



FIELD ANALYSIS OF ELEMENTAL SULFUR IN DRYWALL BY GC/ECD



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ABSTRACT

Complaints of rotten egg odors had been reported by residents in houses built with Chinese drywalls. This occurred during 2003-2006 primarily in Florida and Louisiana. Preliminary studies indicated that the Chinese drywalls were tainted with reduced sulfur containing compounds. The Environmental Protection Agency's Environmental Response Team along with its Response Engineering and Analytical Contract (ERT/REAC) analyzed a limited number of drywall samples using a modified routine GC/MS method for semi volatile organic compounds and found that elemental sulfur was present in most of the Chinese drywall samples. Elemental sulfur was observed as a hump on the chromatogram which interfered with the routine Base Neutral and Acid Extractable (BNA) analysis. The detection of sulfur achieved by integrating the hump using specific sulfur ions such as 64, 128, and 192; however, detection was limited to the ppm (mg/kg) level. In order to quickly quantify sulfur concentrations, the laboratory developed a quick screening method using hexane extraction in conjunction with GC/ECD analysis. With this technique elemental sulfur was observed as a distinct peak in the chromatogram at low ppb (µg/kg) levels, thus making quantification easier than integrating the total area under a hump during the GC/MS analysis. Elemental sulfur can easily be extracted from drywall samples and analyzed down to a detection limit of 125 µg/kg. The extraction and analysis procedures can be completed within 30 minutes per sample per instrument. The result of this study and analytical method are presented in this paper.

INTRODUCTION

Drywall from China was imported to address the shortage of essential construction materials created following 2004 and 2005 hurricane seasons and a national demand for new home construction. Based on press report estimates, as many as hundreds of thousands of houses may have been affected; it is uncertain how many homes have imported drywall in them. According to the print media newspapers, the imported drywall contains reduced sulfur compounds, which are suspected to be the basis for odor and health complaints. It has been reported that as the drywall is exposed to elevated levels of humidity, as well as elevated temperatures, the product may be off-gassing sulfur-based compounds that have been associated with the odor, health, and corrosion complaints. Compounds reported to be detected in the suspect drywall include reduced sulfur compounds.

METHOD SUMMARY

A piece of the drywall is ground using a mortar and a pestle, and approximately 1 gram (g) of the powder is transferred to a 10 ml vial with 5 ml of hexane and it is mixed for about 2 minutes using a Vortex. After the extract is settled for about 10 minutes, 1 ml of aliquot is transferred to 1 ml injection vial. The extract is analyzed for elemental sulfur using GC/ECD after adding 20 : 1 of 5 µg/mL 4,4'-dibromobiphenyl internal standard. In comparison, methanol was also tested to extract sulfur from the drywall samples.

MDL Study for Elemental Sulfur in Drywall	
Spiked at 250 µg/Kg, n =	7
Average Conc.(µg/Kg) =	194
Average Recovery (%) =	78
Standard Deviation. (d) =	11
Theoretical MDL (3 d) =	32

GC/ECD OPERATING CONDITIONS

The operating conditions used for standards and sample analysis on the Agilent 6890 GC/ECD are listed below. Other conditions may be used as long as QA/QC and peak identification criteria are met.

Column ID	Zebron ZB-XLB & RTX-CLP, 30 meter x 0.32 mm 0.5 microns (: m) film thickness
Injector Temperature	250EC
Temperature Program	120EC for 1 minutes 9EC/min to 285EC hold for 10 minutes
Injection Mode	1 : L Pulse Injection

DISCUSSION

The GC/MS method can easily identify the elemental sulfur compound with its mass spectra and the shape of the compound shown in the chromatogram. However, the tailing hump shape of sulfur would require manual integration and the detection limit is at the mg/Kg level. The GC/ECD method has the sulfur linear response at $r^2 = 0.997$ and the theoretical method detection limit at 32 µg/L in the hexane extract. Using our quick screening method, the drywall can be reported with 125 µg/Kg reporting limit. One drywall was extracted with hexane and methanol separately in 5 replicates. These

Elemental Sulfur Analysis By GC/ECD Using Different Solvents		
	Solvent Used	
	Hexane	Methanol
Average Conc (mg/kg)=	247	240
Number of Samples=	5	5
Standard Deviation=	26.5	26.6

extracts were analyzed using GC/ECD method and the results of sulfur concentrations did not show any significant difference. To develop a field screening method for determining the sulfur compound in drywall, the GC/ECD is more compact in term of lab space requirement and less sophisticated to operate. The total of the analytical time can be trimmed to half of an hour or less per sample on the GC/ECD run. In conclusion, GC/ECD in combination with either hexane or methanol extraction is an ideal method for sulfur screening in the drywall if the need of analysis is warranted.

