the CDS Sampler
Product News & Application Notes From CDS
WINTER 2009

High Pressure Pyrolysis from CDS Facilitates Bio-fuel Research

As pressure builds to cut greenhouse gas emissions and reduce reliance on foreign oil, interest in cellulosic bio-fuels continues to grow. Cellulosic bio-fuels are derived from a wide variety of plant materials as well as non-food based feedstocks and energy crops. However, developing cost-effective means of producing cellulosic bio-fuels on a national scale poses major scientific challenges. Regardless of crop type or feedstock under consideration, analytical research tools are required to find new sources of biomass, bio-engineered crop types, and additives.

CDS 5000 Series pyrolysis instruments, including the new CDS Model 5200HP, offer an effective way of meeting one of these analysis challenges. The Model 5200HP is a high-pressure pyrolysis reactive gas instrument that mimics mini-reactor outputs. Whether you use atmospheric or high-pressure pyrolysis, the 8-step temperature programmable range and 1400°C maximum temperature allow users to see the activation energy for the decomposition of the biomass. When coupled with your GC or GC/Mass Spectrometer, CDS 5000 Series pyrolysis instruments can help you better understand the synergies of crop cultivation, harvesting, and processing of biomass energy crops and non-food feedstocks during their thermo chemical conversion.

The system is built on our field-proven model 5200 pyrolyzer, modified with a backpressure regulator. Samples can be pyrolyzed at elevated pressures (500PSI max) and collected onto the built-in trap. After pyrolysis of the sample is complete, the analytes are transferred to the GC at normal operating conditions. Any reactant gas can be used, including H_2, O_2, CH_4 and air. Maximum pyrolysis temperature is 1400°C at 500PSI — the system is compatible with all makes of GC and GC-MS.

The example below illustrates a pyrolysis run on switchgrass. Plant materials like switchgrass, wood, and straw are composed of two important biopolymers-cellulose and lignin. Cellulose is a polysaccharide made from glucose, while lignin is a complex aromatic polymer with considerable phenolic functionality. Each of these biopolymers produces characteristic pyrolysis products, with cellulose making a series of substituted furans and levoglucosan, and lignin producing phenolic products. A natural material containing both cellulose and lignin will produce both sets of products in the program.

Figure 1

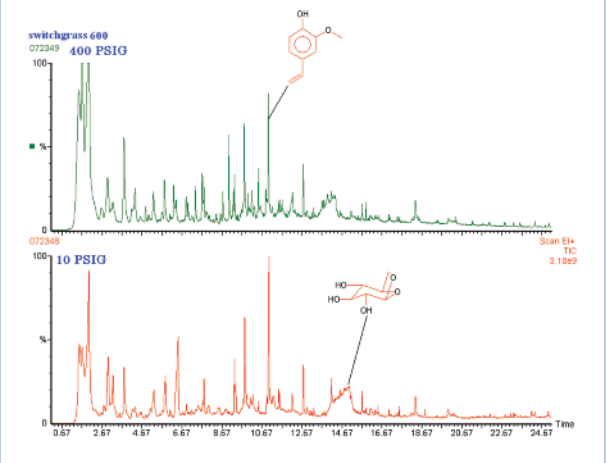


Figure 2

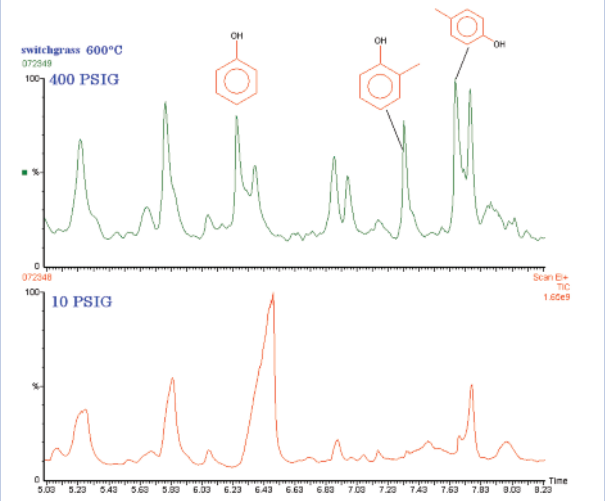


Figure 1 shows programs at low pressure and elevated pressure (400°C) for a sample of switchgrass. Each run contains typical compounds like levoglucosan (shown in lower run) from cellulose and the vinyl methoxyphenol (shown in the upper run) from lignin. There are considerable differences in the two runs, as shown in Figure 2, which expands the region from 5 to 8 minutes. The run performed at 400PSI now reveals more aromatic structures, including phenol and methyl phenols. All samples were pyrolyzed at 600°C.

