271.9872) using an external one-point calibration for a GC column background ion (*m/z* 207.0329). These results clearly show that the AccuTOF GCv 4G is a powerful tool for the qualitative analyses of samples using the NIST library search and the accurate mass measurements for GC x GC data.

Next, the diesel fuel and crude oil samples were analyzed using similar GC x GC conditions (See Table 1). The GC x GC results showed a large number of chromatographic peaks present in the TICs for both samples (Figure 3 and 4). Each analysis showed good peak shapes and good peak separations as a result of the 50 Hz acquisition capability of the AccuTOF-GCv 4G. Further inspection of the mass spectral data allowed us to denote where the major hydrocarbon compound groups are located in the 2D plots, as labeled in Figures 3 and 4. Figure 5 shows a comparison between the GC x GC chromatograms, which visually highlights the qualitative differences between each sample.

Conclusion

In this work, we showed the 50 Hz acquisition capability of the AccuTOF-GCv 4G for GC x GC measurements. This system can provide high sensitivity, high resolution, high mass accuracy and high speed data acquisition measurements, all simultaneously. Additionally, the GC x GC/EI measurements showed very good sensitivity that was on the order of a few picograms. Furthermore, these results showed that the AccuTOF GCv 4G is a powerful tool for the qualitative analyses of samples using the NIST library search and the accurate mass measurements, even when using an external one-point calibration, for GC x GC data.

