

## BACKGROUND

Two (2) TPE331-10AV engines were torn down at Product Integrity (PI) in support of a NTSB accident investigation. The engines had been removed from the left and right sides of the same aircraft. The left engine was identified as ESN P106025C. The right engine was identified as ESN P106026C.

## PROBLEM STATEMENT

- Perform non-destructive evaluations of the fractured torsion shafts and starter gearshafts to determine the modes of separation and orientation of the fracture features.
- Analyze the submitted debris samples from the planet gears.
- Filter the oil samples from ESN P106025C and analyze any collected debris.
- Analyze the oil samples for oil type and determine if any contaminants are present.

## SUMMARY/CONCLUSIONS

- The separations of the torsion shafts from the left and right engines were the result of torsional overload. Overload dimples were observed on both fracture surfaces that were elongated in the clockwise direction (forward looking aft), which indicated that the aft sections separated as a result of torsional loading in the counter clockwise direction.
- The separations of the starter gearshafts from the left and right engines resulted from torsional overload. Overload dimples were observed on both fracture surfaces. The dimples on the left and right starter gearshaft fracture surfaces were elongated in the counterclockwise and clockwise directions, respectively, which indicated that torsional loading on the two starter gearshafts had occurred in opposite directions.

See Findings.

**PART NAME:** Torsion Shaft  
**PART NUMBER:** 3108200-1 (Engine)  
**PART S/N:** Unknown

**EQUIP/ENGINE:** TPE331-10AV  
**ENGINE S/Ns:** P106025C/P106026C  
**OPERATING TIME:** Not Reported  
**OPERATING CYCLES:** Not Reported

**MATERIAL:** AM355  
**MATERIAL SPEC:** AMS5743

**SUPPLIER:** Not Reported  
**SUPPLIER P/N:** Not Reported

**REQUESTOR:** [REDACTED]  
**INVESTIGATOR:** [REDACTED]  
**REVIEWER:** [REDACTED]

**CHARGE #:** [REDACTED]  
**LIMS REQUEST:** [REDACTED]  
**RECEIVED DATE:** 11/18/2013

**COMPLETION DATE:** 04/03/2014

## Findings

The aft ends of the torsion shafts from TPE331-10AV Engine S/Ns P106025C (left engine) and P106026C (right engine) were found separated during an engine teardown. The starter gearshafts from both engines were also found to be separated. The separated ends of the torsion shafts and gearshafts were submitted to the Phoenix Materials Lab for non-destructive examination along with debris samples collected from the planet gears from each engine, and an oil sample from ESN P106025C.

### TORSION SHAFT SEPARATIONS

The torsion shafts from ESNs P106025C and P106026C had separated 1.20" and 1.25" from the aft ends, respectively (Figure 1). The fracture surfaces of the separations were oriented normal to the shaft axes and were flat in appearance.

The fracture surfaces of the torsion shafts were examined in the scanning electron microscope (SEM), as shown in Figures 2 and 3. Examination revealed distinct overload dimples on both fracture surfaces. The overload dimples were elongated in the clockwise direction (forward looking aft), which indicated that the aft sections of the torsion shafts from both engines had separated as a result of torsional loading in the counter clockwise direction. Secondary smearing damage obscured areas on both fracture surfaces.

No material defects were observed on the fracture surfaces.

Non-uniform contact/wear patterns were observed on the aft spline teeth on both shafts (Figure 4). The

contact/wear patterns were fairly light in appearance and were noted on both sides of the teeth.

No additional work was performed on the torsion shafts from ESNs P106025C and P106026C per customer request.

### STARTER GEARSHAFT SEPARATIONS

The starter gearshafts from ESNs P106025C and P106026C had separated 0.70" and 0.67" from the ends of the teeth within the reduced diameter sections of the shafts, respectively (Figure 5). The fracture surfaces were oriented normal to the shaft axes and were flat in appearance.

Digital microscope examinations of the gearshafts revealed evidence of deformation in the reduced sections in the areas adjacent to the fracture surfaces (Figures 6-7). The deformation was noted around the circumferences of both shafts and was indicative of torsional loading.

The orientation of the deformation on the gearshafts from ESNs P106025C and P106026C indicated that the separated ends of the shaft were rotating in the clockwise and counterclockwise directions, respectively, as viewed from the fracture surfaces. Secondary smearing damage obscured areas on both fracture surfaces.

The reduced diameter sections of the starter gearshafts exhibited shot peened appearances.

SEM examination of the starter gearshaft separations revealed distinct dimples on both fracture surfaces (Figures 8 and 9). The dimples were elongated and indicative of torsional overload.

The overload dimples on the fracture surfaces on the gearshafts from ESNs P106025C (Figure 8) and P106026C (Figure 9) were elongated in the counterclockwise and clockwise directions, respectively, as viewed from the fracture surfaces. Elongation of the overload dimples indicated that torsional loading on the two starter gearshafts had occurred in opposite directions.

No material defects were observed on the fracture surfaces.

Wear was noted on the loaded sides of the gear teeth on both gearshafts (Figure 10). The wear patterns on the teeth of the gearshaft from ESN P106025C were generally uniform from tooth to tooth and spanned the lengths of the teeth. The wear patterns on the teeth of the gearshaft from ESN P106026C also spanned the lengths of the teeth; however, the wear patterns varied slightly from tooth to tooth.

No additional work was performed on the starter gearshafts per customer request.

#### **OIL ANALYSIS**

Approximately 1 gallon of oil was collected from ESN P106025C for analysis (Figure 11A). The oil sample exhibited a reddish hue at the time of submission. The oil was filtered using 8  $\mu\text{m}$  Millipore filters. A total of 482 grams of material was removed from the oil (Figure 11B).

The filtered debris was submitted to the SEM for characterization using backscattered electron (BSE) and energy dispersive x-ray (EDX) techniques. Results of the analysis are presented in the "Chemistry" section of this report.

Viscosity and flash point information was measured on a filtered sample of the used oil. Viscosity was measured to be 24.56 cSt at 104°F (40°C), which exceeded the specified minimum requirement for a MIL-PRF-23699F type oil (23 cSt). Flash point was measured to occur at 509°F, which also exceeded the specified minimum requirement for MIL-PRF-23699F type oil (475°F).

A sample of the filtered oil was sent to Analyze Inc. to verify oil type, and whether or not any contaminants were present. The sample was analyzed using gas chromatography-mass spectroscopy (GC-MS).

The chromatogram generated from the analysis of the used oil sample was compared to chromatograms obtained from virgin samples of Mobile Jet Oil II and BP-2380 (Figure 12). Comparison of the chromatograms showed good correlation between the used oil and BP-2380 (Type II) engine oil (Figure 12B).

No distinct evidence of a contaminant was detected in the oil sample by GC-MS.

The full report provided by Analyze Inc. is attached in Appendix A.

#### **CHEMISTRY**

Analysis of the filtered debris samples from the oil collected from ESN106025C detected various metallic and non-metallic particles. The larger metallic particles were typically rich in iron and associated with lesser concentrations of chromium and traces of other various elements. Aluminum, titanium, nickel, and copper rich particles were also detected amongst the debris. The results of the analyses were summarized and tabulated in Table 1. BSE images and EDX spectra of some of the particles are shown in Figures 13-17.

Debris collected from the planet gears on ESN 106025C and 106026C (Figure 18) were characterized using BSE and EDX techniques. Analysis revealed no distinct similarities in the debris constituents between the two engines. The results of the analyses were summarized and tabulated in Table 2 by ESN. BSE images and EDX spectra of the debris are shown in Figures 19-24.

No additional work was performed on the submitted hardware per customer request.

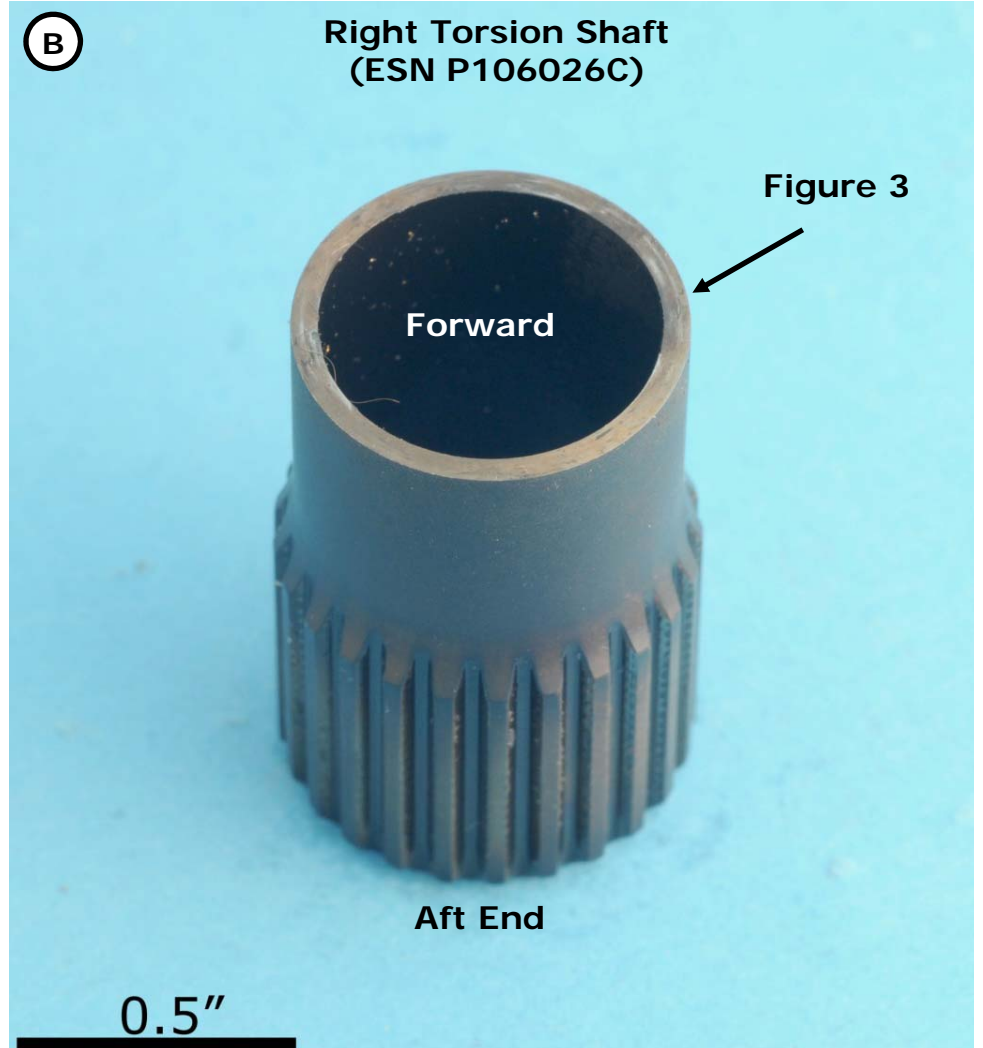
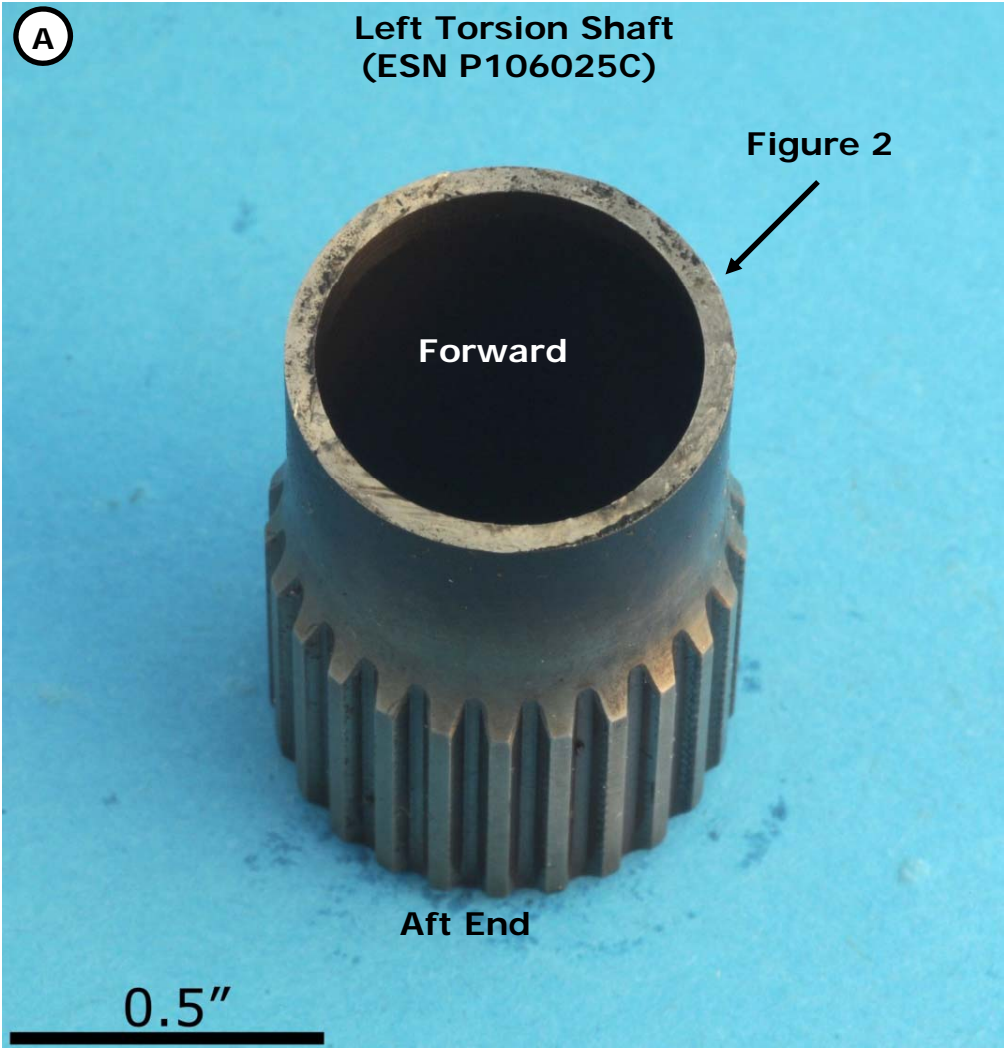


**Table 1. Filtered Debris Samples from ESN P106025C**

Elemental Constituents
1. Ag-rich
2. Al-rich with lesser Cr, C, Cu, and Fe. Traces of Si, Pb, S, Sn, O, and Cd
3. Al-rich with traces of Mg and Ag or Si
4. Ba-rich with Ti, Ca, Cl, and traces of other elements
5. Ca-rich with lesser P
6. Ca-rich with traces of Fe, Cu, Si, and Al
7. Cd-rich with Ba and traces of others
8. Cl-rich with lesser Na (salt)
9. C-rich with traces of Ca, Si, Al, Mg, and O
10. Cu-rich
11. Cu-rich with lesser Fe
12. Cu-rich with traces of Ag, Si, Al, C and O
13. Fe/Si/Al/C/S-rich with lesser Cr, Cu, Ni, Cl, Pb, Si, Al, Cd, Zn, Mg, O and C
14. Fe-rich with lesser Cr and traces of Mn, Ni, Si, and Al
15. Fe-rich with lesser Cr, Ni, Cu, P, S, Pb, Si, Al, Cd, Zn, Mg, O and C
16. Fe-rich with lesser Cr, P and Pb. Traces of other various elements
17. Fe-Rich with lesser Cr, Sn, Zn, Si, and Pb
18. Fe-rich with lesser Cr. Ni and Cu
19. Fe-rich with lesser S and O
20. Fe-rich with lesser Ti and traces of V, Cr, Mn, Si, Al, Mg, and NA
21. Fe-rich with traces of other elements
22. Ni-rich with lesser Cr and Fe. Traces of Ti, Mo, Al, and Si
23. Ni-rich with traces of Ca, Cl, Al, O, and C.
24. Pb-rich
25. Sb-rich
26. Si/Al-rich with lesser K and O with traces of Na, Mg, Pb, Fe, and Cl
27. Si/Al-rich with lesser K and traces of Fe, Mg, Ca, and Ti
28. Si-rich with lesser Mg
29. Si-rich with lesser Ti and Ca with traces of Fe, Cr, Mg, Al, Na, F, O, and C
30. Si-rich with S, Ba, Al and other various elements
31. Si-rich with traces of Al, and Mg
32. S-rich with Si, Ba, Mg and traces of others
33. Ti-rich with V, Si, Al, Cr, Fe, Mg, C, and O
34. Zr-rich with lesser Si

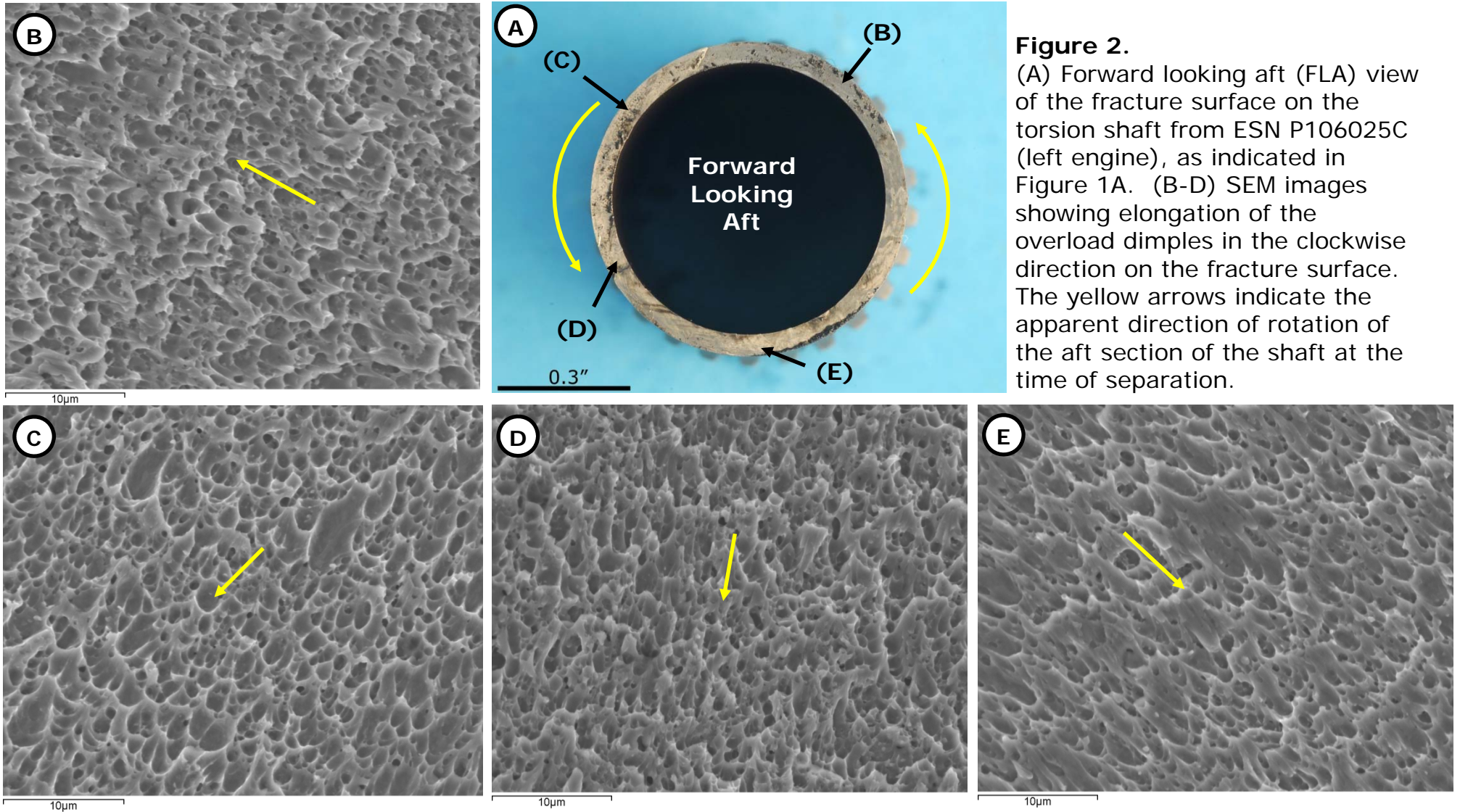
**Table 2. Planet Gear Debris**

Engine	Elemental Constituents
Left (ESN P106025C)	1. Al-rich with traces of Mg and Si
	2. Fe-rich with lesser Cr and traces of Ni, Ti, Si, and Al
	3. Fe-rich with lesser Si and traces of Zn, Al, Ti, Su, Cl, O, and Ca
	4. Si/Al-rich with lesser K and traces of Fe, Mg, Na, and Ti
	5. Ti-rich with V, Si, and Al
Right (ESN P106026C)	1. Ag-rich
	2. C-rich with lesser Cl, Si, P, Na, Al, Mg, F, Cd, K, Fe, Ti, Cr, Cu
	3. F/C/Si-rich with traces of O, Cl, Cd, and S
	4. F/C-rich
	5. F/C-rich with traces of Cd, Cl, S, and Si
	6. F/C-rich with traces of Cl, Ti, Fe, Cu, K, Si, Al, P, and Mg
	7. Si-rich with lesser Al, Ca, Fe, K and traces of Mg, Ti, Cl, O and C
	8. Si-rich with traces of Al, O, and C

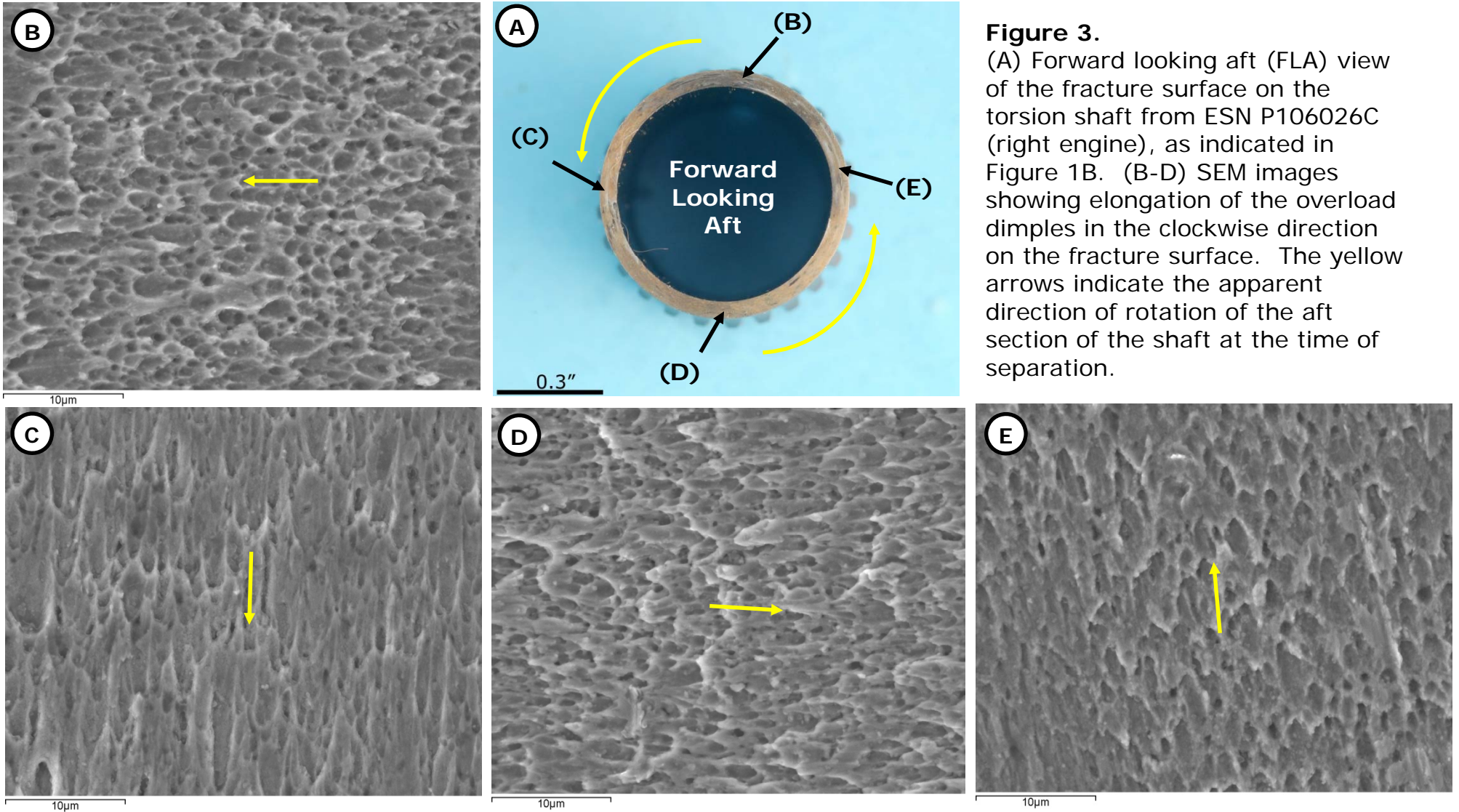


**Figure 1.**

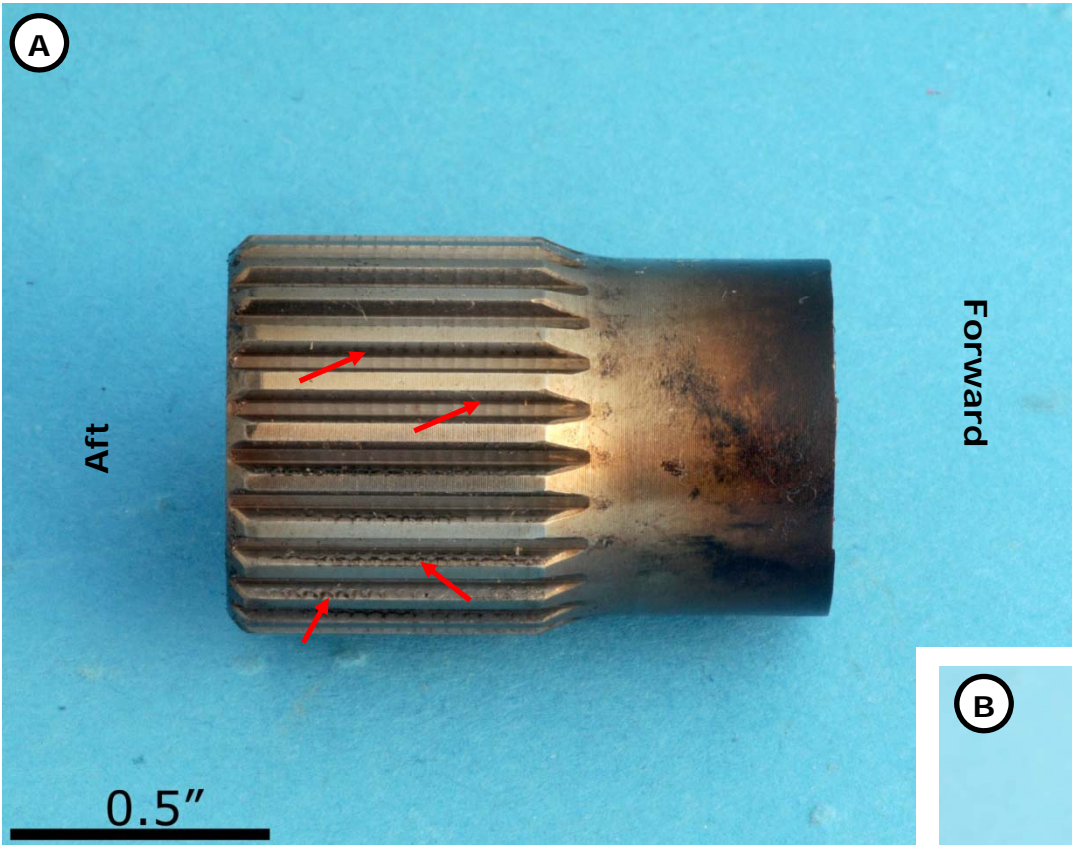
Overall photos showing the separations of the (A) left (ESN P106025C) and (B) right (ESN P106026C) torsion shafts. The fracture surfaces of the torsion shaft separations are shown again in Figures 2 and 3, as indicated.





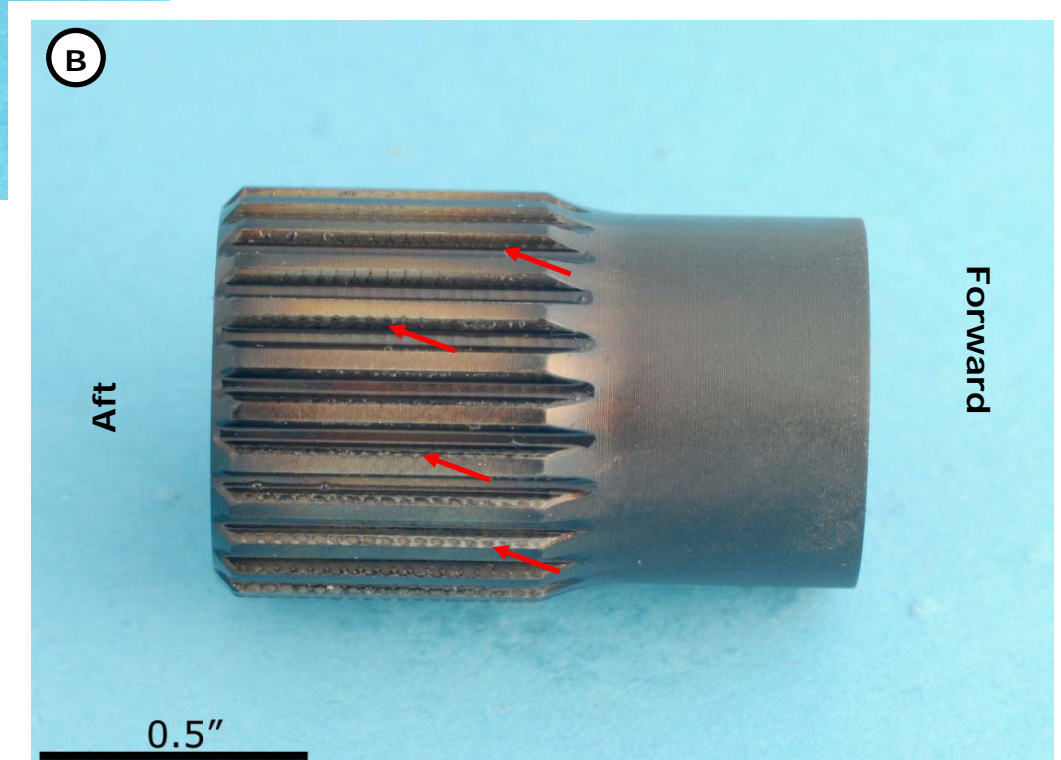


**Figure 3.** (A) Forward looking aft (FLA) view of the fracture surface on the torsion shaft from ESN P106026C (right engine), as indicated in Figure 1B. (B-D) SEM images showing elongation of the overload dimples in the clockwise direction on the fracture surface. The yellow arrows indicate the apparent direction of rotation of the aft section of the shaft at the time of separation.

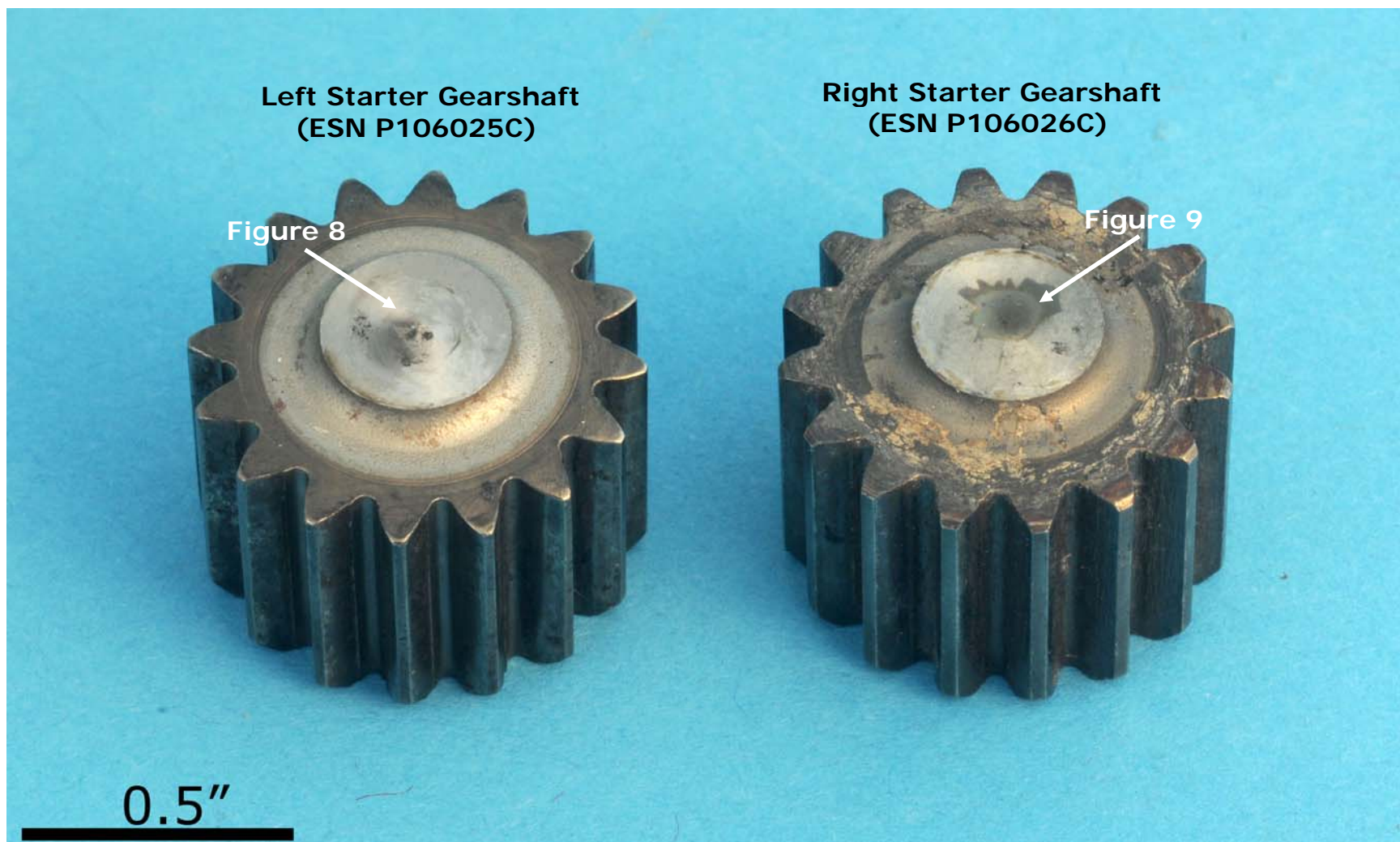


**Figure 4.**

Side views of the aft sections of the torsion shafts from (A) ESN P106025C (left engine), and (B) ESN P106026C (right engine) showing the light wear/contact patterns (red arrows) on the spline teeth.



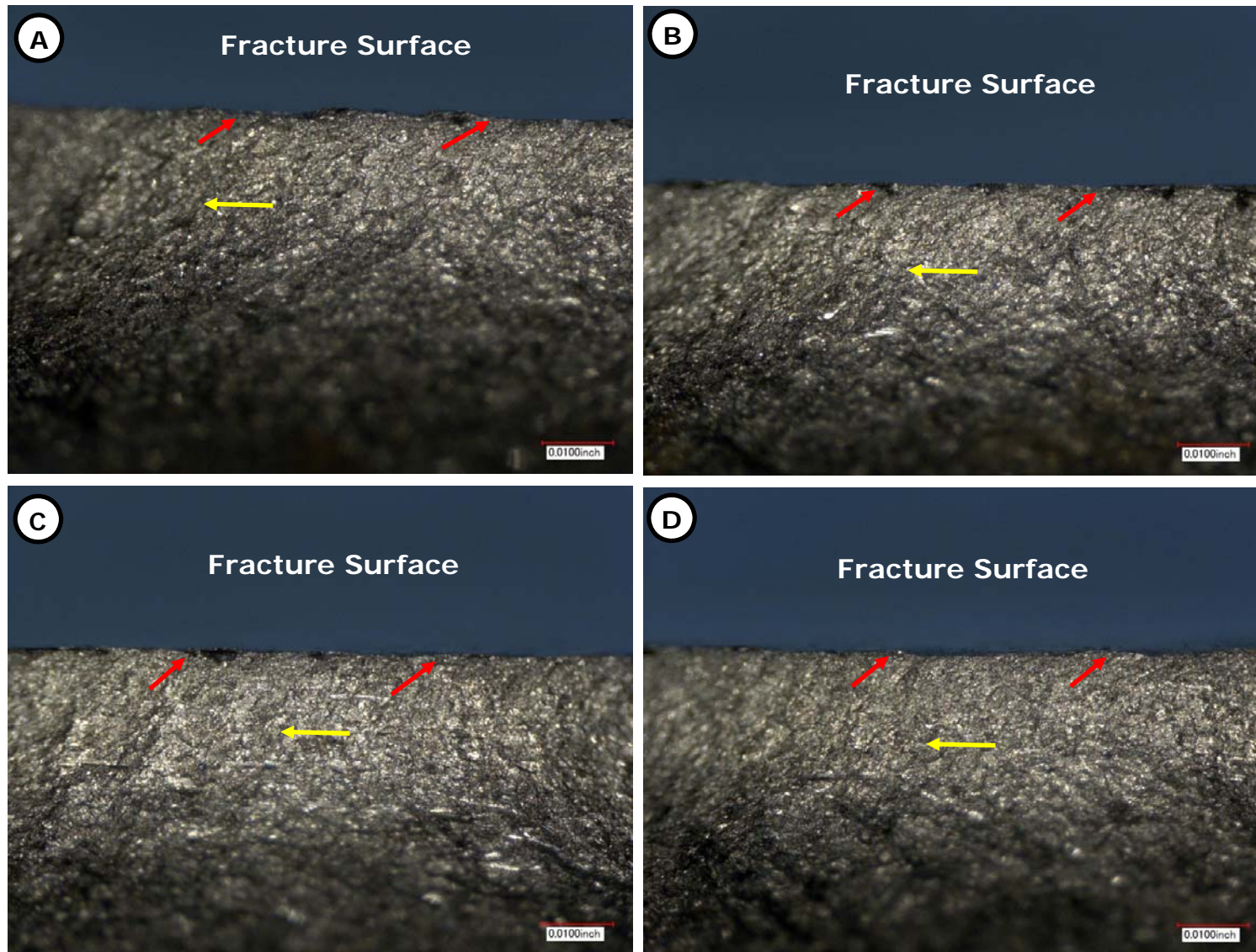




**Figure 5.**

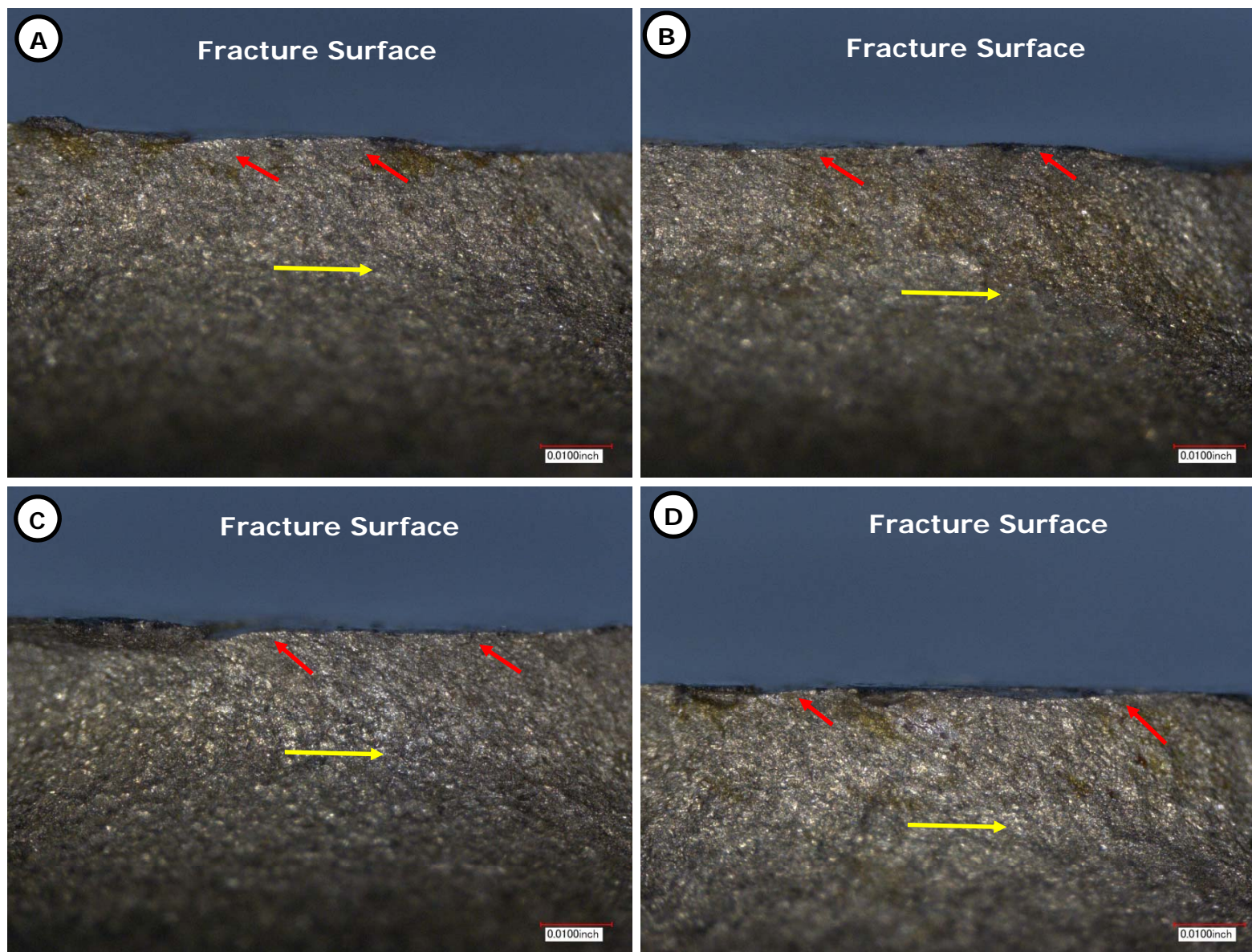
Overall photos showing the separations of the starter gearshafts from (A) ESN P106025C (left engine), and (B) ESN P106026C (right engine). Digital microscope images showing side views of the reduced diameter sections adjacent to the fracture surfaces are shown in Figures 6 and 7. SEM images of the fracture surfaces are shown in Figures 8 and 9.