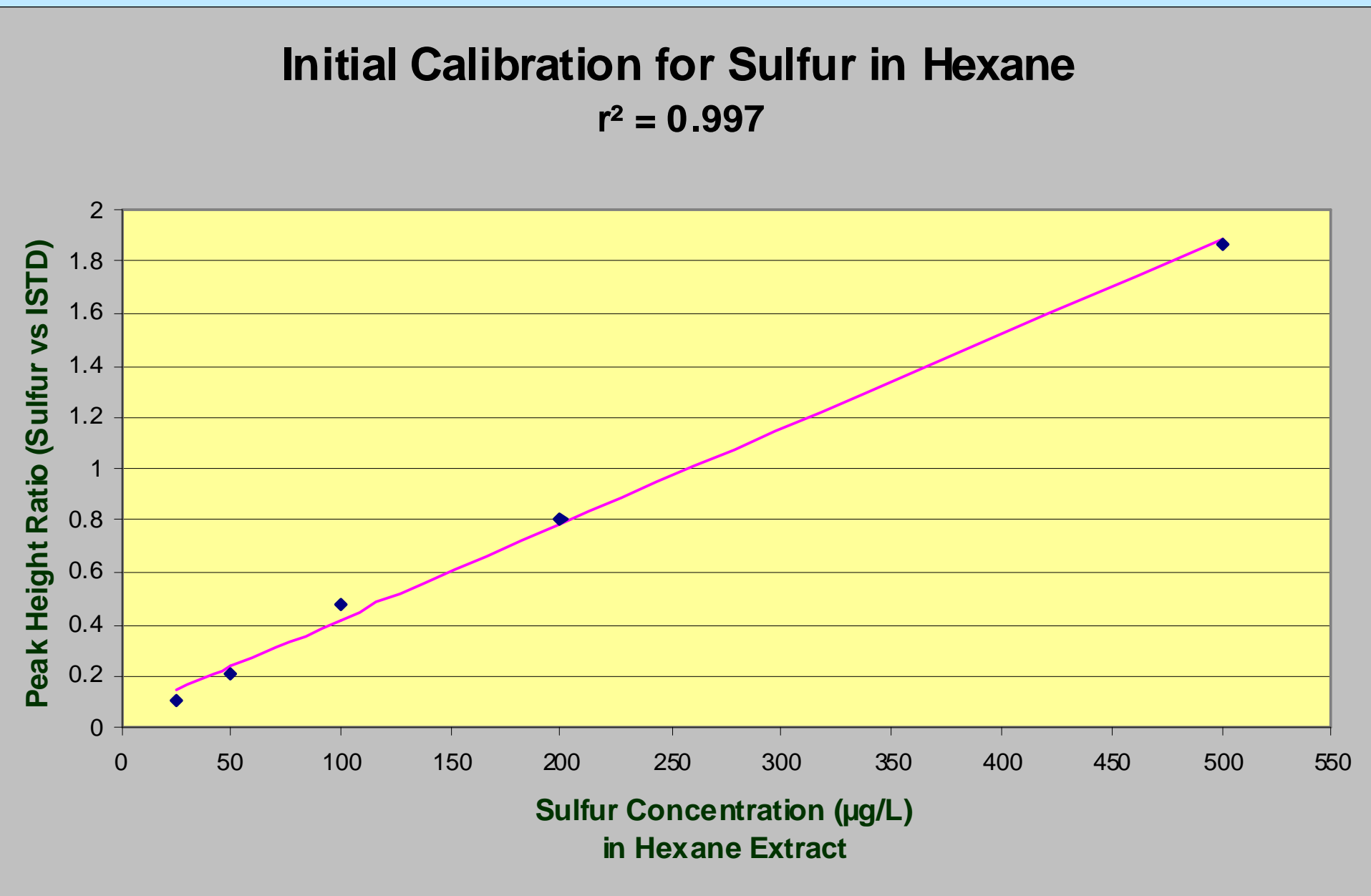
FUTURE WORK

The future work for this screening method is to further reduce the analysis time in the field by modifying the temperature program for the oven and using only one internal standard in the extracts. Portable GC equipped with ECD detector is also being considered for field operation.

