



# U.S. EPA Method 8270 for multicomponent analyte determination

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## Introduction

Multicomponent analytes are compounds that yield several peaks when chromatographed. The pattern of peaks is characteristic, but the relative intensities can vary due to weathering of the sample. They are typically measured using gas chromatography (GC) with a specific detector, such as an electron capture detector (ECD), which is especially sensitive for compounds containing halogens. High sensitivity is necessary to measure low levels of these compounds because the area is distributed among multiple peaks. U.S. EPA Methods 8081A and 8082 prescribe methodology for this approach (1,2). While the ability to positively identify sample analytes can be accomplished with the use of two columns, with the second used for confirmation, it is not necessarily the most desirable of options. In many cases, the confirmational column alone is not sufficient and additional cleanup procedures must be performed to eliminate co-eluting analytes. The additional equipment and analysis time required places productivity burdens on a laboratory.

Gas chromatography/mass spectrometry (GC/MS) is widely used in environmental analyses because its selectivity enables positive identification without additional sample processing. Along with the ability to make qualitative determinations, GC/MS is an invaluable tool for

providing quantitative results. Mass spectrometry methods, however, are generally considered less sensitive than conventional detector methods, although sensitive enough for most applications. The analysis of multicomponent analytes, such as Toxaphene and the Aroclors is more of a challenge. U.S. EPA Method 8270C is a direct injection method for extracted semivolatile compounds. The method states, "In most cases, Method 8270 is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. When these analytes have been identified by another technique, Method 8270 is appropriate for confirmation of the presence of these analytes when concentration in the extract permits." (3)

The development of more sensitive quadrupole mass spectrometry technology, along with innovative sample introduction techniques, allows for the quantitation of many of these analytes at levels previously not achievable. This work illustrates the ability of quadrupole mass spectrometry to quantitate multicomponent analytes at these lower levels. The ability to accurately identify and quantitate using GC/MS can eliminate the need for additional confirmatory analyses and reduce the amount of sample preparation required.

## Experimental

Identical standards were analyzed using two sets of experimental conditions. A 50-mL large-volume injection using the solvent purge mode was utilized in both cases. The sample is injected into the Programmable Split/Splitless (PSS) liner and held at 55°C for 4 minutes to allow the solvent to be purged through the open split vent. The less volatile analytes remain in the liner. The split vent is closed and the injector is rapidly heated to 250°C, vaporizing the analytes and transferring them to the chromatographic column.

One set of standards was analyzed using the GC/MS Full Scan mode (FS-50) and the second using the Selected Ion Recording mode (SIR-50), generally referred to as SIM. Table 1 lists the chromatographic conditions used for both experiments, while Tables 2 and 3 list the mass spectrometer conditions used for each scan mode. The results are evaluated with respect to the accepted standard analytical techniques.

**Table 1. Chromatographic Condition**

<b>PerkinElmer AutoSystem XL™</b>	
Column	PE-5MS 30 m x 0.25 mm; 0.25 mm film thickness
Pre-Column	1 m x 0.32 mm deactivated fused silica
Oven Temperature Program	55°C for 5 min., 45°C/min. to 160°C; 6°C/min to 320°C
Programmable Pneumatic Control (PPC)	Helium 1.0 mL/min.
Programmable Split/Splitless (PSS) Injector	55°C for 4 min.; ballistic to 250°C; Solvent Purge Mode
Injection Volume	50 µL
Solvent	Hexane

**Table 2. Full Scan Mass Spectrometer Conditions**

<b>FS-50 PerkinElmer TurboMass™ Mass Spectrometer</b>	
Mass Scan Range	50 - 350 m/z
Scan Speed	2.0 scans/sec
Filament Delay	5 min.
Ion Source Temperature	150°C
Transfer Line Temperature	250°C
Ionization Mode	El

