



## Calibration Curve Linearity for DDT Using GCxGC-ECD

### **Introduction**

Linear dynamic range can be defined as a detector's response increasing proportionally with increased quantity of detected material. Linear dynamic range is important in any detection system for gas chromatography (GC) to minimize sample handling (i.e. dilutions and concentrations) and number of analyses. The linear dynamic range of an electron capture detector (ECD) is usually defined as being around  $10^4$ . Recently an evaluation was performed to test the linearity of a LECO GCxGC-ECD system for 4,4'-DDT, an important organochlorine pesticide that is routinely monitored in environmental samples.

GCxGC (comprehensive two-dimensional GC) is a way to increase peak capacity by applying two independent separations to a sample in one analysis with one detector. GCxGC involves serially connected columns (differing phases) separated by a thermal modulator. A separation is performed on the first column, and then effluent from the first column is continually (and quickly) focused and "injected" onto the second column. By keeping the second column short, a series of high-speed chromatograms are generated, and the first column separation can be maintained. GCxGC has the power to improve sensitivity for a compound, since thermal focusing near the detector results in peak sharpening.

This performance note defines the linearity of GCxGC-ECD with a calibration curve generated for 4,4'-DDT.

### **Standards**

DDT was in an organochlorine pesticide mix from Restek Corporation. Pentachloronitrobenzene used as an internal standard was also obtained from Restek.