**Figure 6.**

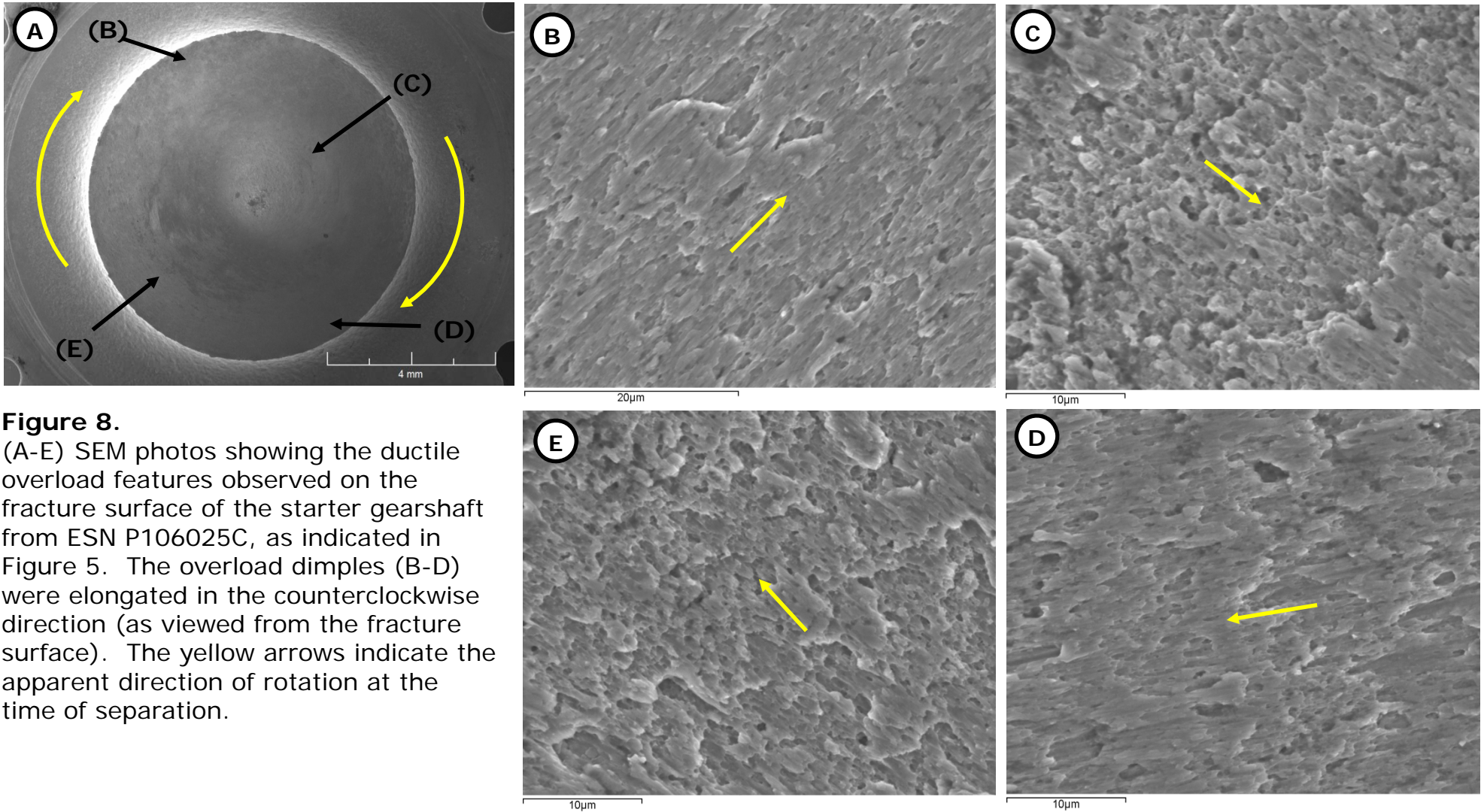
(A-D) Digital microscope images showing the deformation noted in the reduced diameter section adjacent to the fracture surface on the starter gearshaft from ESN 106025C, as indicated in Figure 5. The shafts were deformed in the direction indicated by the red arrows which indicated that the separated section of the shaft was rotating in the clockwise direction (yellow arrows), as viewed from the fracture surface.



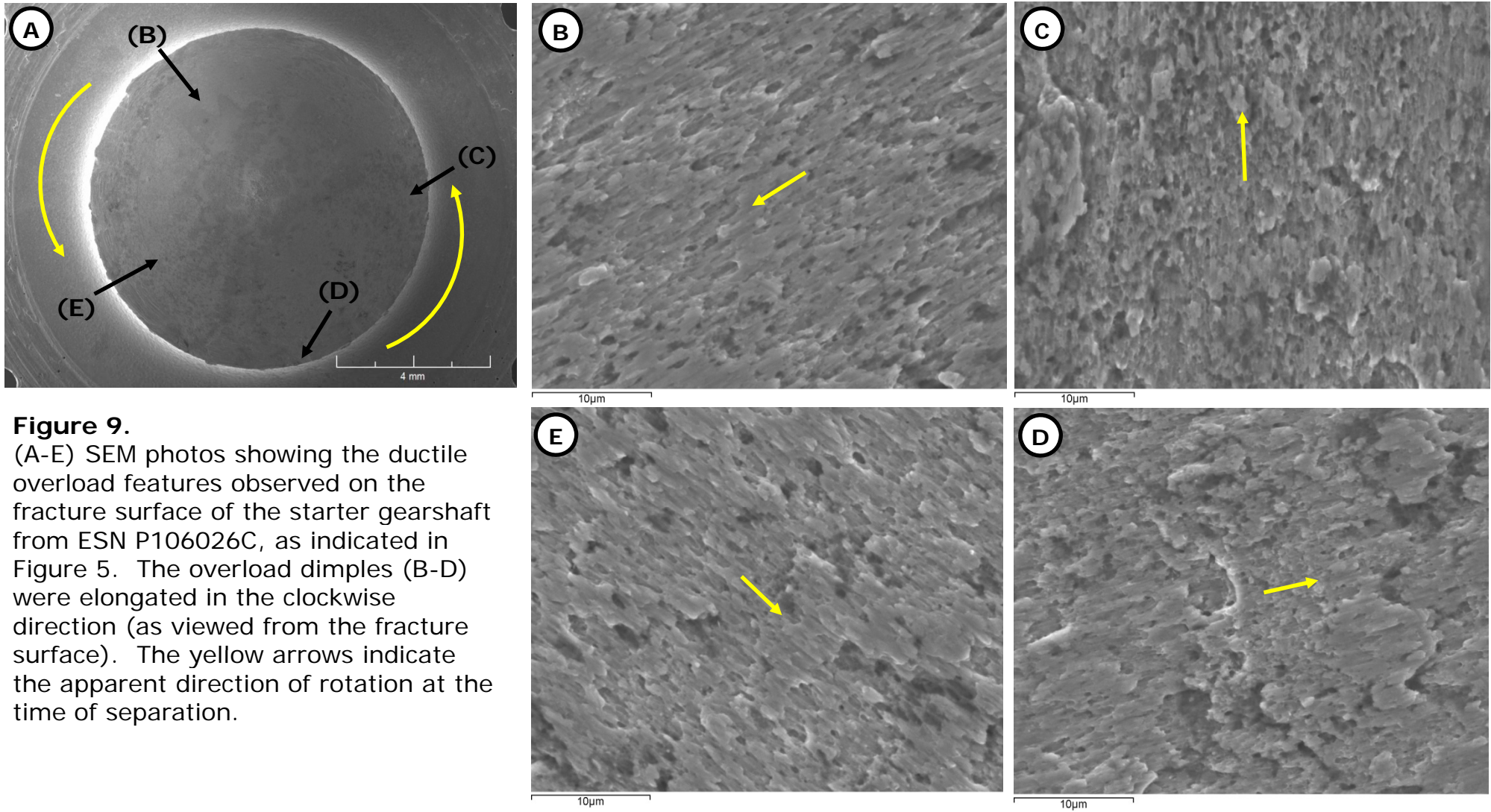
**Figure 7.**

(A-D) Digital microscope images showing the deformation noted in the reduced diameter section adjacent to the fracture surface on the starter gearshaft from ESN 106026C, as indicated in Figure 5. The shafts were deformed in the direction indicated by the red arrows which indicated that the separated section of the shaft was rotating in the counterclockwise direction (yellow arrows), as viewed from the fracture surface.



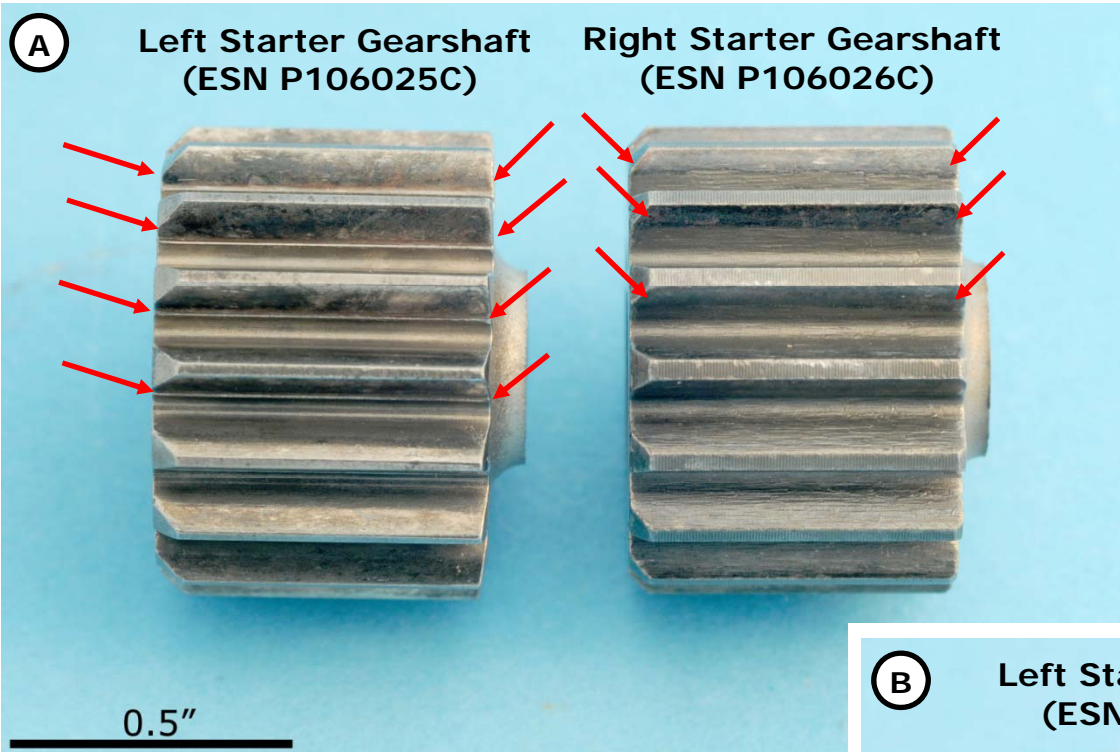


**Figure 8.** (A-E) SEM photos showing the ductile overload features observed on the fracture surface of the starter gearshaft from ESN P106025C, as indicated in Figure 5. The overload dimples (B-D) were elongated in the counterclockwise direction (as viewed from the fracture surface). The yellow arrows indicate the apparent direction of rotation at the time of separation.

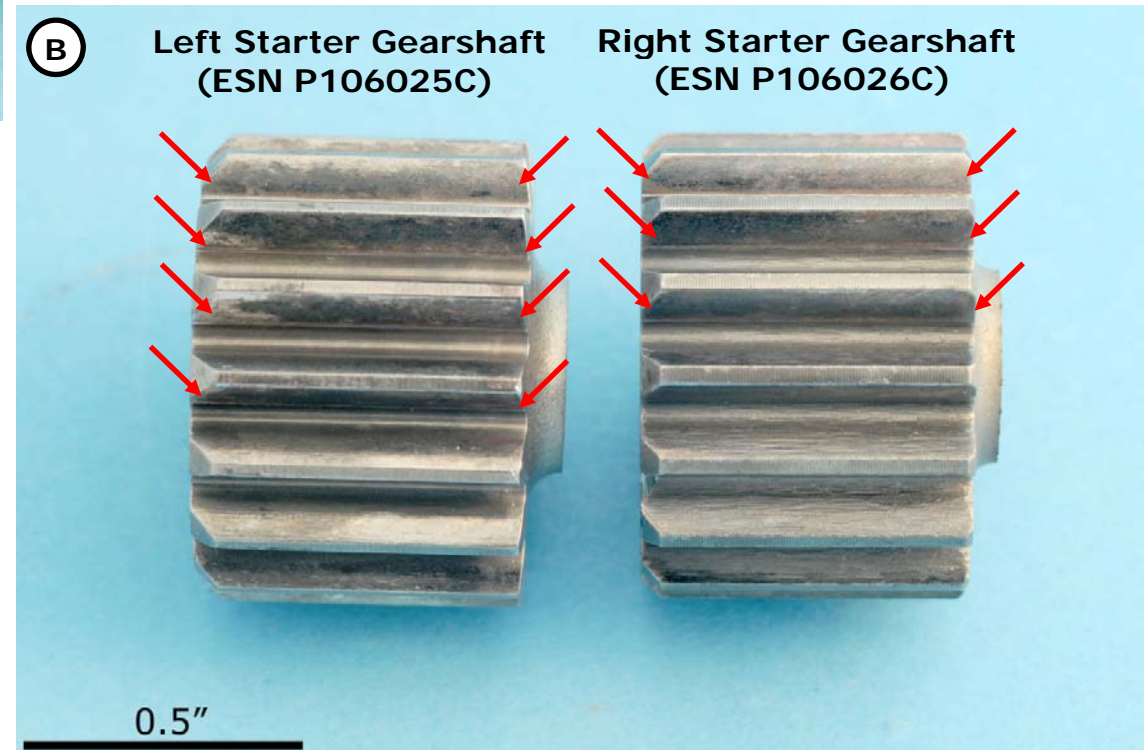


**Figure 9.** (A-E) SEM photos showing the ductile overload features observed on the fracture surface of the starter gearshaft from ESN P106026C, as indicated in Figure 5. The overload dimples (B-D) were elongated in the clockwise direction (as viewed from the fracture surface). The yellow arrows indicate the apparent direction of rotation at the time of separation.





**Figure 10.**  
(A and B) Side views of showing the wear patterns (red arrows) noted on the loaded sides of the gear teeth on the starter gearshafts from ESNs P106025C and P106026C.

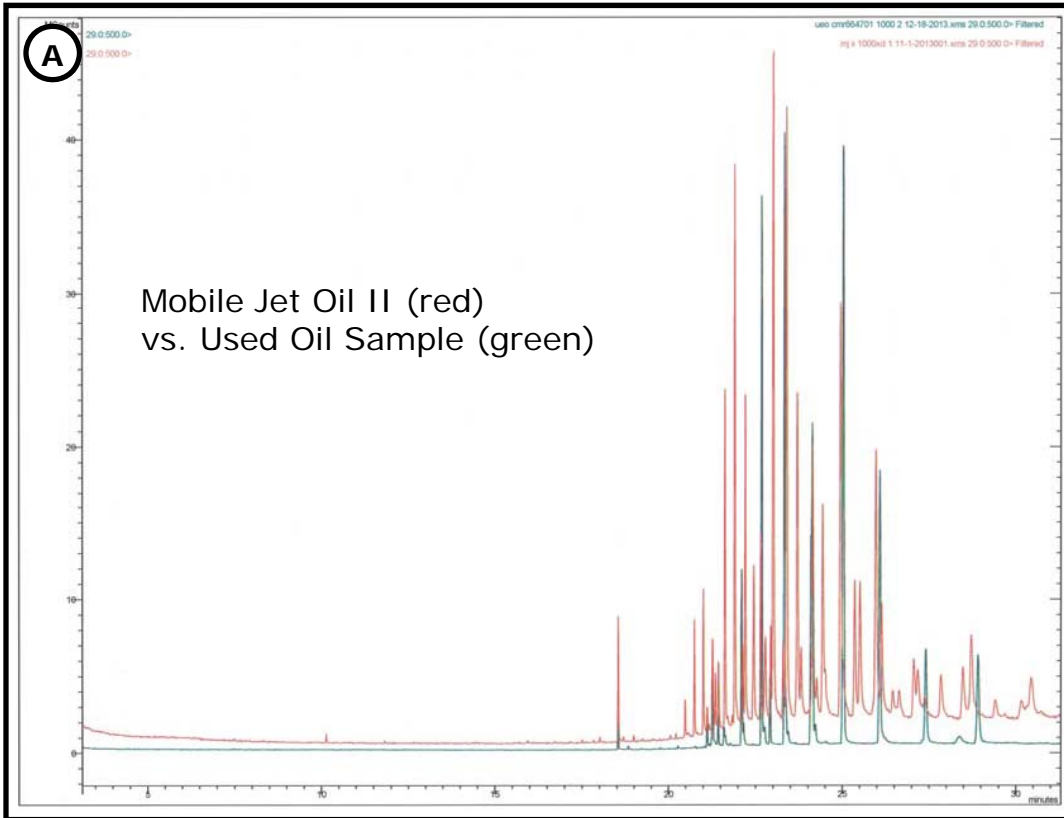




**Figure 11.**

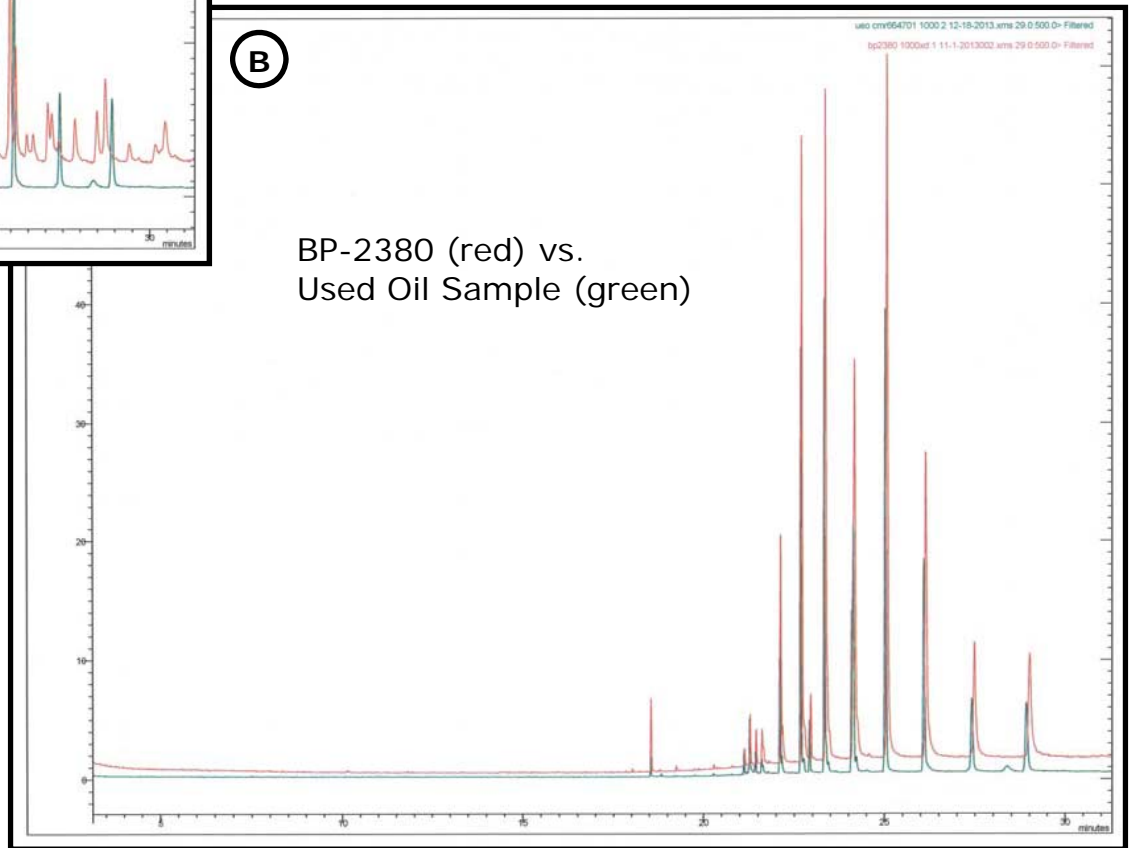
(A) Overall photo showing the oil sample collected from ESN P106025C.  
(B) Close-up photo showing the debris collected from the filtered oil sample.





**Figure 12.**

GC-MS total ion chromatograms (TIC) comparing the use oil sample from ESN P106025C (green in (A) and (B)) to virgin samples of Mobile Jet Oil II and BP-2380 engine oil (red in (A) and (B), respectively). Note that the TIC for the use oil sample showed good correlation to the TIC for the virgin BP-2380 sample.





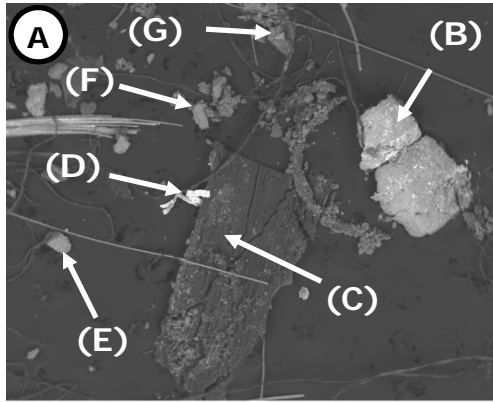
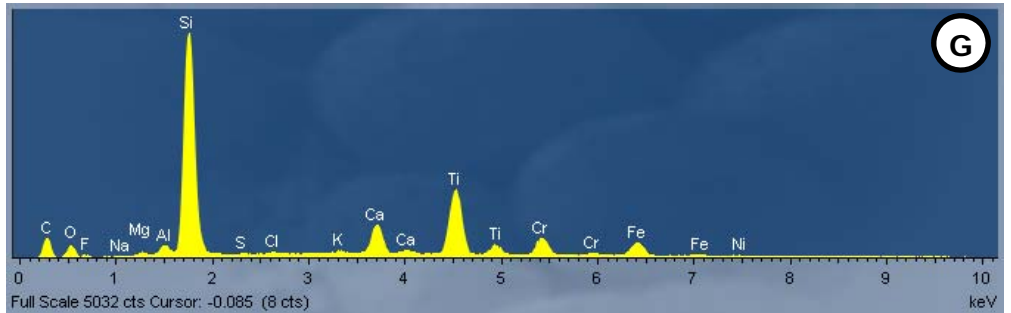
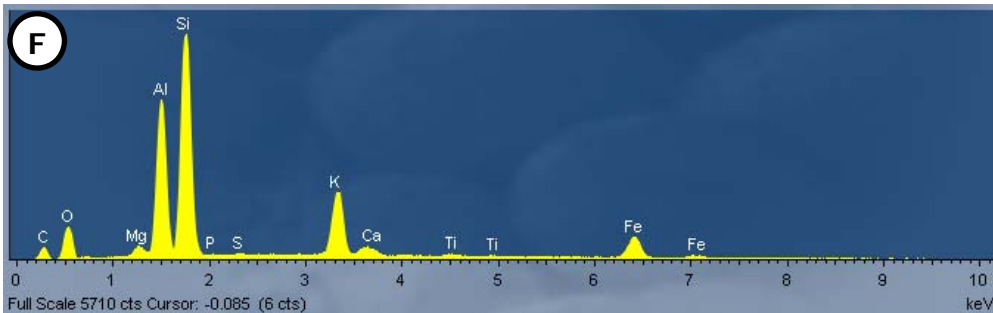
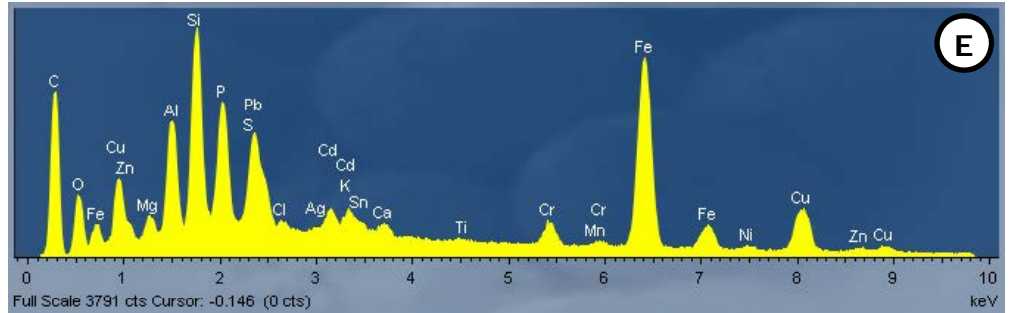
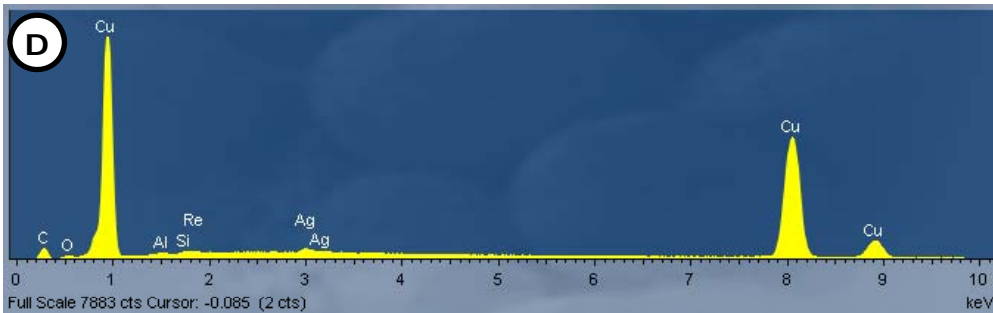
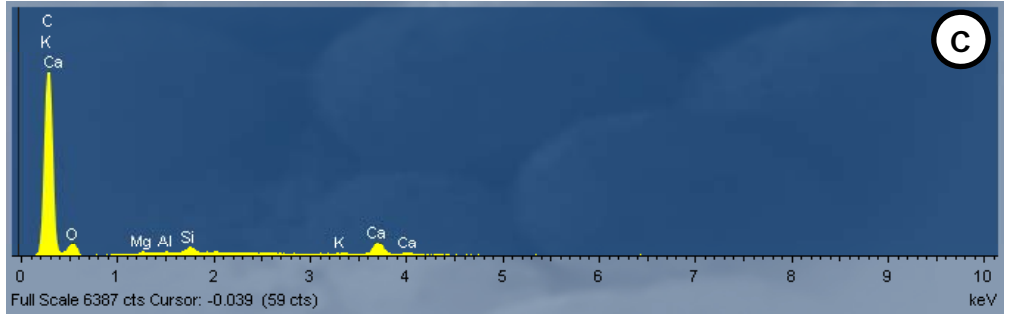
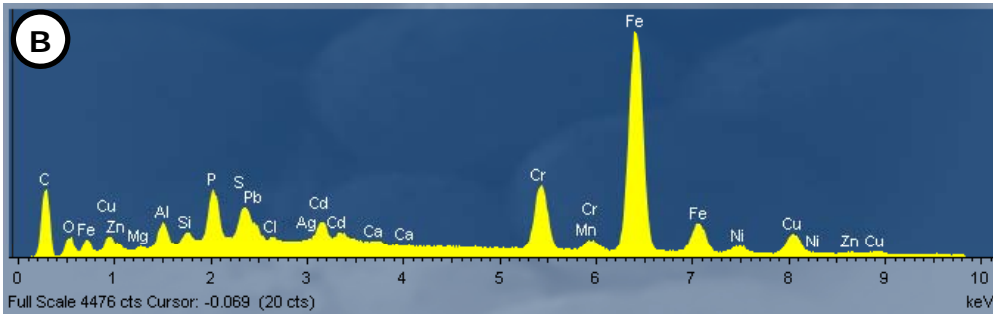
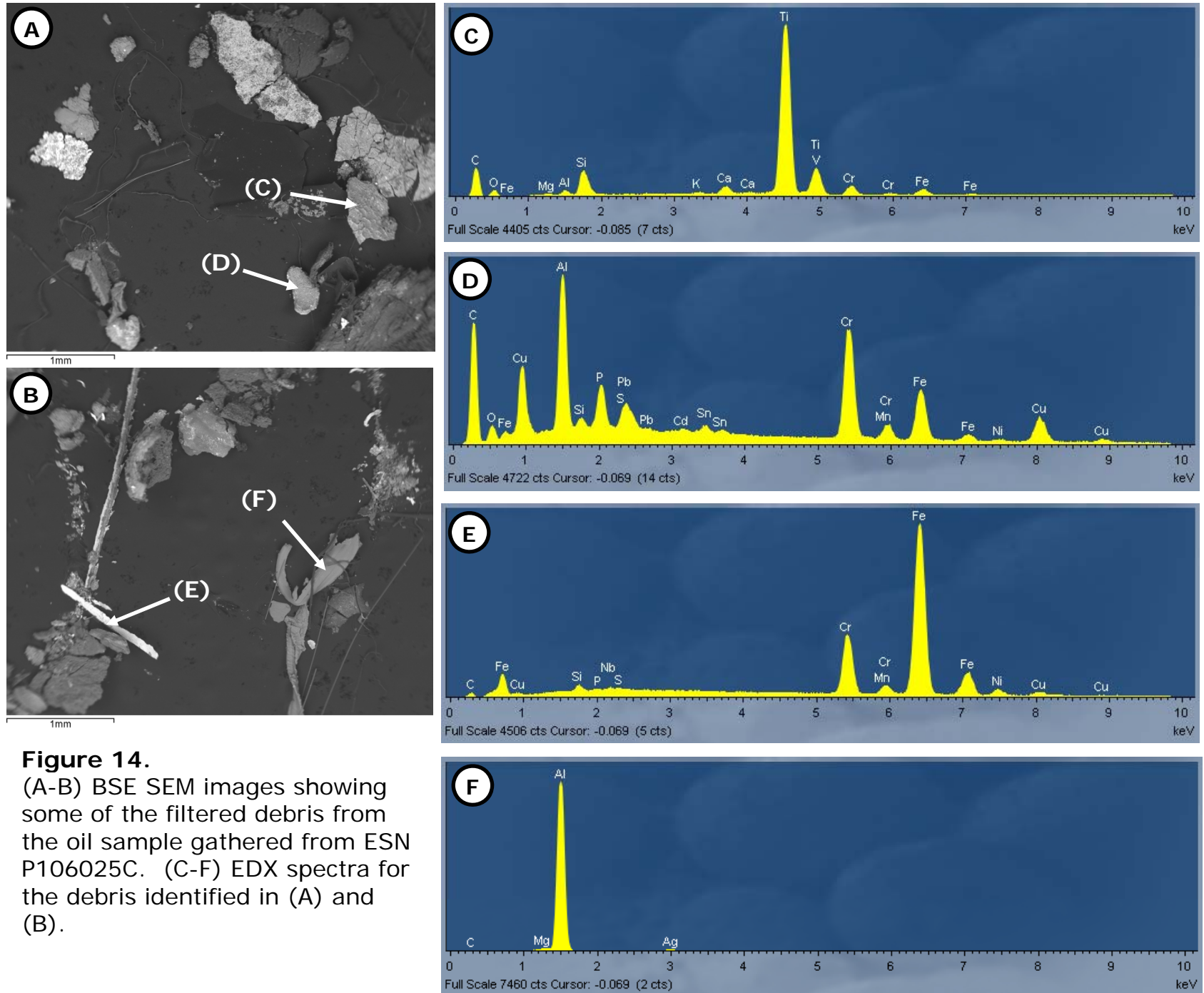


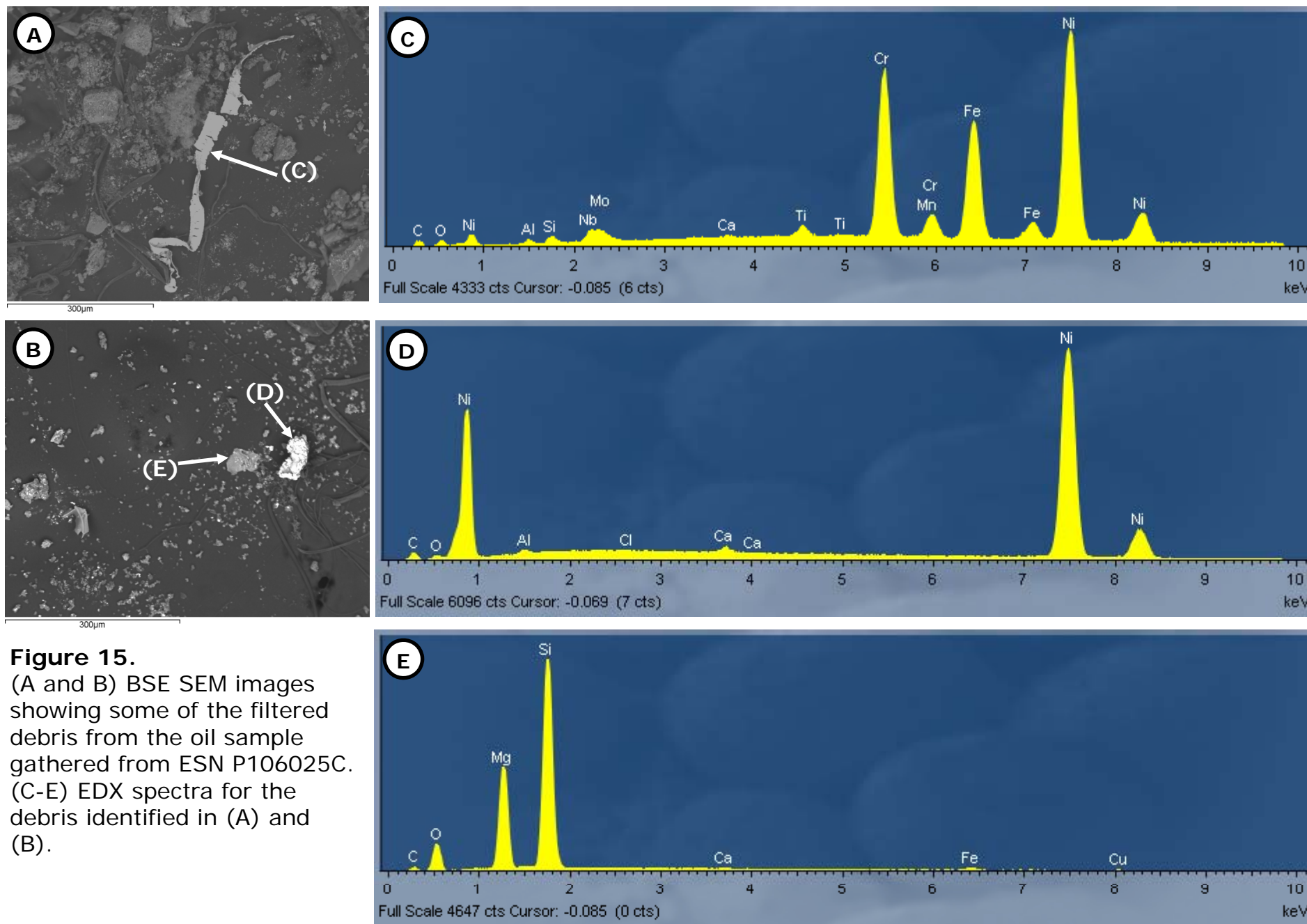
Figure 13.

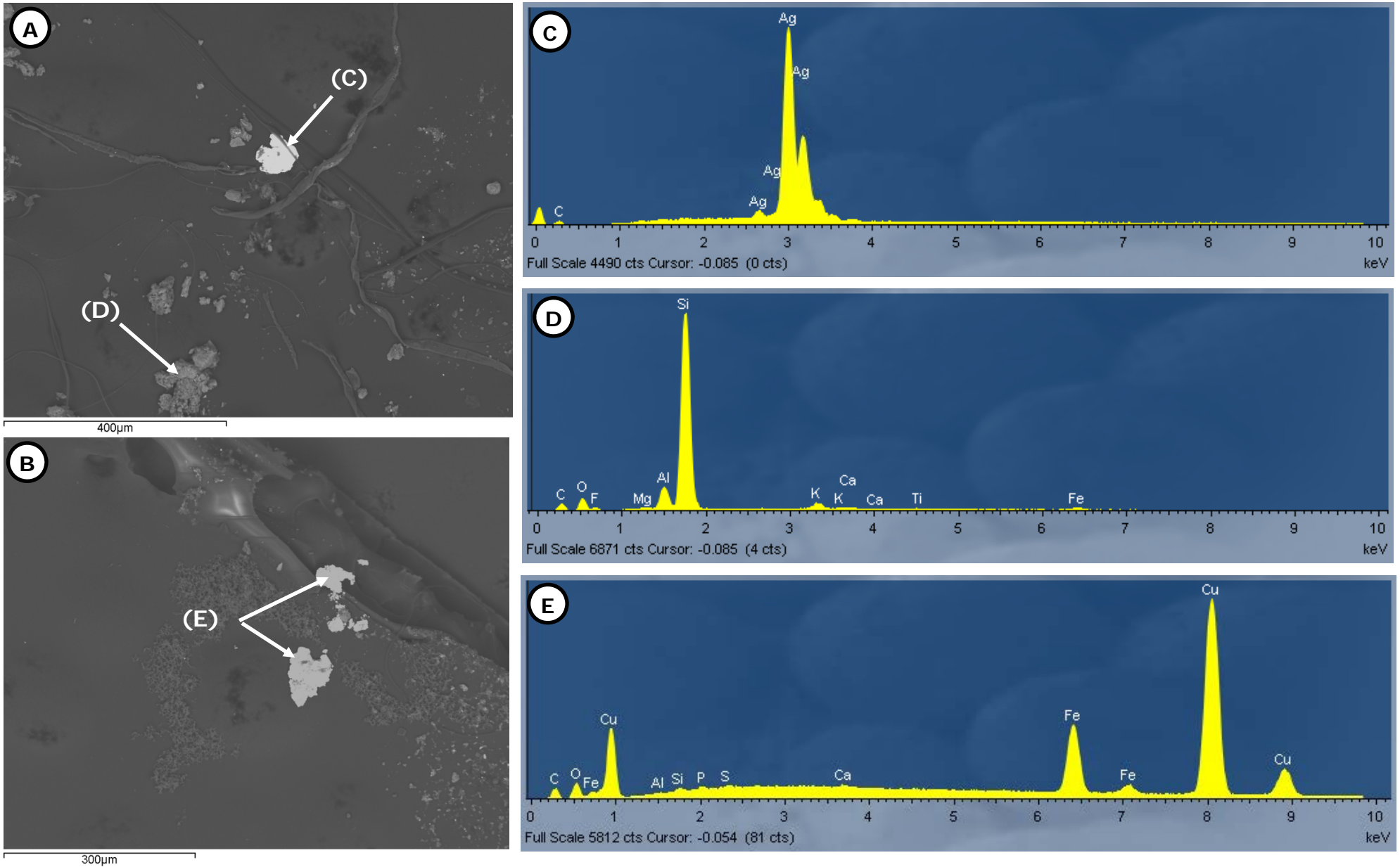
(A) Backscattered electron (BSE) SEM image showing some of the filtered debris from the oil sample gathered from ESN P106025C. (B-G) EDX spectra for the debris identified in (A).





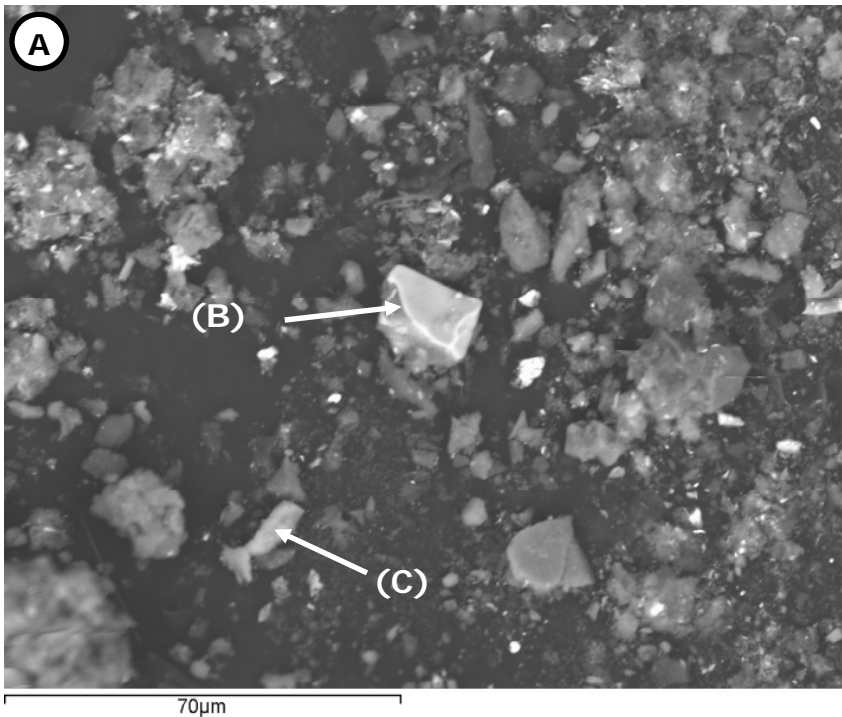
**Figure 14.**  
 (A-B) BSE SEM images showing some of the filtered debris from the oil sample gathered from ESN P106025C. (C-F) EDX spectra for the debris identified in (A) and (B).





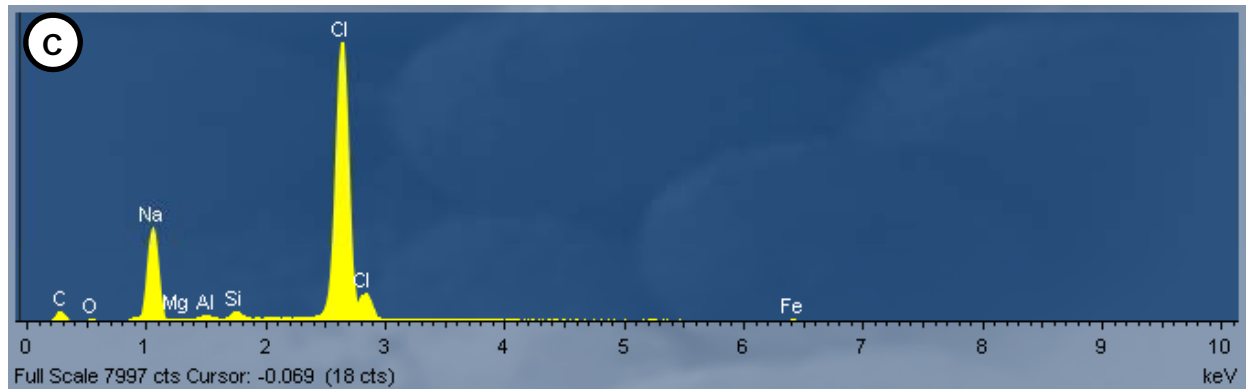
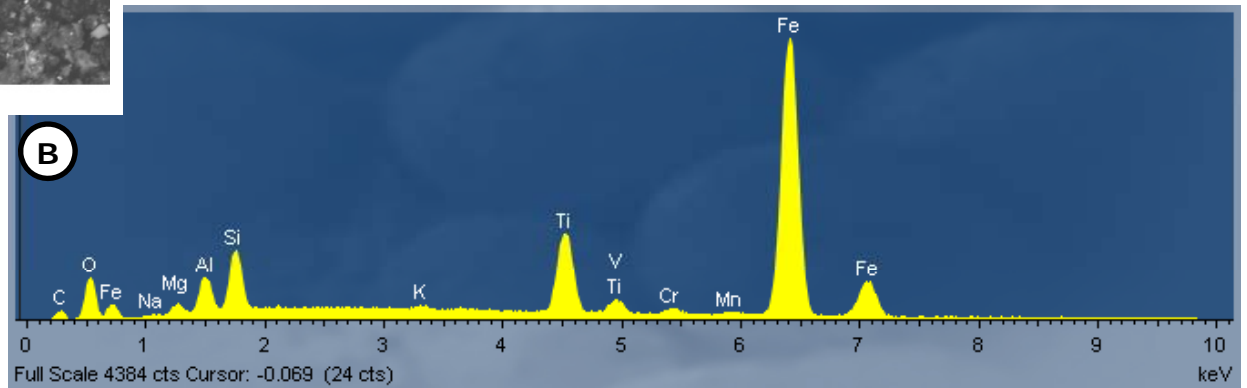
**Figure 16.** (A and B) BSE SEM images showing some of the filtered debris from the oil sample gathered from ESN P106025C. (C-E) EDX spectra for the debris identified in (A) and (B).