**Table 3. Selected Ion Recording (SIR) Mass Spectrometer Conditions**

<b>SIR-50 PerkinElmer TurboMass Mass Spectrometer</b>	
Selected Scan Masses	159, 231, 233 m/z
Scan Speed	2.0 scans /sec.
Filament Delay	5 min.
Ion Source Temperature	150°C
Transfer Line Temperature	250°C
Ionization Mode	El

## Results and Discussion

Traditionally, a single GC peak is available and used for quantitation of a compound. Retention time identifies the peak and the height or area is compared to a known standard for quantitation. In GC/MS, retention time and spectral matching identifies the analyte and a single characteristic ion is compared to a standard for quantitation. With a multicomponent analyte, it is more accurate to integrate several chromatographic peaks to determine a representative value. The relative size of the peaks may vary due to weathering, but the average will remain relatively constant. Since Method 8270 does not address multicomponent analyte quantitation, criteria from Method 8081 were combined with criteria from Method 8270 to demonstrate adequate performance.

Toxaphene standards at 0.10 ng/mL, 0.20 ng/mL, 0.50 ng/mL, 1.00 ng/mL, and 5.00 ng/mL concentrations in hexane were analyzed using both measurement modes. The chromatograms shown in Figure 1 were obtained using the SIR mode. All the calibration standards clearly exhibit the characteristic Toxaphene pattern. Four chromatographic peaks were selected and determined as representative of the multicomponent analyte Toxaphene. Calibration factors (CF) were calculated based on the integrated peak areas and the known standard concentrations. From these results, the relative standard deviation (RSD) for each multilevel concentration range was determined. These results were averaged providing a final Toxaphene RSD. Correlation coefficients were calculated in a similar fashion and are illustrated in Figures 2 and 3.

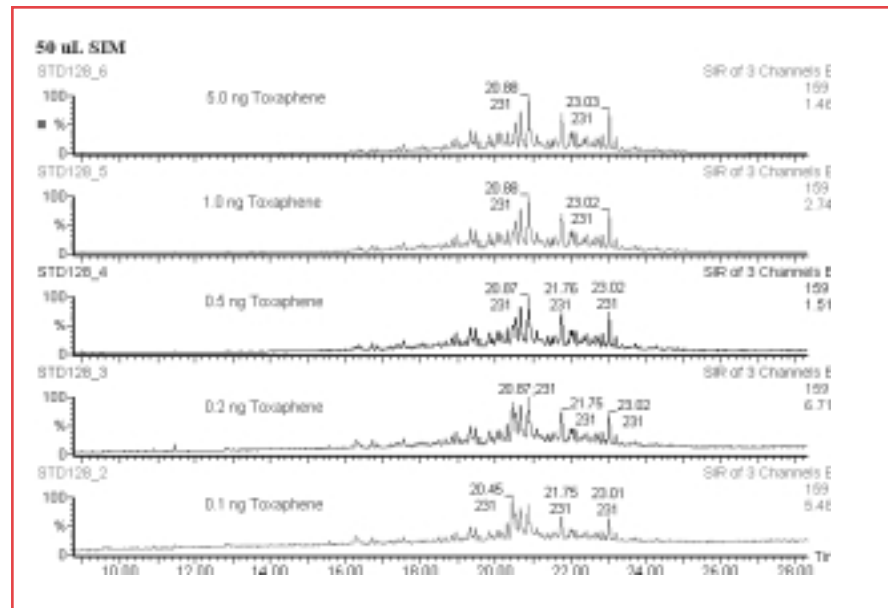


Figure 1. Calibration Standards show recognizable pattern for all levels.

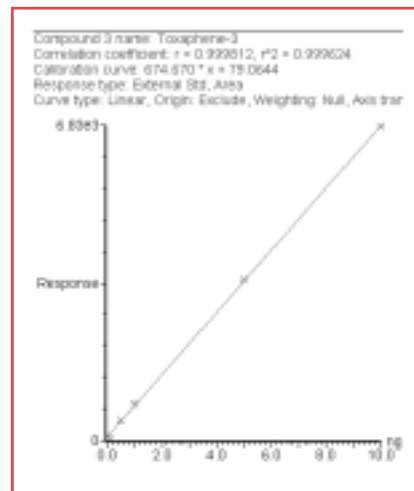


Figure 2. Toxaphene peak #3 calibration curve.