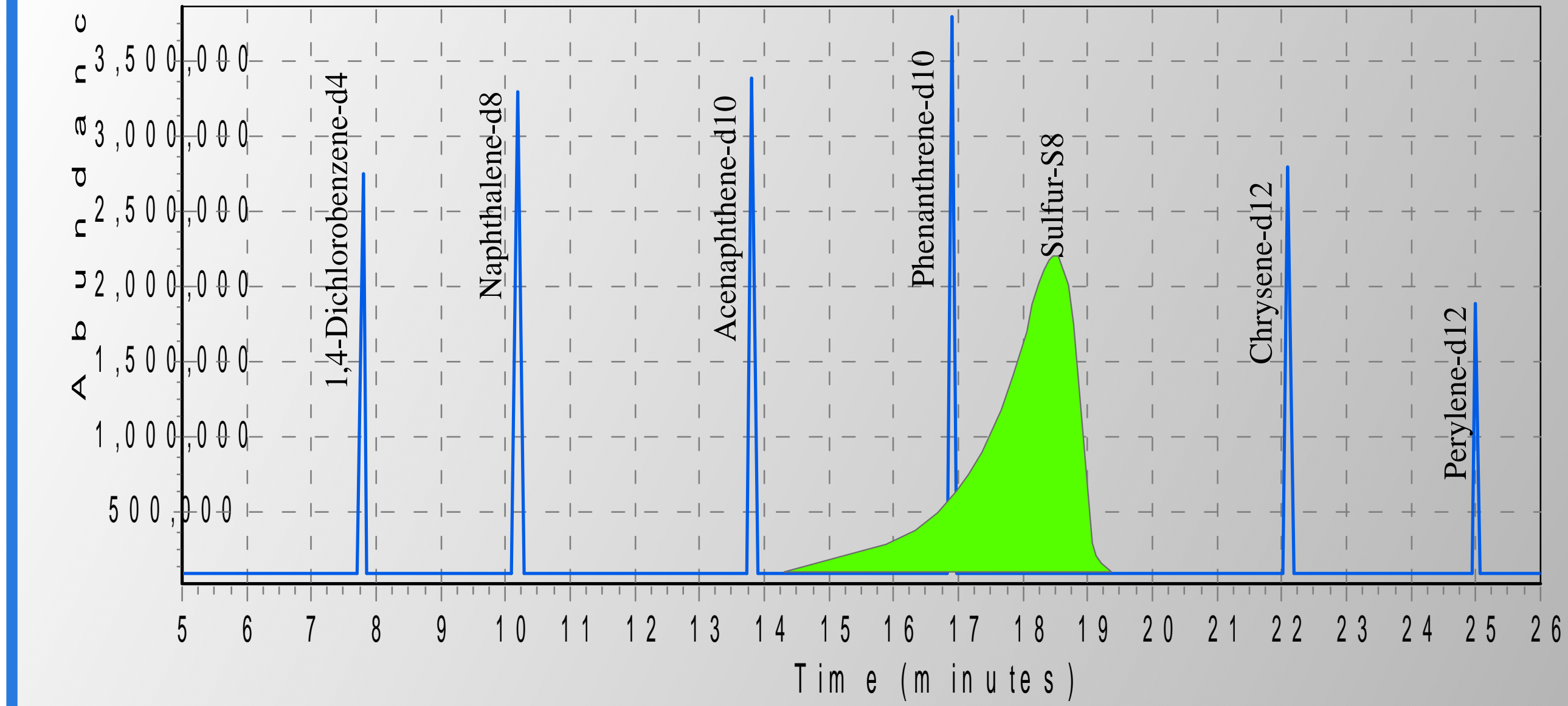
ACKNOWLEDGEMENT AND DISCLAIMER

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GC/ECD Elemental Sulfur Linearity



