the CDS Sampler
Product News & Application Notes From CDS
WINTER 2010

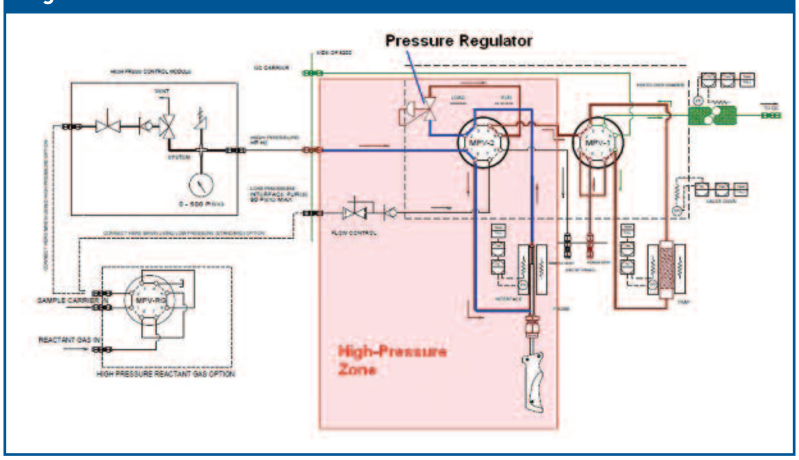
Another CDS First: High Pressure Pyrolysis with Catalytic Reactor

The Pyroprobe 5200 HP-R, our small-scale high-pressure pyrolysis reactor system, isn't just the first of its kind; it's one of a kind. This dynamic instrument incorporates analytical pyrolysis with the ability to process samples in reactant gas, at elevated pressure, and then send the products made through an independently-controlled catalytic reactor.

Scientists studying new biomass feedstock, coal, oil, or polymer reactions, need to understand how these materials break down in a reactor under various conditions. The 5200 HP-R allows them to study both high temperature and high pressure on a small scale.

This innovative system offers scientists, and chemical engineers, time and money efficiencies by increasing their reaction chemistry understanding before making the financial commitment required for a large laboratory or a pilot reactor. The instrument is highly flexible; for example, background gas can be GC carrier gas or a reactant gas such as air, hydrogen, or oxygen.

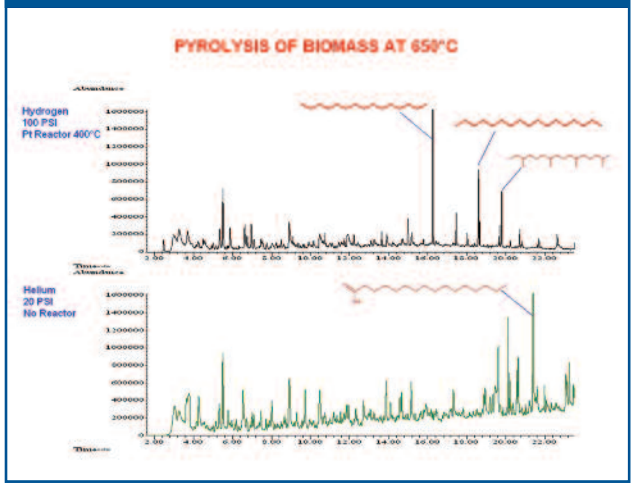
Figure 1



Pyroprobe 5200 HP-R pneumatics. One system valve controls whether Pyroprobe is on-line or off-line with the high pressure/reactor system, while the other determines if trap is on-line or off-line from the GC.

Traditionally, pyrolysis occurs in the GC carrier gas. The HP-R system enables users to pyrolyze samples at an elevated pressure of any other gas, for example 300 PSI of hydrogen or oxygen, then send the products through a catalyst bed, such as platinum at 500°C, and analyze the final products by GC.

Figure 2



Comparison of pyrolyzing a sample of biomass at 20 PSI helium in the GC carrier and through the Pt reactor in hydrogen at 100 PSI.

The Py-GC run is dominated by pyrolysis products of cellulose and lignin, and has a large peak for a fatty acid. Using the reactor system, the fatty acids are converted to normal hydrocarbons, which are now the major peaks seen in the chromatogram. The 5200 HP-R permits varying the pressure and flow of the reactant gas, as well as the temperature of the catalyst reactor. Having the ability to change these variables allows users to study the effects on products generated.