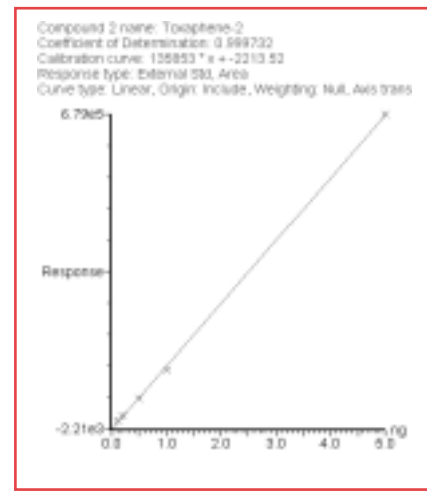


Figure 3. Toxaphene peak #2 calibration curve.

The results of the calibration data and acceptance criteria are listed in Tables 4 and 5. Both experimental results easily comply with method performance specifications.

The Method Detection Limits (MDLs) listed in Table 6 are the result of seven (7) replicate injections of a 0.10-ng/mL standard using the standard deviation and the *t*-statistic. The reported MDL is an average of the value for each of the four peaks chosen to represent the compound.

**Table 4. Comparison with Calibration Acceptance Criteria Using Full Scan Mode**

Calibration Peaks	Full Scan-50			
	RSD (%)		Correlation Coefficient	
	Actual	Acceptance Limit	Actual	Acceptance Limit
Peak #1	11.0		0.99934	
Peak #2	13.6		0.99949	
Peak #3	12.6		0.99962	
Peak #4	8.4		0.99948	
Toxaphene (Average of 4 peaks)	11.4	15.0	0.9995	0.99

**Table 5. Comparison with Calibration Acceptance Criteria Using Selected Ion Mode**

Calibration Peaks	SIR-50			
	RSD (%)		Correlation Coefficient	
	Actual	Acceptance Limit	Actual	Acceptance Limit
Peak #1	10.4		0.99936	
Peak #2	8.2		0.99973	
Peak #3	10.8		0.99967	
Peak #4	7.5		0.99934	
Toxaphene (Average of 4 peaks)	9.2	15.0	0.9995	0.99

**Table 6. Calculated Detection Limits Based on a 1-liter Sample Concentrated to 1mL**

Calibration Peaks	Calculated Analytical Detection Limits (ppm)		Maximum Allowable Concentration Limit (ppm)
	FS-50	SIR-50	MCL
Peak #1	0.073	0.065	
Peak #2	0.089	0.009	
Peak #3	0.105	0.021	
Peak #4	0.035	0.014	
Toxaphene (Average)	0.076	0.027	3.

Integrated peaks representative of the entire calibration range can be seen in Figure 4. The bottom chromatogram was obtained from a 0.05-ng/mL standard which is below the lowest calibration standard of 0.10 ng/mL. The peaks are readily discernible above the noise and can be easily integrated.

What sets the mass spectrometer apart from other forms of detectors is the ability to selectively identify individual masses. Figure 5 shows the Total Ion Chromatogram (TIC) of a mixture of 100 ng/mL Toxaphene and 0.10 ng/mL pesticide mix. The Extracted Ion (EI) mass 159 is Toxaphene, and the Extracted Ion (EI) mass 66 is Aldrin, which was confirmed by a NIST library search as seen in Figure 6. Aldrin is easily identified and integrated without additional preparatory procedures.

