

# CDSolutions

## APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

### GC/MS Detection of Formaldehyde in Aggressively Heated Resin-Coated Sands

Resin-coated sands are used in hydraulic fracturing of rock, and the metal casting industry. In both applications, this sand can be heated to very high temperatures, degrading the resin, and outgassing potentially hazardous components. Used to make formaldehyde-phenol resins, formaldehyde has known health risks, and its analysis by GC/MS is difficult due to its volatility and difficulty to be distinguished in a MS detector. However, if this carbonyl is collected onto a sorbent trap coated with derivatizing reagent, it is amenable to GC analysis.

First, resin coated sand was heated to a temperature of 550°C. Typical breakdown products of phenolic resins are seen, like phenol and methyl phenols (Figure 1). In a second analysis, a pyrolysis-derivatization method was performed. Pentafluorophenyl hydrazine (PFPH) reacts with carbonyls like formaldehyde to make GC friendly hydrazones. A sorbent trap was soaked in 2.90mg/mL solution of PFPH in hexane for 10 minutes. Afterwards, the trap was placed in the CDS Model 5200 in direct pyrolysis (no trapping) mode; its rest temperature was set to 65°C, and left to purge for 10 minutes to remove any remaining solvent. After 10 minutes, the Pyroprobe was placed in trapping mode.

As before, the sand was heated to 550°C using the Pyroprobe coil, and analytes were collected onto the trap. Desorption of the reagent-coated trap shows a formaldehyde-PFPH derivative, indicating that when the resin is heated, formaldehyde is released (Figure 2).

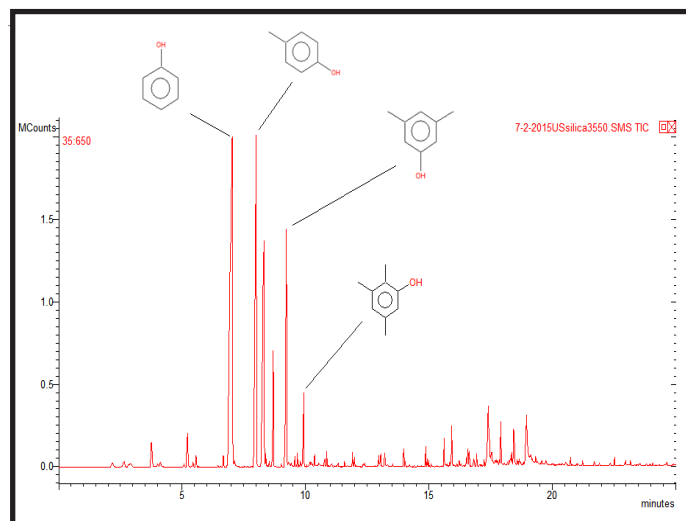


Figure 1: Pyrolysis GC/MS of resin coated sand

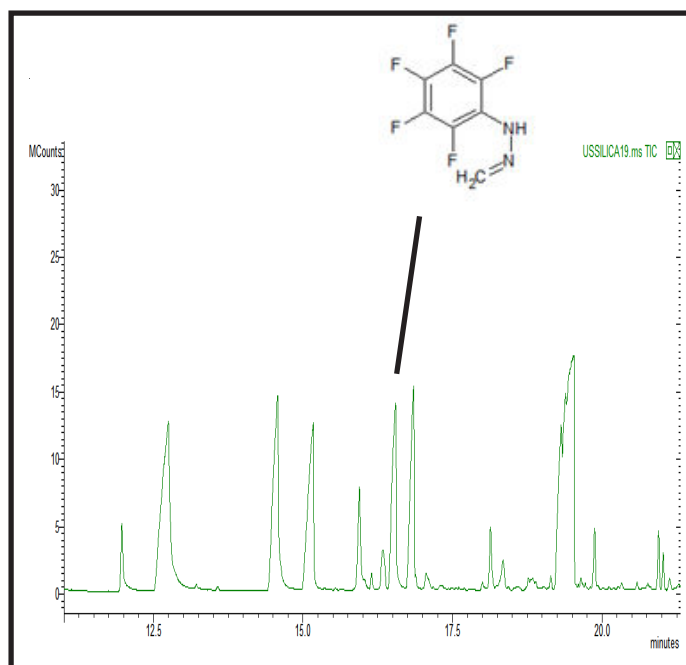


Figure 2: Pyrolysis-Derivatization of resin coated sand

## Equipment

This sample was analyzed using a CDS Model 5200, interfaced to a gas chromatograph/mass spectrometer.

### Model 5200 Conditions

Valve Oven: 325°C  
Transfer Line: 325°C  
Pyrolysis: 550° for 60 seconds  
Interface Final: 300°C for 4 minutes  
Trap Final: 300°C for 4 minutes  
Trap Contents: Tenax TA

### GC/MS Conditions

Carrier: Helium  
Injector: 325°C  
Split: 50:1  
Column: 5% Phenyl (30m X 0.25mm)  
Detector: Quadrupole MS  
Range: 35 - 550  
Sol Delay: 11 min

### GC Program:

Initial: 30°C for 4 minutes  
Ramp 1: 7°C/min to 100°C  
Ramp 2: 8°C/min to 250°C hold 2 min

FOR MORE INFORMATION  
CONCERNING THIS APPLICATION,  
WE RECOMMEND THE  
FOLLOWING READING:

Ho and Yu, Environ. Sci. Technol.  
2004, 38, 862-870

Additional literature on this and related applications may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

CDS Analytical, LLC has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of pyrolysis, CDS manufactures the Pyroprobe. 5000, 5150, 5200 and 5250 autosampler for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications as well as purge & trap instruments for drinking and waste water. CDS also manufactures the Dynatherm line of thermal desorption instruments including the 9300 series for air monitoring and the 7500 autosampler. Our customers, their requirements and applications are important to us. To help meet your needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, call us at 1 800 541 6593, or log onto [www.cdsanalytical.com](http://www.cdsanalytical.com).