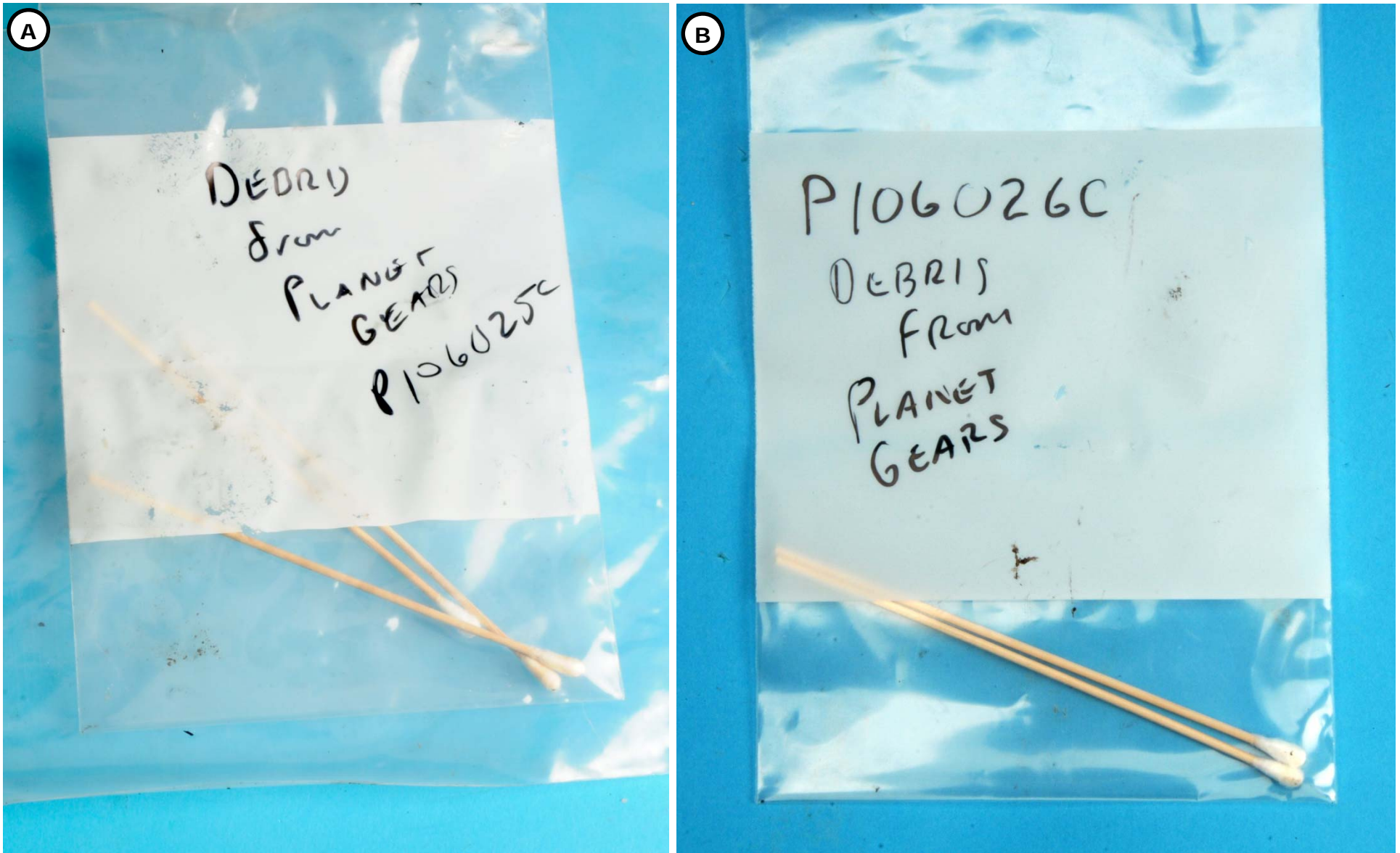




**Figure 17.**

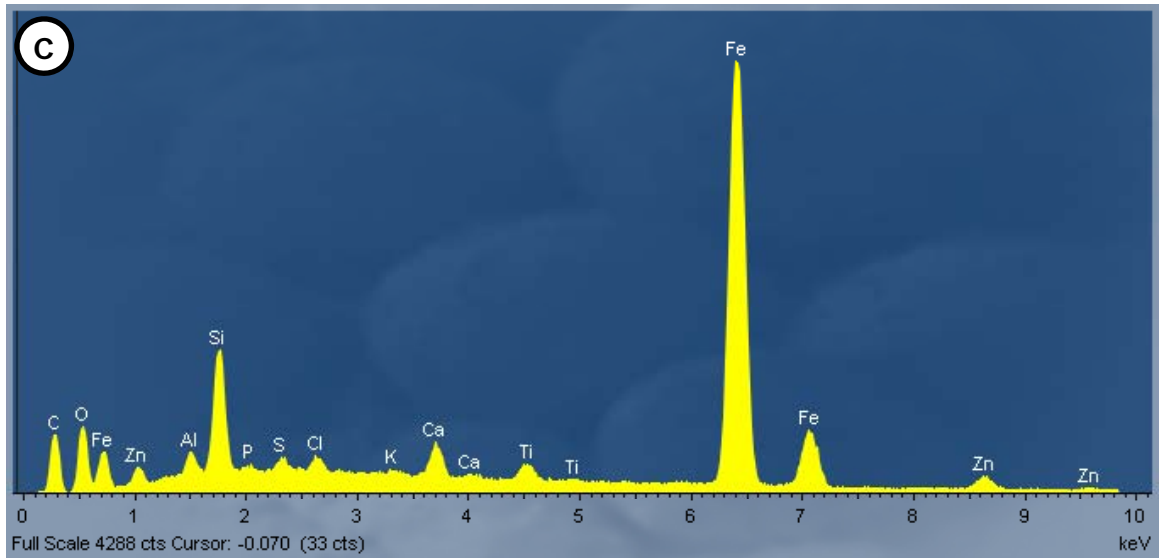
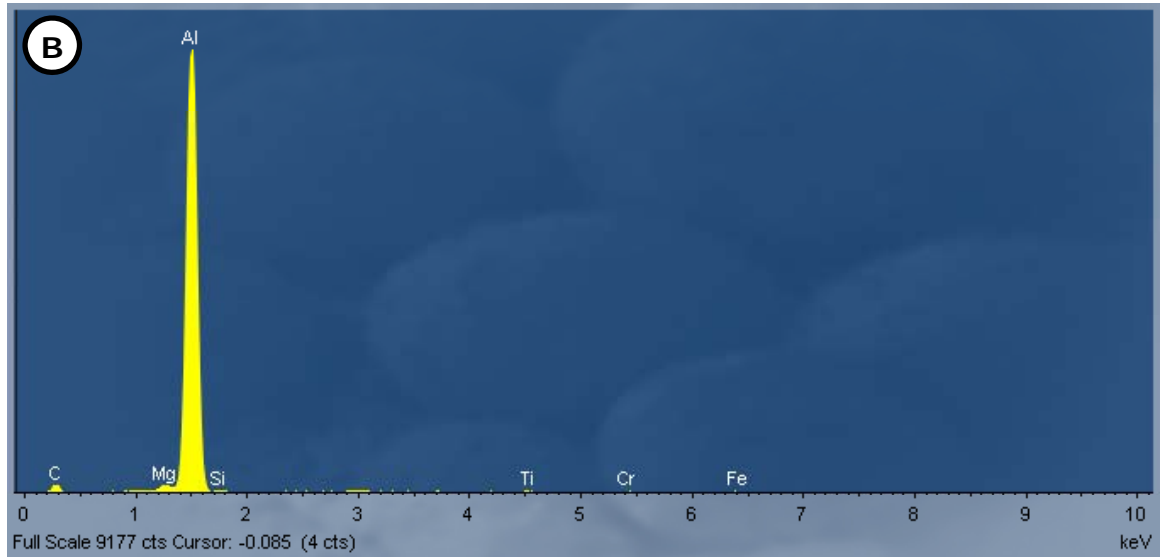
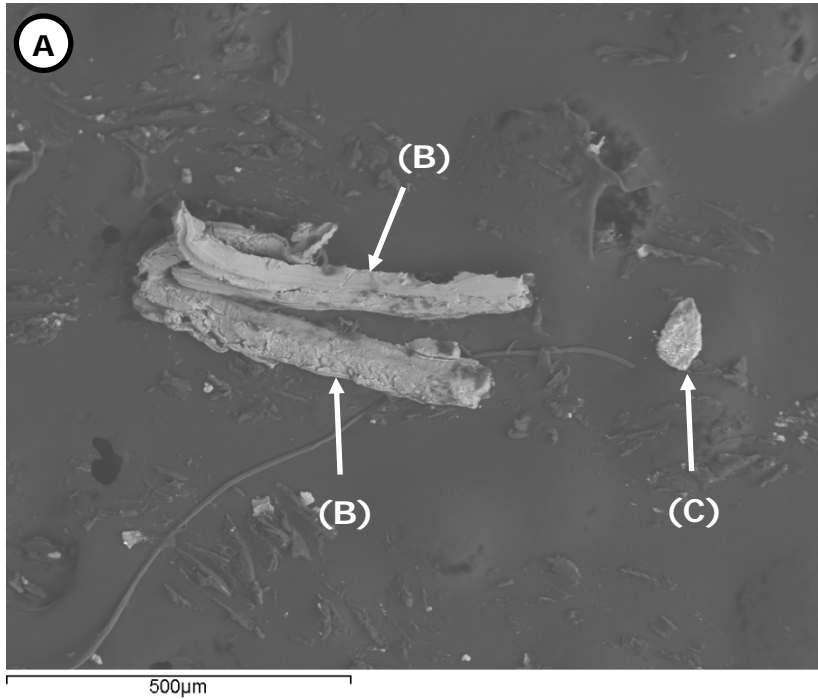
(A) BSE SEM image showing some of the filtered debris from the oil sample gathered from ESN P106025C. (B and C) EDX spectra for the debris identified in (A).





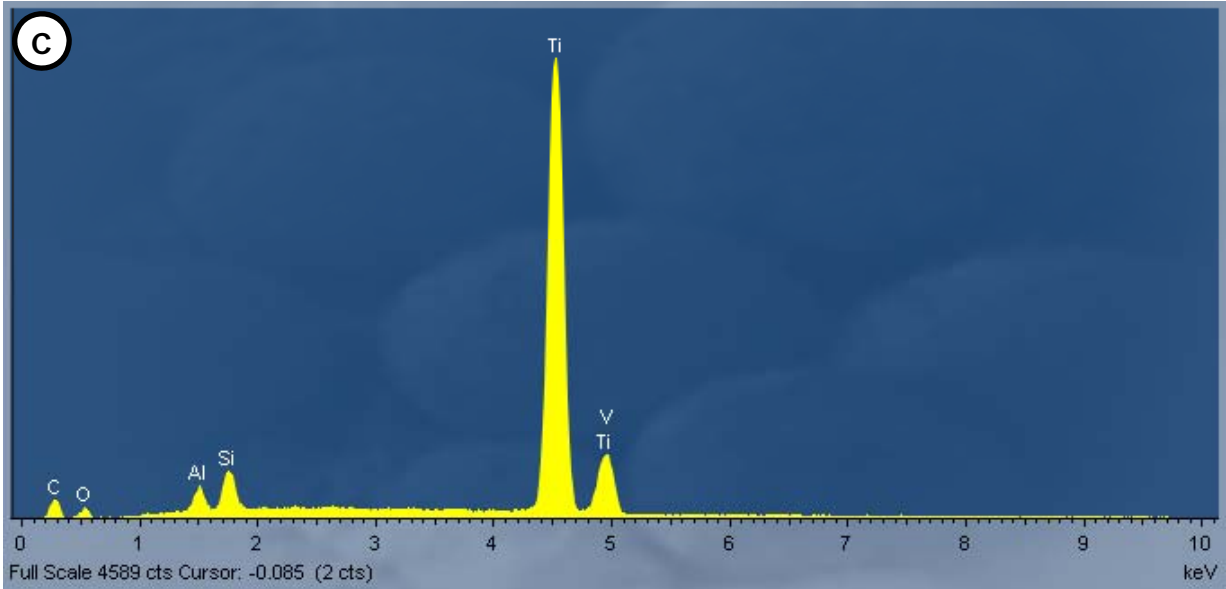
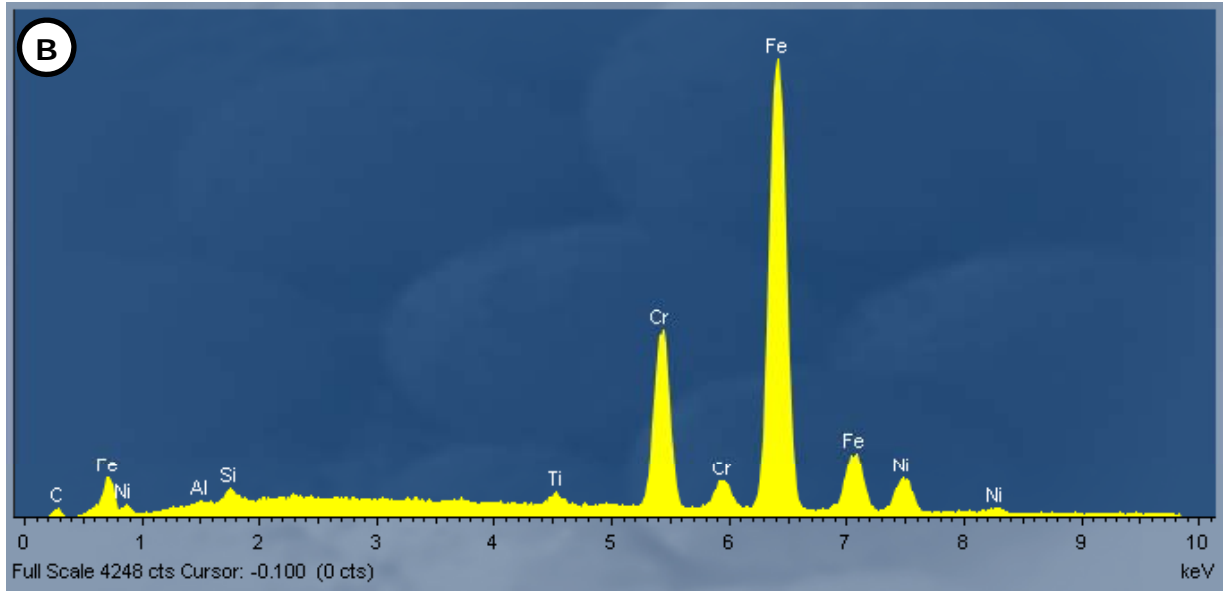
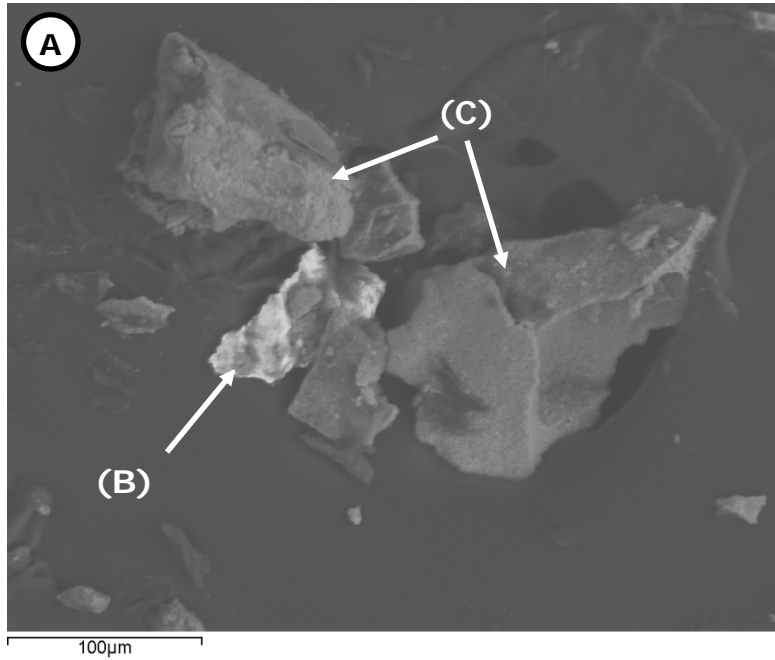
**Figure 18.**

Overall photos showing the as-received condition of the planet gear debris collected from ESNs (A) P106025C and (B) P106025C. The debris was transferred from the swabs/bags to carbon tabs for characterization in the SEM.

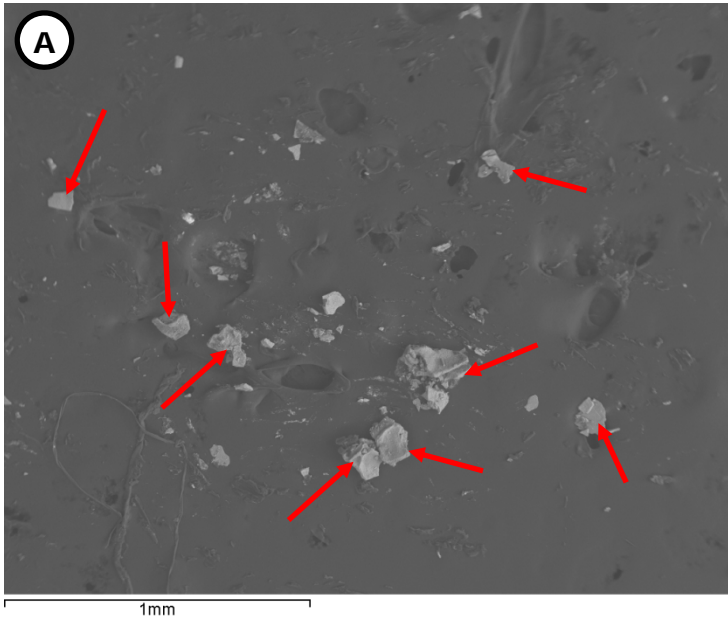


**Figure 19.** (A) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106025C. (B and C) EDX spectra of the debris indicated in (A).



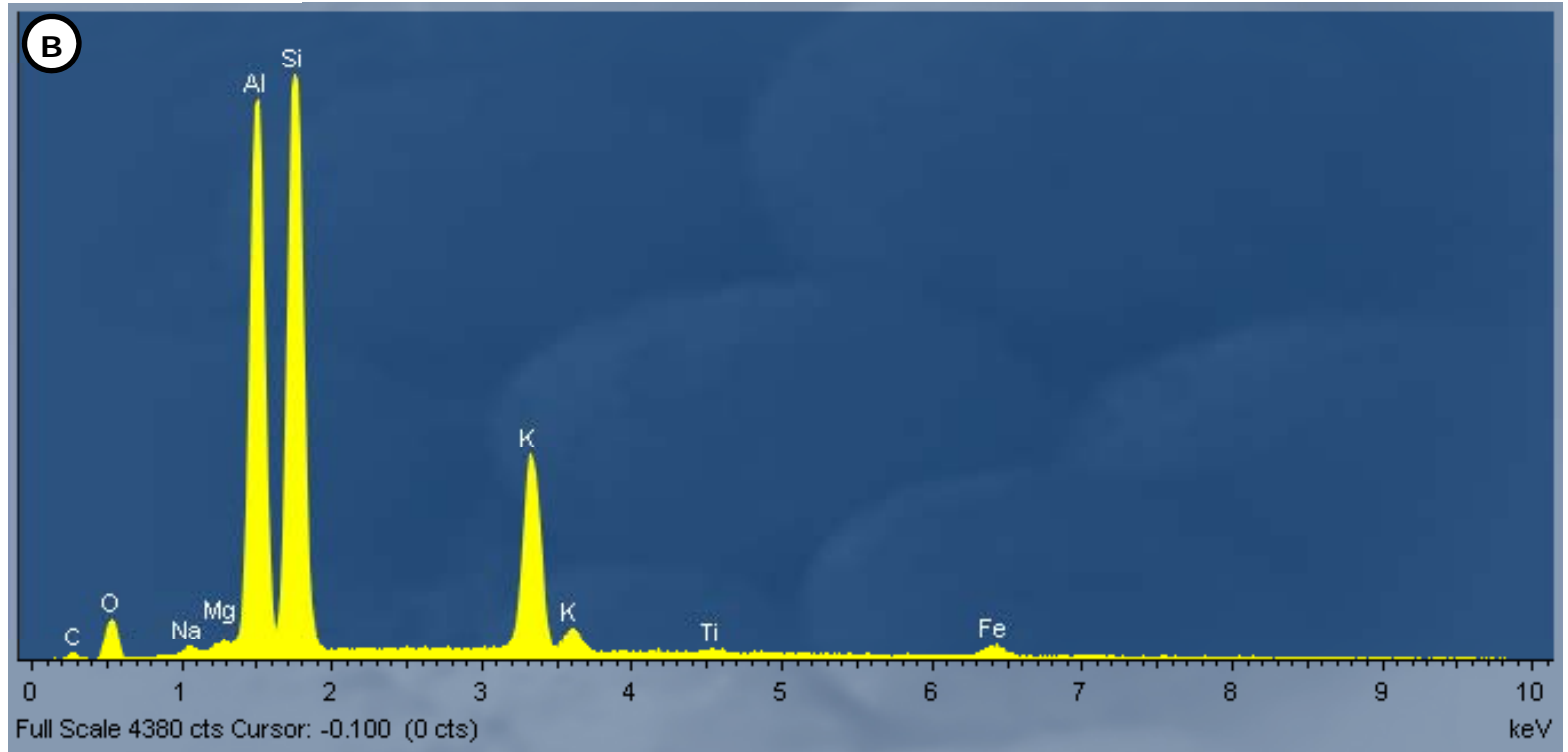


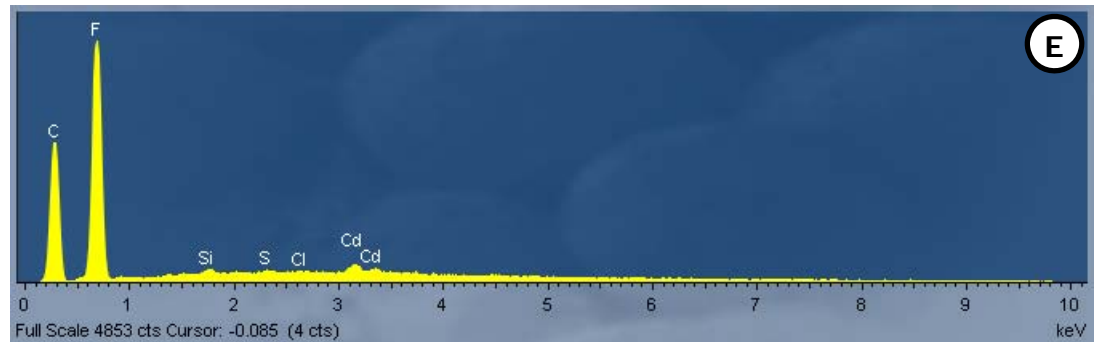
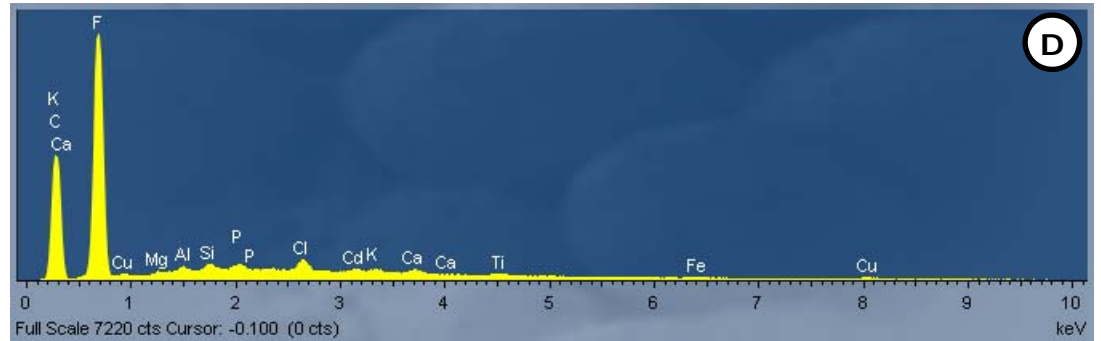
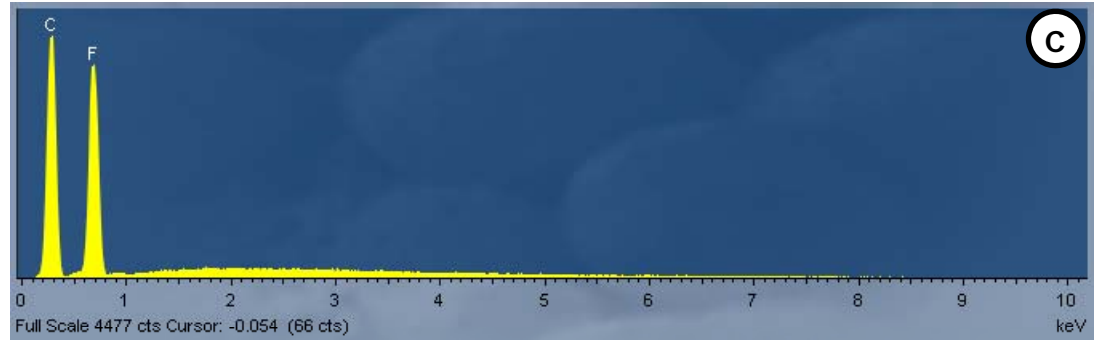
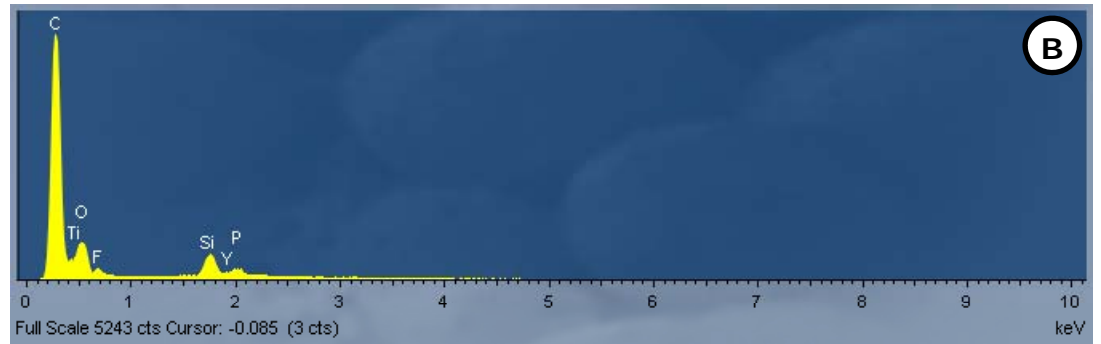
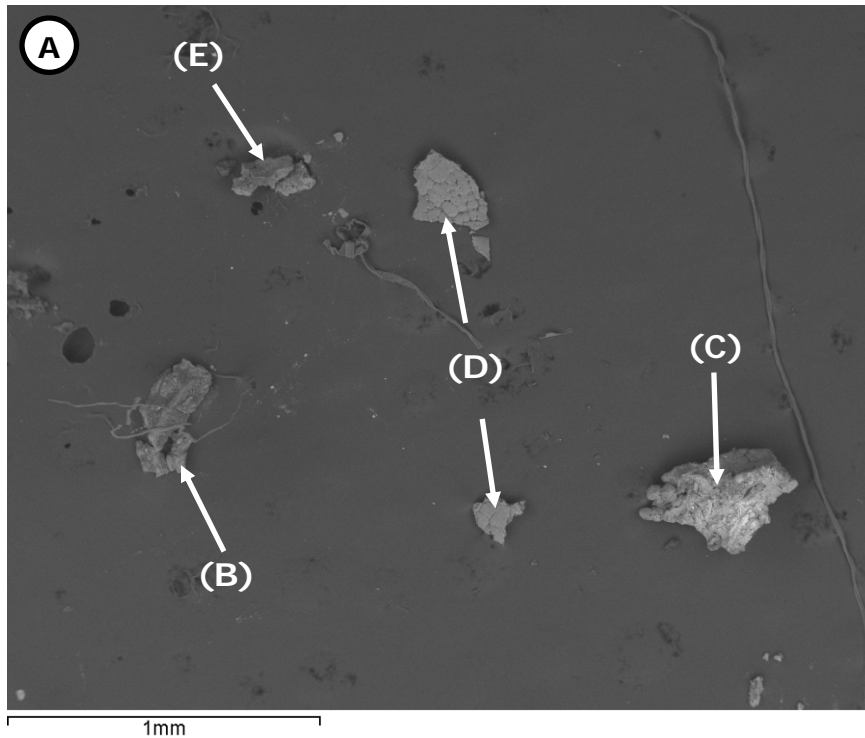
**Figure 20.**  
(A) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106025C. (B and C) EDX spectra of the debris indicated in (A).



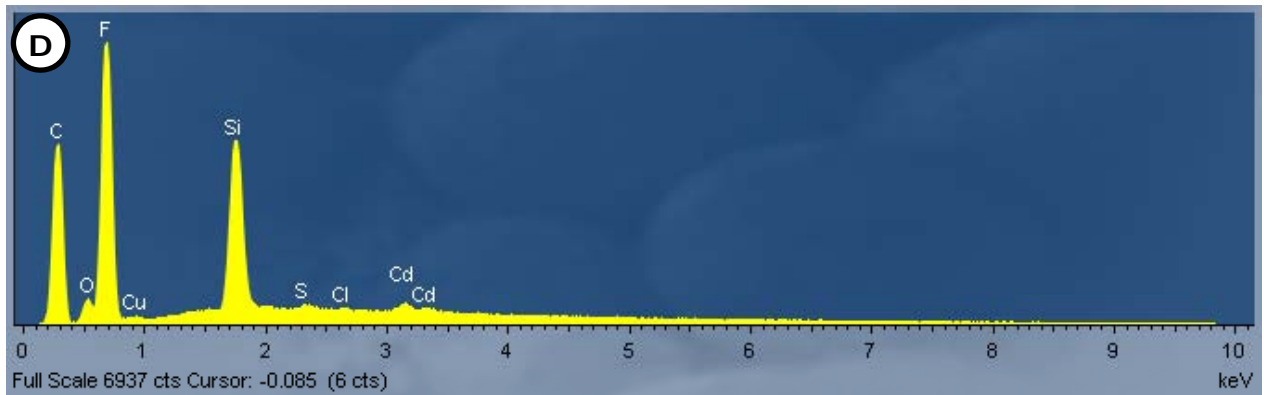
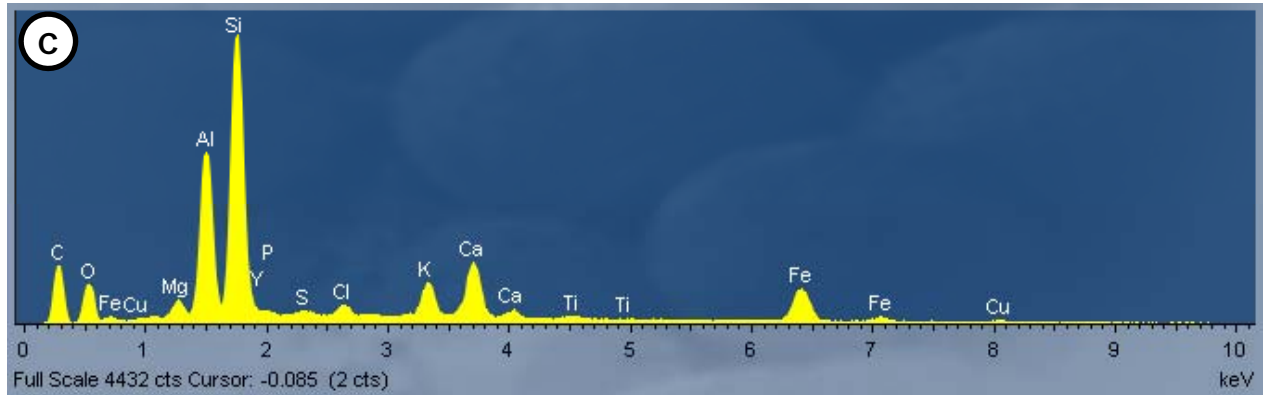
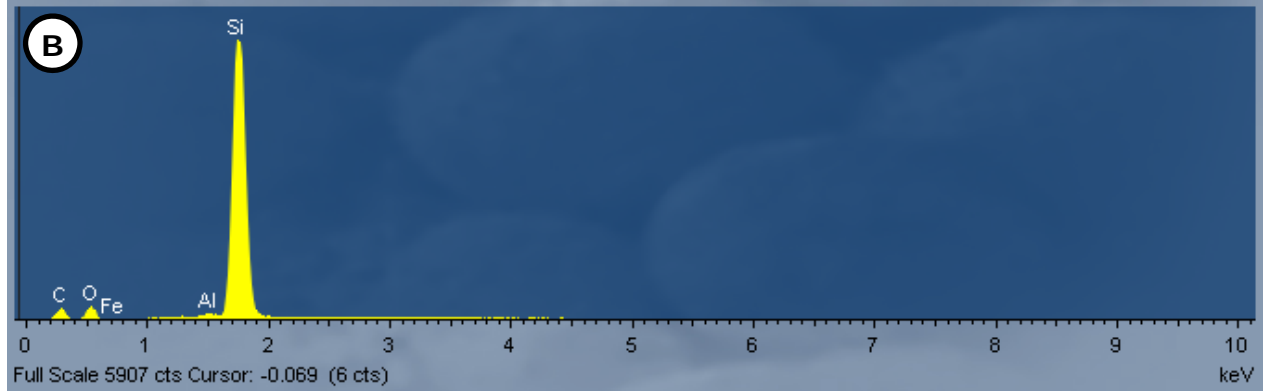
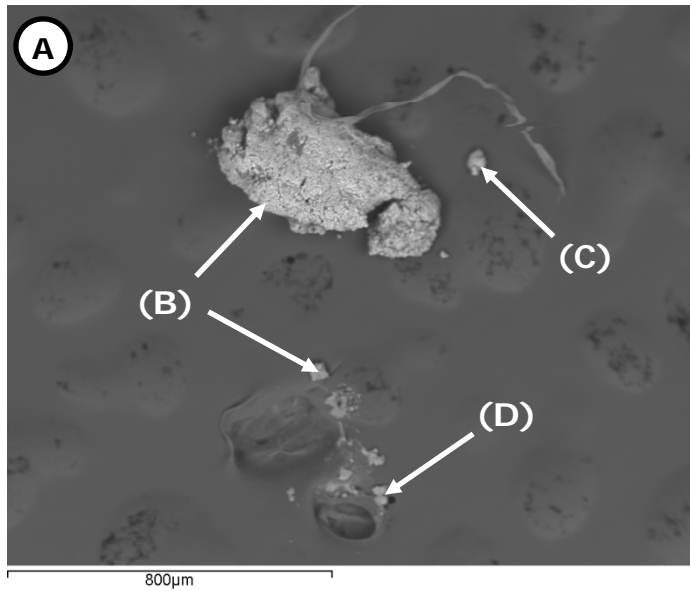
**Figure 21.**

(A) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106025C (red arrows). (B) Representative EDX spectrum for the debris indicated in (A).

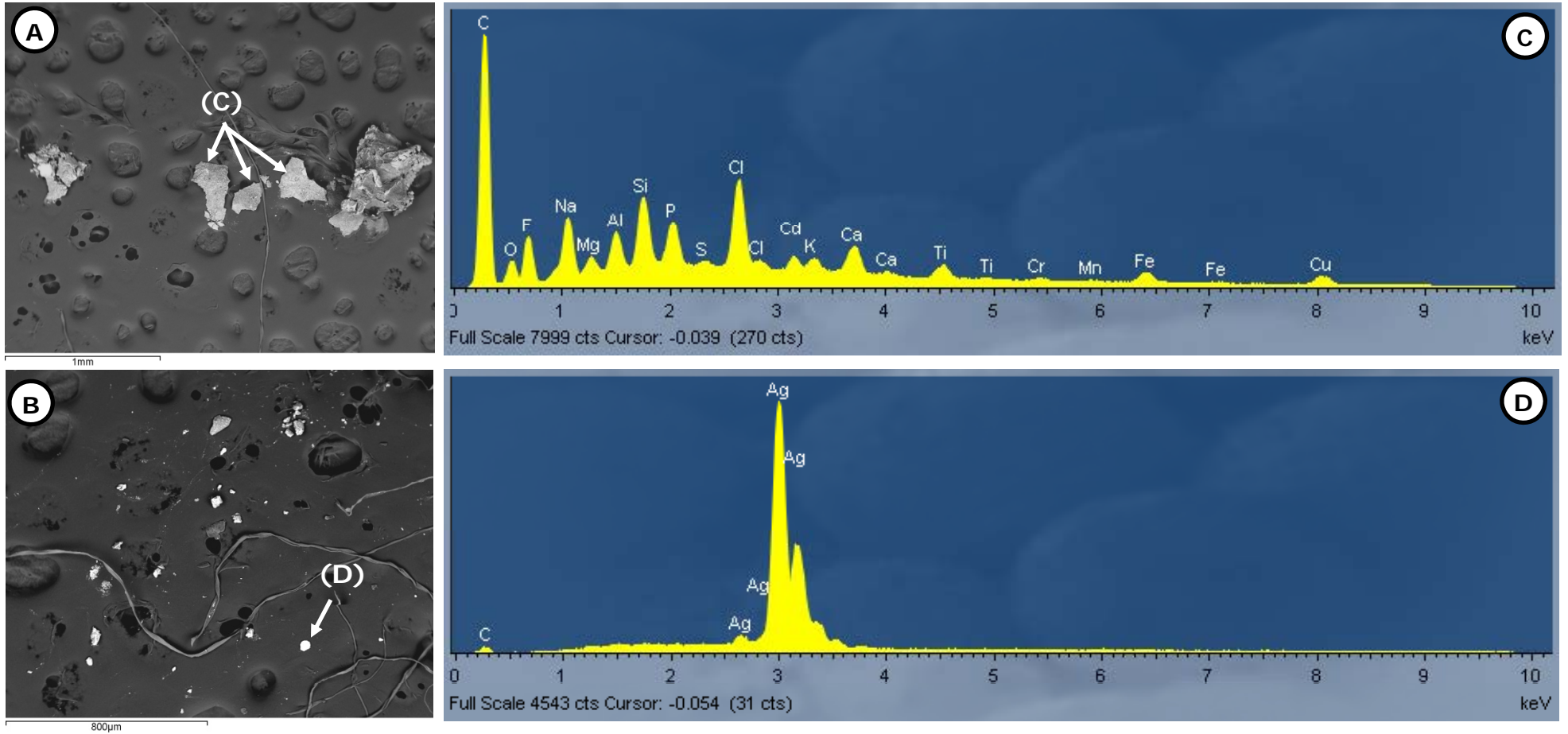




**Figure 22.**  
(A) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106026C.  
(B-E) EDX spectra of the debris indicated in (A).



**Figure 23.** (A) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106026C. (B-D) EDX spectra of the debris indicated in (A).



**Figure 24.**  
(A and B) BSE SEM photo showing some of the debris collected from the planet gears on ESN P106026C.  
(C and D) EDX spectra of the debris indicated in (A) and (B).

**Appendix A**  
**Gas Chromatography-Mass  
Spectroscopy (GC-MS) Report  
from Analyze Inc.**



## MATERIALS CHARACTERIZATION REPORT

**Report No.:** [REDACTED] **Date:** December 20, 2013

**Customer:** [REDACTED]  
Honeywell  
M/S 302-101  
PO Box 52181  
Phoenix, AZ 85072

**Customer P.O.:** [REDACTED]

**Sample:** Used Engine Oil CMR 664701

**Objective:** Compare the Used Engine Oil to MJII and BP2380 Reference Samples Using Gas Chromatography-Mass Spectroscopy (GC-MS)



## SUMMARY

1. The Used Engine Oil was diluted with methylene chloride at various concentrations and analyzed by GC-MS. The Used Engine Oil appeared to be completely soluble with no undissolved material remaining in the vials.
2. All of the volatile/semi-volatile compounds present in the BP 2380 turbine oil reference chromatogram were contained in the Used Engine Oil. The retention times, relative peak heights and mass spectra of the volatile compounds detected in the reference BP2380 chromatogram provide excellent matches with the majority of volatile and semi-volatile components detected in Used Engine Oil. The mass spectra of many components are consistent with synthetic esters based on C5 to C10 aliphatic carboxylic acids. Two aromatic amines (anti-oxidants) and tricresyl phosphate isomer admixture (boundary lubricant) were identified.

Low levels of C5-C10 carboxylic acids, 1H-benzotriazole, toluene, decane, undecane and methylphenol were also detected in the Used Engine Oil sample.

The presence of low levels of free carboxylic acids and methylphenol in Used Engine Oil but not in the reference BP2380 oil suggests that some hydrolysis of the ester fluid and the tricresyl phosphate boundary lubricant has occurred in the Used Engine Oil sample.

3. It was noted that methylene chloride blank runs after the 10X dilution of the Used Engine Oil sample contained a considerable amount of carryover of unknown high molecular weight components. It is possible high molecular weight contaminant(s) high boiling contaminants are present in the Used Engine Oil sample which cannot be properly analyzed by GC-MS using these conditions. If non-volatile components are present, liquid chromatography will need to be employed to determine the presence of these unknown component(s).

## INTRODUCTION

One sample, identified as Used Engine Oil CMR [REDACTED] was received from [REDACTED] on December 17<sup>th</sup>.

The objective of the analysis is to compare the Used Engine Oil to MJII and BP2380 reference samples using Gas Chromatography-Mass Spectroscopy (GC-MS).

## ANALYSIS

**Gas Chromatography-Mass Spectrometry (GC-MS).** GC-MS is a valuable analytical technique for the separation and identification of volatile organic compounds. The compounds are separated on the GC column and identified by matching their mass spectra to those of reference mass spectra contained in the 390,000 spectra Wiley digital library (7<sup>th</sup> Edition).

**Instrumentation.** The analysis was performed using a Varian CP-3800 Gas Chromatograph (GC) equipped with a Varian 1200L Quadrupole Mass Selective Detector and a CTC Analytics CombiPAL autosampling system. A Varian Factor Four 5-MS capillary GC column (30 m x 0.25 mm with 0.25  $\mu$ m film thickness) and deactivated glass wool split inlet liner were used for the chromatography under the following conditions:

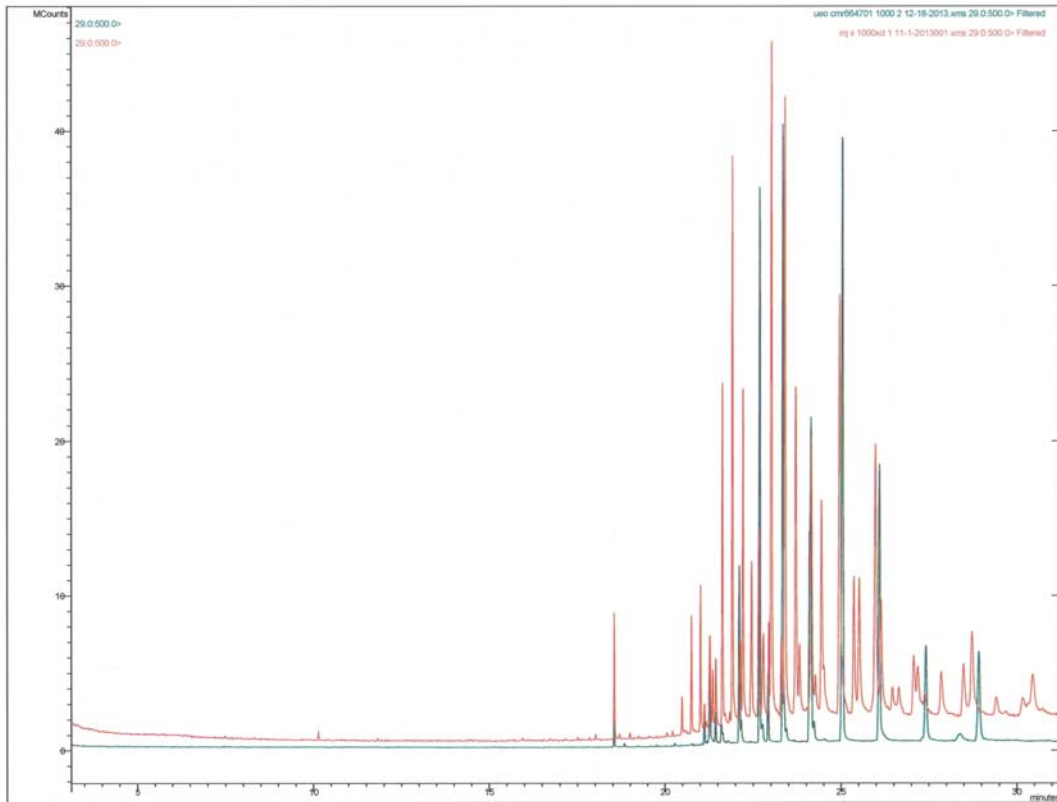
Injector Temperature:	275 °C	Split Ratio:	20:1
Transfer Line Temperature:	280 °C	Injection Volume:	1 ul
He Flow Rate:	1.0 ml/min		
Column Oven:	40 °C hold 4 min		
	ramp from 40 °C to 300 °C @ 15 °C/ min		
	300 °C hold 10 min		
MSD:	m/z 29 – 500		

**Sample Preparation.** Methylene chloride was chosen as solvent for the Used Engine Oil. Sample dilutions of ca. 10X, 100X, 1000X and 10,000X were analyzed. The Used Engine Oil sample was vortexed for 30 seconds prior to analysis. None of the samples were filtered prior to analysis.