GCMS Libraries For Polymers, Additives & Biofuel Compounds

Pyrolysis-GCMS is an effective tool for analyzing unknown industrial polymers and/or naturally formed organic solids. However, sometimes small, volatile peaks are lost among the pyrolyzed polymer fragments, and interpreting data can be tricky. To maximize applicability and ease-of-use, CDS has developed the most comprehensive set of libraries available.

CDS personnel have assembled several libraries including polymer additives, polymers, and even biofuel compounds. For polymers, the library is a collection of spectra generated by averaging all spectra for a given sample. The standard GC/MS software can be used to identify the averaged spectra run for a sample against polymer compounds in the CDS library.

In the case of volatile additives and biofuel compounds, customers can use one of several industry standard deconvolution software programs with these libraries to search for individual compounds or groups of compounds. The libraries are currently compatible with AMDIS, Ion Signature, and Agilent ChemStation. These programs are used to search for user-specified compounds in a complex program and then identified against our library of individual spectra. Compounds are broken into several categories including anti-oxidants, flame-retardants, HALS, lubricants, and plasticizers.

There are currently over 500 additives, and 400 polymers, in the library – with no end in sight! We offer free library upgrades for three years.

Figure 3: TIC of rubber pyrolyzed at 750°C.

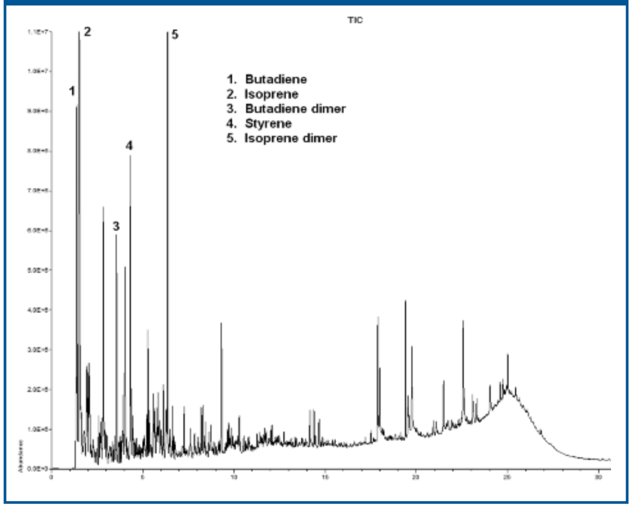
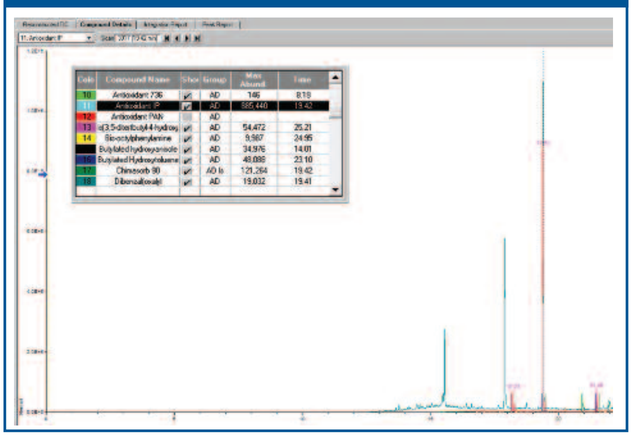


Figure 4: Search results for rubber, indicating Antioxidant IP at 19.42 minutes.



TDA-9300 Autosampler Delivers Knowledge To Build On

As building materials and basic consumer products come under increased public scrutiny, scientists are on the lookout for simple, reliable, and cost-effective ways to screen for contaminants and potentially toxic compounds. The recent controversy regarding sulfur compounds in Chinese wallboard reinvigorated a perennial debate, while the presence of formaldehyde continues to be a concern in "green" building initiatives. Formaldehyde analysis is typically done separately using HPLC. Now, thermal desorption/thermal extraction GC/MS analysis from CDS offers a quick, effective, and flexible screening technique for both toxins.

