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Analysis of a Complex Garlic Extract for Chlorinated Pesticides Using the OI Analytical Model 5360 Halogen Specific Detector

Application Note 15460700

Keywords
Chlorinated Pesticides
Garlic
Gas Chromatograph
Halogens
Sulfur Interference
XSD

Complex sample matrices present one of the most common problems associated with the analysis of food products. The resulting extracts typically contain an unusually high number of co-extractants as interference. Analysis of garlic extracts for chlorinated pesticides is an example of such a case. Garlic extracts contain many suflur-containing compounds as co-extractants, which can obscure the chlorinated pesticides on an ECD chromatogram. Figures 1 and 2 are characteristic of the types of complex ECD chromatograms that can be expected from a garlic extract. It is clear that the presence of multiple sulfur co-extractives will create a complex ECD chromatogram, and it is difficult to discriminate between the target compounds and the background interferences.

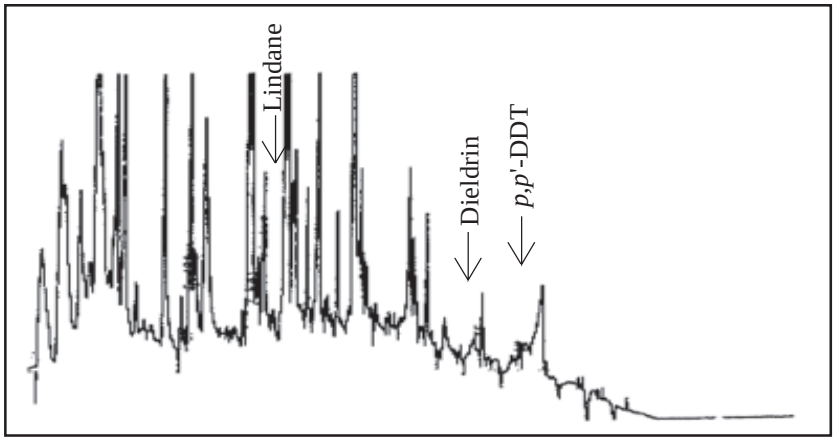

Figure 1. ECD Chromatogram of a "Typical" Garlic Extract on a 15-m DB17 Column Following Florisil Cleanup (FDA methodology). Expected Retention Times (RT) for Three Chlorinated Pesticides are Indicated. (1-uL injection, split 9:1)

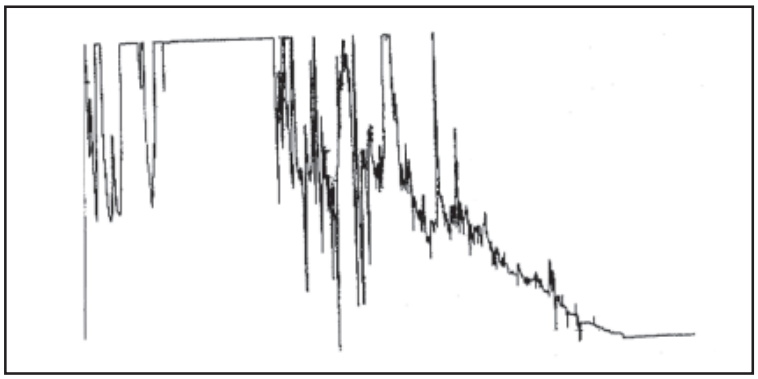

Figure 2. ECD Chromatogram of a "Worst Case" Garlic Extract Following Florisil Cleanup (1-uL injection, split 9:1)

A highly selective detector that responds to the chlorinated pesticides but does not respond to the sulfur interference is the ideal solution for this case. The OI Analytical Model 5360 Halogen Specific Detector (XSD™) has been specifically developed for the quantitation of halogenated compounds on a gas chromatograph. The XSD is operated in an oxidative mode that converts the halogens to free halogen atoms, which are then adsorbed onto an activated platinum probe, resulting in an increased thermionic emission and a measurable output current. In this case, the XSD responds only to the halogens in the chlorinated pesticides, and all interference from the sulfur co-extractives is completely eliminated. Figure 3a shows a chromatogram of the same "worst case" garlic extract shown in Figure 2 run on an OI Analytical XSD. The sulfur interference is totally eliminated, and the target pesticide peaks are now easily identified. In Figure 3b, the extract has been spiked with a pesticide standard containing atrazine, lindane, and chlorpyrifos at 6.9–9.2 pg. The negative spike at ~7.5 minutes is due to elution of a large volume of hydrocarbon. Figure 4 shows an XSD chromatogram of the OI Analytical pesticide standard with the detectivity calculated for atrazine, lindane, and chlorpyrifos at less than one picogram chlorine per second (< 1 pg Cl/sec).