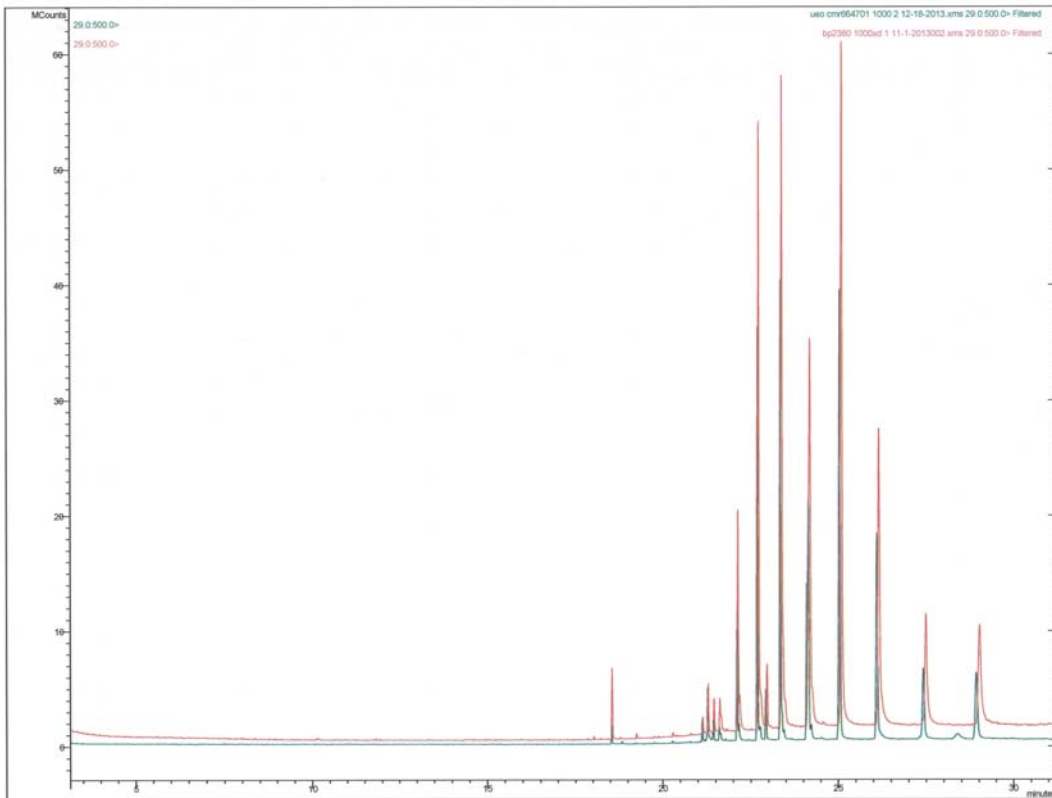
**Analyses.** The instrument was tuned prior to analysis. A blank solvent injection was made before each sample analysis to verify cleanliness of the system. All samples were run in duplicate at various dilutions. All tune files, chromatograms and spectral matches are collected in the Appendix.

The total ion chromatograms (TIC) of the Used Engine Oil with the MJII and BP2380 Reference Samples are shown in Figures 1-2.

**Figure 1 – TIC of MJ II and Used Engine Oil**



While a few of the major peaks are present in both the Used Engine Oil (green) and MJII (red), the majority of the peaks in the TIC of the MJII are different or not present in the Used Engine Oil suggesting MJII is not detectable in the Used Engine Oil.

**Figure 2 – TIC of BP2380 and Used Engine Oil**

With the exception of a few minor components, the BP2380 TIC shows excellent correlation with the Used Engine Oil and comprises the majority of volatile and semi-volatile compounds detected in the sample.

Table I list the retention times and component identities (where possible) of each of the samples analyzed.



**Table I – Reference and Used Engine Oil Component Identities**

Retention Time (min)	Component Identity	Used Engine Oil CMR 664701	BP2380	MJII
1.15	m/z 32, 45, <u>73</u> , 74, 103, 193, 267, 355			x
4.70	Toluene	x		
6.89	Pentanoic acid	x		
8.63	Decane	x		
9.53	Heptanoic acid	x		
9.62	Methylphenol	x		
9.85	Undecane	x		
10.59	Octanoic acid	x		
10.94	m/z 29, 41, 4357, 71, 85, 99	x		
11.91	m/z 29, 41, 57, 71, 85, 99	x		
12.47	Decanoic acid	x		
12.83	m/z 29, 43, 57, 71, 85, 99	x		
13.55	1H-Benzotriazole	x		
17.81	m/z 41, 57, 85, 113, 156, 172, 231	x		
18.57	N-Phenylnaphthalenamine (or similar anti-oxidant, chelator)	x	x	x
19.23	m/z 41, 92, 194, 210, 281	x		
19.75	m/z 43, 85, 113, 131, 198	x		
20.26	m/z 29, 43, 69, 85, 98, 113, 127, 145, 198, 229	x		
20.50	m/z 41, 57, <u>85</u> , 156			x
20.75	m/z 41, 57, <u>85</u> , 156			x
20.79	m/z 29, 43, 57, 99, 127, 155, 212, 271, 329, 436	x		
21.01	m/z 41, 57, <u>85</u> , 156			x
21.13	m/z 32, <u>65</u> , 91, 165, 179, 208, 243, 271, 341, 368, 431	x	x	
21.31	Tris(3-methylphenyl) phosphate (o, m, p isomers)	x	x	x
21.36	m/z 41, 57, <u>85</u> , 113, 156			x
21.48	Tris(3-methylphenyl) phosphate (o, m, p isomers)	x	x	x
21.63	m/z 43, 57, <u>85</u> , 113, 156			x
21.63	m/z 29, 43, 57, 85, 99, <u>113</u> , 368	x	x	
21.77	m/z 29, 43, 98, 127, 155, 173, 212, 243, 271	x		
21.91	m/z 43, 57, <u>85</u> , 113, 156			x
22.14	m/z 29, 57, <u>85</u> , 127, 156			x
22.16	m/z 43, 57, 85, <u>113</u> , 341	x	x	
22.21	m/z 43, 57, <u>85</u> , 113, 156			x
22.46	m/z 41, 57, <u>85</u> , 127, 156			x
22.67	m/z 43, 57, <u>85</u> , 113, 156			x
22.74	m/z 29, 43, 57, 68, 85, <u>113</u> , 127, 198, 229, 355	x	x	
22.79	m/z 29, 43, 57, <u>85</u> , 127, 156			x
22.99	4-Octyl-N-(4-octylphenyl)benzeneamine (anti-oxidant, chelator)	x	x	x
23.02	m/z 29, 43, 57, <u>85</u> , 113, 156, 184			x
23.29	m/z 43, 57, 85, 113, 156			x
23.40	m/z 43, 57, <u>85</u> , 113, 156, 184			x
23.42	m/z 29, 43, <u>57</u> , 113, <u>127</u> , 212, 243, 355	x	x	
23.71	m/z 43, 57, <u>85</u> , 113, 156			x
23.81	m/z 41, 57, <u>85</u> , 155			x
24.15	m/z 29, 43, 57, <u>85</u> , 113, 127, 156			x
24.23	m/z 29, 43, 57, 85, <u>113</u> , 127, 155, 200, 229, 271, 341	x	x	
24.44	m/z 29, 43, 57, <u>85</u> , 113, 156, 184, 315			x
24.95	m/z 29, 43, 57, 85, 113, 156, 184, 315			x
25.16	m/z 29, <u>43</u> , 57, 71, 85, 98, 113, 127, 155, 200, 229, 271, 355, 383	x	x	
25.38	m/z 43, <u>57</u> , 85, 113, 127, 156, 184, 315			x
25.54	m/z 29, 43, 57, <u>85</u> , 113, 155			x
25.99	m/z 29, 43, 57, <u>85</u> , 113, 127, 16, 184, 315			x
26.15	m/z 29, 43, 57, <u>85</u> , 113, 155			x
26.22	m/z 29, 43, 57, 98, <u>127</u> , 155, 200, 243, 369, 397	x	x	
27.08	m/z 32, 43, 85, <u>113</u> , 184, 343			x
27.19	m/z 43, <u>57</u> , 85, 113, 127, 184			x
27.55	m/z 29, 43, 57, 85, 99, 113, <u>155</u> , 200, 229, 271, 383	x	x	
27.86	m/z 43, 57, <u>85</u> , 113, 155, 184, 315			x
28.50	m/z 43, 57, <u>85</u> , 113, 343			x
28.67	Biphenyl-4,4'-dicarboxylic acid, bis-(4-pentyl-phenyl ester)	x		
28.74	m/z 57, <u>85</u> , 113, 155, 184, 315			x
29.09	m/z 29, 43, 57, 71, 81, 99, 127, <u>155</u> , 200, 243, 271, 397	x	x	
30.45	m/z 43, 57, <u>85</u> , 113, 155			x

\*Pink denotes common components with Used Engine Oil

The compounds contained in the BP2380 constitute the vast majority of the volatile and semi-volatile material detected in the Used Engine Oil. The main ions of most mass spectra acquired of eluting compounds for both the BP2380 and the Used Engine Oil are 43, 57, 113, 127 and 155 m/z. The relative ion abundances and molecular ions vary depending upon the retention times of the eluting compounds. These mass spectra are consistent synthetic esters based on C5 to C10 aliphatic carboxylic acids.

Two aromatic amines (anti-oxidants) and tricresyl phosphate isomer admixture (boundary lubricant) were identified.

Low levels of C5-C10 carboxylic acids, 1H-benzotriazole, toluene, decane, undecane and methylphenol were also detected in the Used Engine Oil sample. The presence of low levels of free carboxylic acids and methylphenol in Used Engine Oil but not in the reference BP2380 oil suggests that some hydrolysis of the ester fluid and the tricresylphosphate has occurred.

As questions arise during your review of this report, please do not hesitate to call us.

[REDACTED]

[REDACTED]

[REDACTED]

# **GC-MS APPENDIX**

Tune Report FC-43 EI (Tuned on 18 Dec 2013) (Reported on 18 Dec 2013 09-45).log

Hardware diagnostics report for Varian Quadrupole Mass Spec

Time: 9:45:26

No errors detected. Proceeding with autotune.

-----  
Tune report for Varian Quadrupole Mass Spec

Time: 9:57:04 Tune file: FC-43 EI (Tuned on 18 Dec 2013)

Ionization mode:EI

Scan Optimization: Standard GC

(EDR)

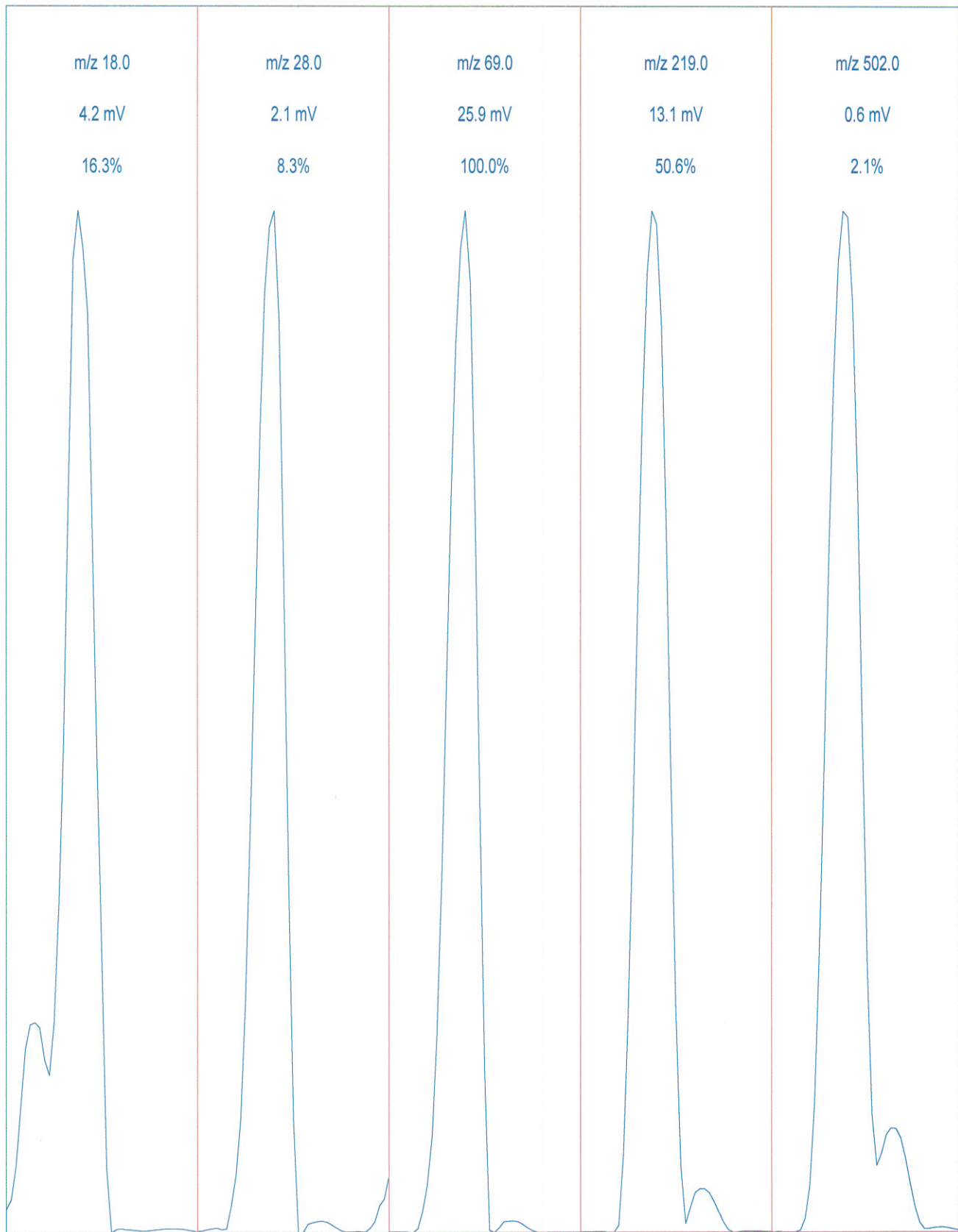
Exact mass (amu)	Measured mass (amu)	Peak height (mV)	Relative height (%)	Peak width (amu @ 50%)	Valley (% of iso)	Resolution (m/delta-m)
18.0	18.0	4.210	16.272	0.67	100	27
28.0	28.0	2.140	8.272	0.70	100	40
69.0	69.0	25.871	100.000	0.69	100	100
219.0	219.0	13.089	50.592	0.73	80	301
502.0	502.0	0.552	2.134	0.77	36	649

(Detector = 1781.765 V)

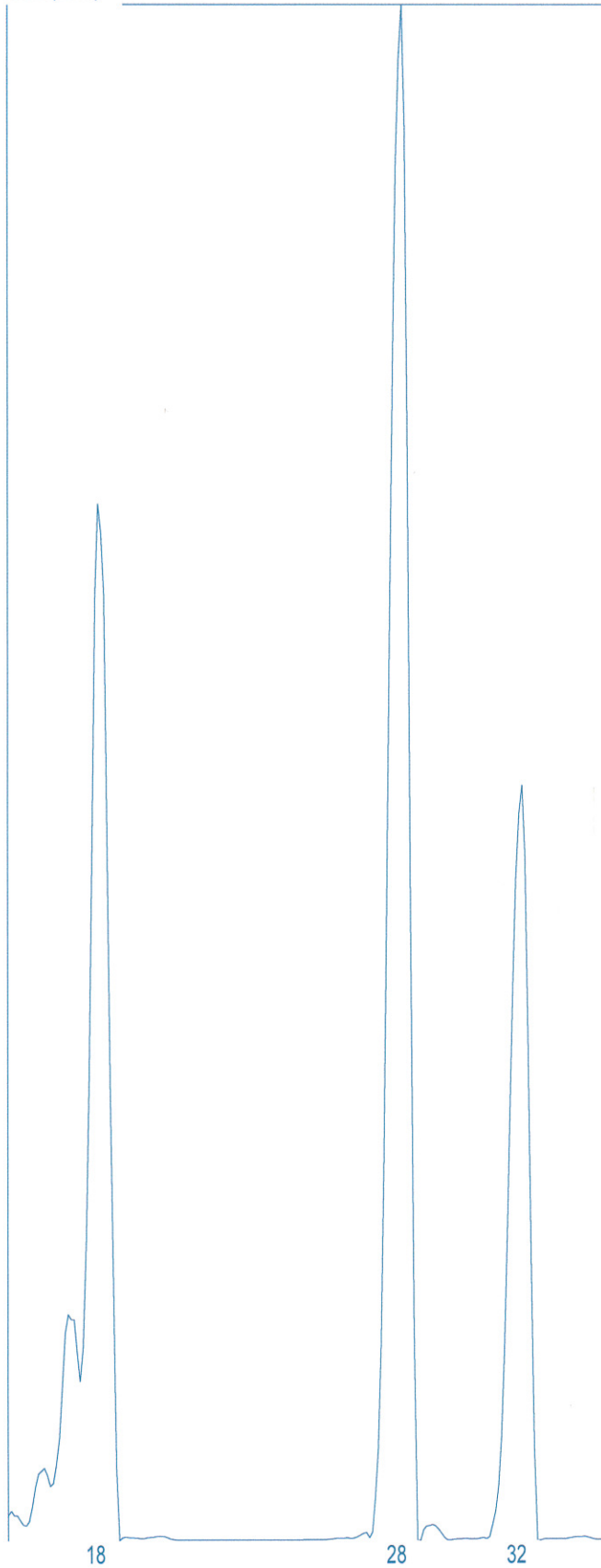
Tuning of quad 1 in positive mode completed.

\*\*\*\*\*  
\*\*\*\*\*





1.9mV full scale (EDR)



28/18 Ratio

- Limit 1.2:1 - Measured 1.5 :1
- Ratio too high!

28/32 Ratio

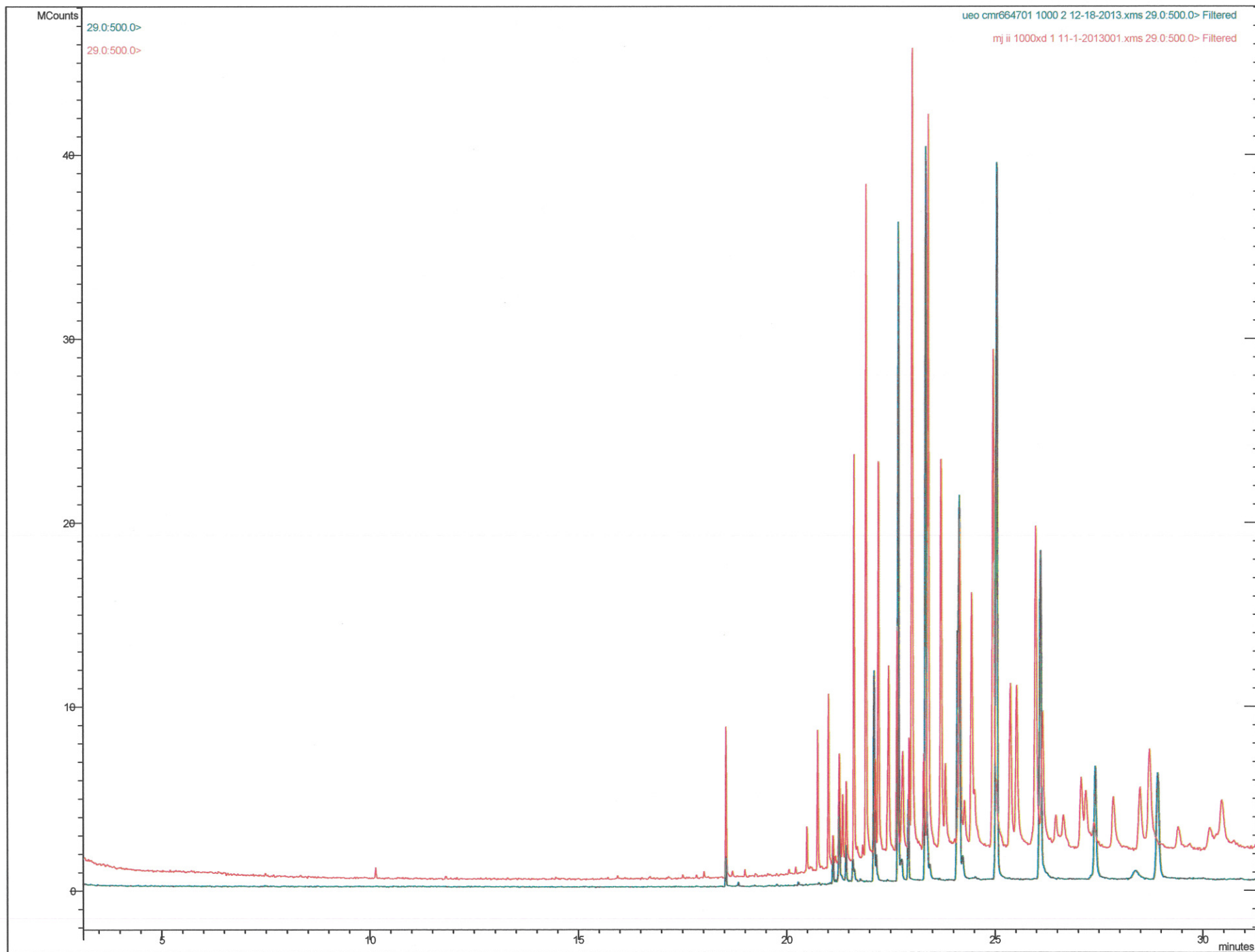
- Air 3.5:1 - Measured 2.1:1
- OK! - Not indicative of Air

28 absolute size (mV - EDR on)

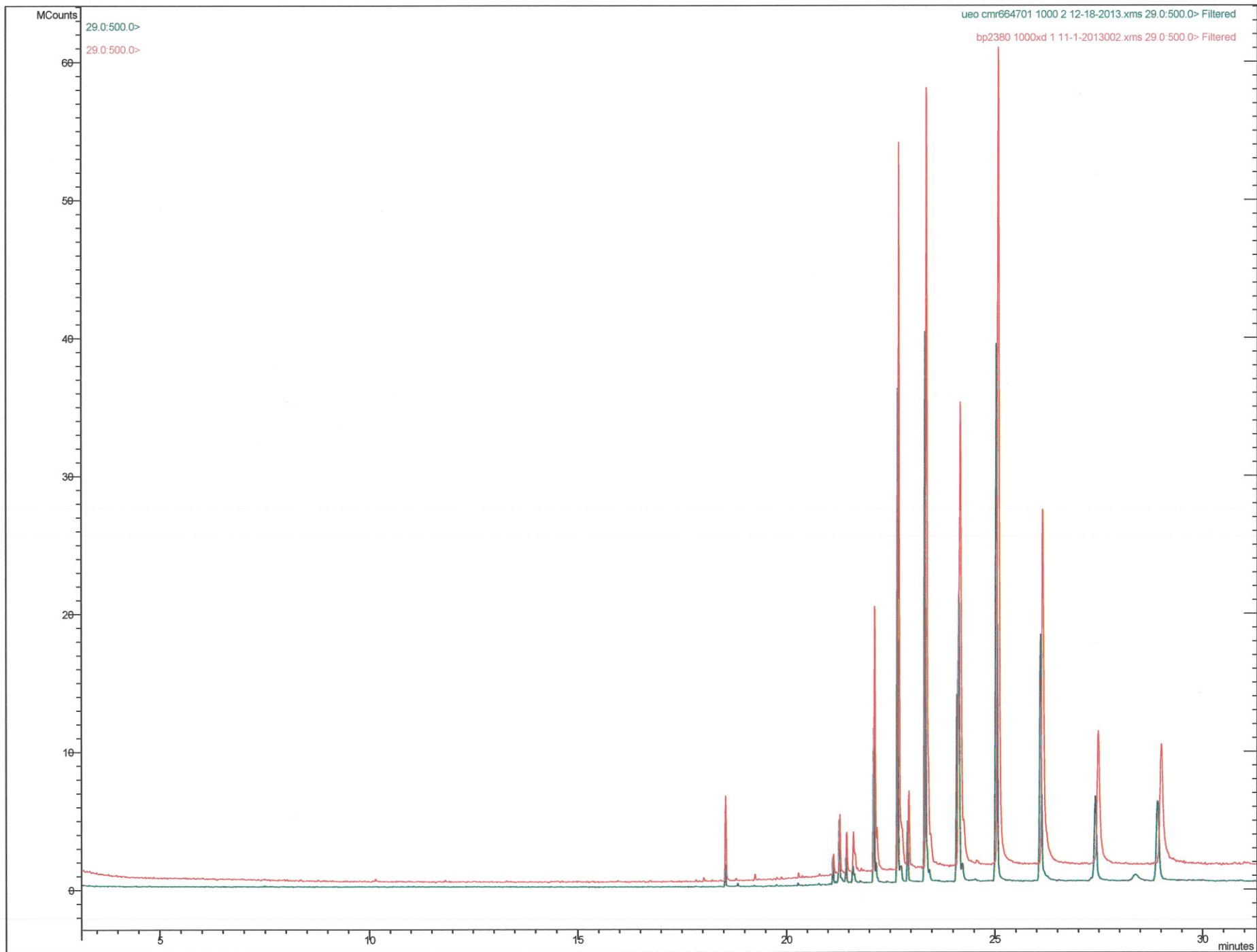
- Limit 20.0 - Measured 1.9
- OK!

- No significant leak detected

Overlaid Chromatogram Plots



### Overlaid Chromatogram Plots





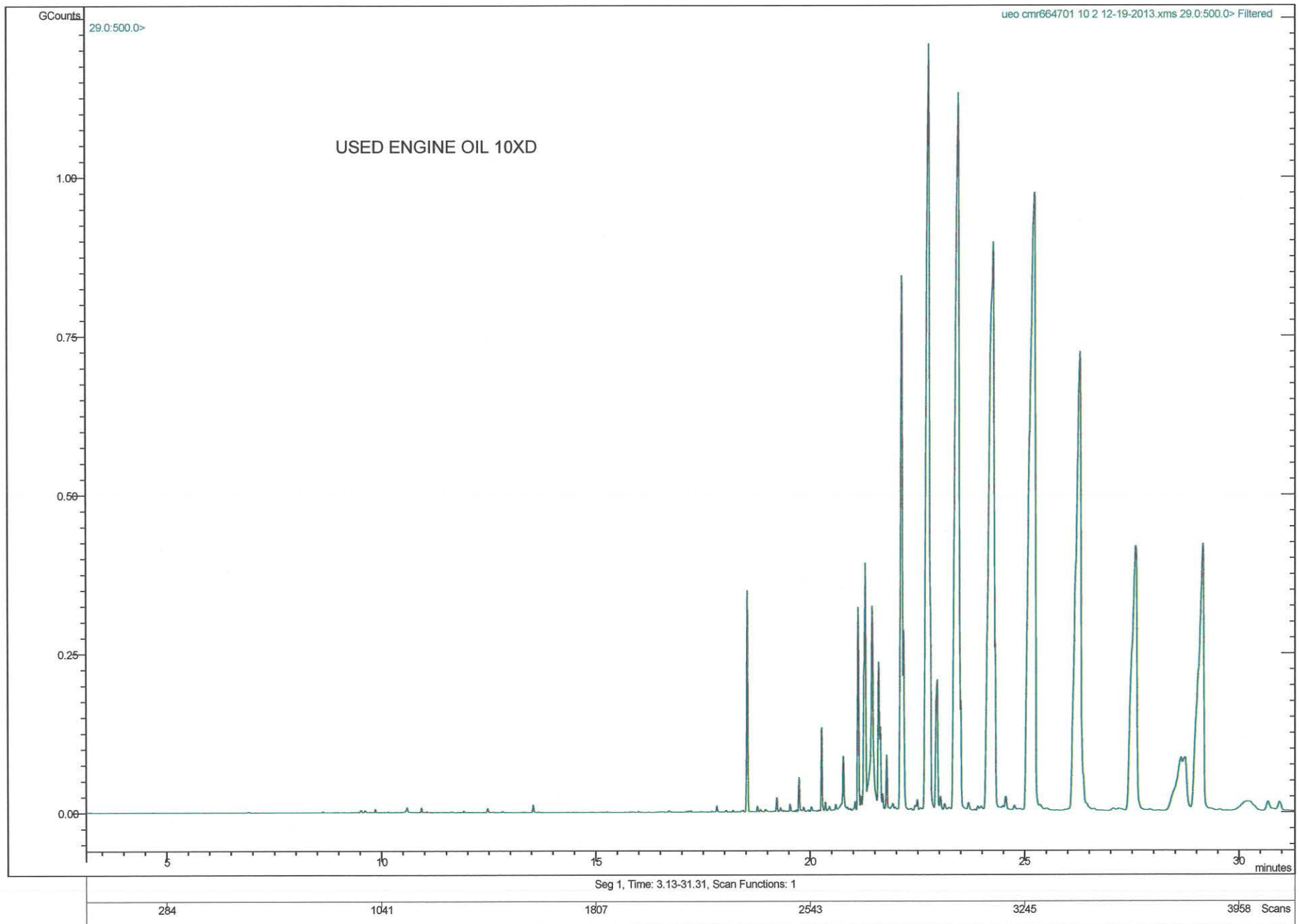
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Sample: [REDACTED]  
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Operator: CG

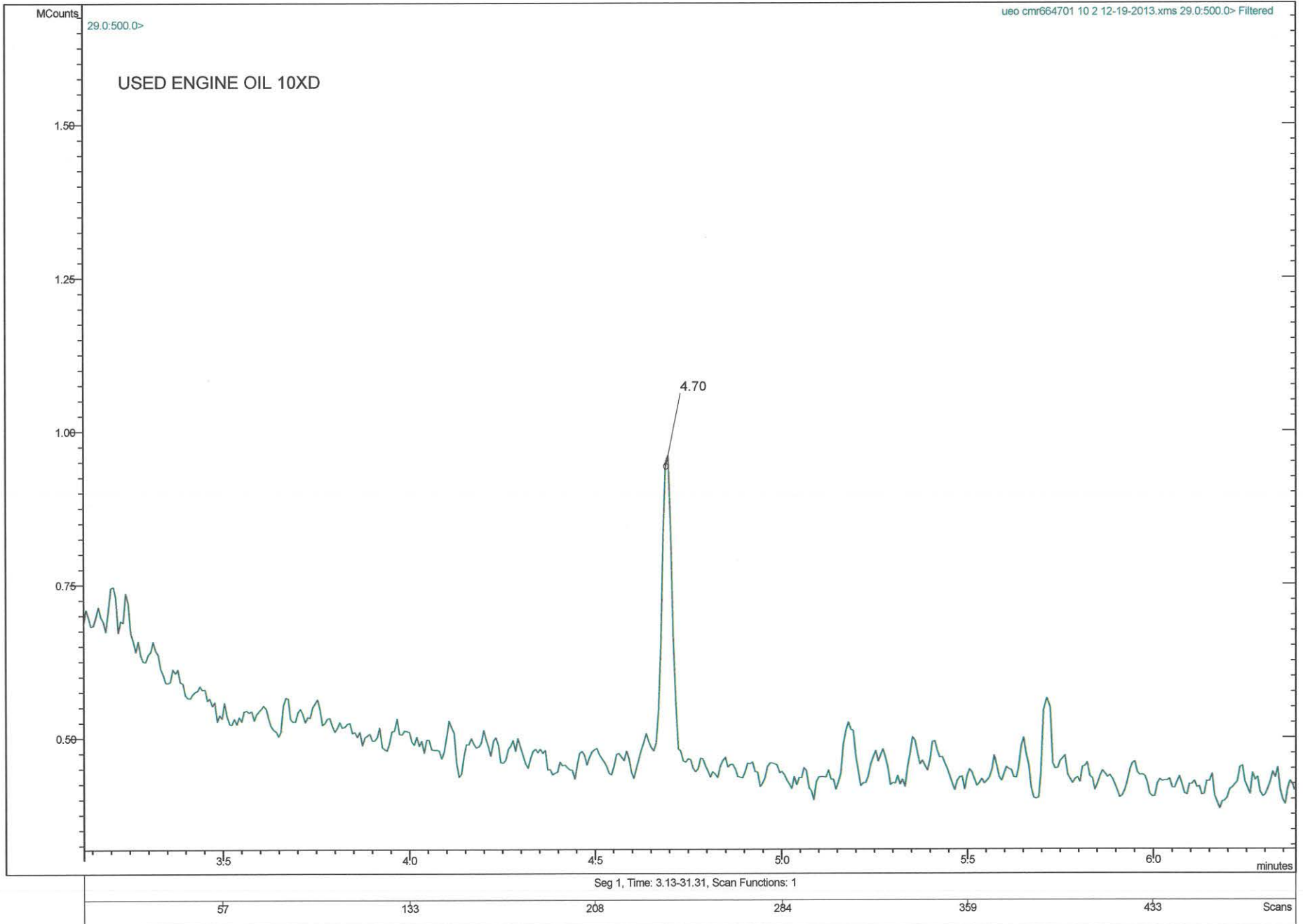
Date: 12/19/2013 5:21 PM



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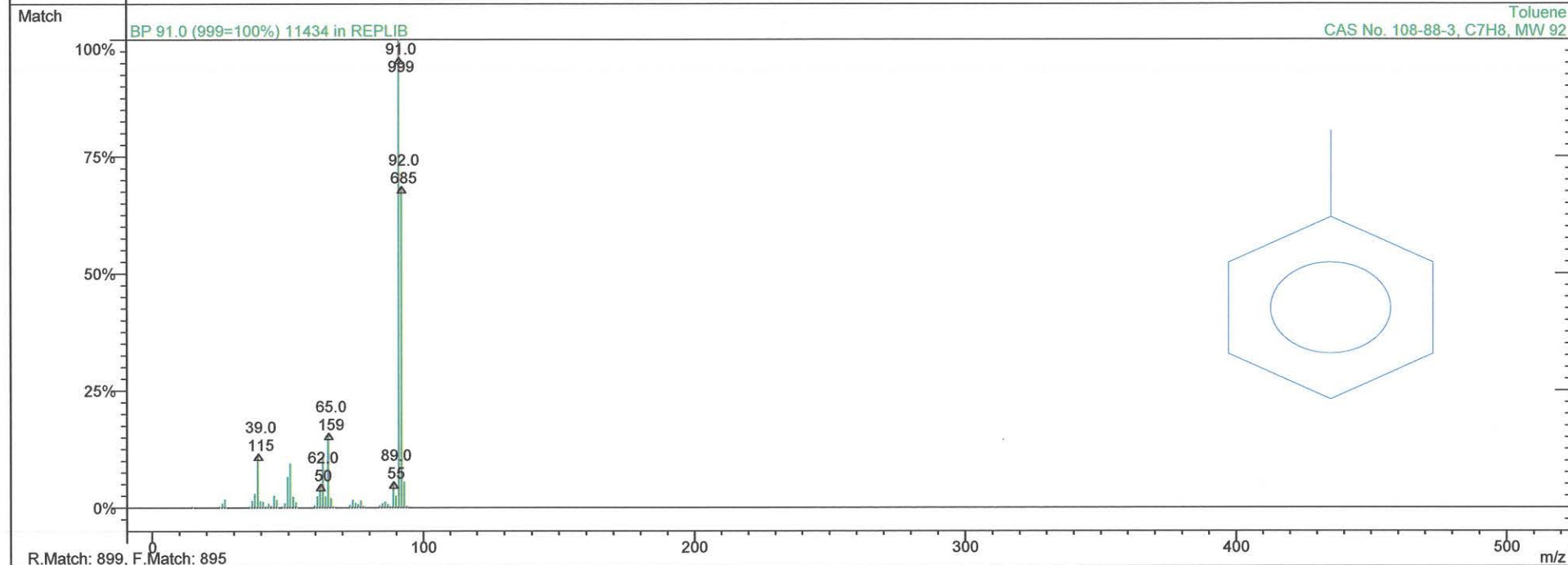
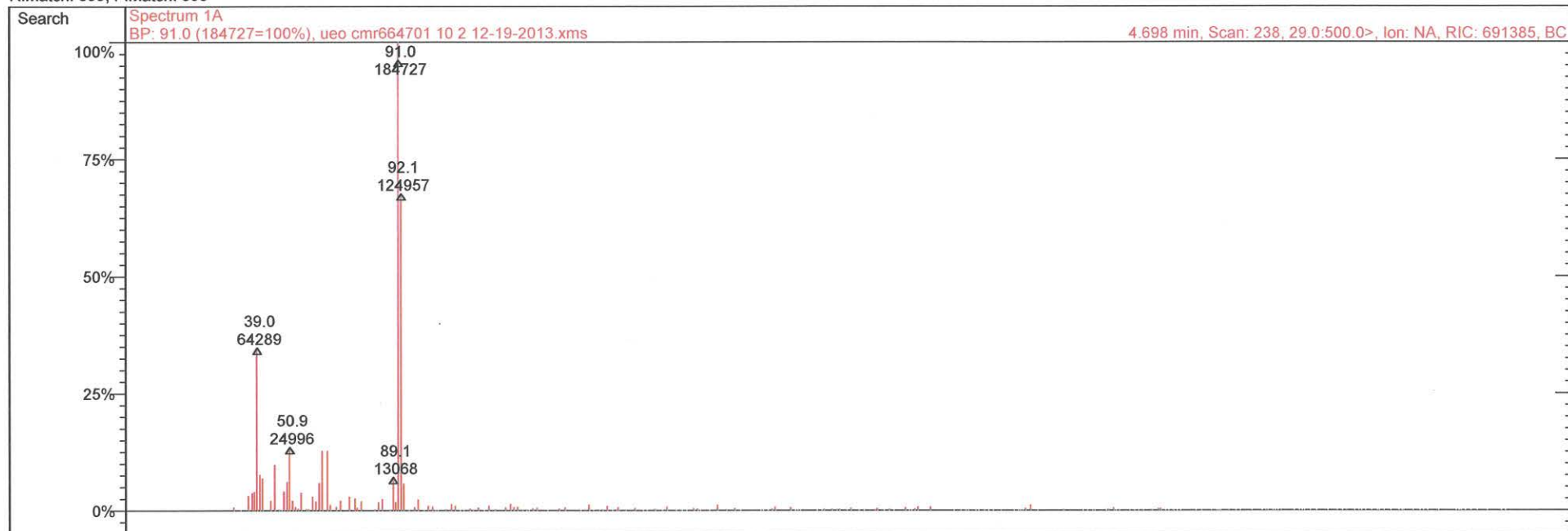
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Date: 12/19/2013 5:21 PM



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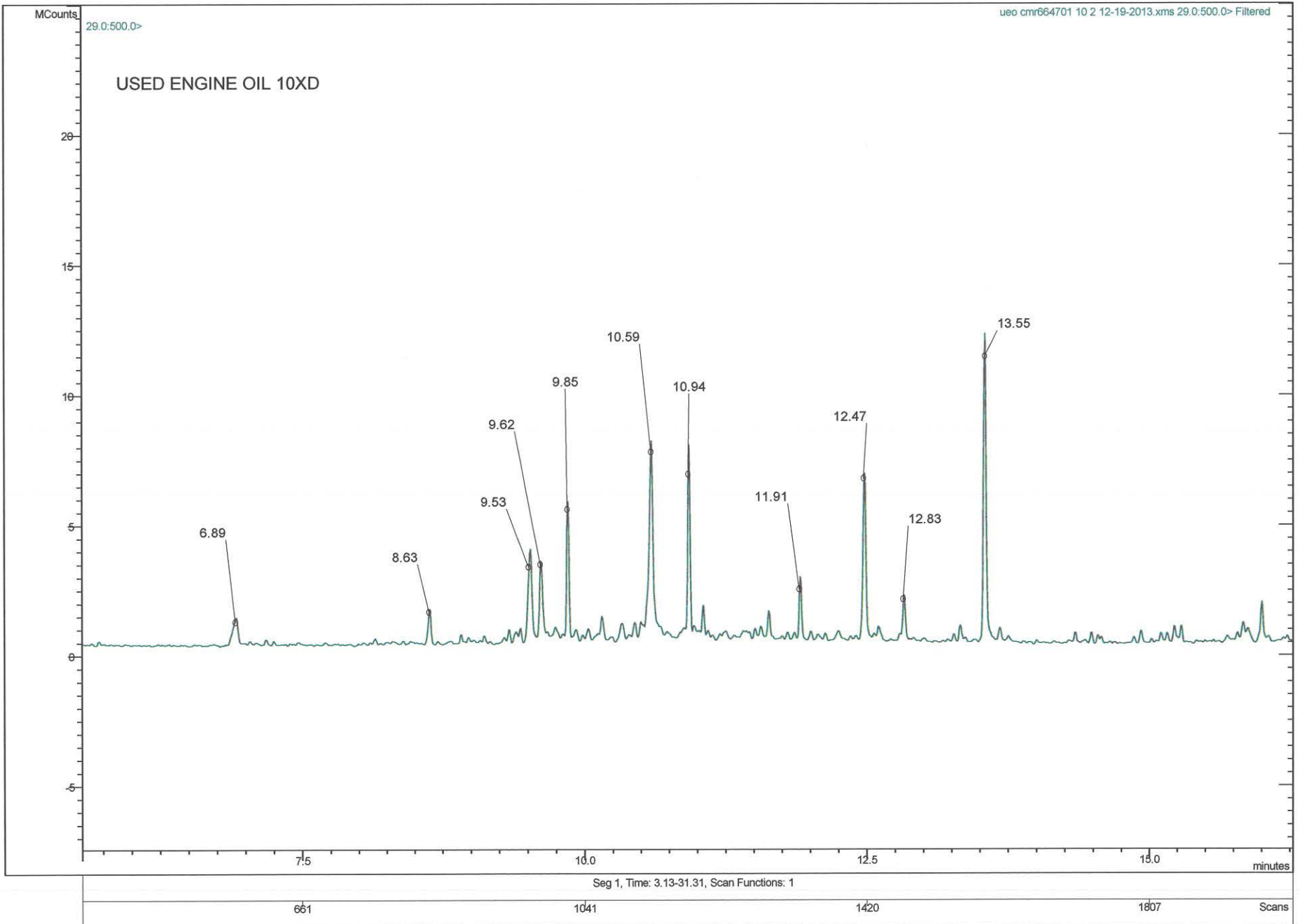
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Sam [REDACTED]  
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Operator: CG  
Date: 12/19/2013 5:21 PM