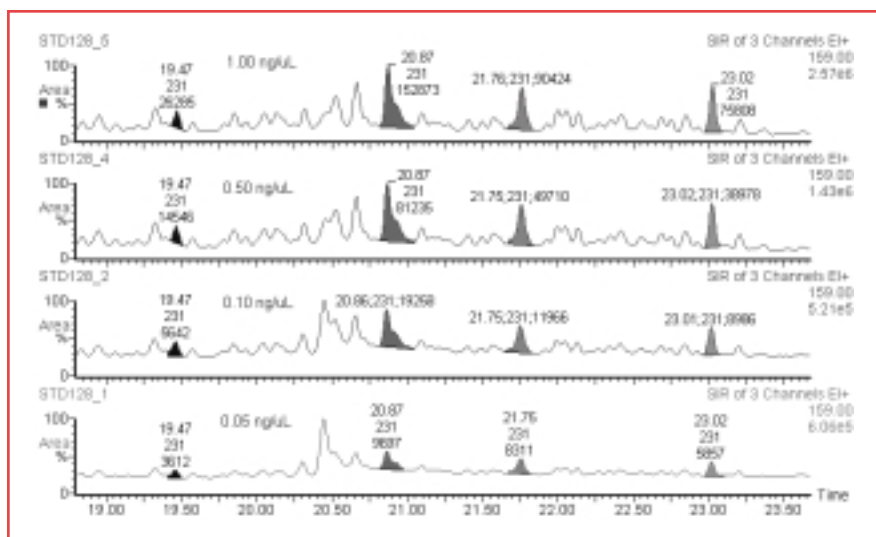


Figure 4. Integrated toxaphene peaks.

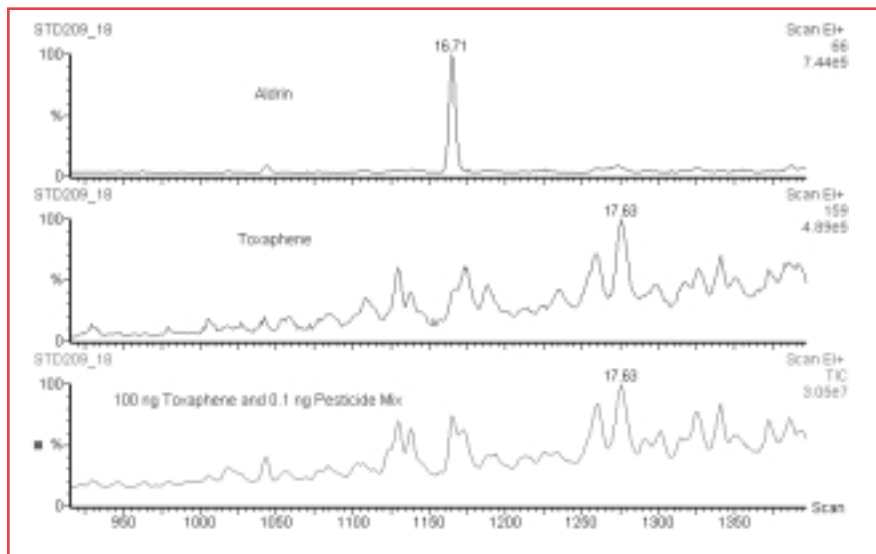


Figure 5. Aldrin and toxaphene extracted ions.