Model 7000 Purge & Trap Now Available Direct From CDS

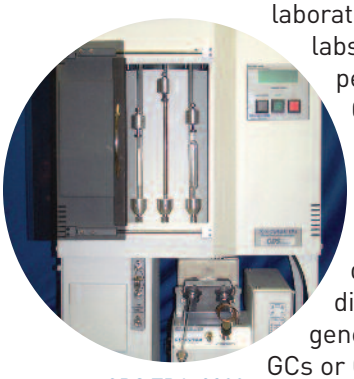
After years of being marketed exclusively through private labels, the model 7000 Purge & Trap instrument is now available direct from CDS. This high performance instrument, long acknowledged as the industry workhorse, offers: lowest cost of ownership, best chromatographic resolution anywhere, integrated GC and on-trap injection ports, first patented foam sensor, full-length wet trap for moisture control, and original exchangeable sample pneumatics system.

CDS 7000 Purge and Trap

We've recently expanded the instrument's capabilities by adding an electronic flow control option. Now, users can control purge and GC flows individually during purge, trap dry, desorb, and bake cycles. The resultant decrease in cycle time allows for more samples per instrument, thereby improving productivity.

TDA-9300 Offers Thermal Desorption and Dynamic Headspace

Today, productivity and efficiency have never been more important; and that's especially true for contract laboratories, production labs and QA/QC labs that must achieve maximum performance from their expensive GC/MS equipment. As you know, when two similar sample introduction techniques must be done separately, twice the amount of instrumentation is required. Thermal desorption and dynamic headspace require two different autosamplers, which generally are connected to two separate GCs or GC/MS systems.



CDS TDA-9300

With the introduction of the TDA-9300, the first thermal desorption and dynamic headspace autosampler, CDS introduces a new level of flexibility, simplicity, and efficiency to your lab.

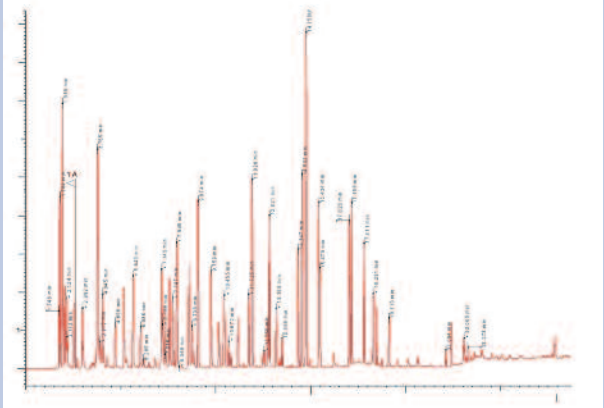
Volatiles in Air by TO-17

The "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Method TO-17" is used to assess volatile organic compounds in ambient air using active sampling onto solid sorbent tubes. The TDA-9300, (Thermal Desorption Autosampler), was developed to analyze any size sorbent tube for volatile organics. This instrument provides excellent sample transfer, automatic leak checking, dry purge of both tubes and traps, and sample-saver capabilities required to meet the most stringent method requirements, all without liquid cryogens or peltier cooling.

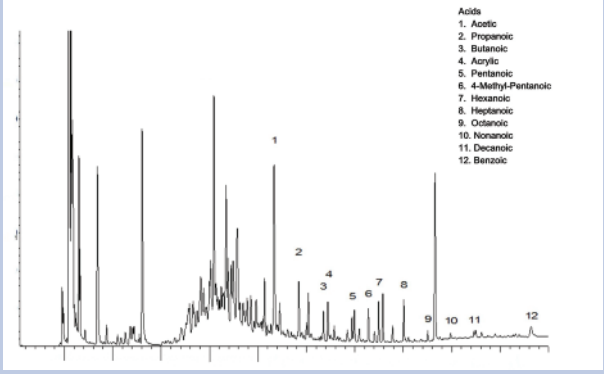
Dynamic Headspace

The TDA-9300 also provides dynamic headspace chambers that allow users to quickly change between thermal desorption and dynamic headspace. This capability enabled one QA/QC lab to monitor air samples for employee health and also analyze polymer samples for traces of organic acids identified as contaminants.

TO-17 Volatile Organic Standard (65 Components)



Polymer Contaminated With Organic Acids



Identifying Additives Using Deconvolution Software

There's a vast array of polymer additives available today, with new ones being introduced all the time. As a result, it is becoming increasingly difficult to find trace additives in unknown polymer samples. To help manage this growing concern, CDS has developed a search library containing hundreds of additives, designed for use with the leading deconvolution software package from Ion Signature Technology (www.ionsigtech.com).

Pyrolysis-GC/MS is a simple technique that may be used to study the composition of complex polymeric materials. For example, tire rubber is made with polymers and additives, such as carbon black, antioxidants, and other processing agents. The pyrogram of rubber shown in Figure 1 reveals that the polymeric material was formulated using styrene, butadiene, and isoprene. Additional information about the additives used is generally also present, requiring the investigation of peaks not related to the rubber pyrolysis products.

Deconvolution software can facilitate this investigation by comparing specific ions in the mass spectra of the various peaks with a library of known compounds. Searching the total ion chromatogram produces a reconstructed TIC made up of likely matches for compounds in the method library. The peaks are identified by compound name and retention time, making it simple to confirm the identification in the original chromatogram.