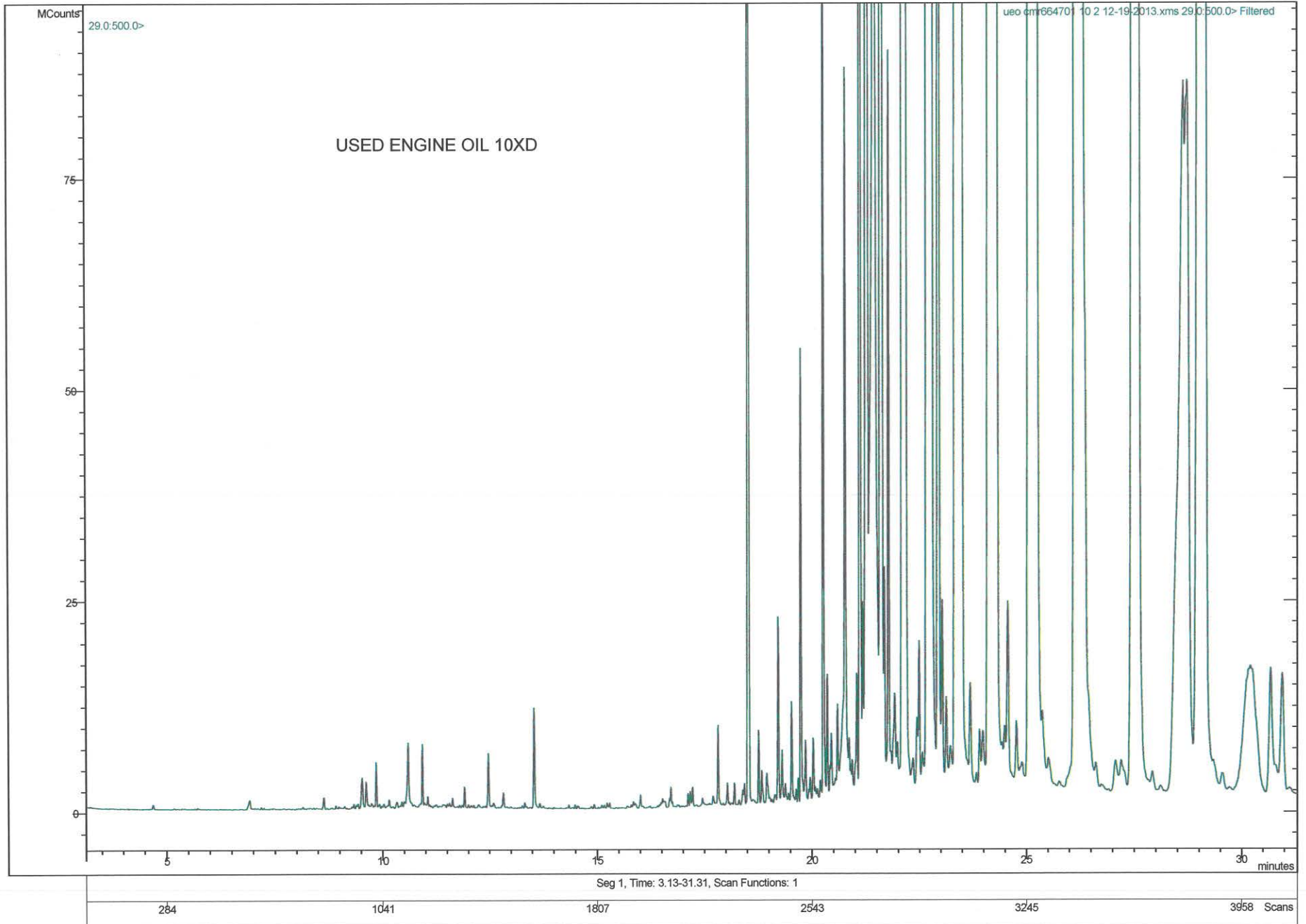




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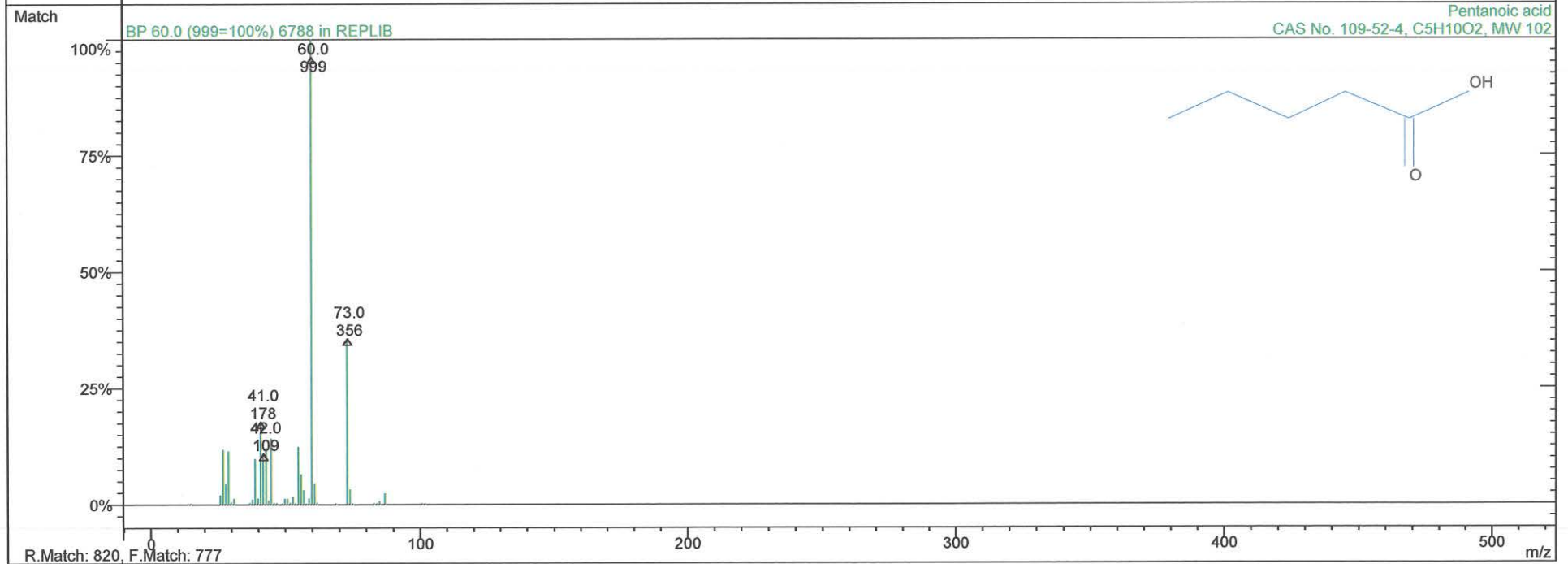
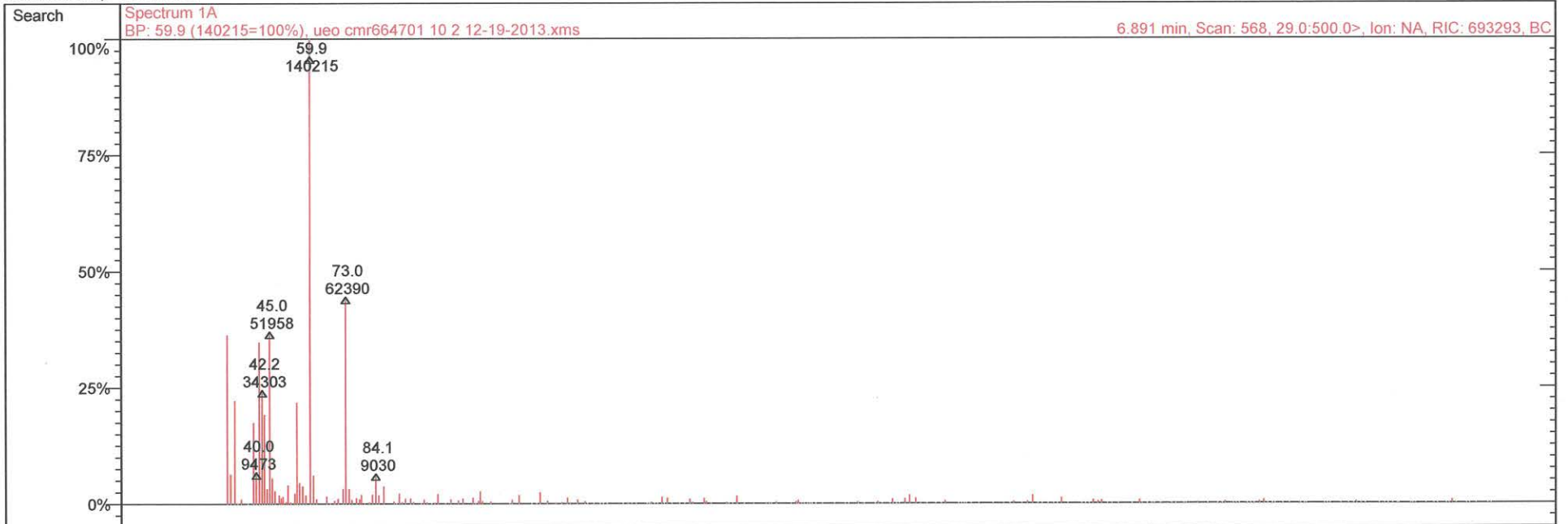
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Sam [REDACTED]  
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Operator: CG  
Date: 12/19/2013 5:21 PM



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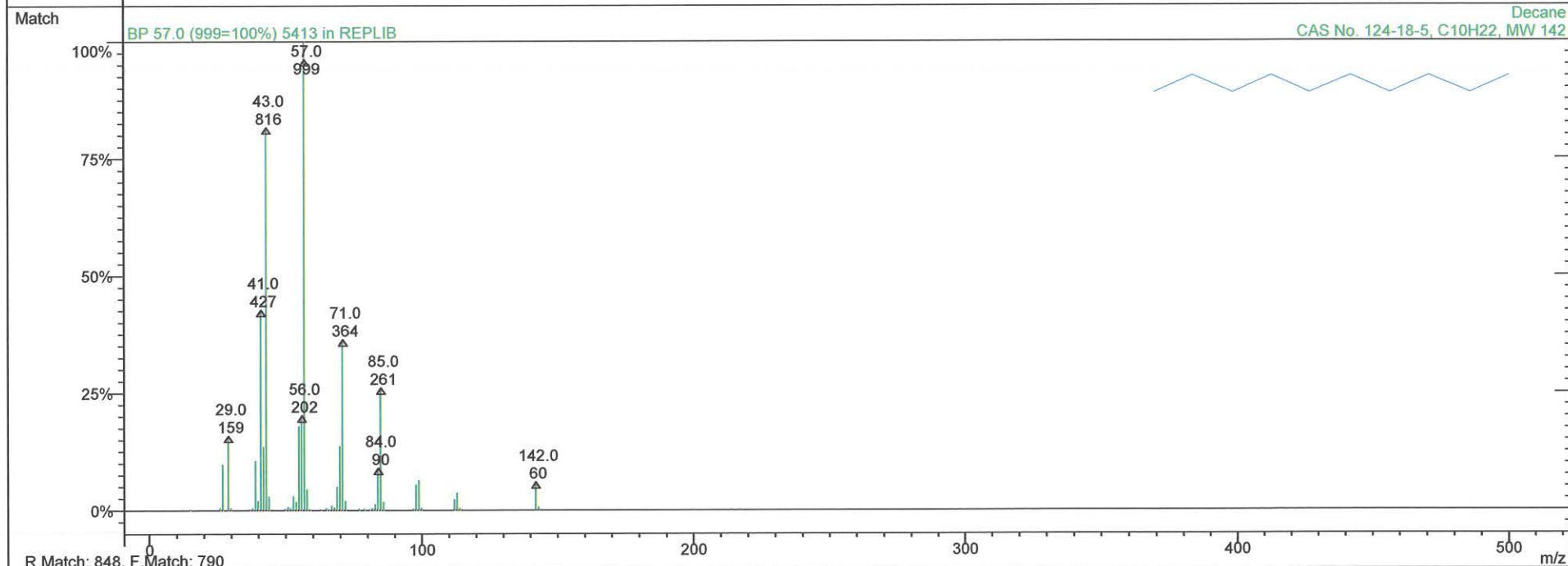
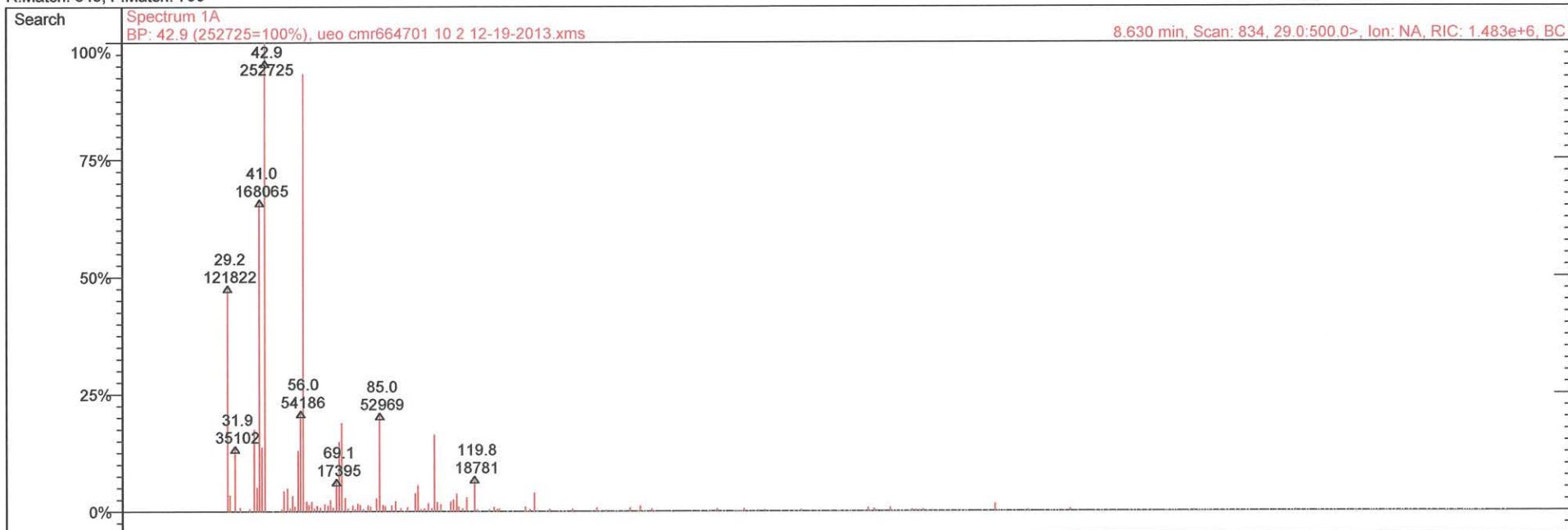
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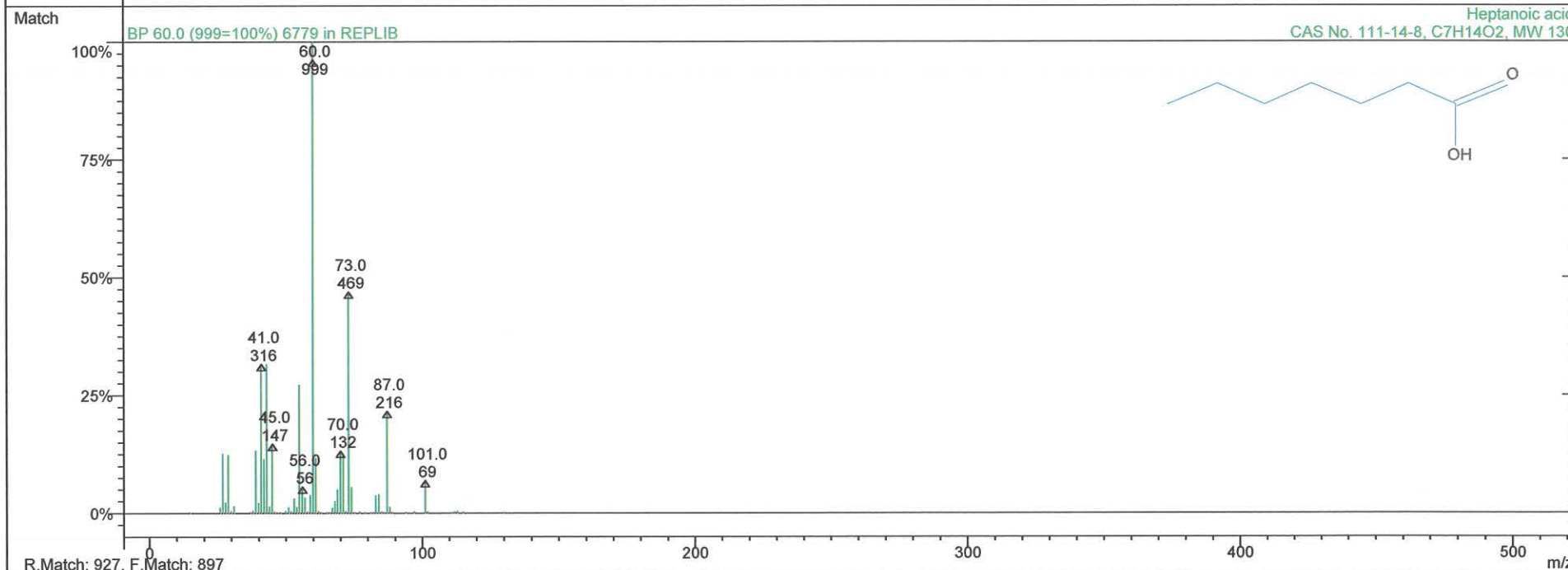
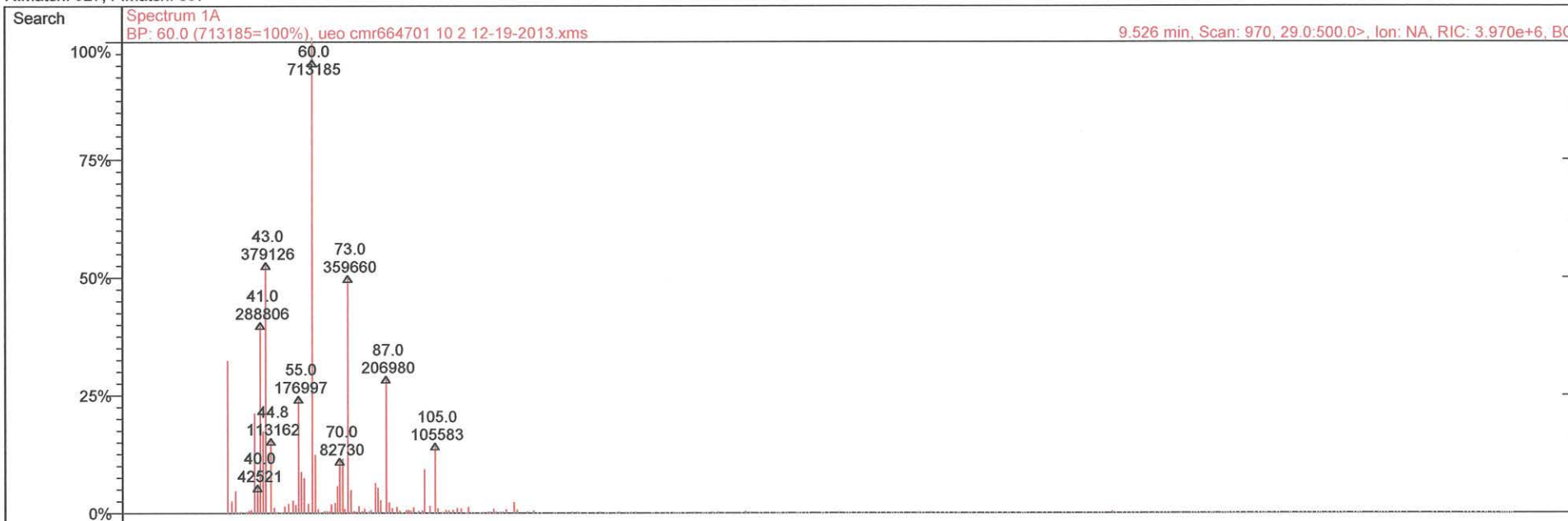
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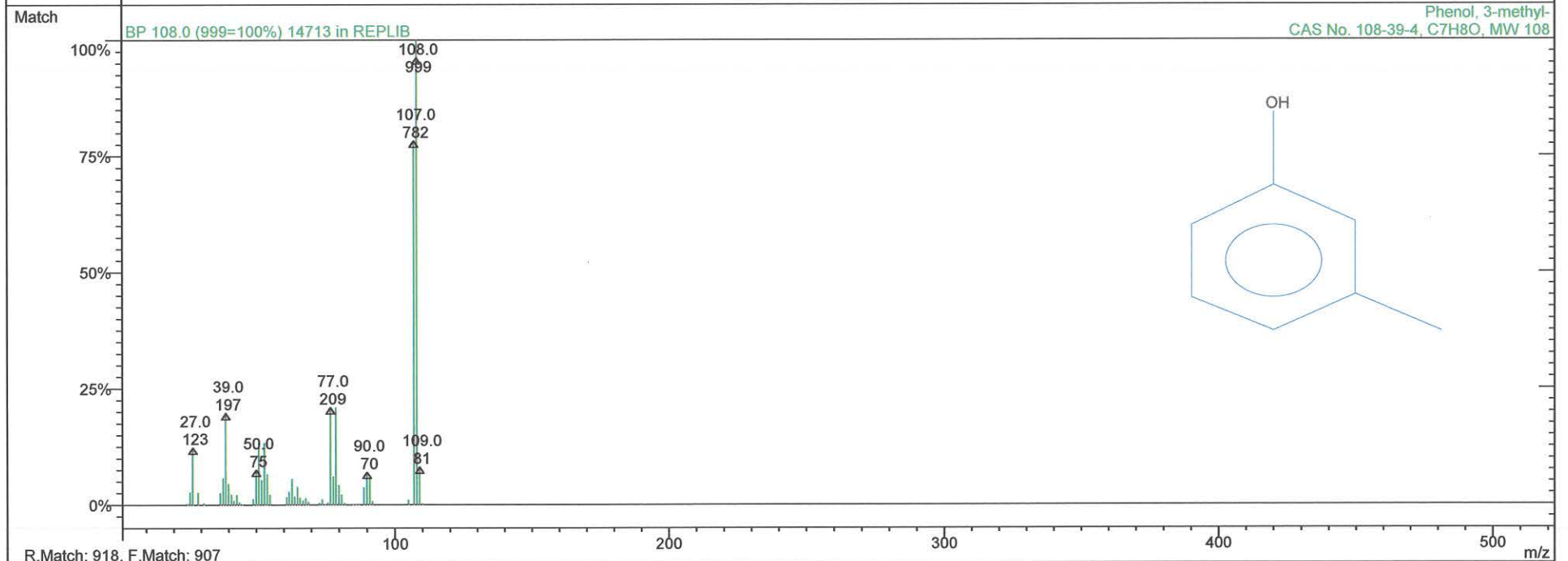
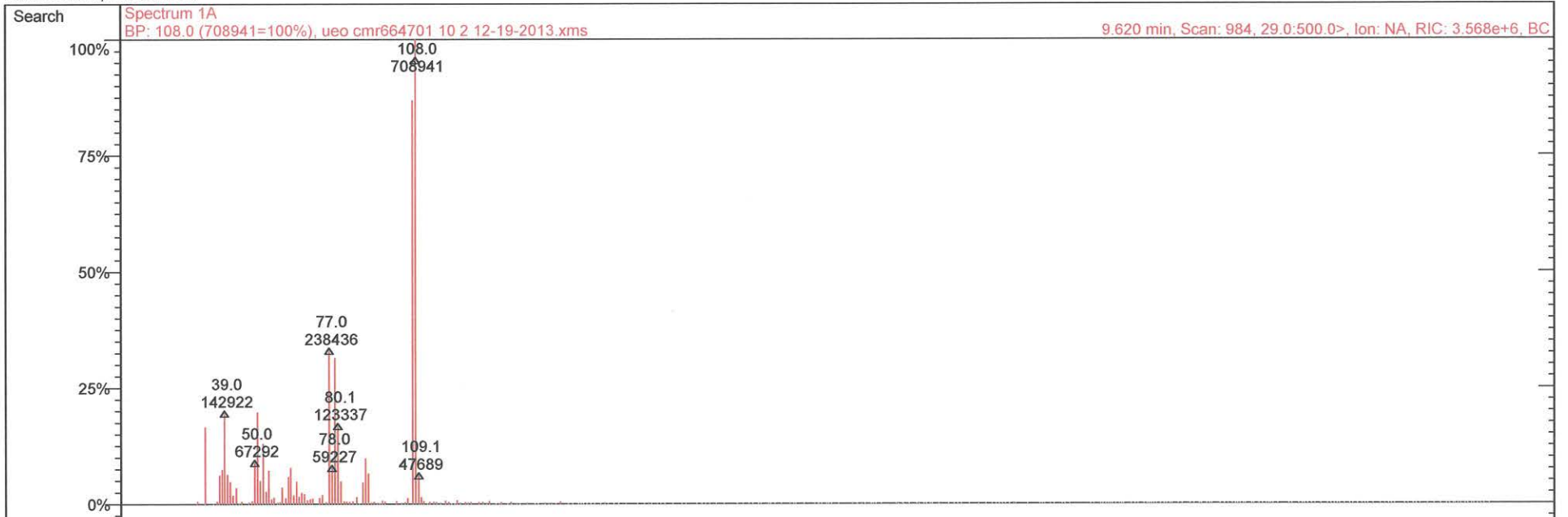




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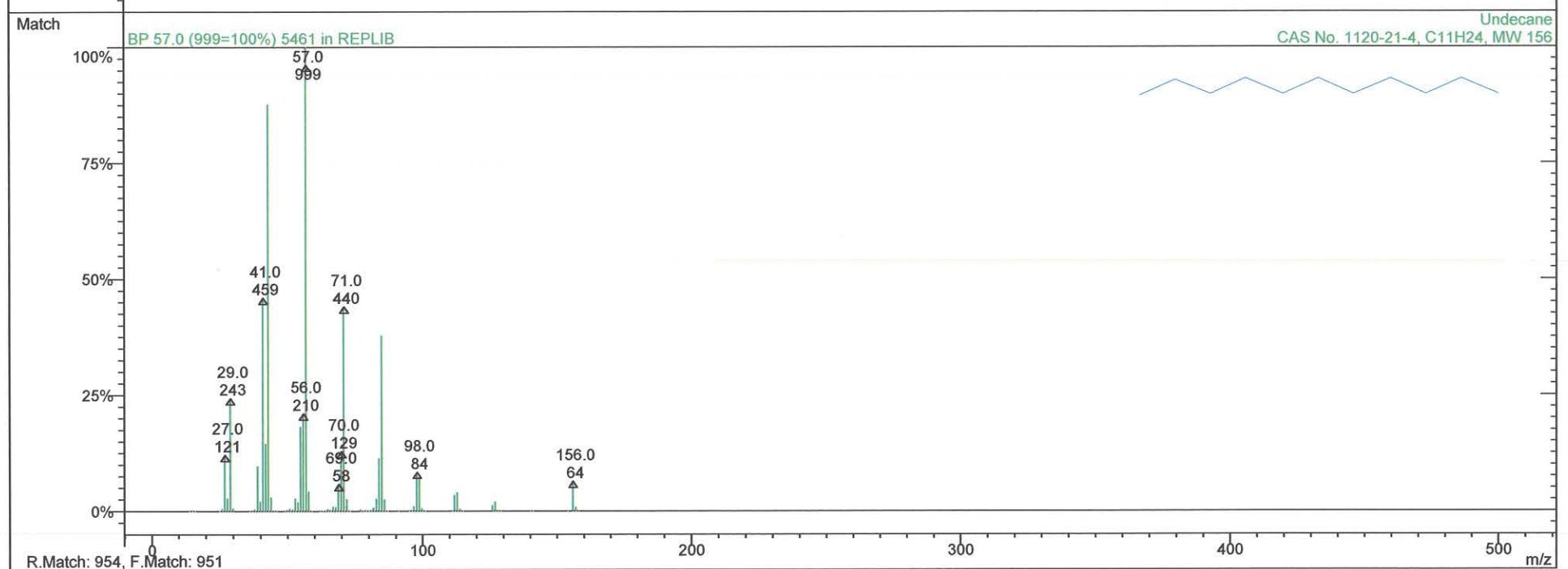
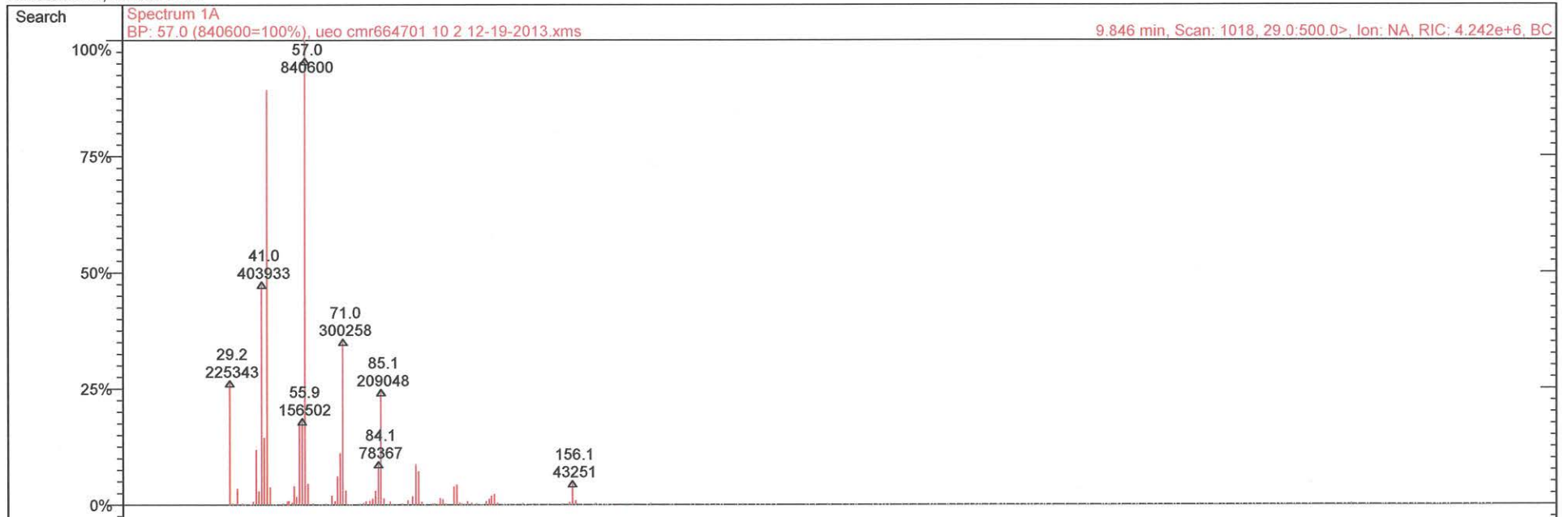
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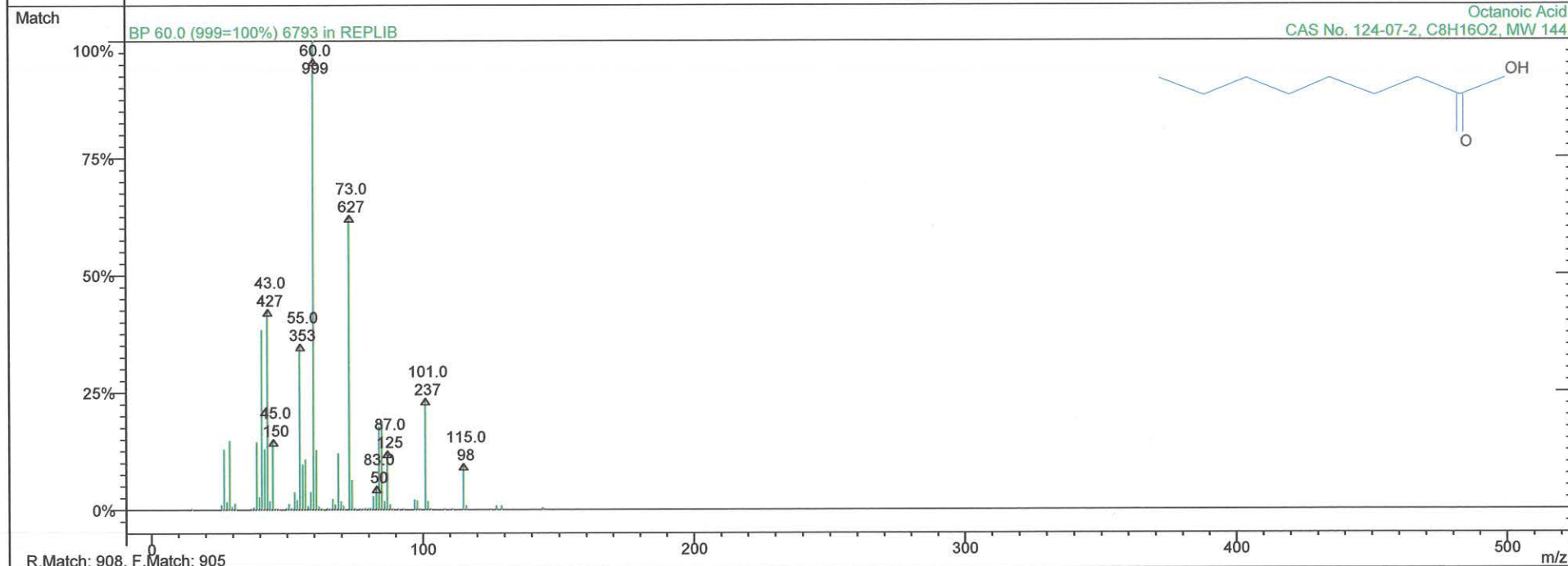
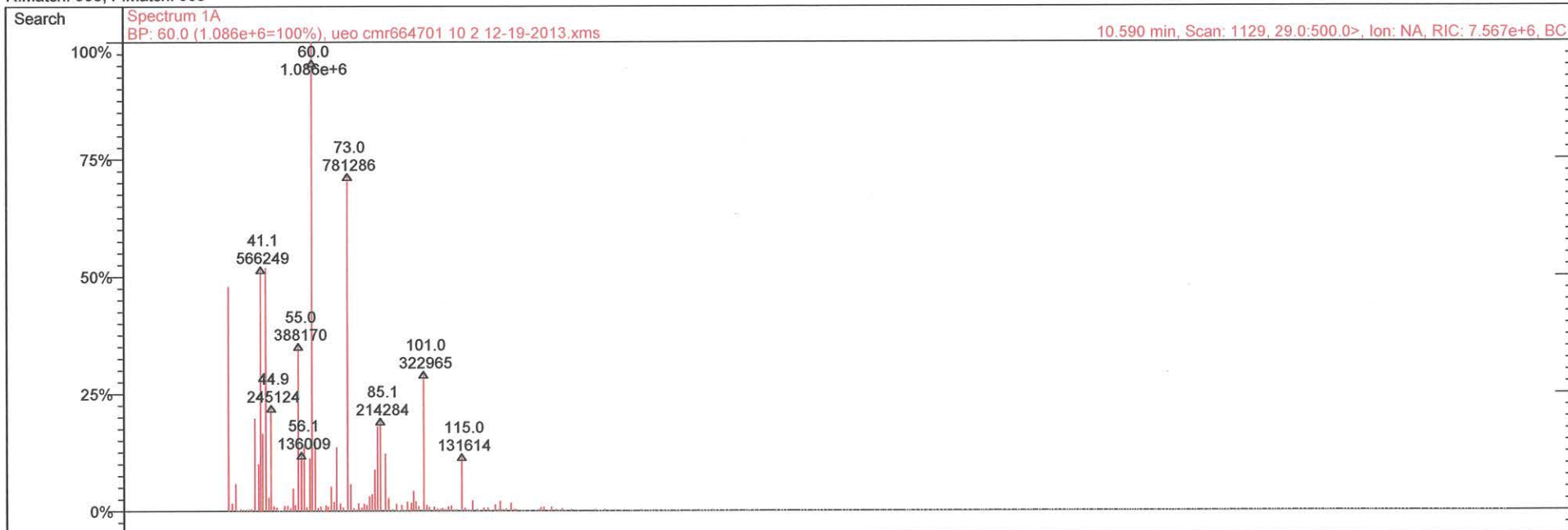


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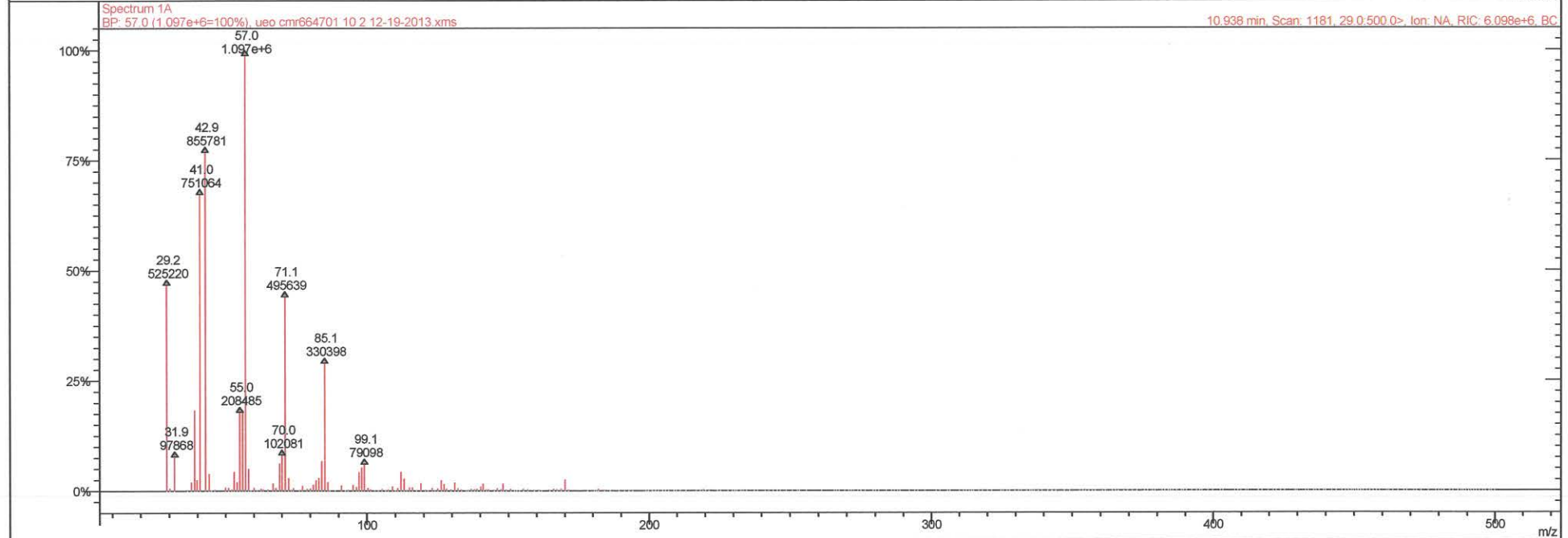
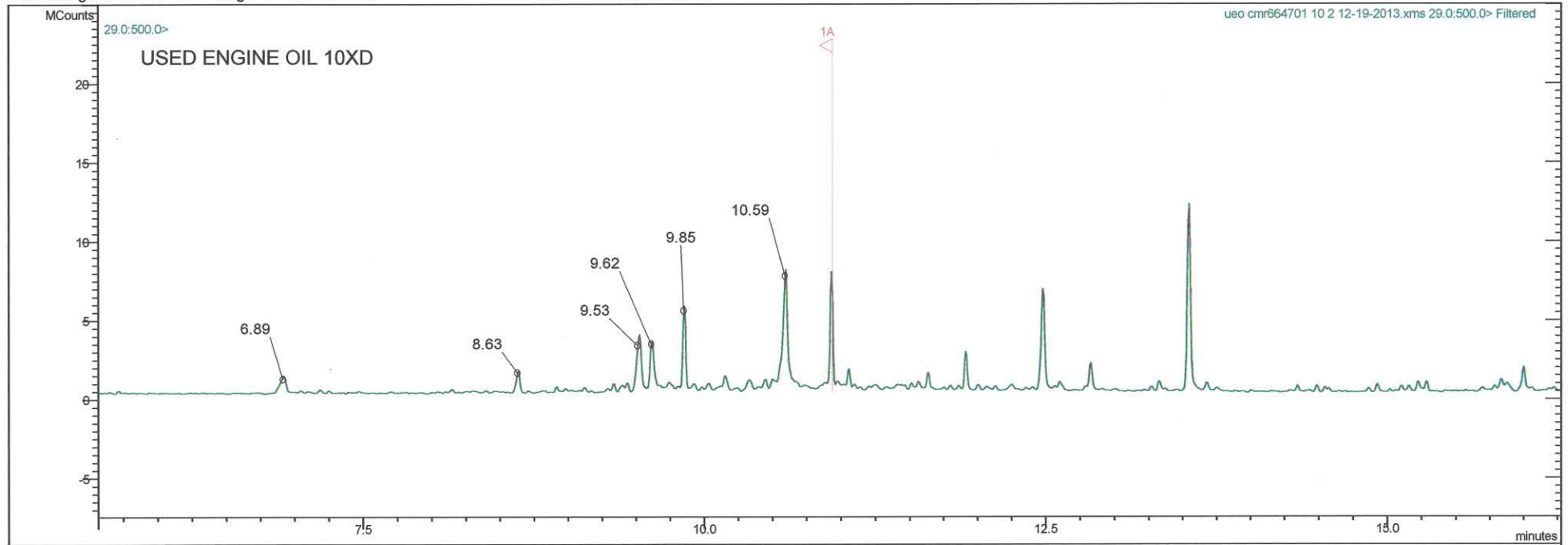


### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:30 AM

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Sam [REDACTED]

