



Multi-Step Analysis of Coal by Pyrolysis-GC/MS

Application Note

Energy

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Although the use of the word “coal” generically assumes all types of “black combustible substances”, there is a great difference in chemical composition from source to source. Coal is a complex, highly aromatic material with varying amounts of sulfur and other trace inorganic constituents. In order to study the organic make-up of a coal sample by GC, it requires considerable energy to degrade the larger organic compounds so they are amenable to GC injection.

Analytical pyrolysis-GC/MS is a powerful tool used for the investigation of complex, solid materials. With the use of a pyrolyzer, a coal sample may be heated to a variety of temperatures with the compounds analyzed at each programmed temperature. This sequential stepping of the sample temperature is useful in removing contaminants before pyrolysis, determining which products are desorbed and which are degradation products, optimizing pyrolysis parameters, as well as the investigation of the behavior of a sample under various thermal conditions. Figure 1 shows the result of heating a sample of coal sequentially to temperatures from 300° to 900°C. Consequently, at 300°, little is seen in the chromatogram, except for some small peaks for aromatics and some aliphatics at the end of the run. At 500°, there is a peak for carbon dioxide, and some small aromatics are produced. Toluene and xylene become more prominent at 600°, and the first evidence of phenol is seen. At 700° more substituted benzenes are present, as well as phenol and methyl phenol. Finally at 900°, the production of gases is reduced and the chromatogram is dominated by aromatics, including naphthalene and substituted naphthalenes.

Optionally, a catalytic reactor can be added into a CDS pyrolyzer as well as different reactant gases and varying pyrolysis pressures. The addition of these variables will allow the researcher to study how best to process coal when the objective is to break it down into syngas or specialty chemicals.

CDS Pyrolyzer 6200 Conditions:

| | |
|----------------|---|
| Pyroprobe | |
| Initial: | 300°, 500°, 600°, 700°, 900°C for 15 seconds |
| Interface | |
| Rest: | 300°C |
| Initial: | 300°C for 2 minutes |
| Iso Zones | |
| Valve oven: | 300°C |
| Transfer line: | 315°C |

GC Conditions:

Column: 30 m x 0.25 mm 5% phenyl MS
Carrier: Helium
Split: 100:1

Oven Program
Initial Temp: 40°C
Initial Hold: 2 minutes
Ramp: 10°C/minute to 325°C

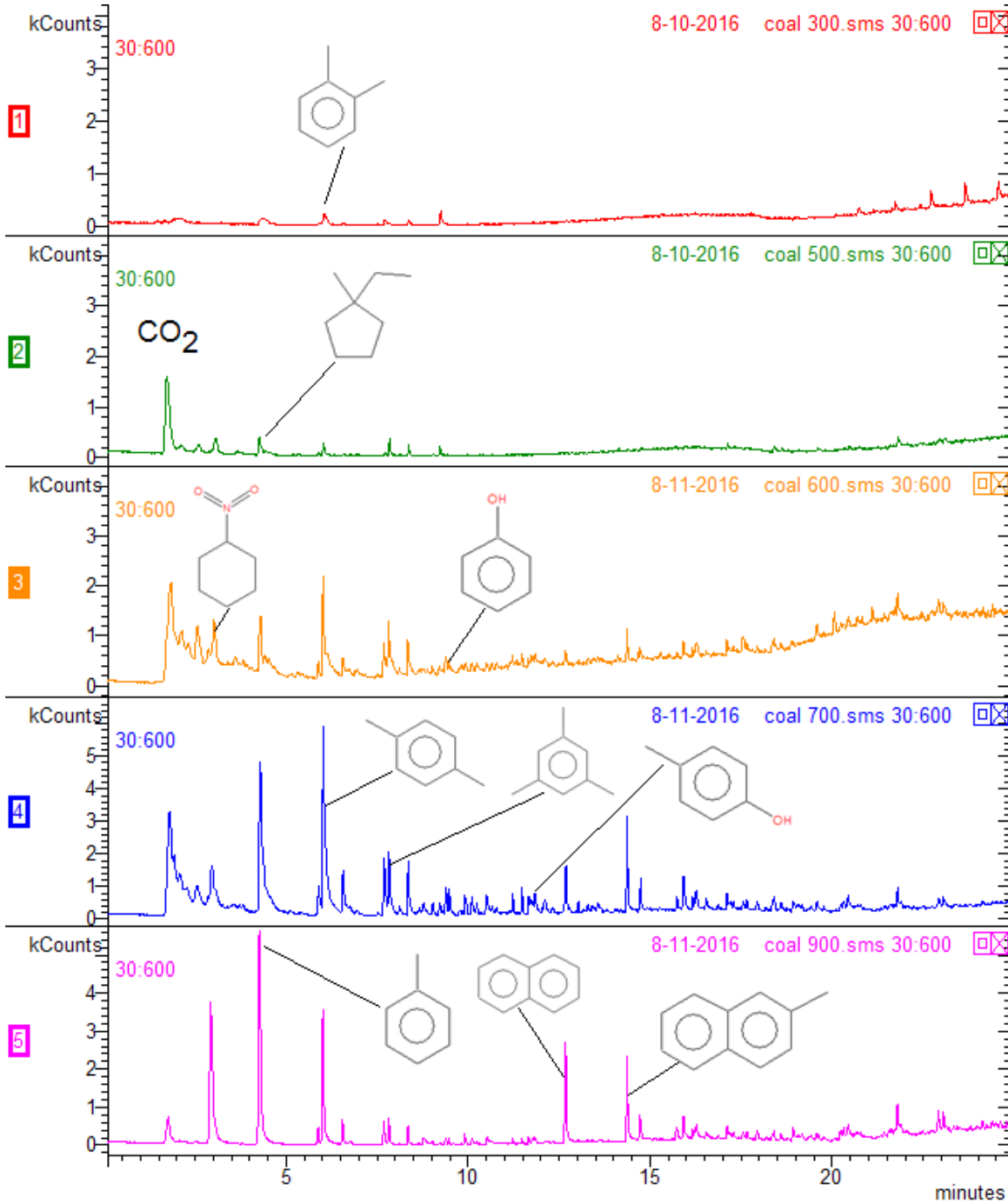


Figure 1. Coal sample heated sequentially to 300°, 500°, 600°, 700°, and 900°C