Scientific and Technical Report on
"Advanced NMR Approaches in the Characterization of Coal"
DE-F22-90PC90290

Report No. 8
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DOE/PC/90290--8

DE93 006009

The paper submitted earlier on the use of (bicyclo[3.2.1]4-pyrrolidino-N-methyl-octan-8-one triflate) (^{13}C -123) as a ^{13}C intensity standard was accepted for publication. Subsequently, ^{13}C -321 was used in this manner for quantitative ^{13}C CP-MAS NMR analysis (including spin counting) of Argonne Premium coals. The cross-polarization time constants, T_{CH} , and the rotating-frame proton spin-lattice relaxation times, $T_{1\rho}^{\text{H}}$, were determined for each major ^{13}C peak of each coal via a combination of variable contact-time and variable spin-lock ($T_{1\rho}^{\text{H}}$) experiments. Two or three components of rotating-frame ^1H relaxation decay and two or three components of T_{CH} behavior were observed for each major ^{13}C peak of each coal. These data were used to determine the number of carbon atoms detected in each coal; these values are in the range between 77% and 87% of the amount of carbon known to be in each coal from elemental analysis data, except for Pocahontas #3, for which only 50% of the carbon was detected.

In an attempt to use ^1H CRAMPS to elucidate chemical functionality in coal, pyridine-saturated samples of the Argonne Premium coals were examined in detail in terms of their ^1H CRAMPS NMR spectra. These spectra were deconvoluted to yield relative concentrations for individual peaks. Six separate peaks, three aliphatic (1 ppm, 1.8 ppm, 3.5 ppm) and

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three aromatic (6.8 ppm, 8.3 ppm, 10.5 ppm), were identified. This allows assignment of the following chemical functionalities: methyl, methylene, methoxy, aliphatic methylene ethers ($-\text{CH}_2-\text{O}-$), aromatic ($\text{sp}^2\text{C-H}$), protons α to aromatic nitrogen in pyridine-type structures, phenolic OH, carboxylic OH, amine NH_2 and o-hydroxypyridine OH. The relative intensity of each peak has been estimated from the deconvolutions. A large diversity of chemical functionality of Argonne Premium coal samples has been found and to some degree characterized. These results were compared in a preliminary way with data obtained via ^{13}C techniques and attempts continue to correlate these two types of data.

Lectures on both the ^{15}C and ^1H NMR results outlined here were prepared for presentation at the International Symposium on Coal Structure in Poland during September, 1992. Manuscripts in this work have been submitted for publication and copies are included with this report.

Manuscripts submitted

Antoni Jurkiewicz and Gary E. Maciel, "Spin Dynamics and Spin Counting in the ^{13}C CP-MAS Analysis of Argonne Premium Coals," *Fuel*, submitted.

Antoni Jurkiewicz, Charles E. Bronnimann and Gary E. Maciel, " ^1H CRAMPS NMR Study of the Chemical Functionality of Argonne Premium Coals," *Fuel*, submitted.

Previously submitted manuscripts that have been accepted.

Vicky Haiou Pan and Gary E. Maciel, "The Analysis of Three Representative Premium Coals by ^{13}C Nuclear Magnetic Resonance," *Fuel*, in press.

Randal A. Hall, Antoni Jurkiewicz and Gary E. Maciel, "A Bicyclic Ketone as a Solid-State ^{13}C NMR Intensity Reference," *Anal. Chem.*, in press.

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