Operator: CG  
Date: 12/19/2013 5:21 PM

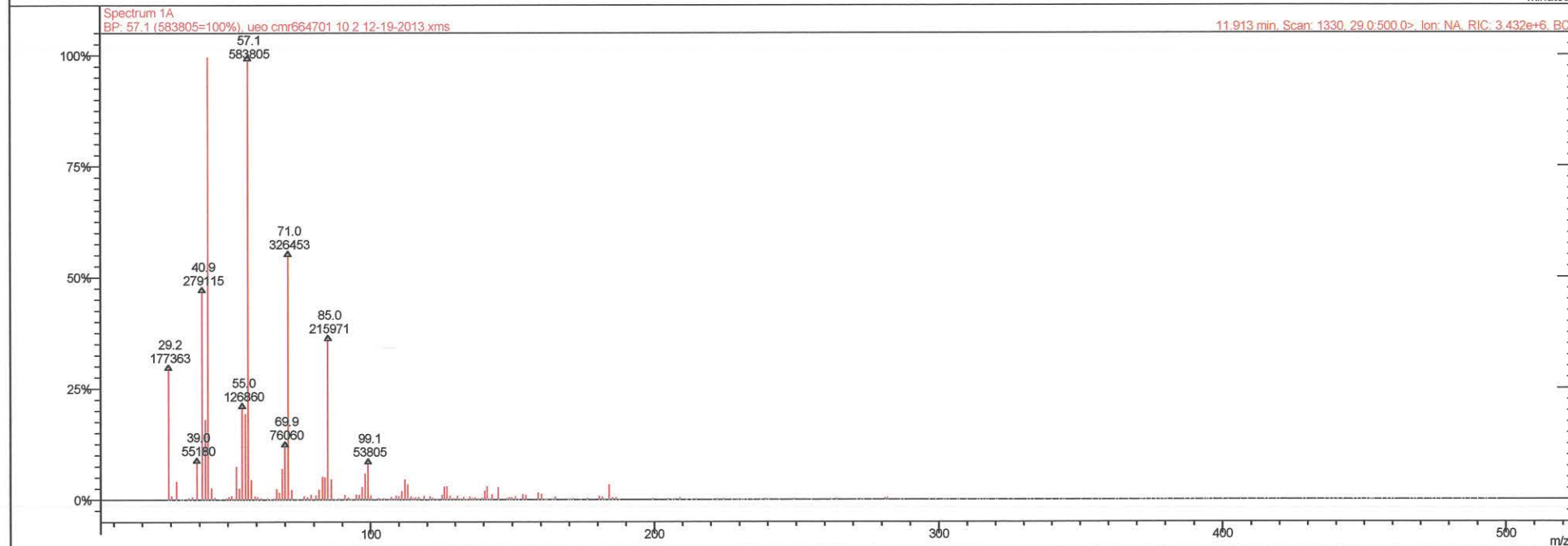
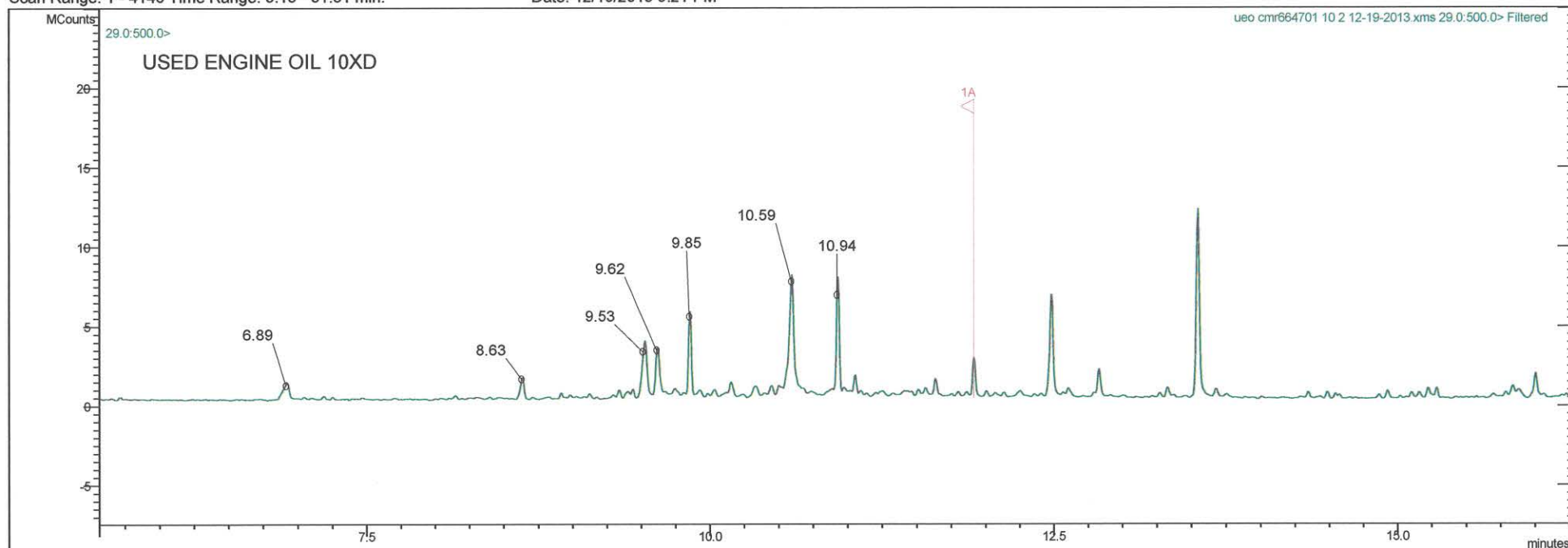
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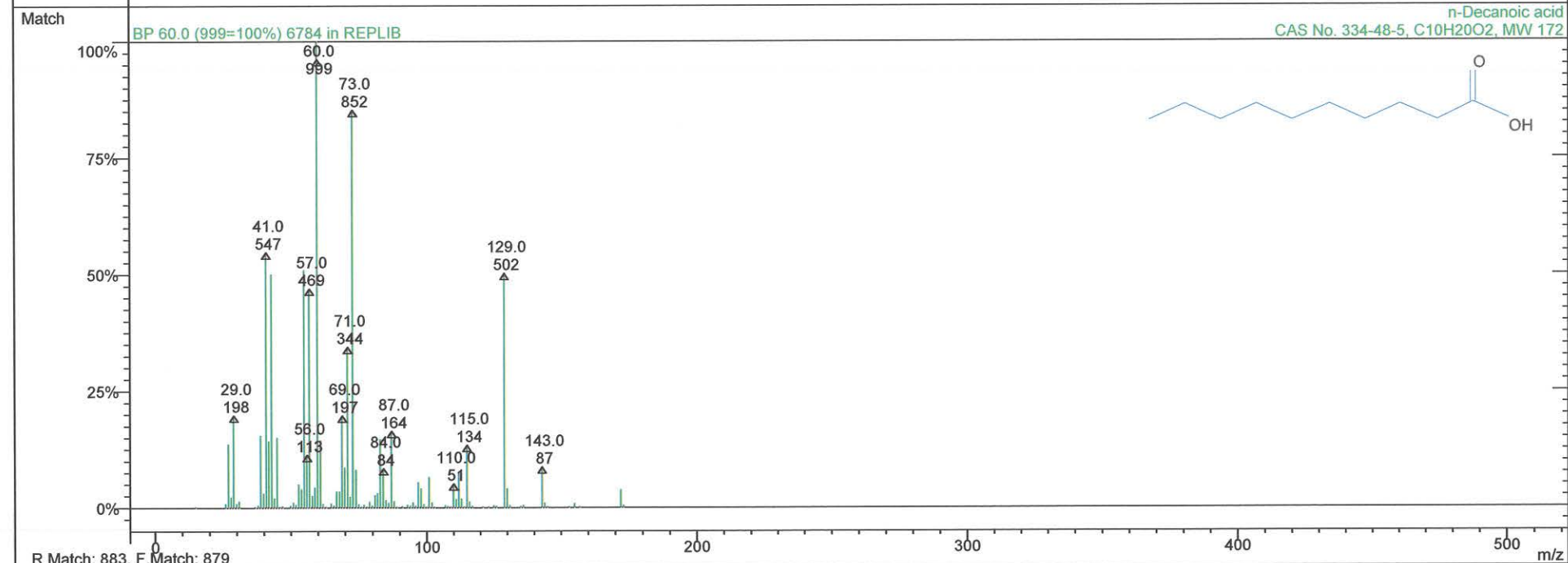
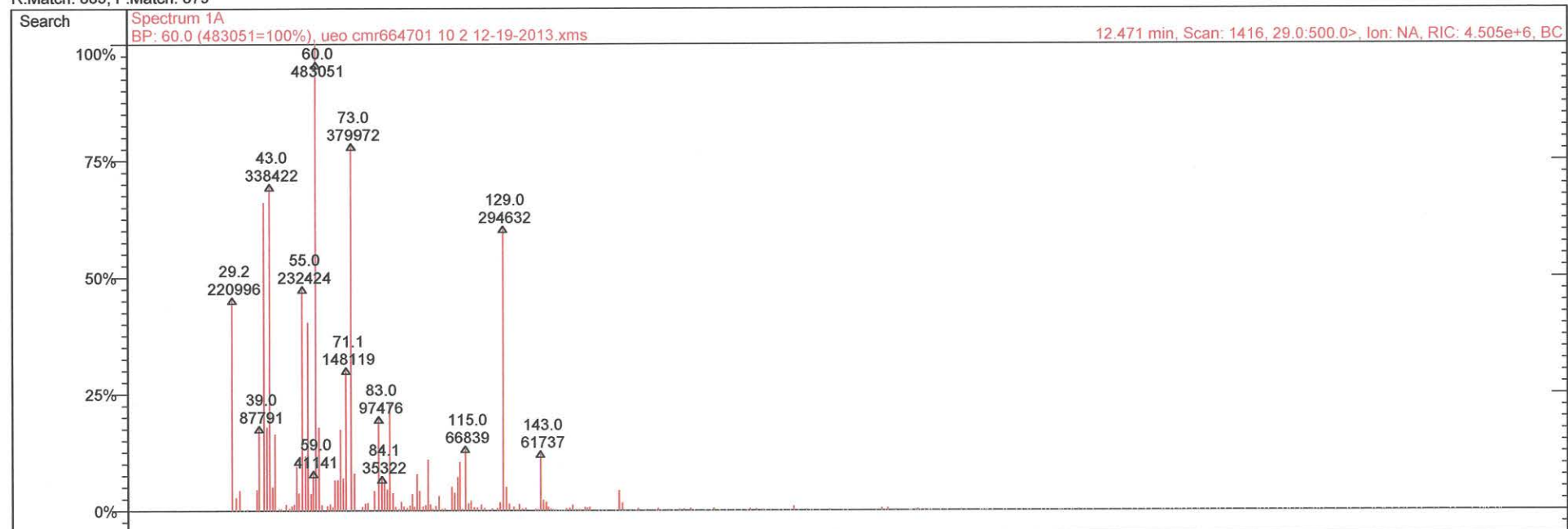
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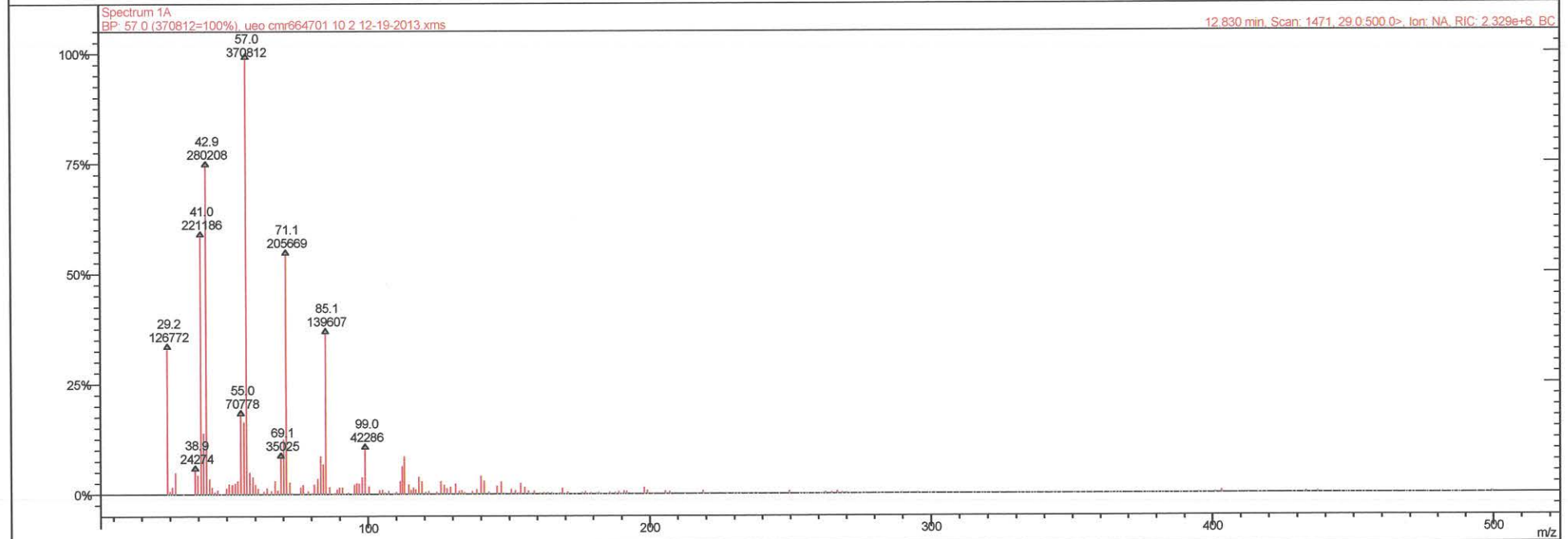
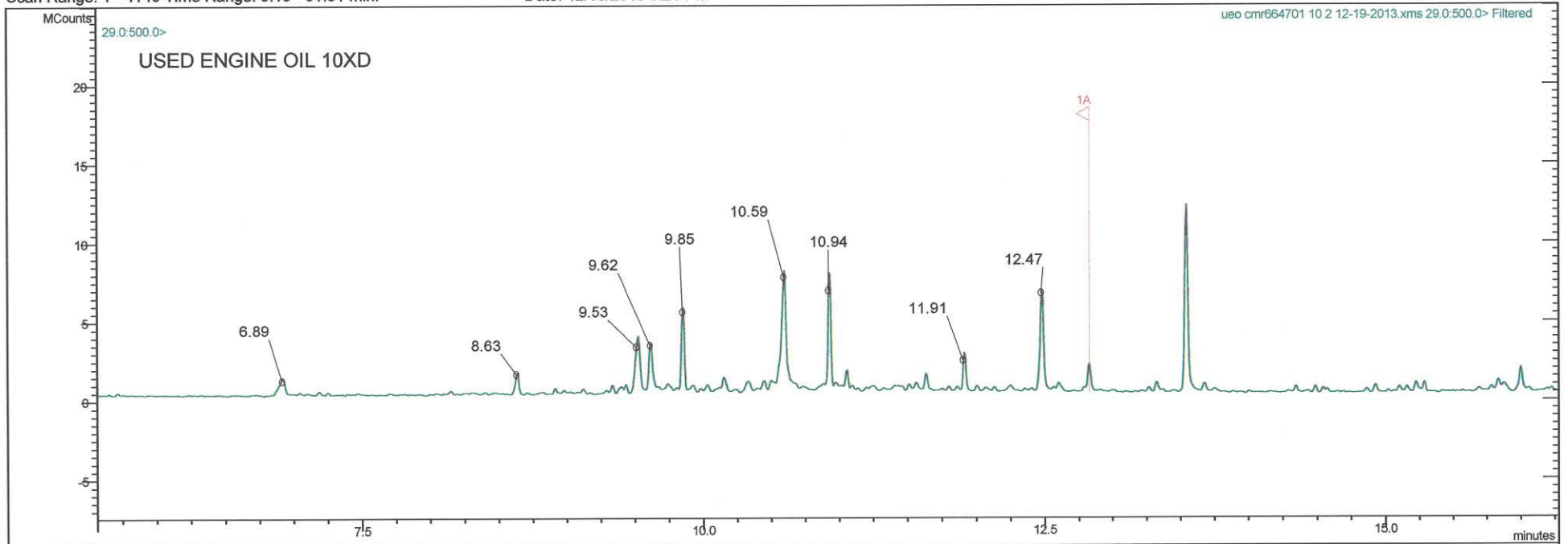
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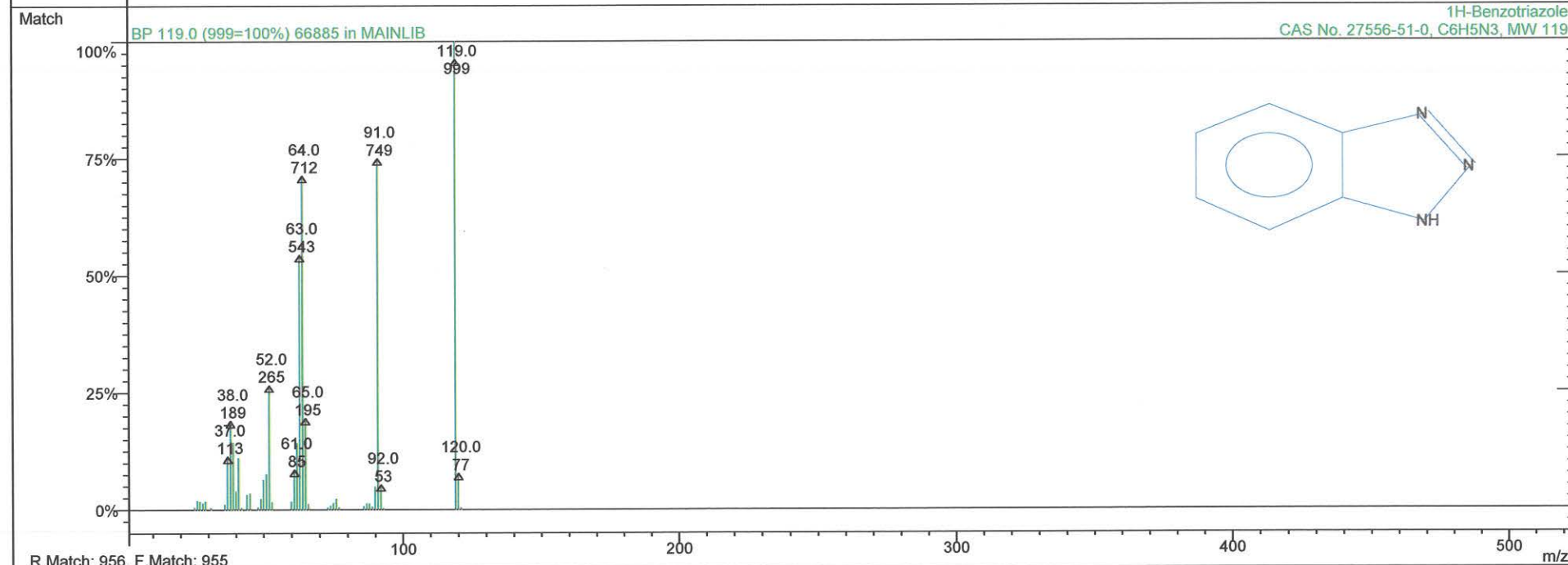
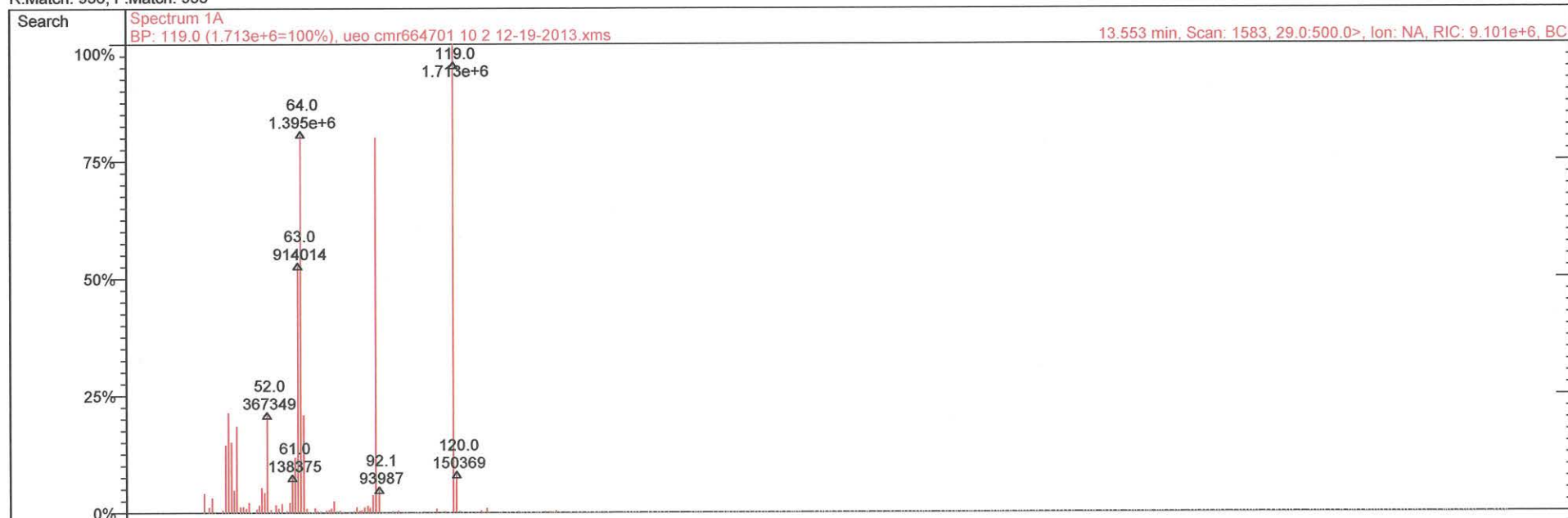
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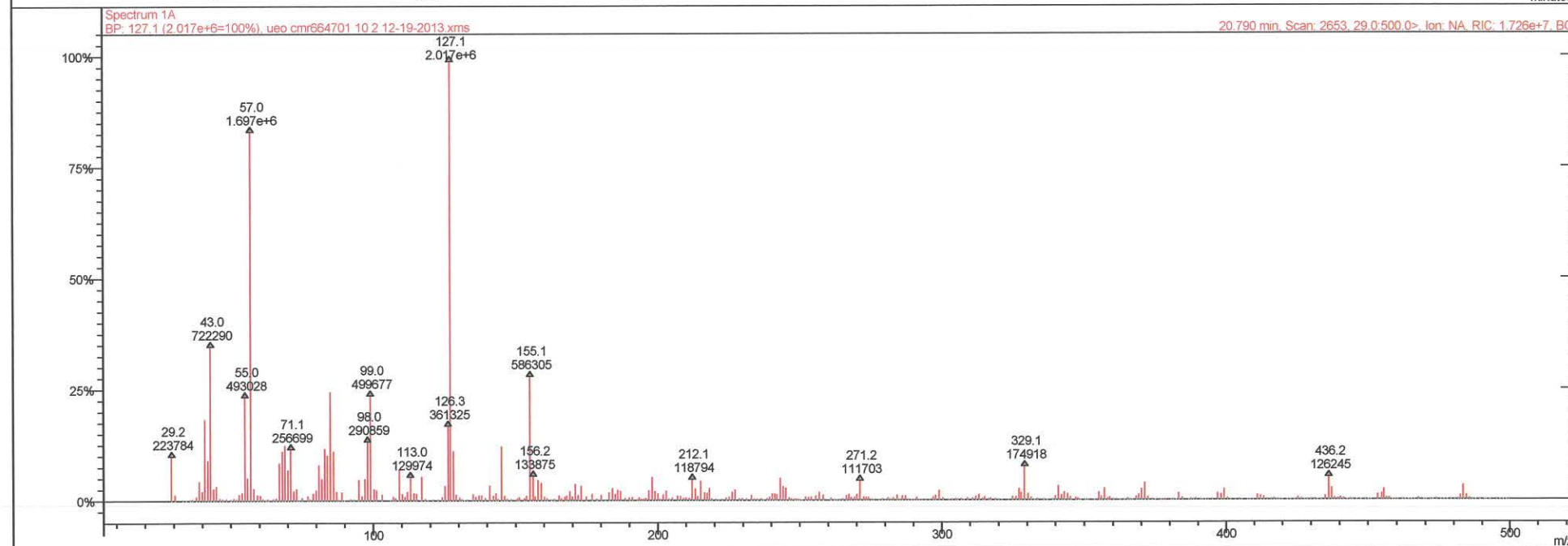
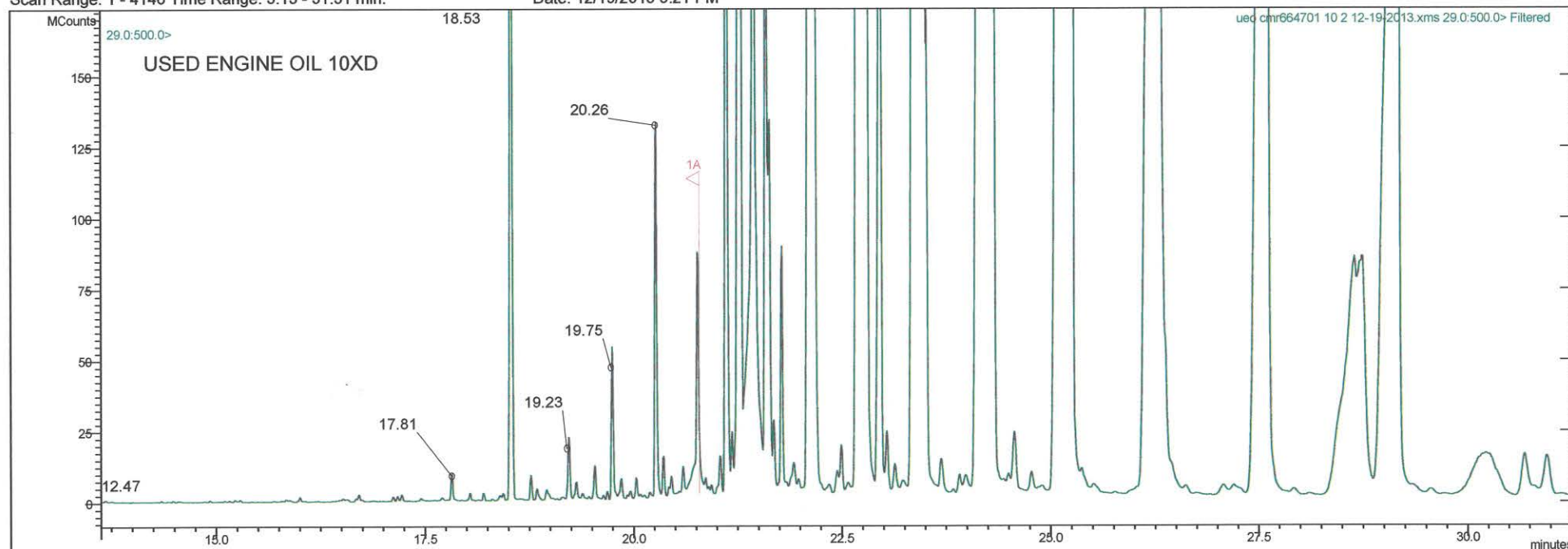
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### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:56 AM

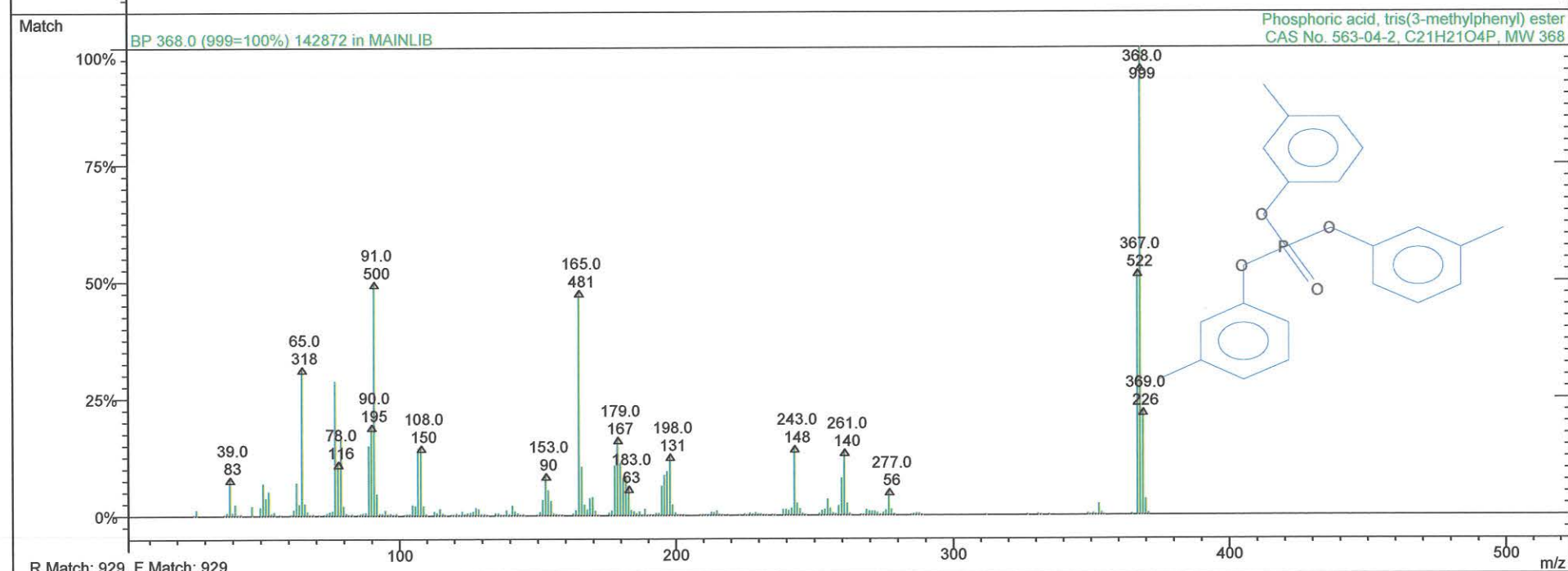
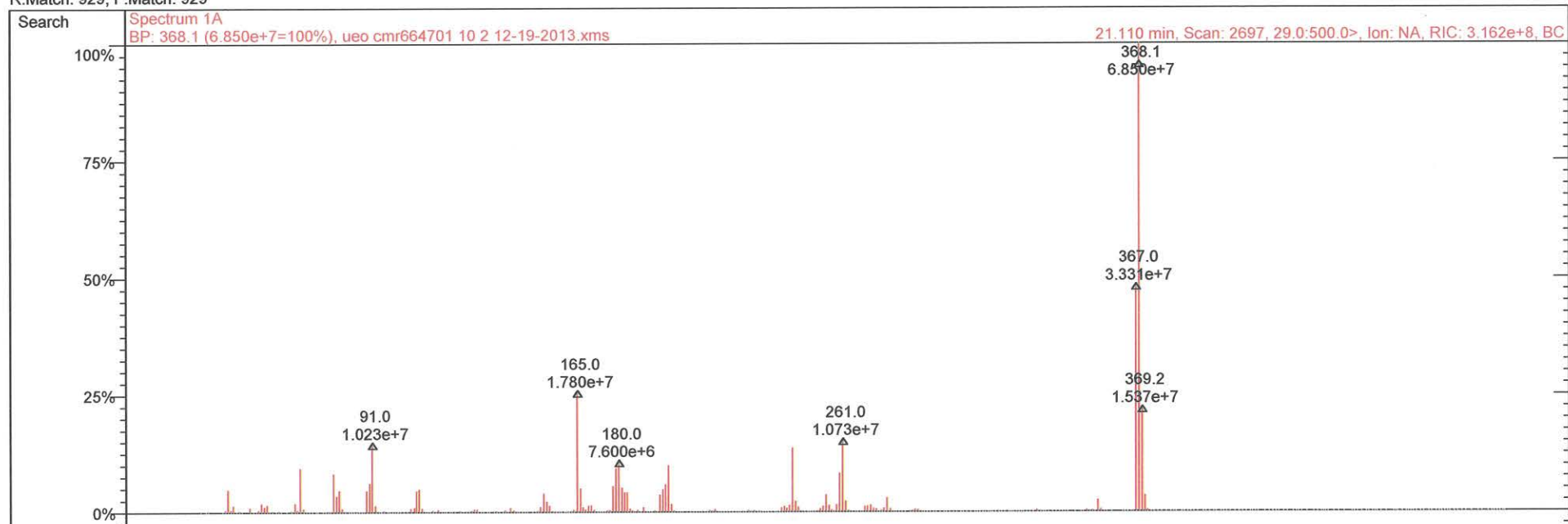
File: XXXXXXXXXX Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



Scan 2697 from e

Entry 142872 from

R.Match: 929, F.Match: 929

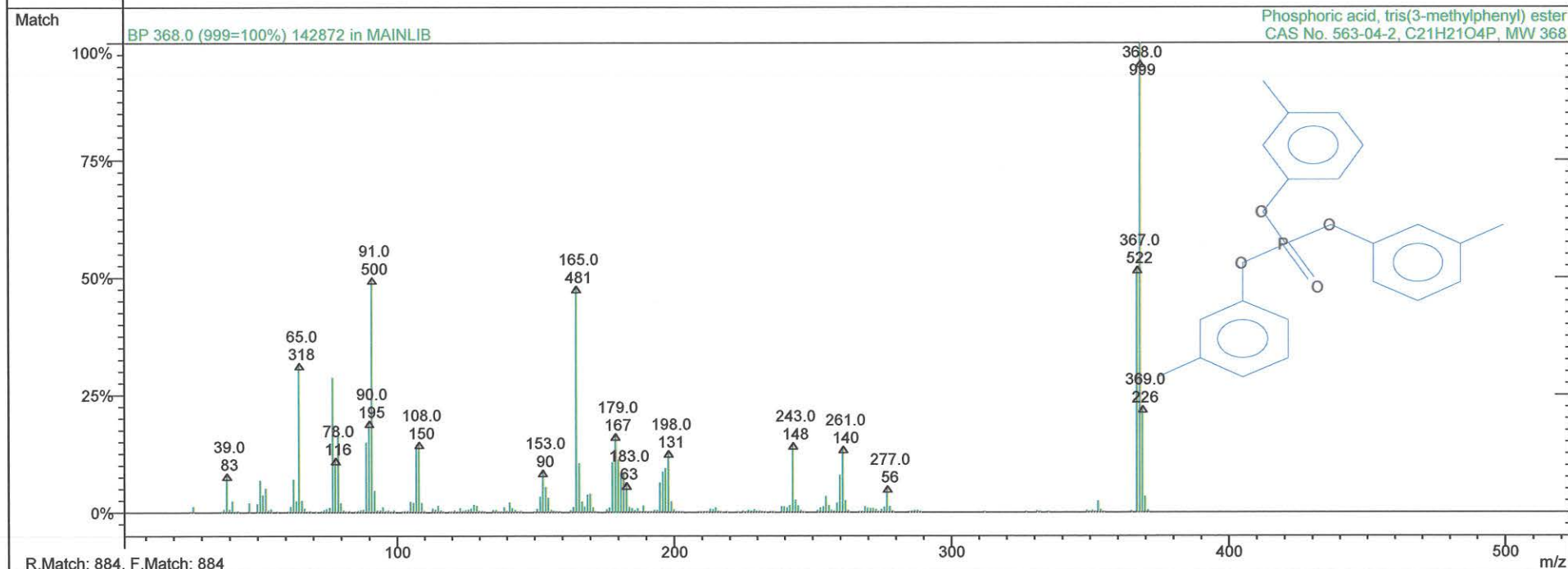
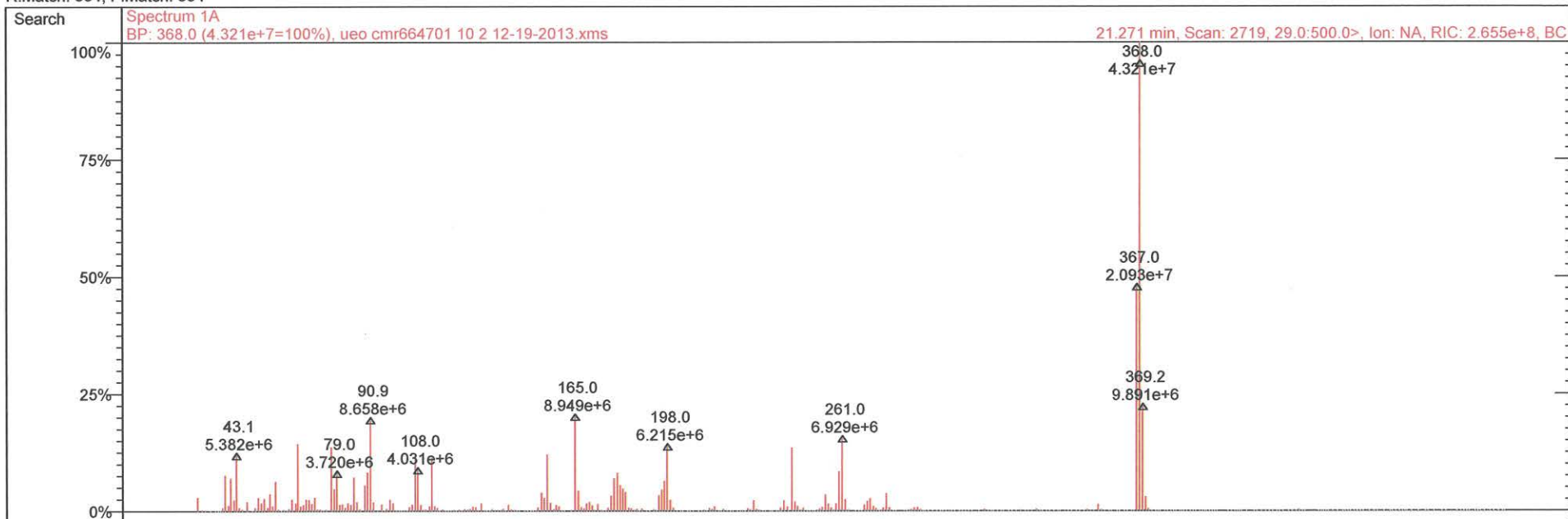


R.Match: 929, F.Match: 929

Scan 2719 from [REDACTED]

Entry 142872 from [REDACTED]

R.Match: 884, F.Match: 884

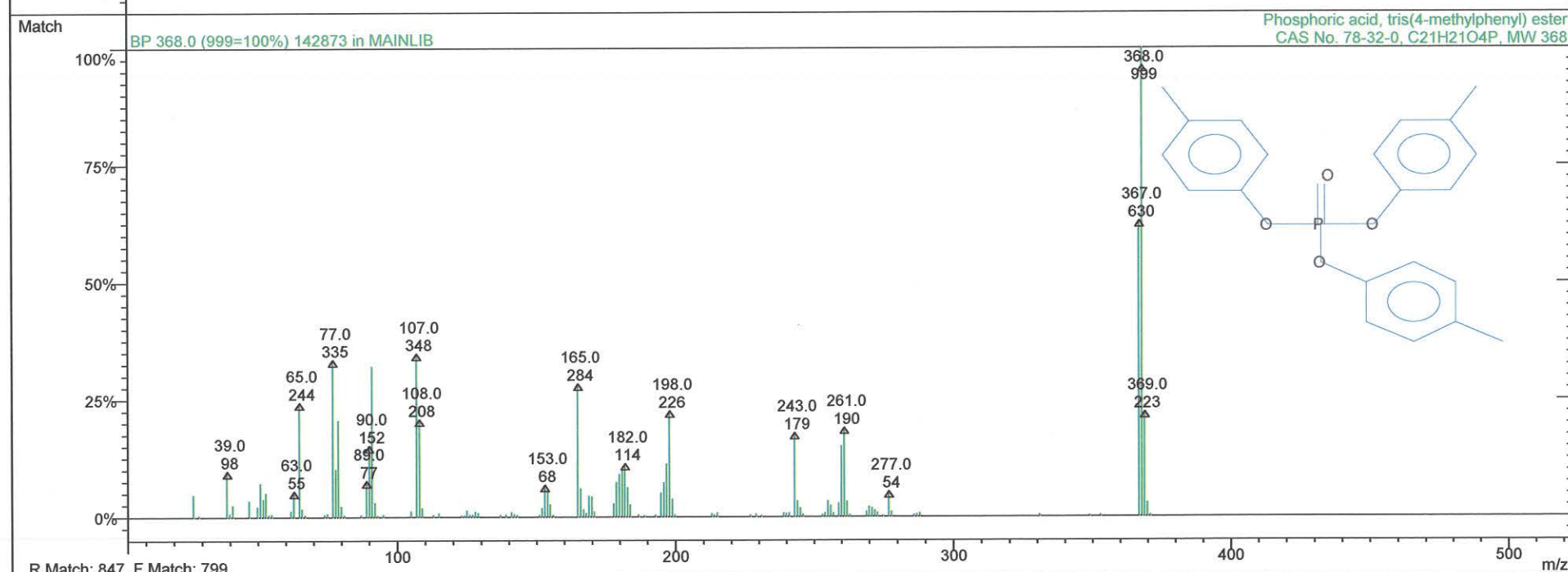
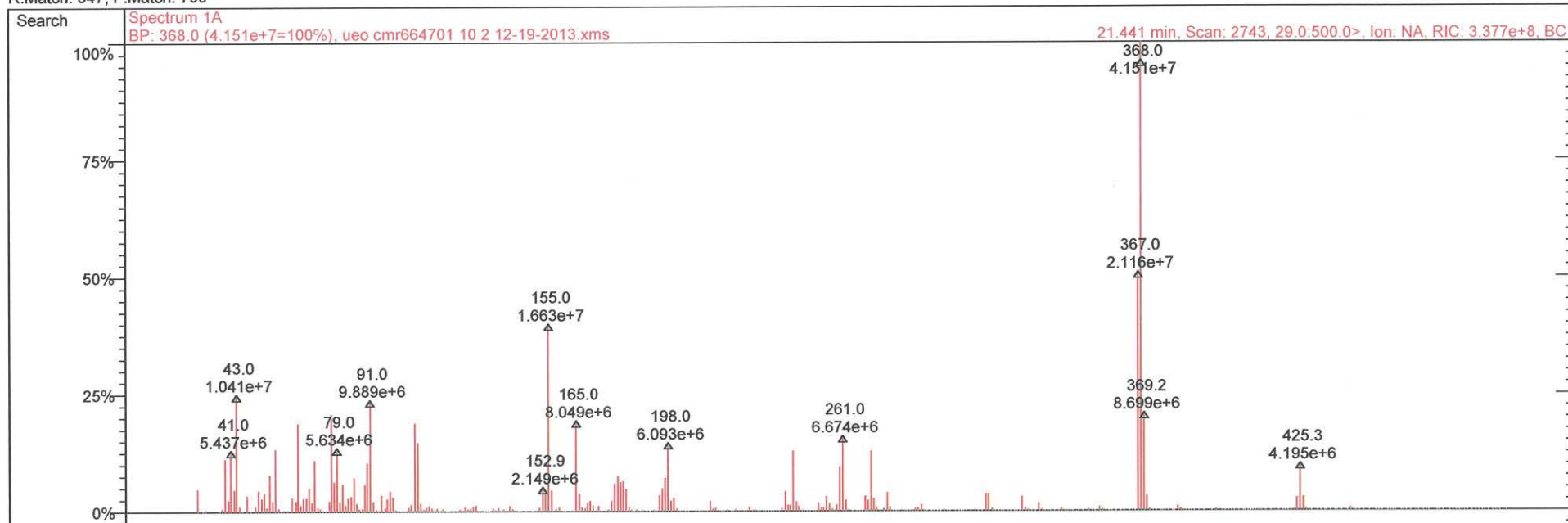


R.Match: 884, F.Match: 884

Scan 2743 from [REDACTED]

Entry 142873 from [REDACTED]

R.Match: 847, F.Match: 799

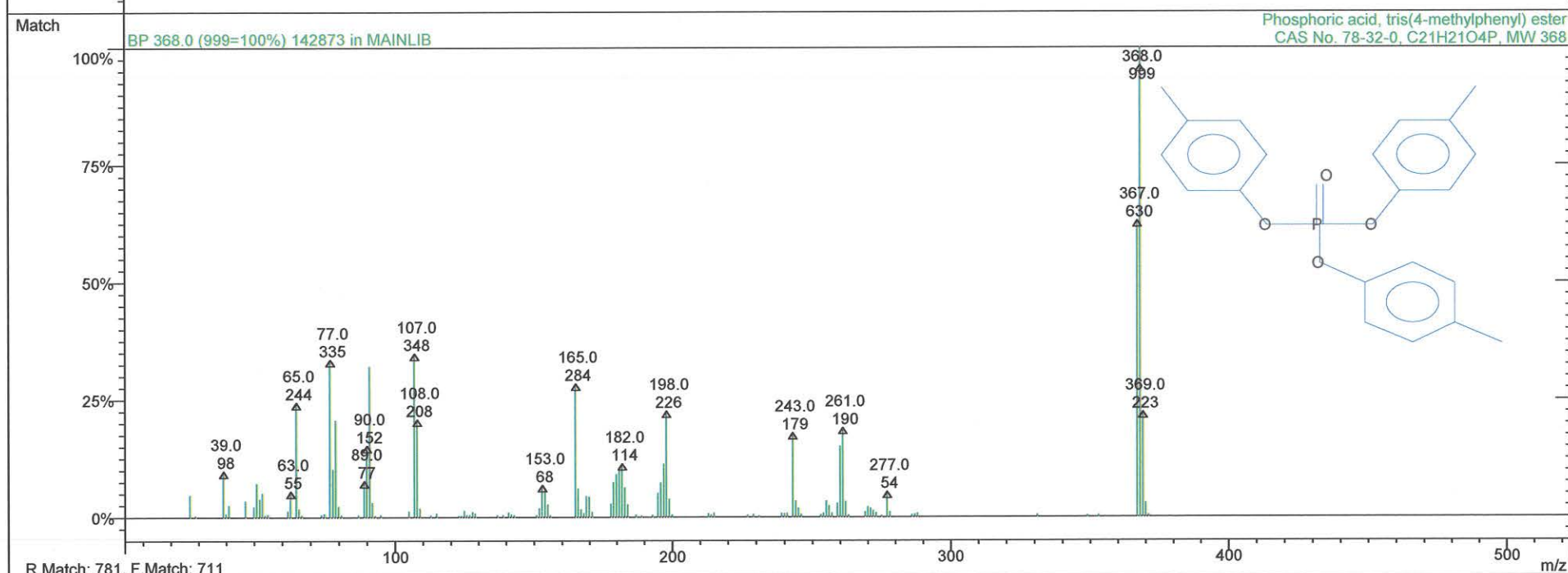
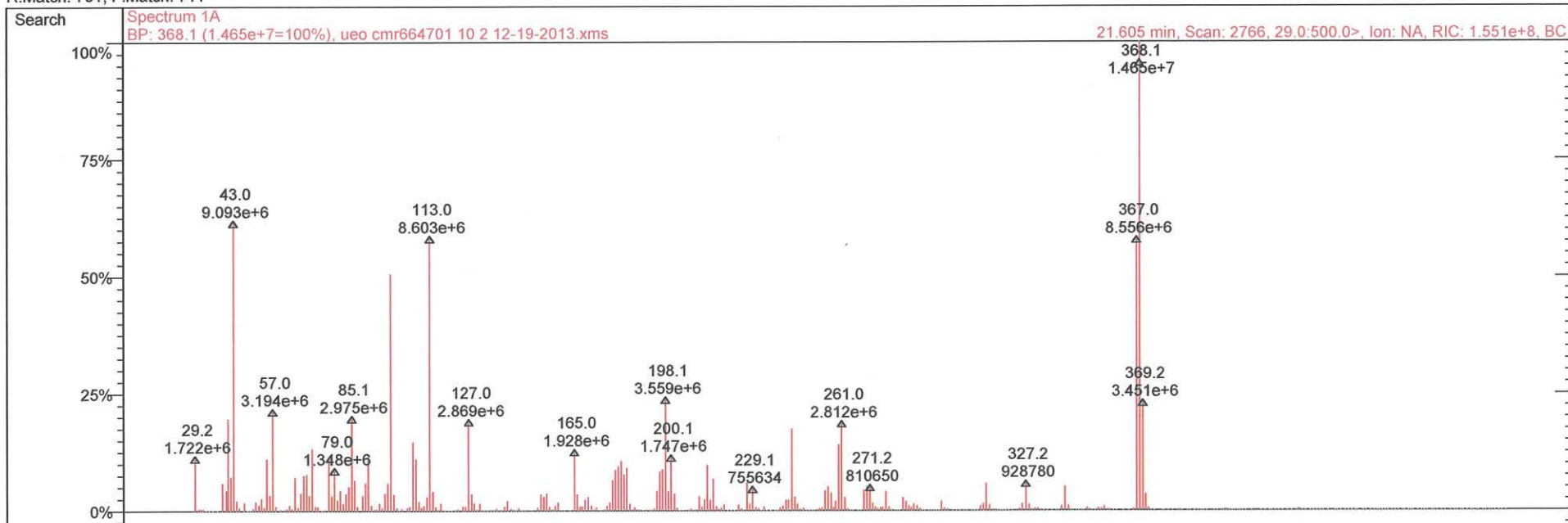