GC/MS Chromatogram : 500 ppm Elemental Sulfur in DC



Drywall Grinding



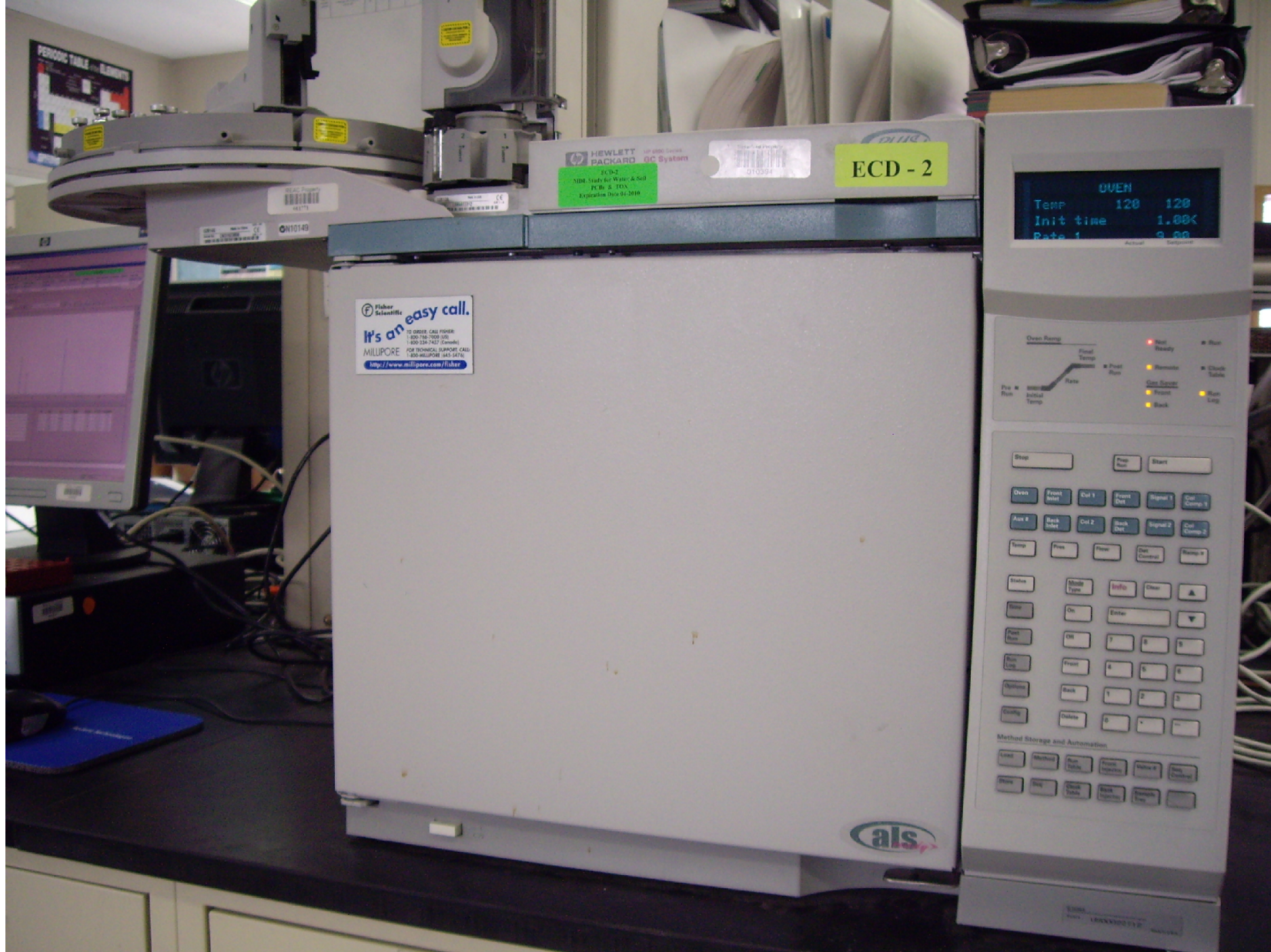
Drywall - 1 gm Aliquot



Vortex Extraction - 1 gm drywall in 5ml hexane



GC/ECD Instrumentation



GC/ECD Chromatogram : 100ppb Elemental Sulfur in Hexane