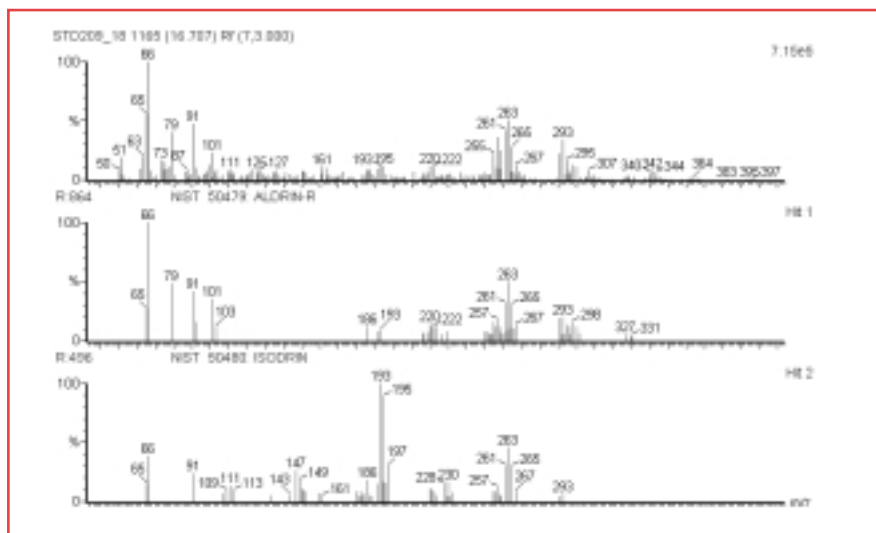


Figure 6. Library searchable spectra.

The Contract Laboratory Program (CLP) lists 0.2 ng/mL as the quantitation limit for Aroclor 1221 using an Electron Capture Detector. Figure 7 shows Aroclor 1221 well above the noise level at the 0.20-ng/mL quantitation level, using GC/MS in the SIR mode and large-volume injection.

## Conclusions

The ability of GC/MS to selectively identify a component based on an extracted ion chromatogram from a mixture of compounds not only assures a positive identification, but also saves time by eliminating additional cleanup and analyses.

Recent technological advances in quadrupole mass spectrometry have increased the instrument's sensitivity. The use of Selected Ion Recording provides further sensitivity enhancements. In addition to the detector and its mode of operation, the use of large-volume injection with a programmable inlet system allows for introduction of larger sample volumes.

The combination of these elements enhances the sensitivity of a GC/MS system so multicomponent analytes can be identified and quantified in an efficient and productive manner.

The TurboMass mass spectrometer allows the collection of EI and SIR data simultaneously. Although this feature was not used here, it can provide further productivity increases by combining the ability to collect library-searchable spectra with sensitive data for quantitation.

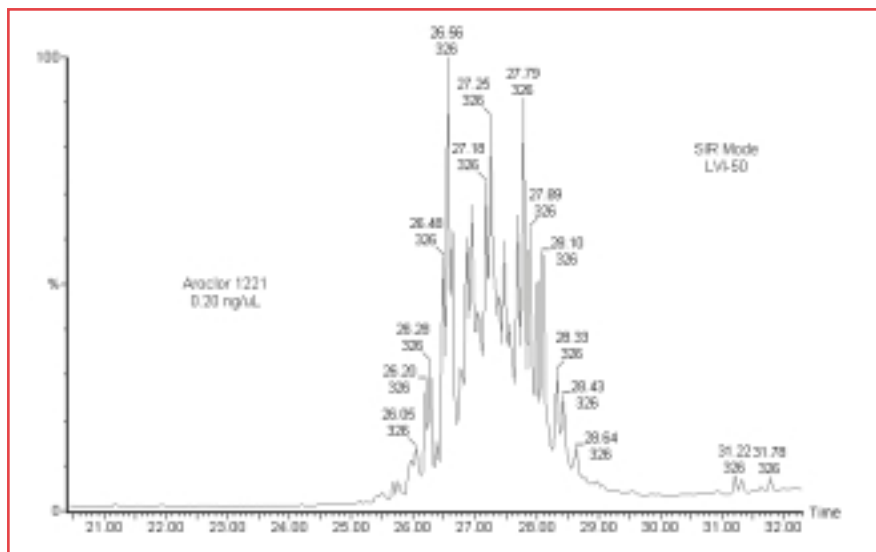


Figure 7. Quantitation limit pattern recognition.

## References

1. Method 8081A, Organochlorine Pesticides by Gas Chromatography, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846).
2. Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846).
3. Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846).

  
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