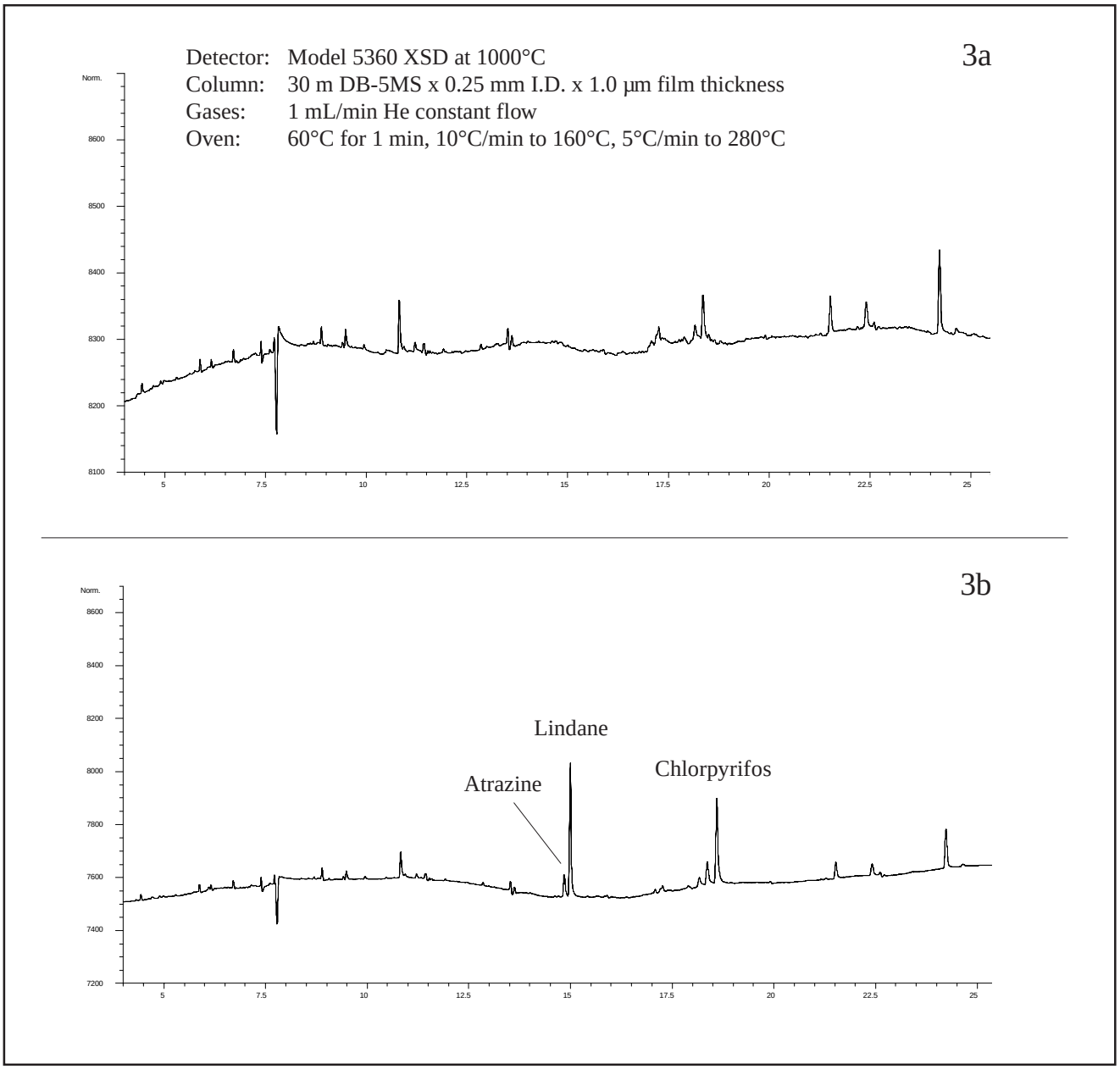

Figure 3. Chromatogram of the Same "Worst Case" Garlic Extract Run on an OI Analytical Model 5360 Halogen Specific Detector (1-uL injection, split 9:1). The Bottom Chromatogram has been Spiked with Atrazine, Lindane, and Chlorpyrifos.

Compound	pg Cl on Column	S/N	Detectivity
1. Atrazine	8.2	16	0.45 pg Cl/sec
2. Lindane	36.6	83	0.41 pg Cl/sec
3. Clhorpyrifos	45.5	69	0.51 pg Cl/sec

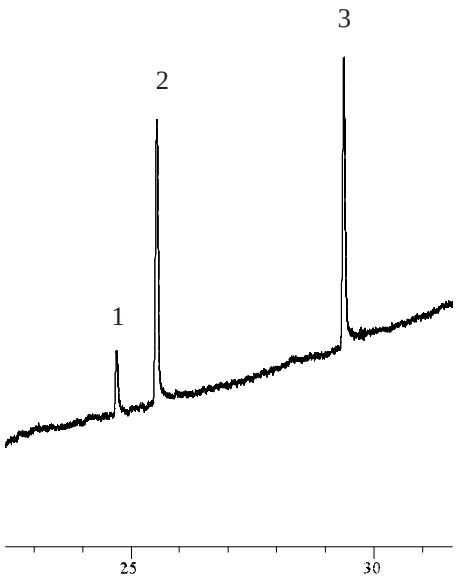

Figure 4. Chromatogram of a Pesticide Standard on the XSD (1-uL injection, split 9:1). Detectivity is Calculated at < 1 pg Cl/sec.

The OI Analytical Model 5360 Halogen Specific Detector (XSD) is an excellent choice for halogen selectivity when sulfur is an expected interference. The XSD is highly selective for halogen containing compounds such as chlorinated pesticides, has a detectivity of less than one picogram chlorine per second, and does not respond to interfering sulfur co-extractives.

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