### **Experimental Conditions**

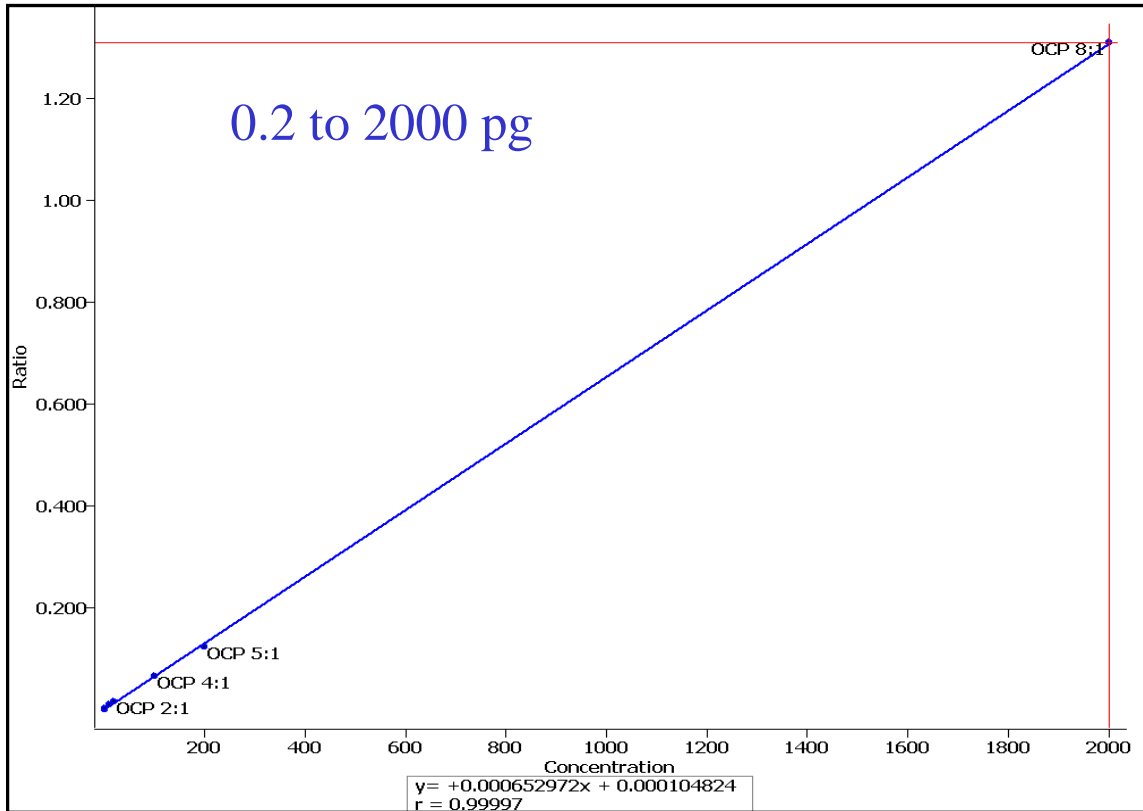
#### **LECO GCxGC-ECD**

*Agilent 6890 GC-ECD equipped with a LECO Quad Jet—Dual-Stage Thermal Modulator*

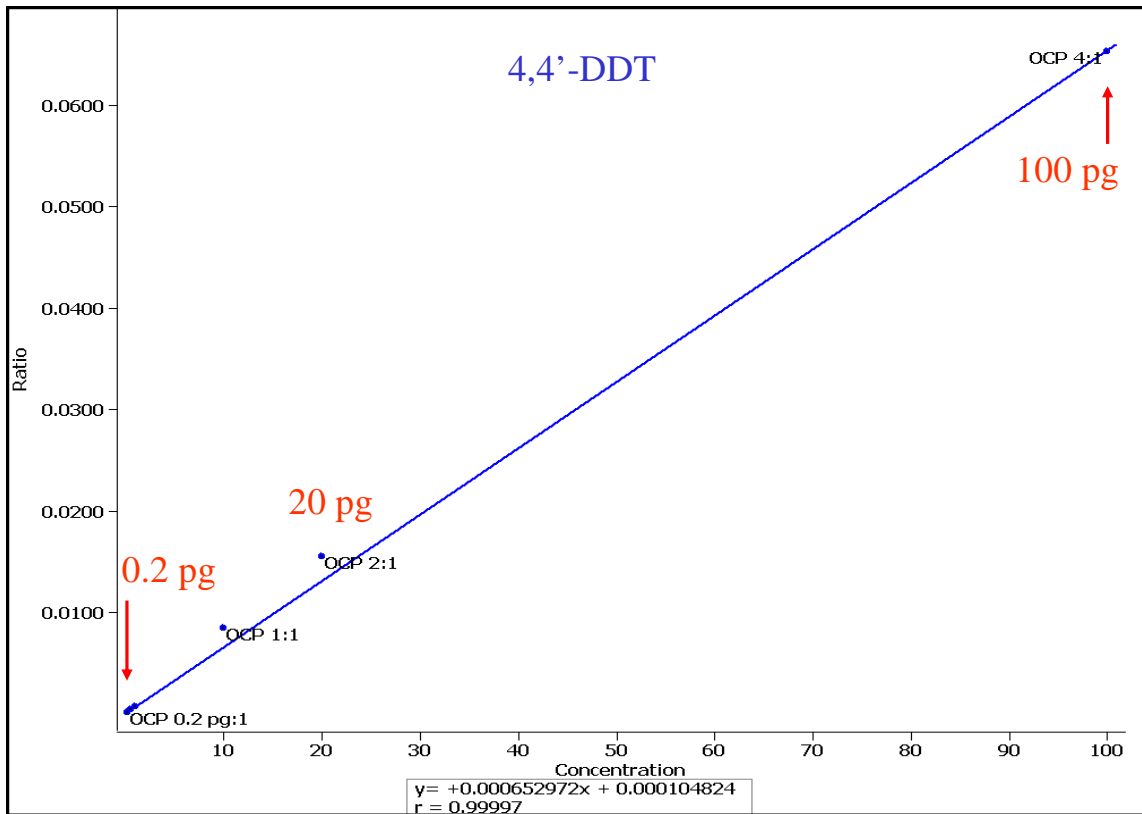
Column 1:	10 m x 0.18 mm x 0.20 $\mu$ m Rtx-5 (Restek)
Column 2:	1.1 m x 0.10 mm x 0.10 $\mu$ m DB-17 (J&W Scientific)
Carrier:	Helium at 2 mL/min, constant flow
Injection:	1 $\mu$ L split at 250°C, split ratio 20:1
Oven 1 Program:	40°C (1 min), 10°/min to 290° (1 min)
Oven 2 Program:	10°C offset from oven 1
Modulation time:	4 sec.
Detector:	ECD, 325°C, N2 makeup gas at 148 mL/min, 50 Hz

## Results and Discussion

Figure 1 is the internal standard calibration curve plotted from the analysis of 0.2, 0.5, 1.0, 10, 20, 100, 200, and 2000 pg/ $\mu$ L 4,4'-DDT standards. Linearity is excellent. Closer inspection of the lower end of the curve is illustrated by Figure 2. It is important to note that since a split injection of 20:1 was used, the actual amount of DDT on column for the lowest calibration point is only 10 fg.



**Figure 1.** Internal standard calibration curve for 4,4'-DDT analyzed using GCxGC-ECD.



**Figure 2.** Zoom of lower end of internal standard calibration curve for 4,4'-DDT analyzed with GCxGC-ECD.

### Conclusion

The linear dynamic range for GCxGC-ECD has been determined to be at least  $10^4$  for 4,4'-DDT.



LECO Corporation • 3000 Lakeview Ave. • St. Joseph, MI 49085-2396  
 Phone: 800-292-6141 • Fax: 269-982-8977 • info@leco.com • www.leco.com  
 ISO-9001: 2000 • No. FM 24045