Figure 2 shows a deconvolution search of the rubber in Figure 1 performed using the software from Ion Signature and a method library prepared by CDS. At 19.42 minutes, a large peak is indicated for N-(-1-methylethyl)-N'-phenyl-1,4-benzenediamine (called Antioxidant IP among other names). The software indicates the masses used for matching as well as the actual and expected abundances of these masses.

Figure 1: TIC of rubber pyrolyzed at 750°C

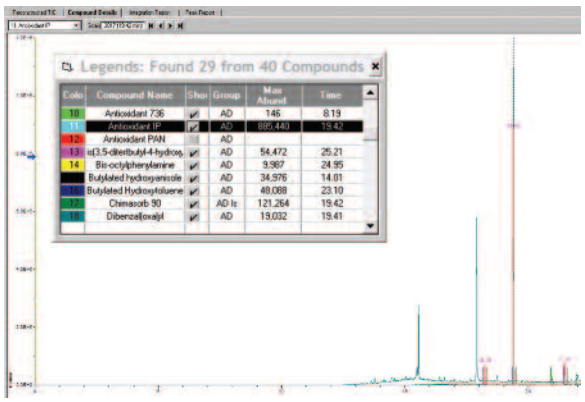
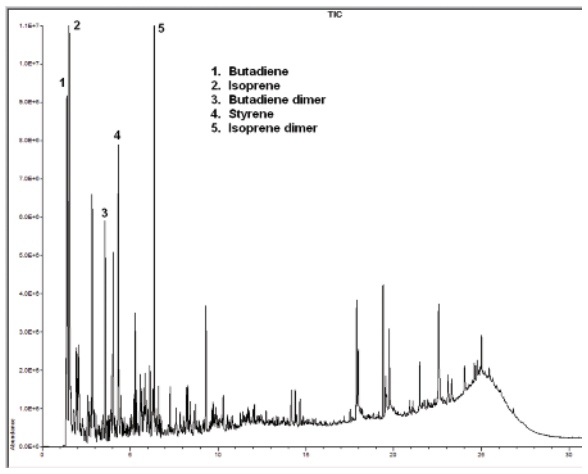


Figure 2: Search results for rubber, indicating Antioxidant IP at 19.42 minutes



Leverage Your Investment with Upgrades

For many of us, today's challenging environment means doing more with less- without sacrificing accuracy or convenience. That's why maximizing on a proven platform already in place is an idea whose time has come. Upgrading your CDS pyrolysis system leverages existing value and greatly increases return on investment.

Several options available to your 5000 series electronics include:

36 Sample Autosampler- Increase your productivity by adding an autosampler to your system. Allows the same 8-step programming per sample that your manual system already has.

Built-in analytical trap- Add a sorbent or cold trap to your system. This will allow you to pyrolyze in air for combustion studies, add a thermal desorber to your system and give you the ability to study gram size samples for off-gassing.

Analyze VOC in air samples- Add the ability to desorb sorbent tubes to the built-in focusing trap.

Add Dynamic Headspace Station- Study semi-volatiles in large samples by adding dynamic head-space capabilities. The vessel can heat samples up to 300C while being purged to the built-in trap. After the purge cycle the sample is rapidly desorbed into the GC.

Cryofocusing- Sharpen those early eluting peaks with an injection port mounted cryofocuser.

Poly Additive Library- Increase your effectiveness in finding unknown and trace additives in your sample. Software can work with most brands of MS instruments.

PITTCON 2009 Papers and Posters

Analysis of Natural and Synthetic Polymers Using High-Pressure Pyrolysis-GC/MS
Abstract Number: 2700 - 8P
Session 2700 - Mass Spectrometry of Bioanalytical & General Interest Samples
Day and Time: Thursday, March 12, 2009,
Morning Poster

Pyrolysis GC-MS of Plant Derived Fabrics
Abstract Number: 2980 - 5
Session 2980 - Polymer Characterization
Day and Time: Thursday, March 12, 2009, 03:05 PM
Room S503b

The Use of Thermal Desorption/Extraction Techniques to Analyze for Pesticides in Vegetables, PCBs in Fish and Phthalates in Packaging
Abstract Number: 780 - 2
Session 780 - Food Science: Product Profiles and Quality II
Day and Time: Monday, March 09, 2009, 01:50 PM
Room S505a

The Use of Thermal Desorption/Extraction as a Solution to a Variety of Unique Industrial Problems
Abstract Number: 1320 - 12P
Session 1320 - New Developments in GC
Day and Time: Tuesday, March 10, 2009,
Morning Poster

Purge & Trap; Getting the Best Chromatography for Your Volatile Gases
Abstract Number: 1980 - 25P
Session 1980 - Gas Chromatography Mass Spectrometry
Day and Time: Wednesday, March 11, 2009,
Morning Poster

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