R.Match: 847, F.Match: 799

Scan 2766 from

Entry 142873 from

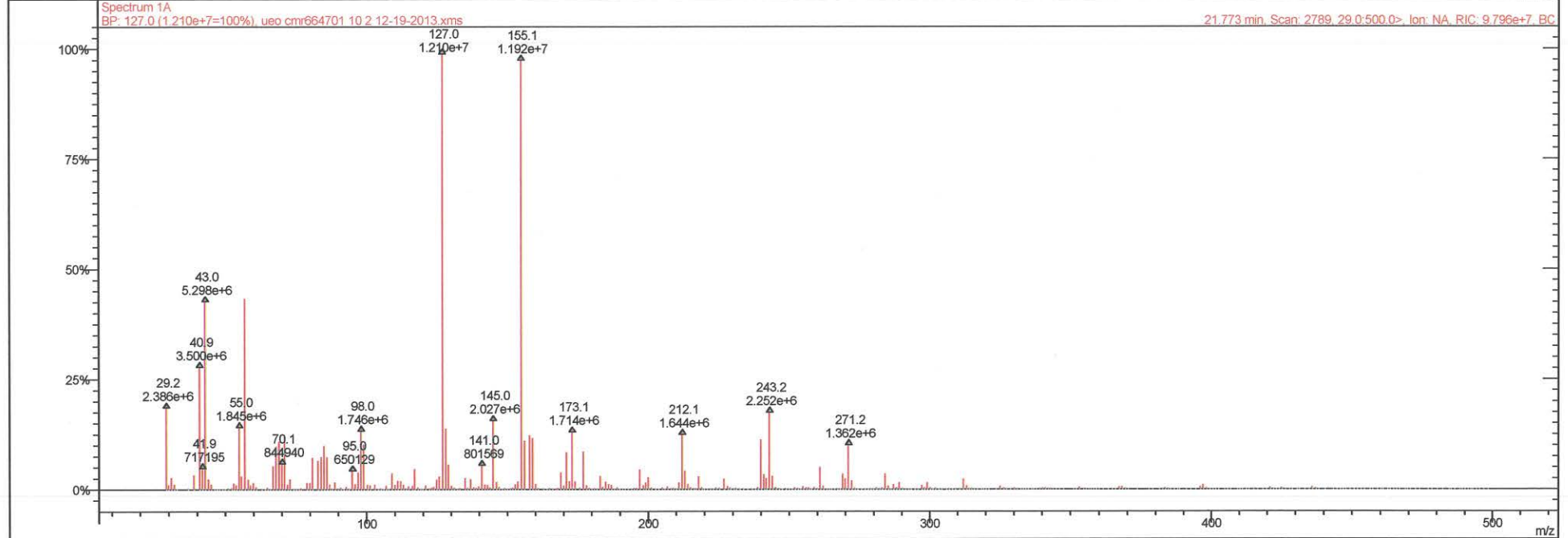
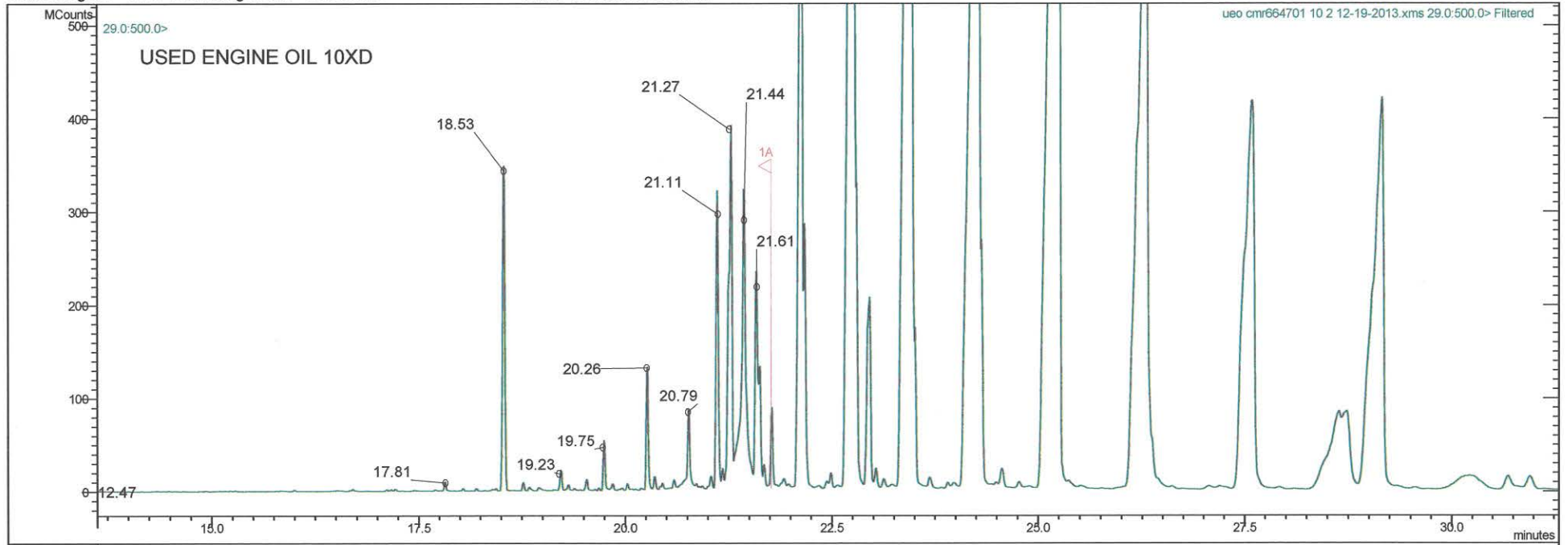
R.Match: 781, F.Match: 711





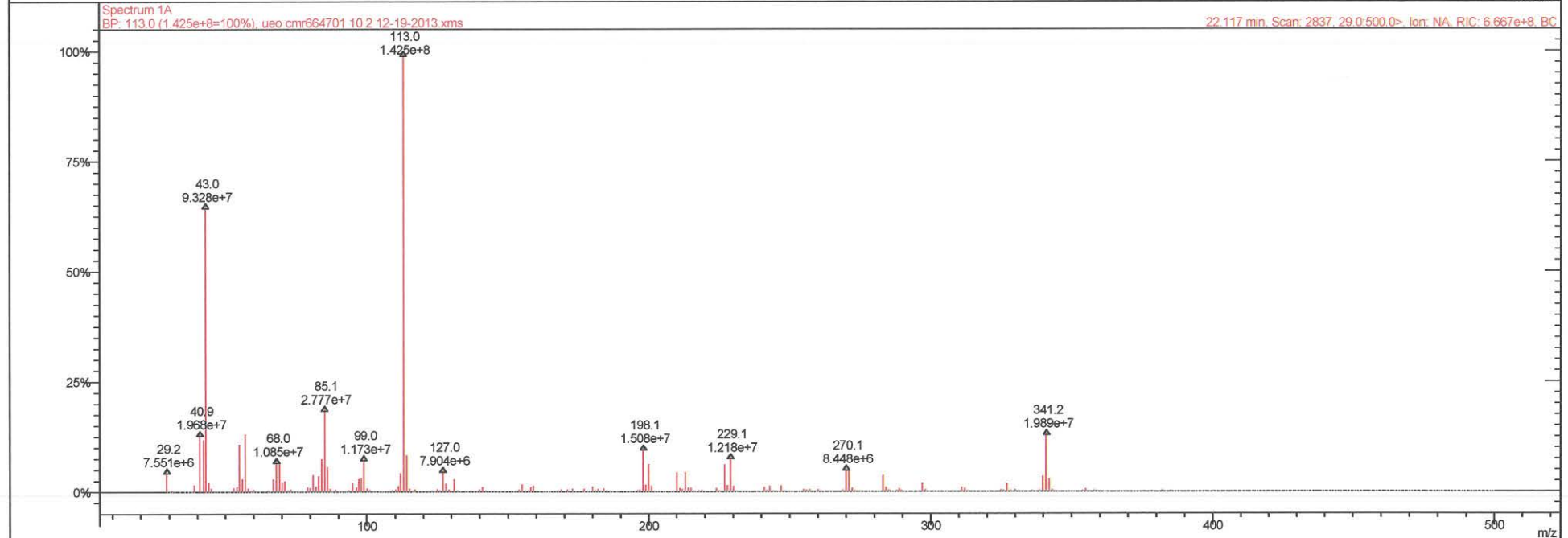
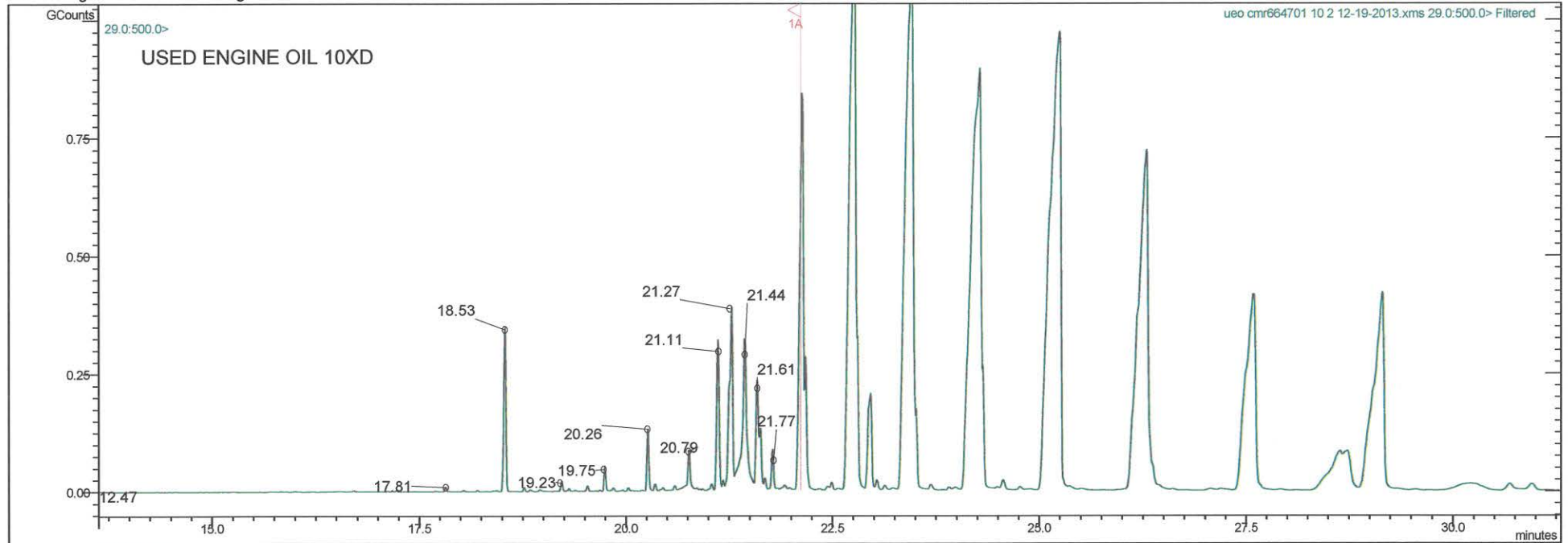
### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:24 AM

File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



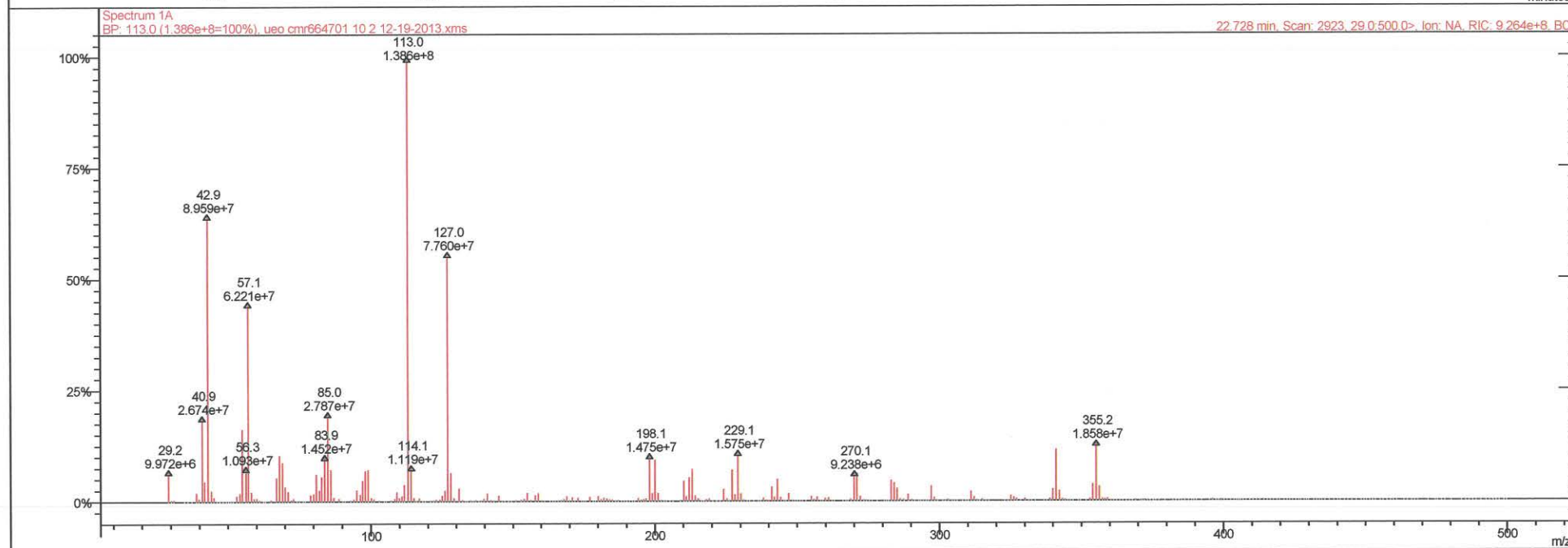
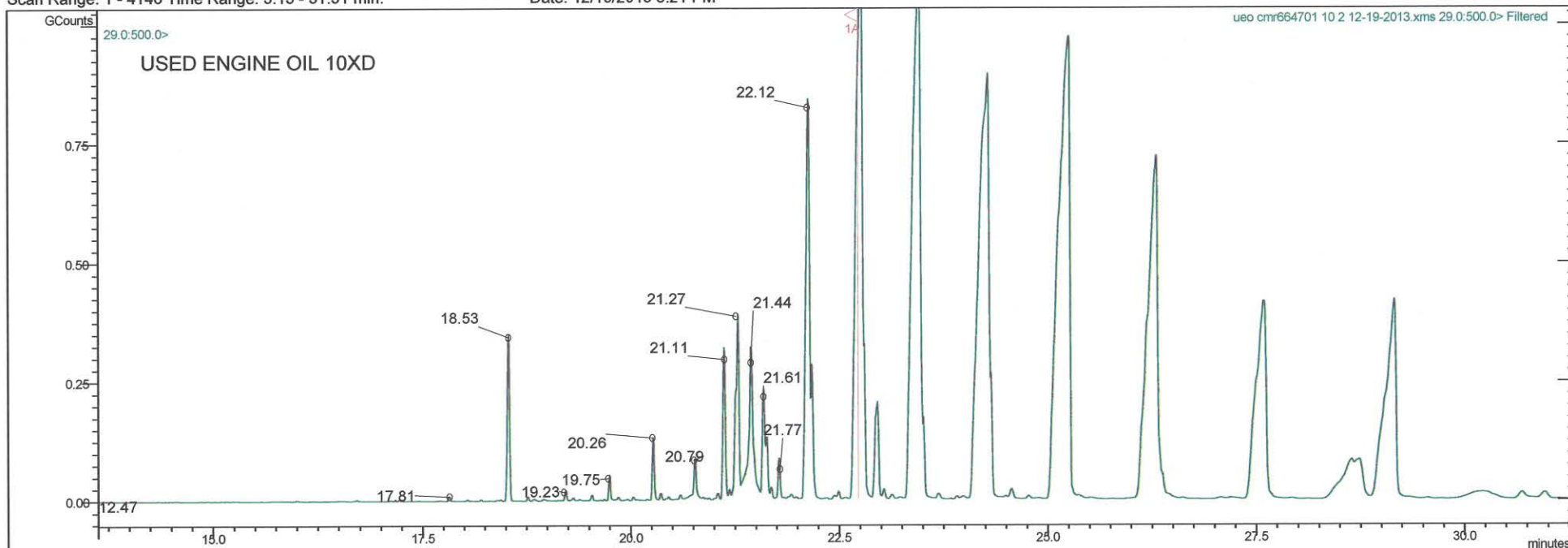
### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:26 AM

File: [REDACTED]  
Sam: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:27 AM

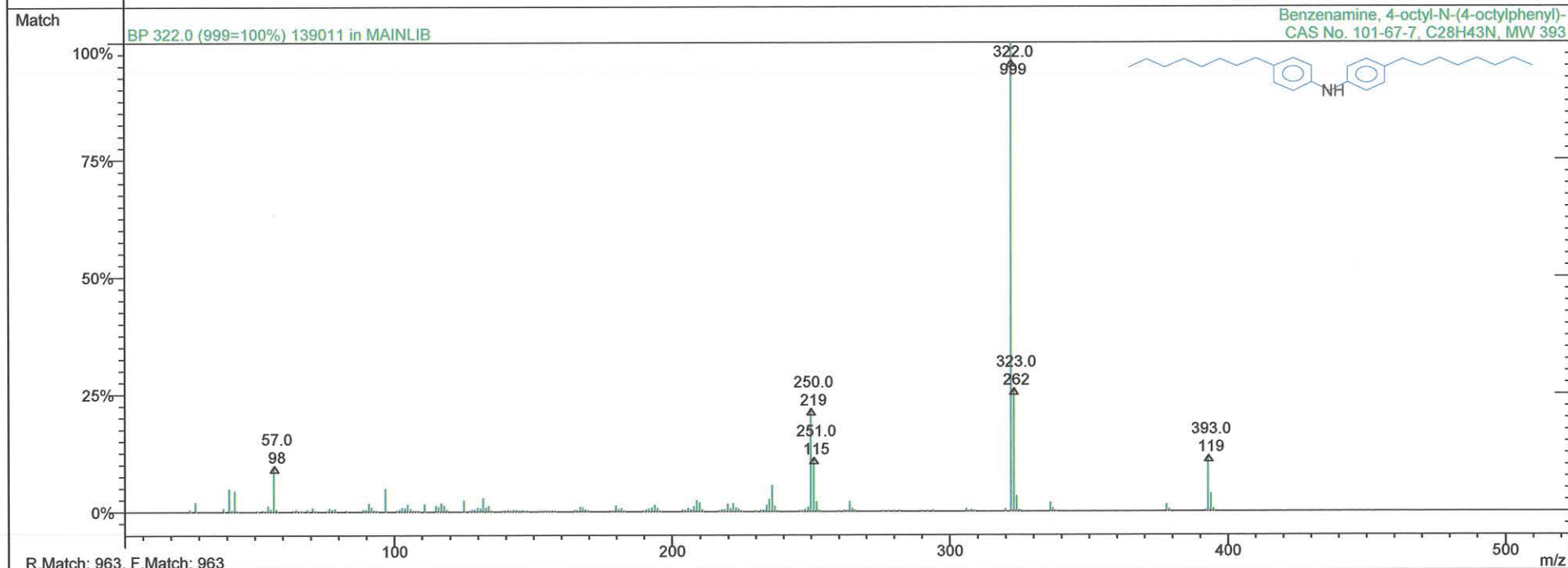
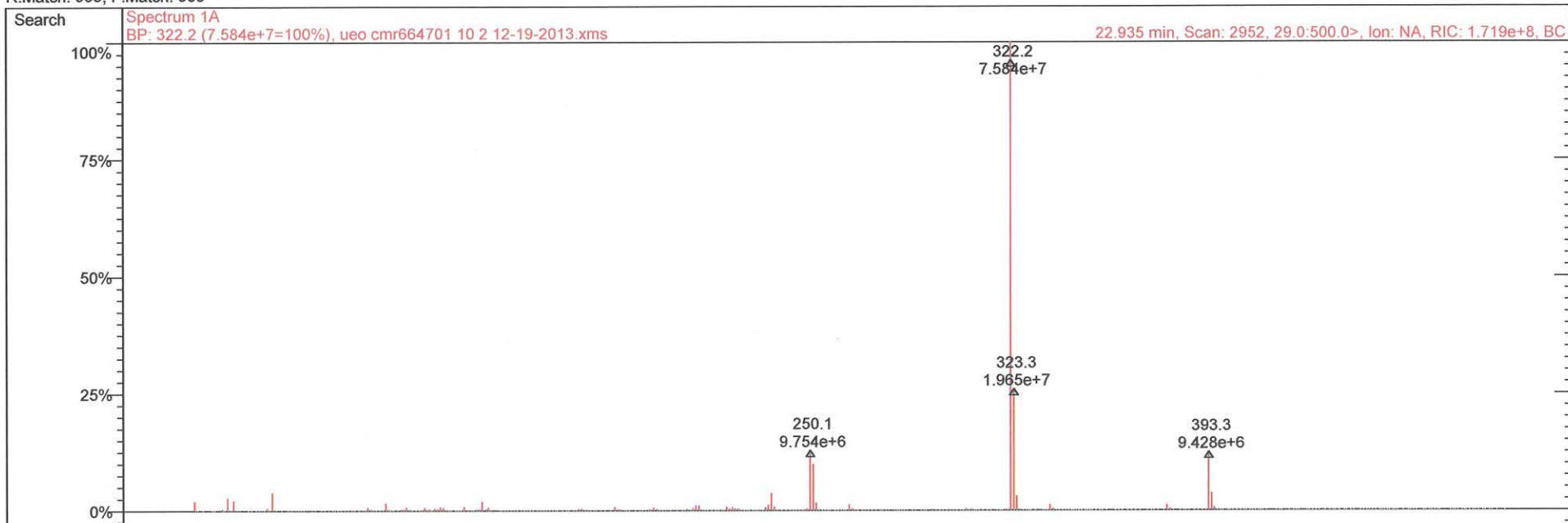
File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



Scan 2952 from [REDACTED]

Entry 139011 from [REDACTED]

R.Match: 963, F.Match: 963

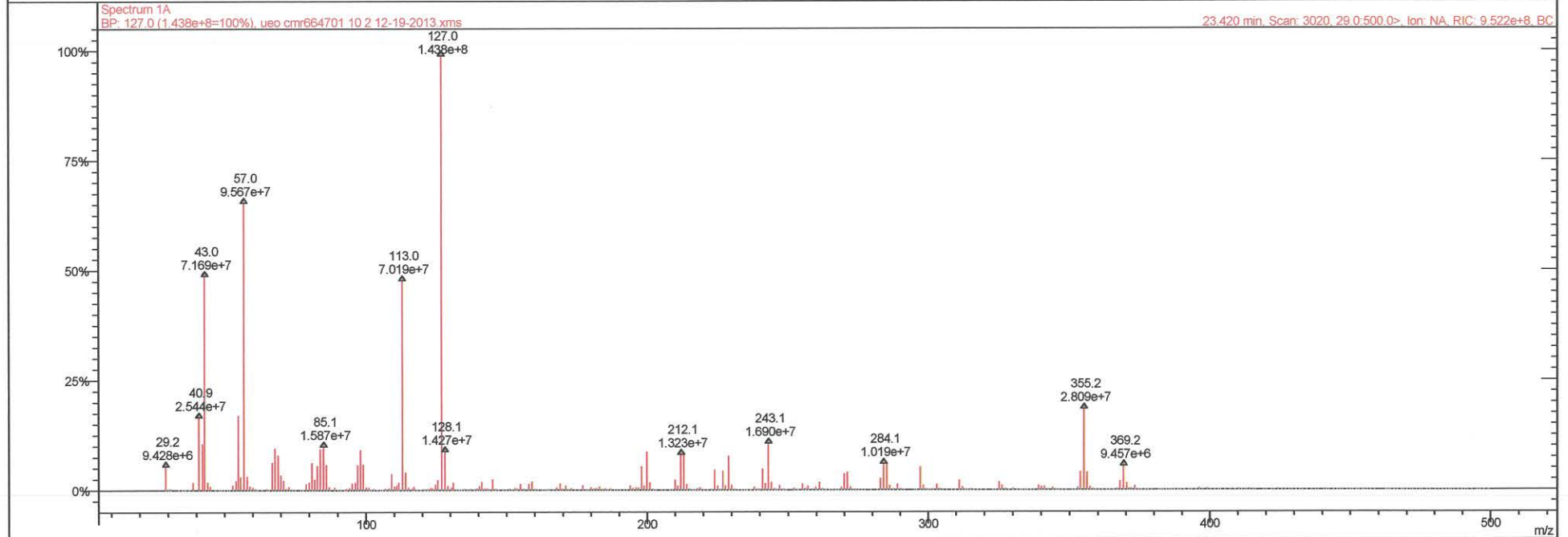
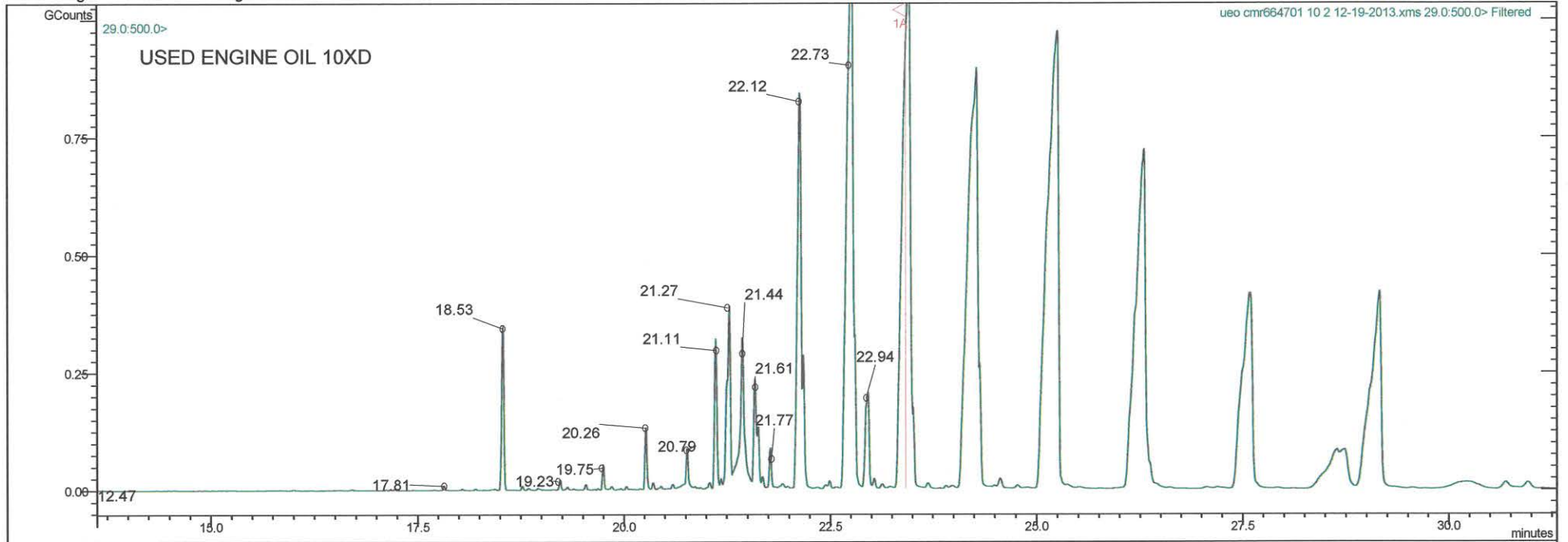


MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:28 AM

File: [REDACTED]  
Sample: [REDACTED]

Operator: CG  
Date: 12/19/2013 5:21 PM

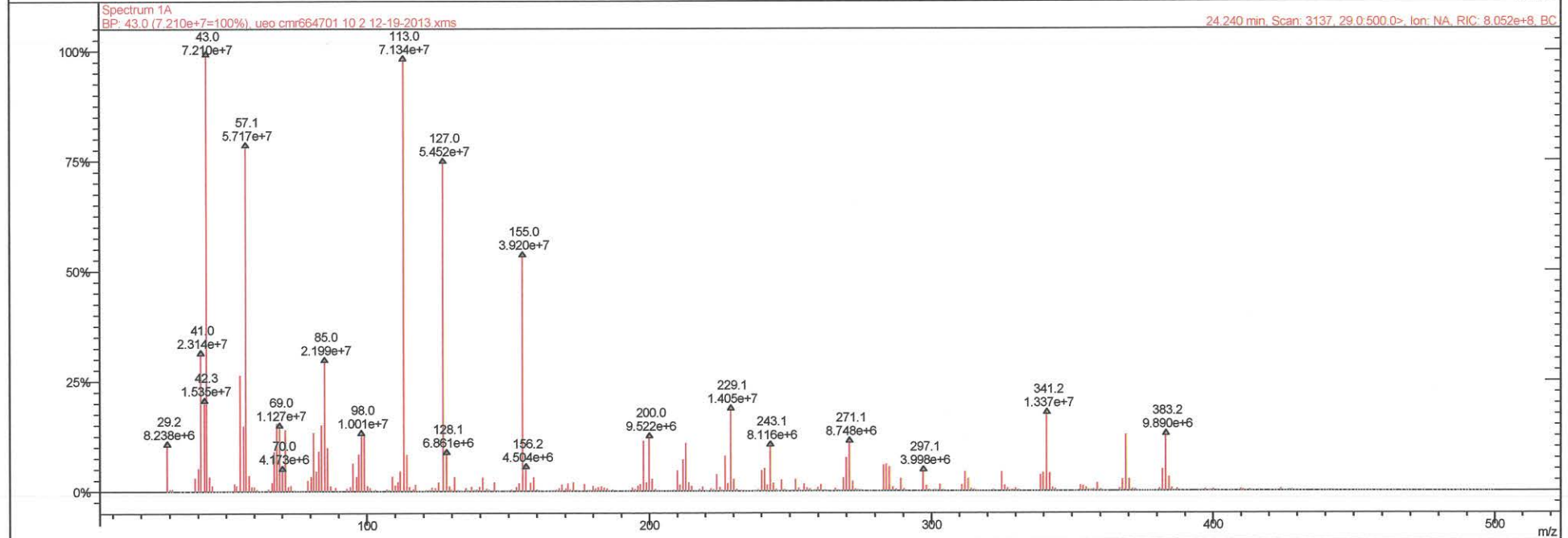
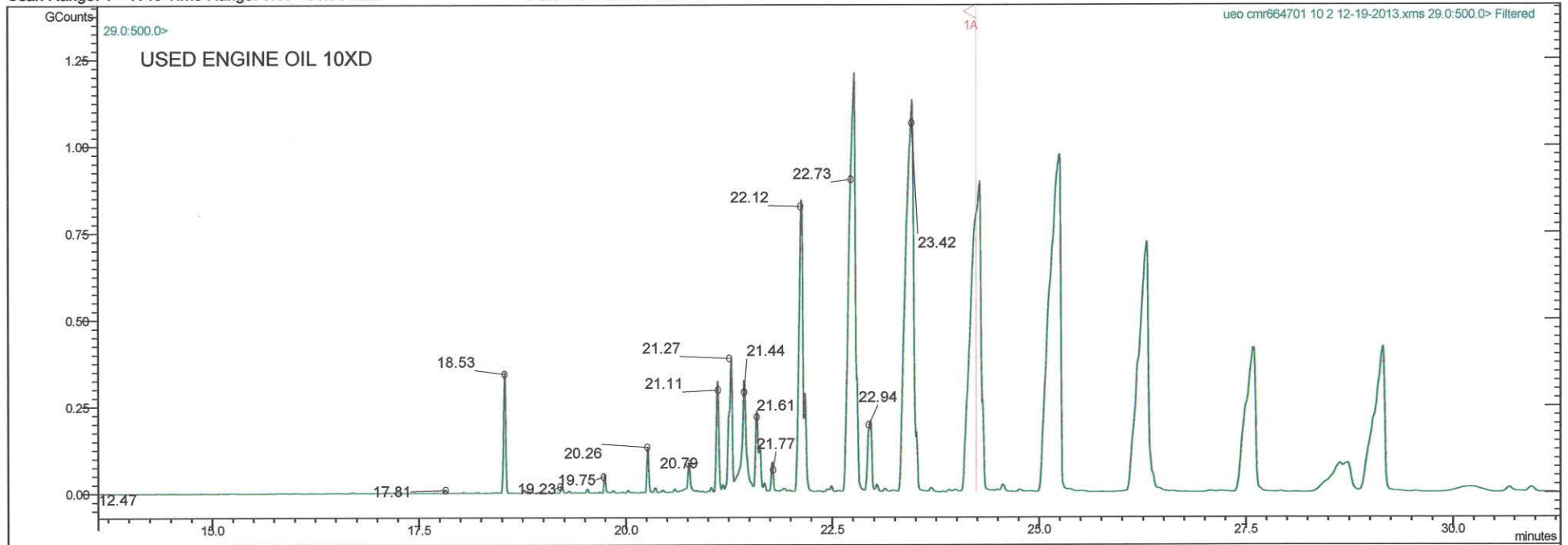
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min.





### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:29 AM

File: XXXXXXXXXX Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM

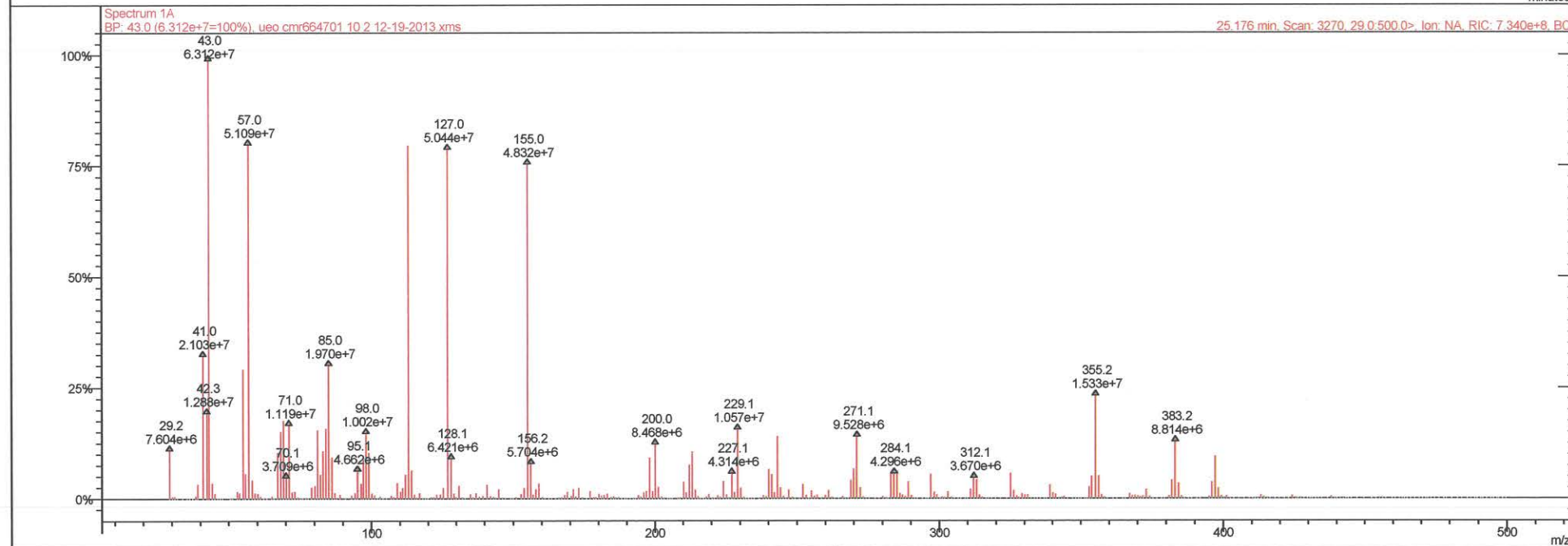
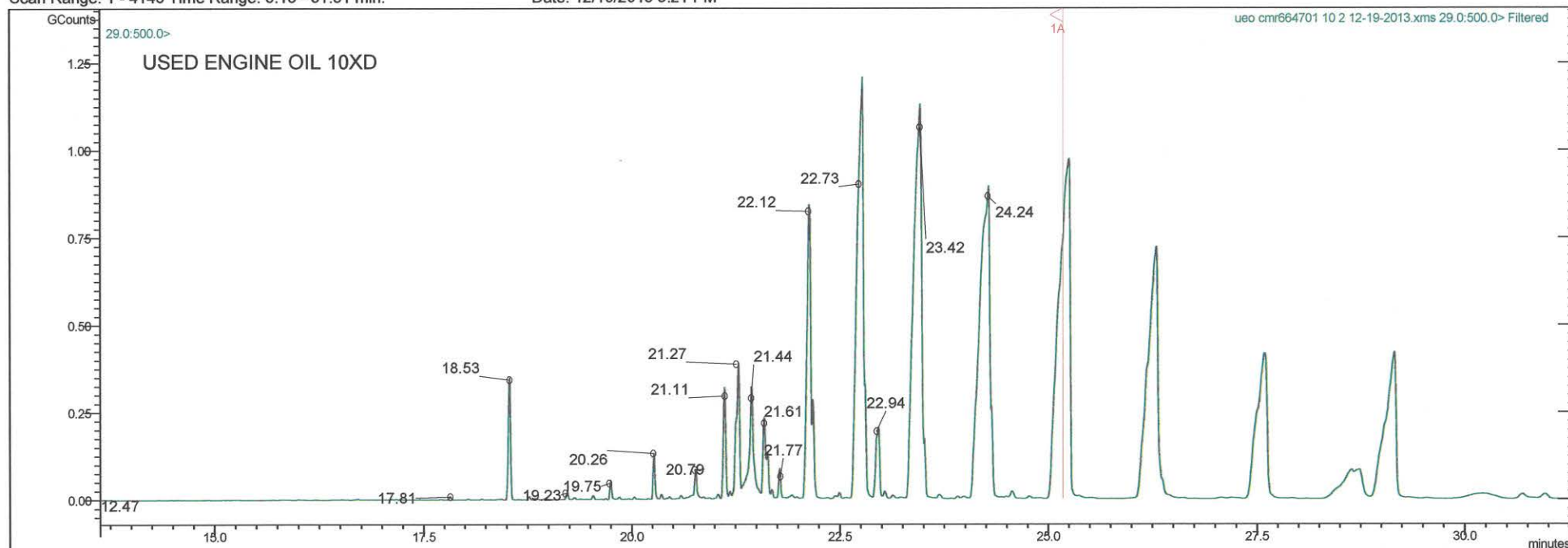


### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:30 AM

File: XXXXXXXXXX  
Sam: XXXXXXXXXX

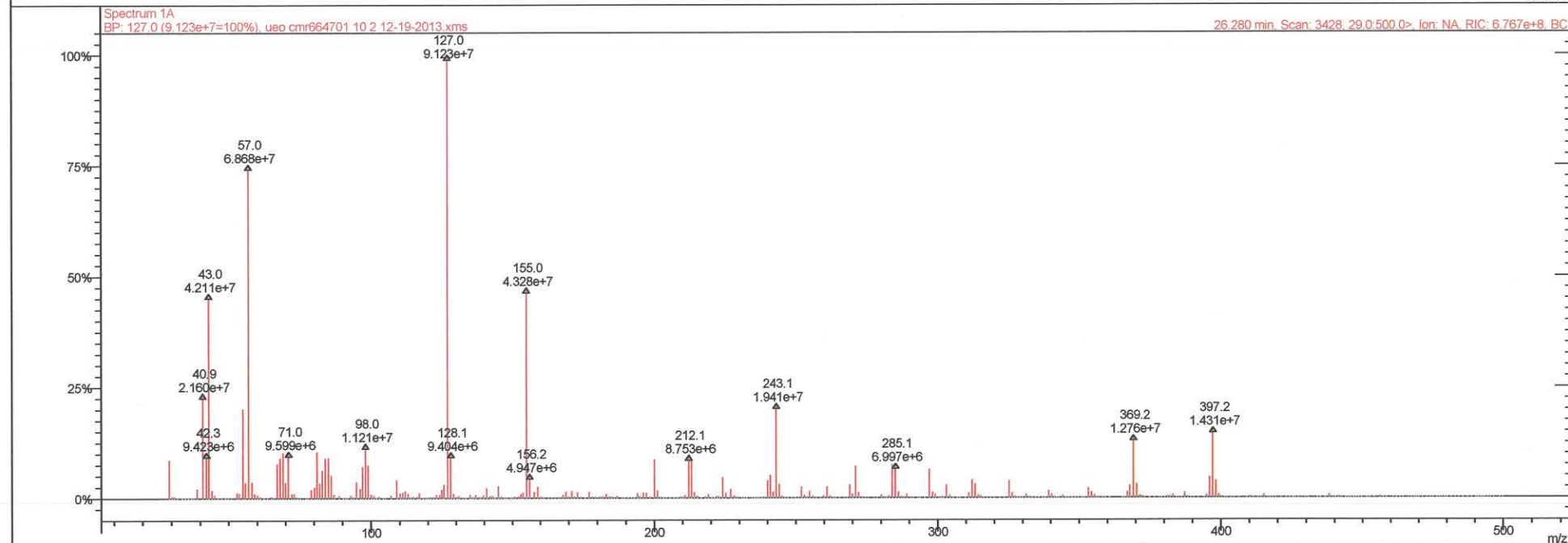
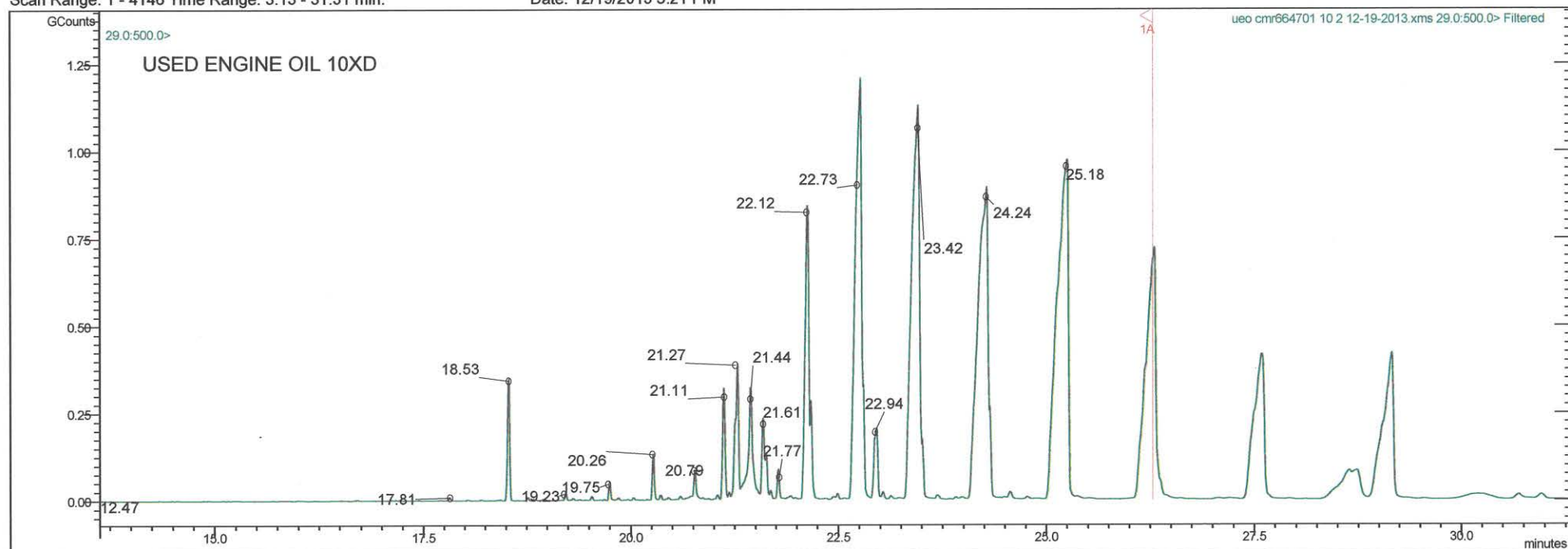
Operator: CG  
Date: 12/19/2013 5:21 PM

Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min.



### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:30 AM

File: [REDACTED]  
Sample: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM

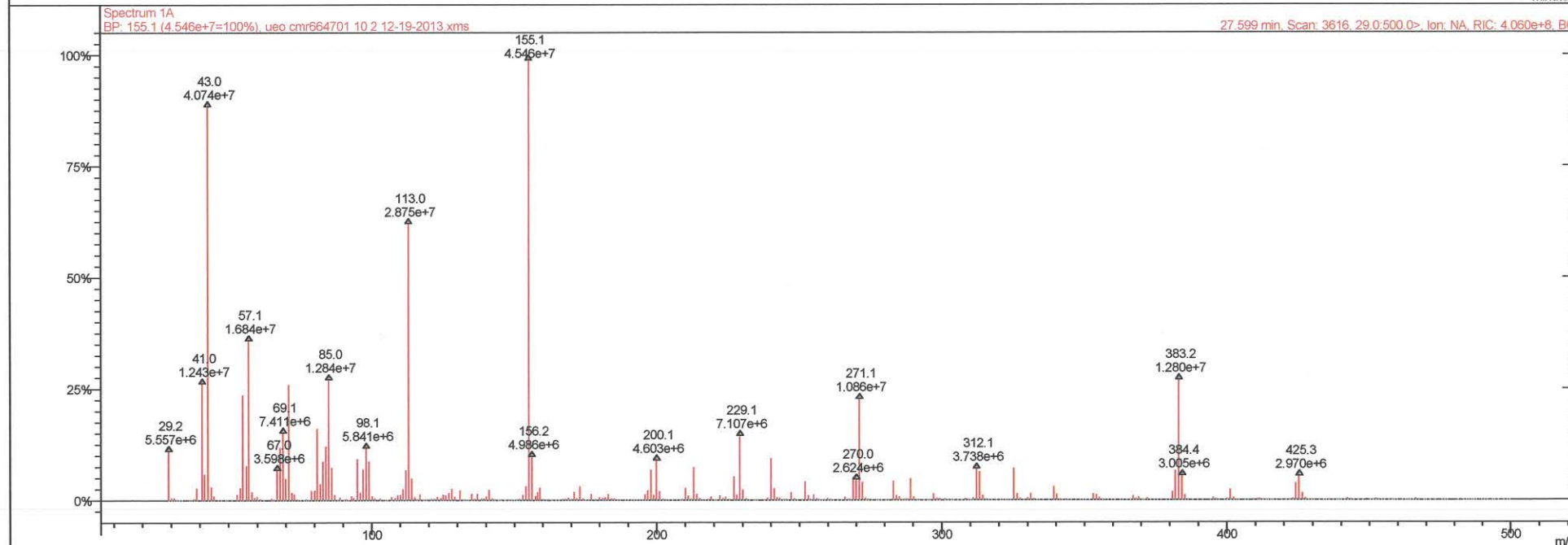
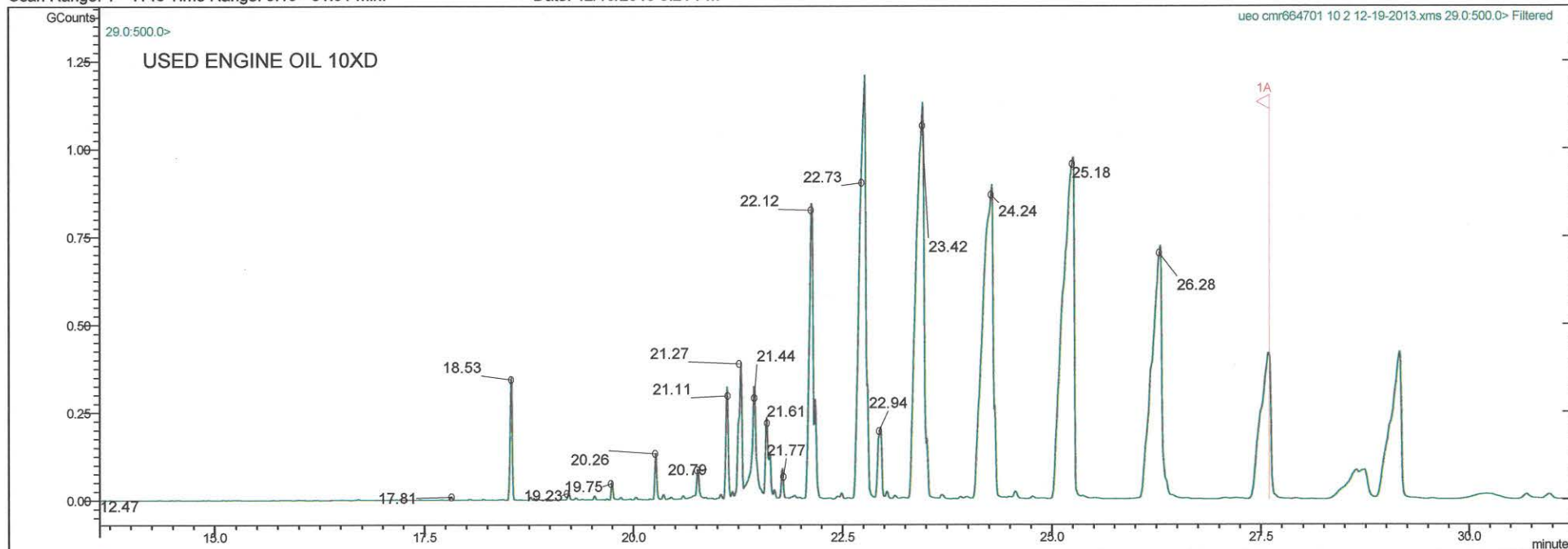


### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:31 AM

File: [REDACTED]  
Sam: [REDACTED]

Operator: CG  
Date: 12/19/2013 5:21 PM

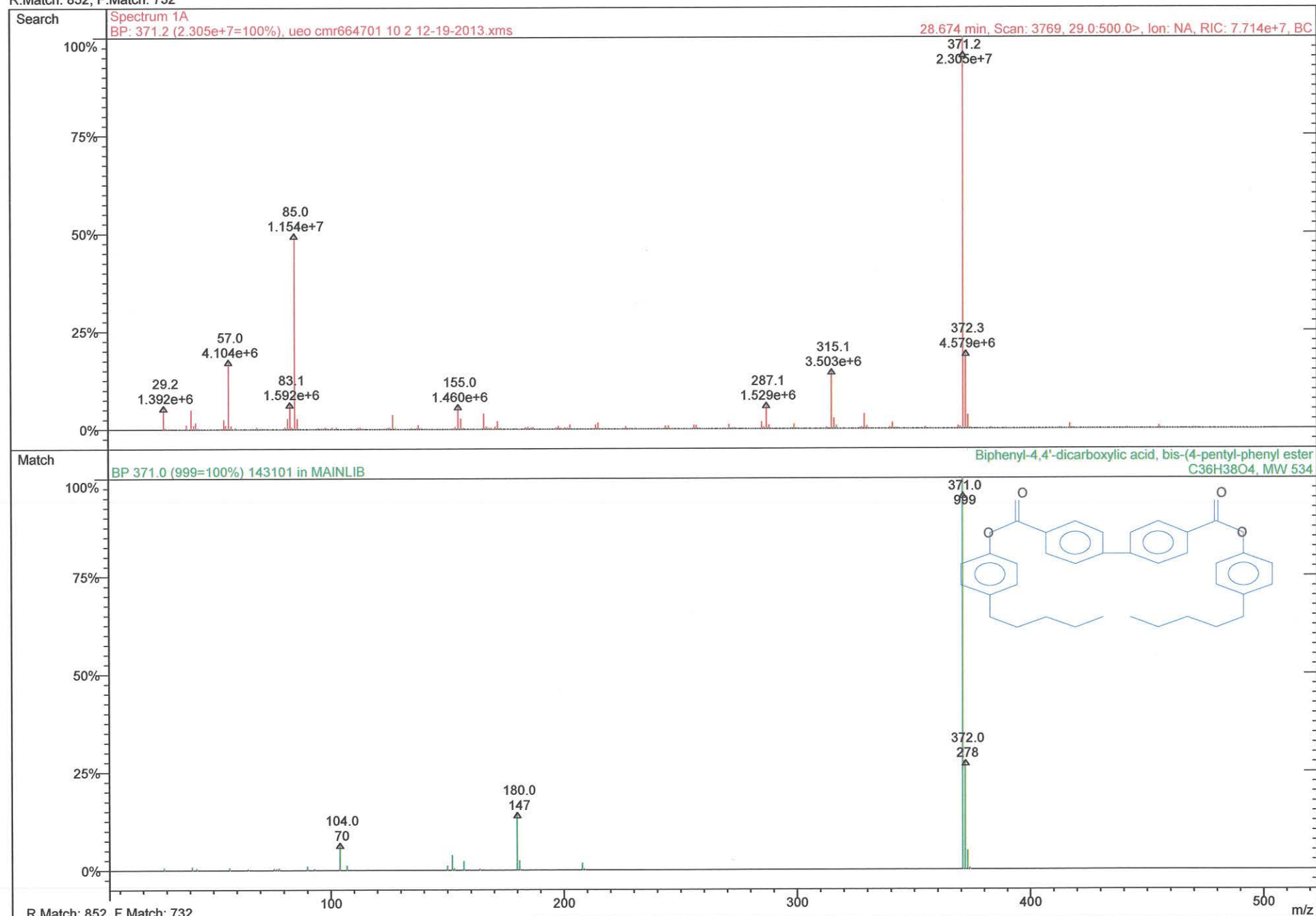
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min.



Scan 3769 from e:\

Entry 143101 from

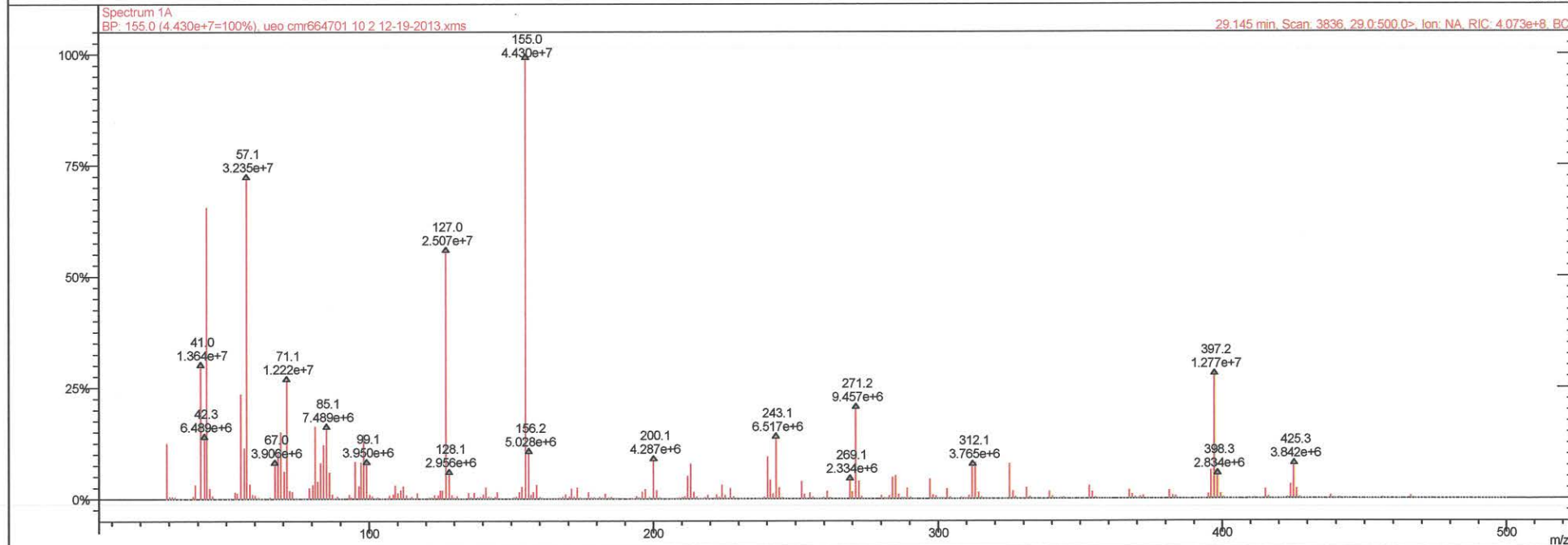
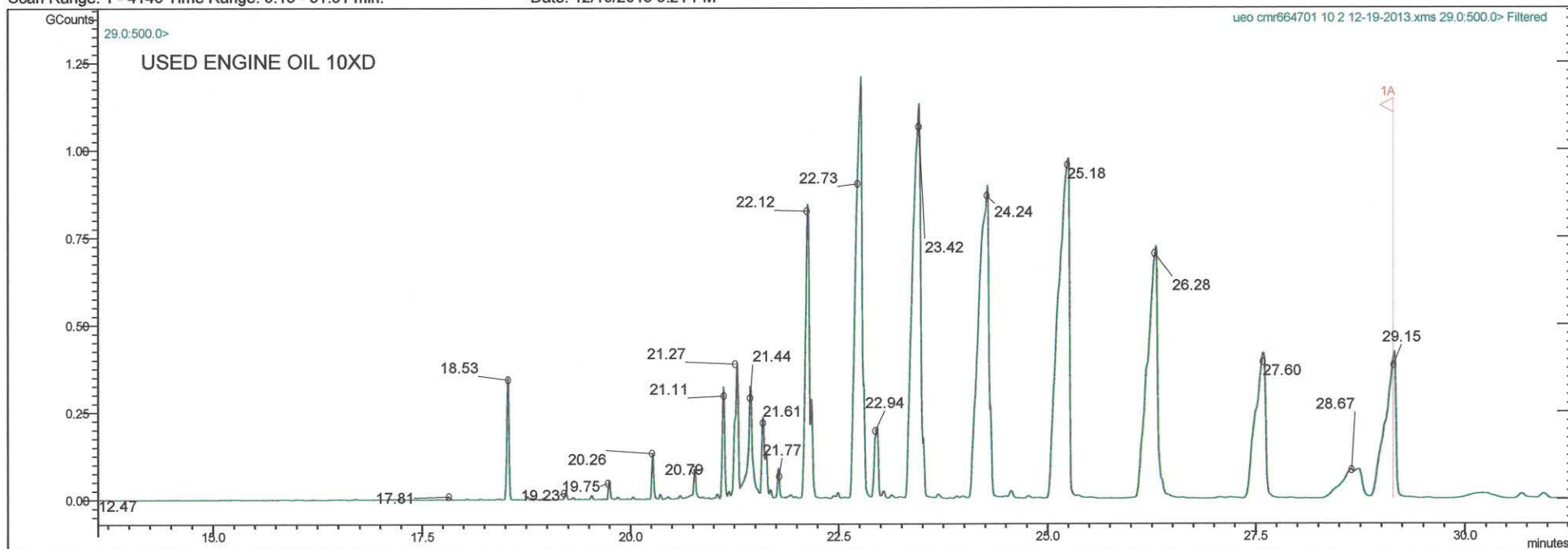
R.Match: 852, F.Match: 732





### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 10:31 AM

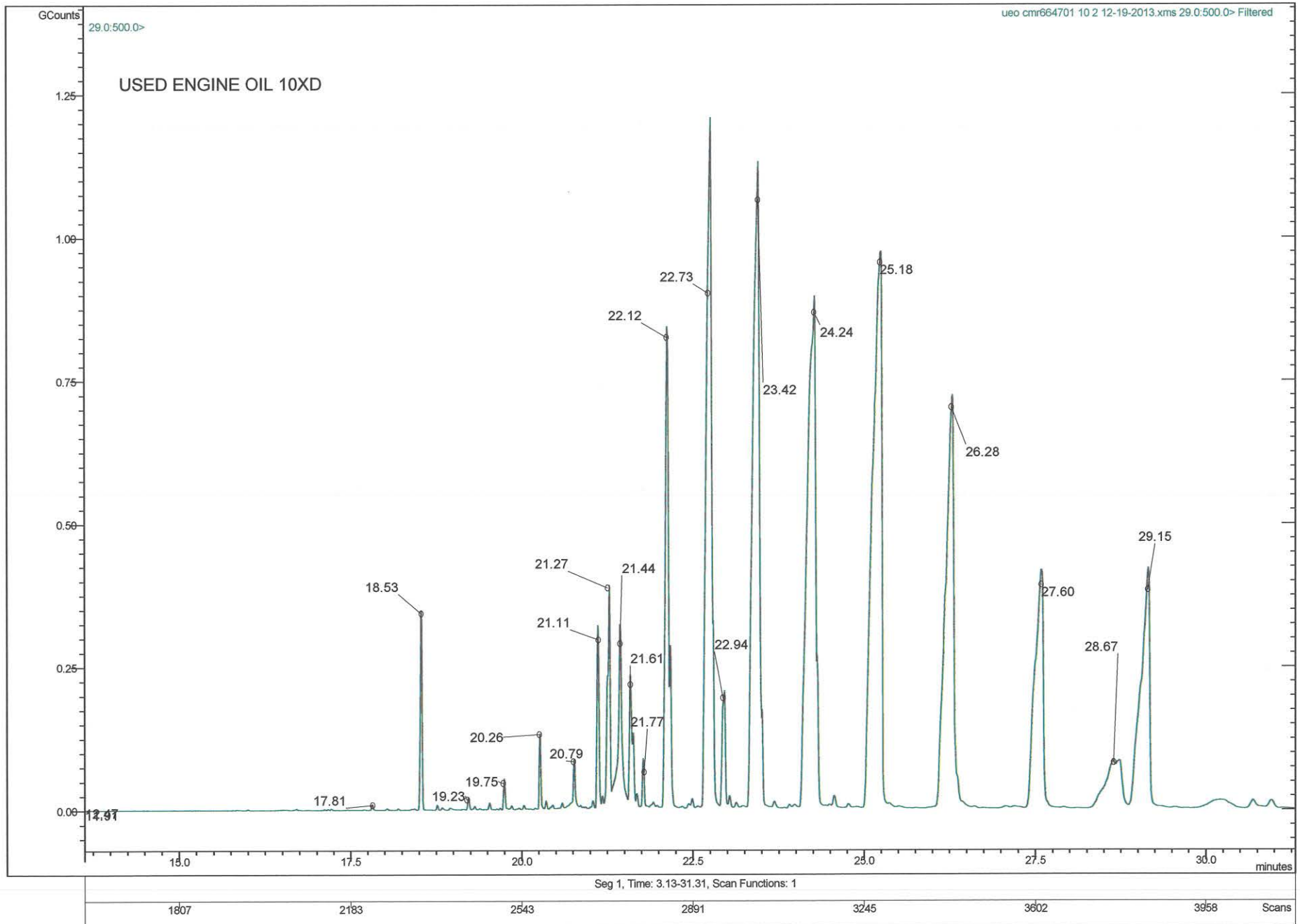
File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



### Chromatogram Plot

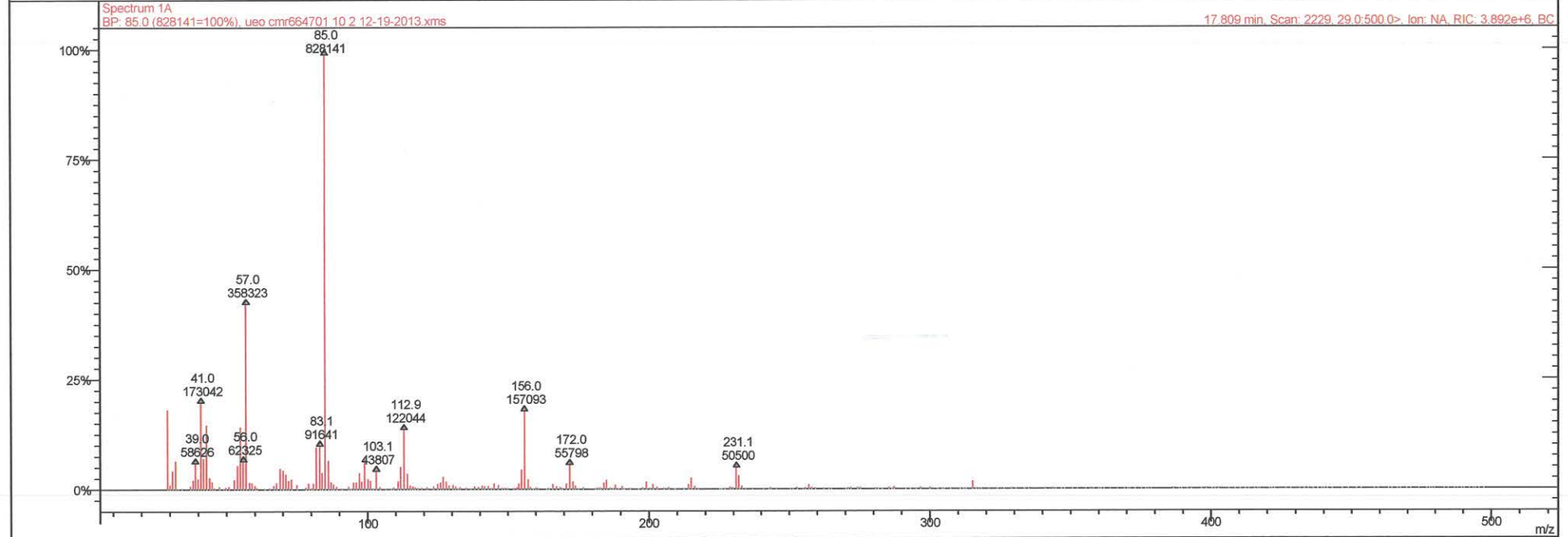
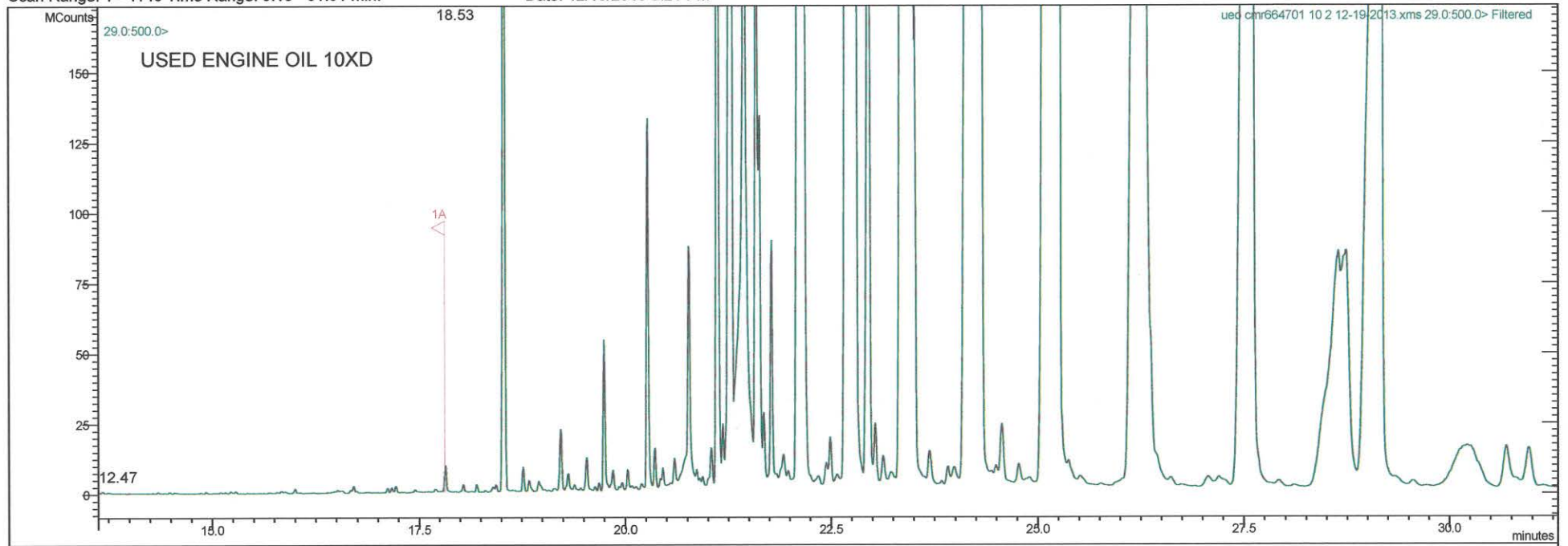
File: [REDACTED]  
Sam [REDACTED]  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min.

Operator: CG  
Date: 12/19/2013 5:21 PM



MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:48 AM

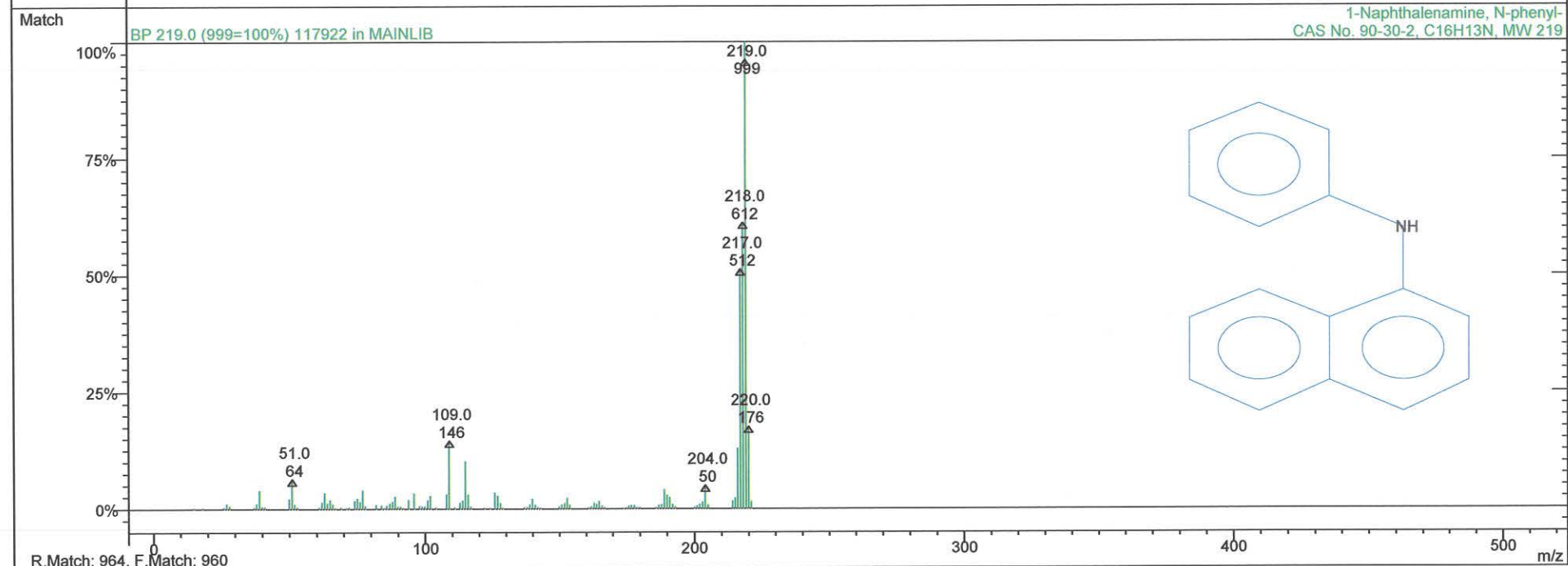
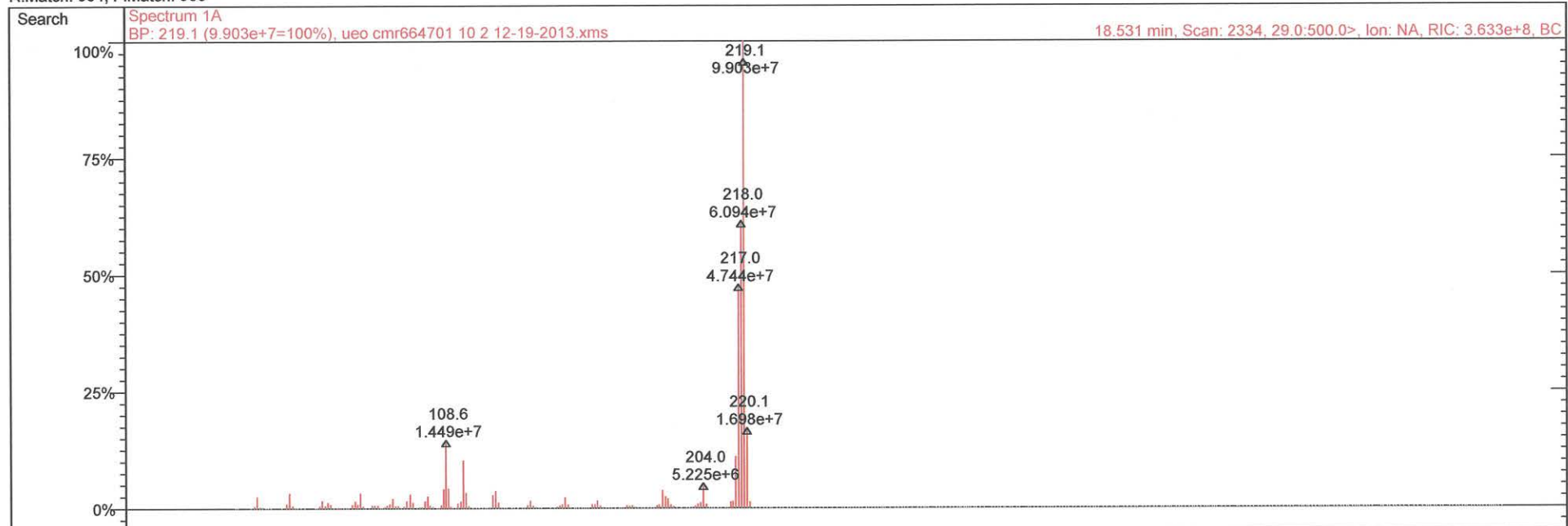
File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



Scan 2334 from e: [REDACTED]

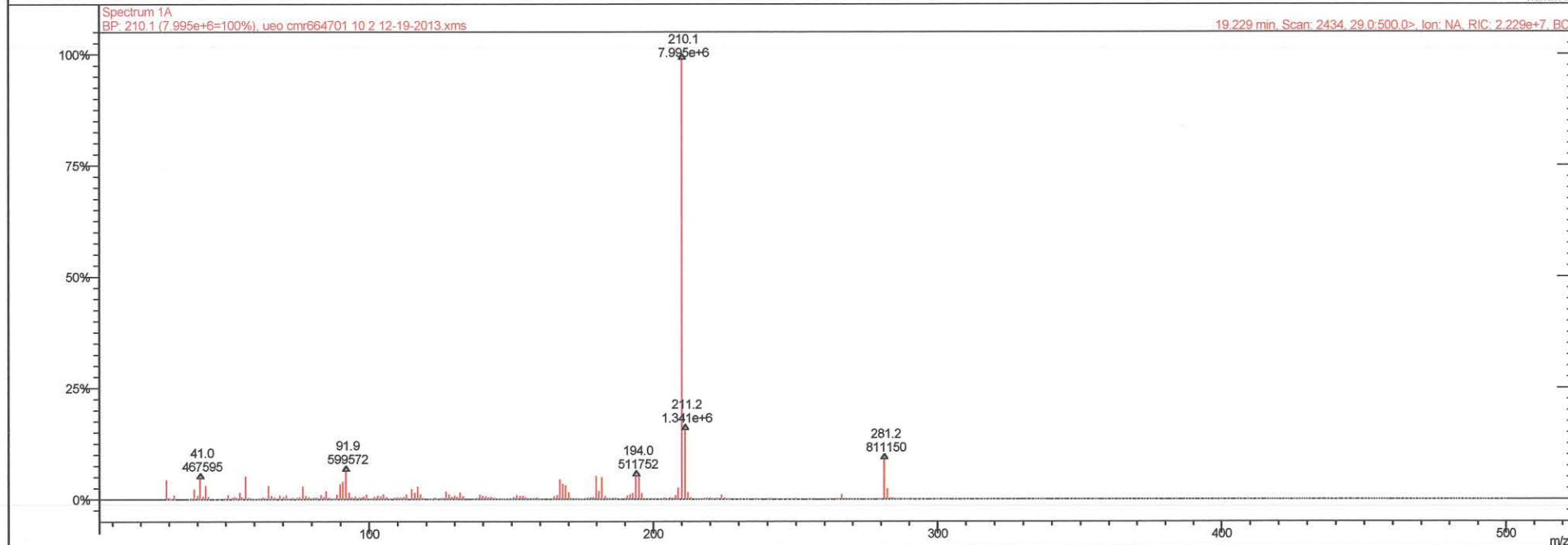
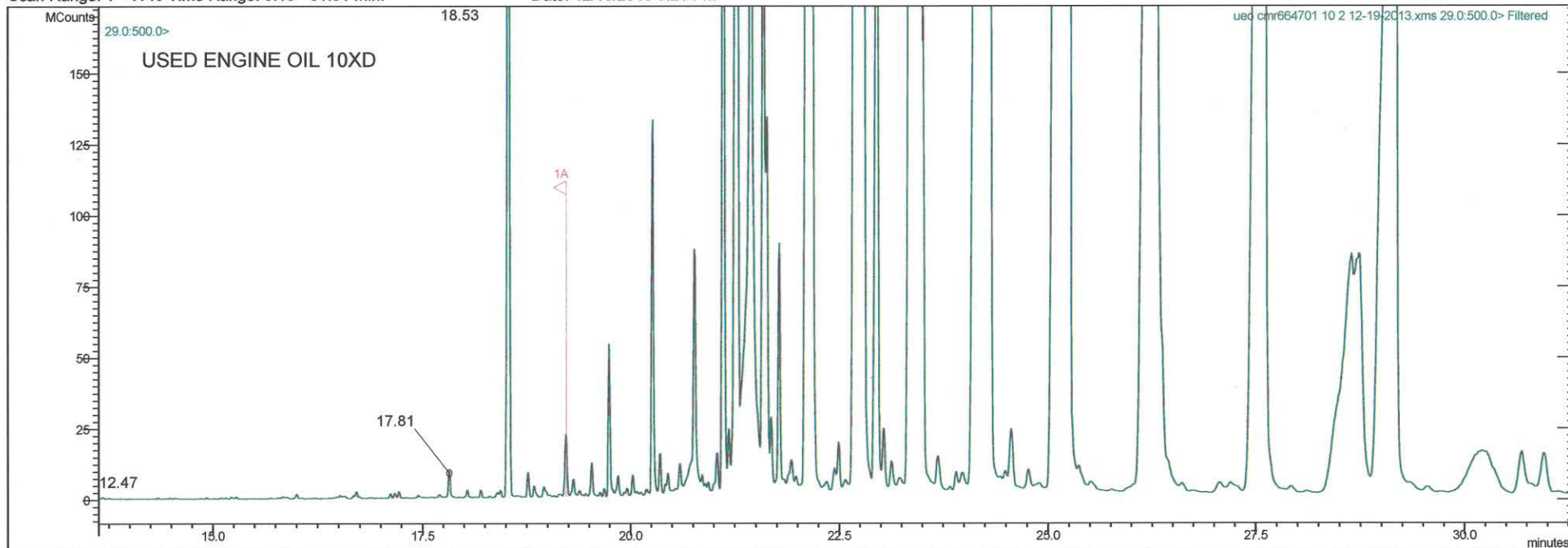
Entry 117922 from [REDACTED]

R.Match: 964, F.Match: 960



MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:50 AM

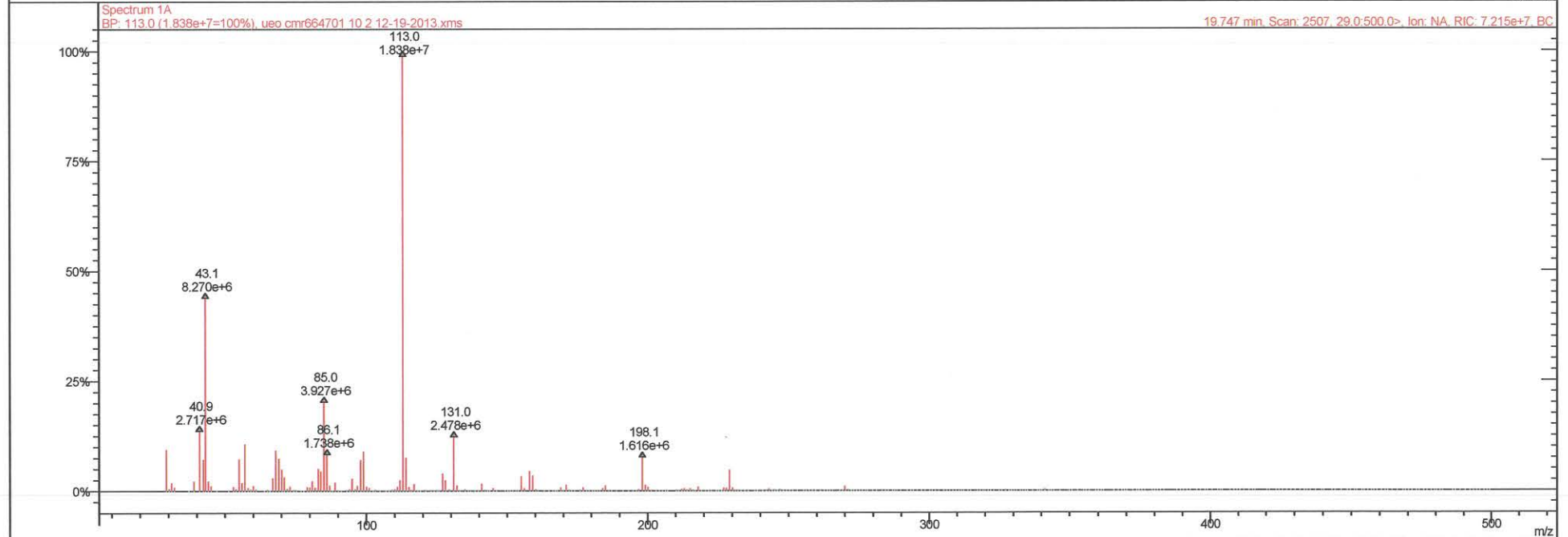
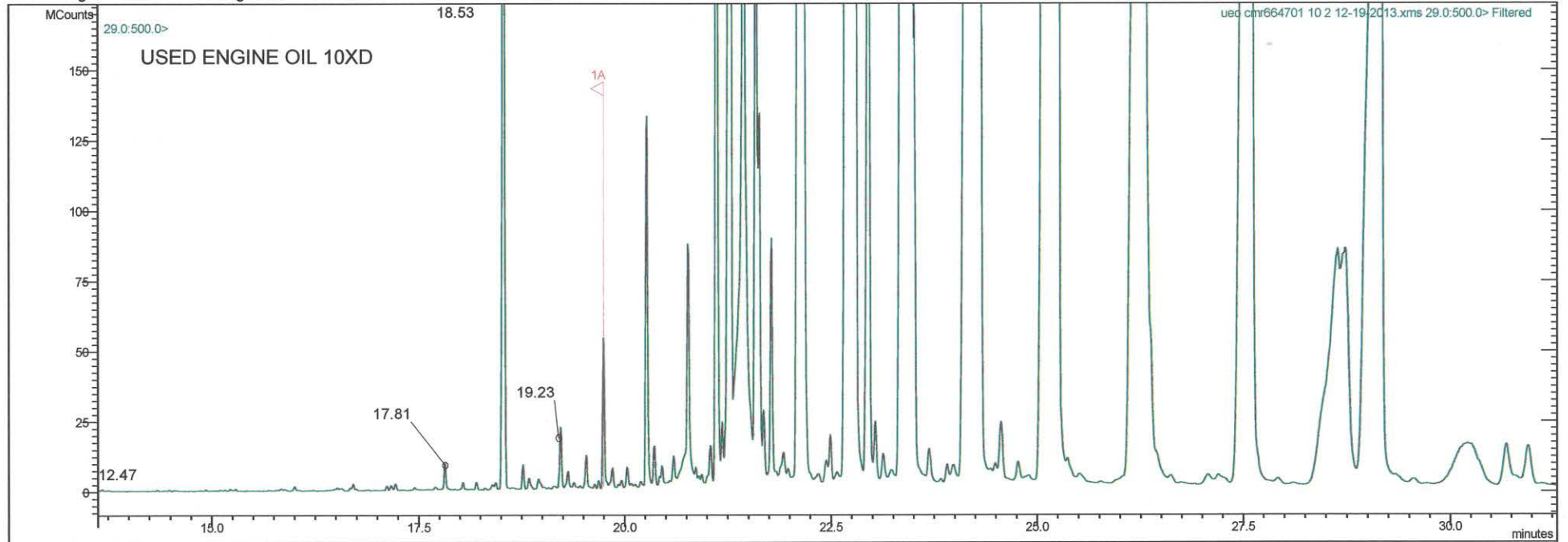
File: [REDACTED]  
Sample: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM





MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:55 AM

File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM



### MS Data Review Active Chromatogram and Spectrum Plots - 12/20/2013 9:55 AM

File: [REDACTED] Operator: CG  
Scan Range: 1 - 4146 Time Range: 3.13 - 31.31 min. Date: 12/19/2013 5:21 PM