The TDA-9300 autosampler lets users choose between thermal desorption, thermal extraction, or dynamic headspace. In the example below, drywall samples can be placed in a dynamic headspace vessel on the autosampler, or in large vessels and sorbent tubes collected from the effluent. Each sample is reduced to a fine powder. Samples weighing ~250 mg are placed into individual test tubes and then into individual headspace stations.

Detection of formaldehyde has historically been done using liquid chromatography and 2,4-dinitrophenyl hydrazine derivatives of the suspected gaseous carbonyl compound. Solvent extraction of the 2,4-DNP-Hydrazones and analysis by HPLC completes the analysis. Utilizing the TDA-9300 Thermal Desorption Autosampler coupled to GC/MS, and PFPH (Pentafluorophenyl Hydrazine) derivitization, CDS was easily able to determine formaldehyde contamination in building materials.

Thermal desorption/extraction has proven to be a very useful sample introduction technique for the analysis of various industrial applications ranging from product emissions to QA/QC of solid materials. With the introduction of PFPH derivatization for Aldehydes and Ketones, the usefulness of the technique has been expanded significantly.

Thermal desorption/extraction has many advantages over the more popular solvent extraction or static headspace methods. Solvent extraction is time-consuming, expensive, and often results in matrix interferences. Static headspace is less sensitive and can give highly variable results. The CDS TDA-9300 thermal desorption autosampler enables users to do both direct thermal desorption and dynamic headspace with the same unit. This provides unparalleled flexibility for various industries where product emissions and/or chemical residuals are evaluated.

Figure 6: Chinese Drywall

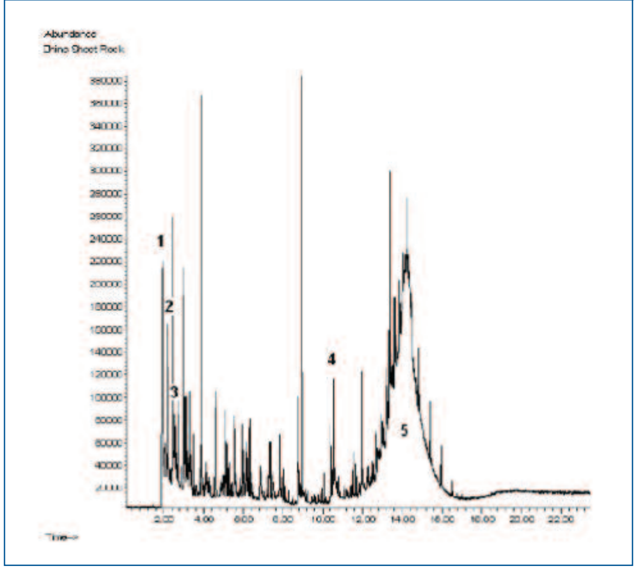
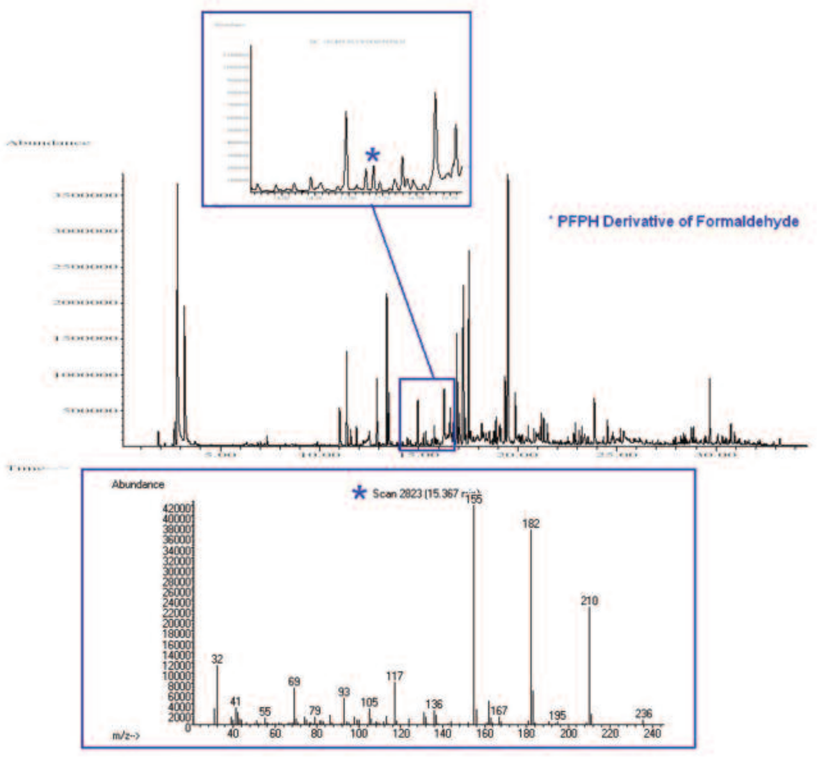


Figure 6 shows the total ion chromatogram of the imported drywall sample. Significant amounts of sulfur compounds were found, including: Sulfur Dioxide, two Alkyl Thiols, and Cyclic Hexa and Octa Elemental Sulfur.

Figure 5



Detection of formaldehyde has been done using liquid chromatography and 2,4-dinitrophenyl hydrazine derivatives of the suspected gaseous carbonyl compound. Solvent extraction of the 2,4-DNP-Hydrazones and analysis by LC completes the analysis.

Figure 7: Domestic Drywall

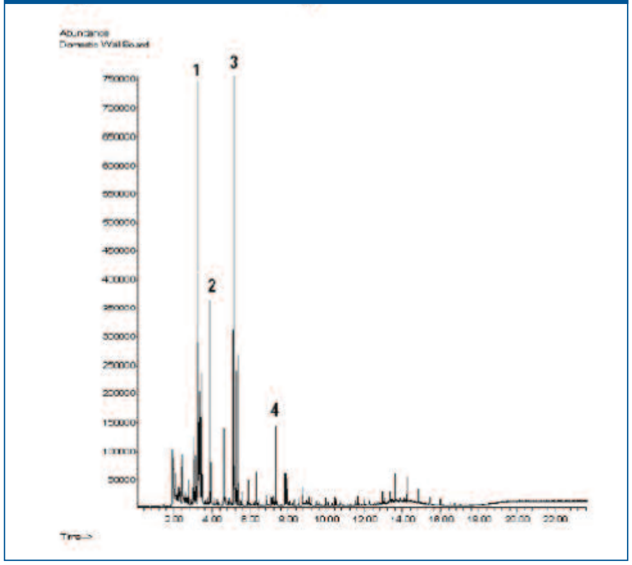


Figure 7 is a total ion chromatogram of a domestic drywall. Examination of this chromatogram shows no detectable elemental sulfur or sulfur compounds.

PITTCON 2010 Papers and Posters

The Use of Thermal Desorption for a Range of Consumer Safety Concerns!

Abstract Number: 190 – 2
Session 190 – GC Sample Introduction/New Columns/ Optimization
Day and Time: Sunday, February 28, 2010, 1:20 PM
Location: Room 207C

Sampling Techniques for the Analysis of Oils from Algae

Abstract Number: 660 – 7P
Session 660 – Sampling & Sample Preparation - LC, GC & MS
Day and Time: Monday, March 1, 2010, Morning
Location: Gray Area – Hall B4, Aisles 3400-3900

Purge & Trap; The Truth in Advertising!

Abstract Number: 600 - 14P
Session 600 – Gas Chromatography
Day and Time: Monday, March 01, 2010, Morning
Location: Blue Area – Hall A2, Aisles 700 – 1300

The Use of Thermal Desorption/Extraction to Screen "Chinese Wall Board" and Other Novel Concerns

Abstract Number: 600 – 13P
Session 600 – Gas Chromatography
Day and Time: Monday, March 1, 2010, Morning
Location: Blue Area – Hall A2, Aisles 700 – 1300

The Use of Pyrolysis Gas Chromatography Mass Spectrometry to Characterize Synthetic, Degradable Polymers

Abstract Number: 2070 – 8P
Session 2070 – GC-MS Methodologies
Day and Time: Wednesday, March 3, 2010, Morning
Location: Blue Area – Hall A2, Aisles 700 – 1300

Effects of Temperature and Pressure on the Pyrolysis of Cellulose

Abstract Number: 2300 - 1
Session 2300 – GC-MS Method Development Continued and Software Development
Day and Time: Wednesday, March 3, 2010, 2:00 PM
Location: Room 205A

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