Analysis Package: Coal characterization

Service benefits:

^{13}C Solid State NMR (SS NMR) is an established tool for structural analysis of solid fossil fuel samples in a non-destructive manner. Our lab offers the determination of the structural parameters listed in table 1 for the characterization of coals.

Parameter Description	Label
Fraction Aromatics	f_a
Corrected Fraction Aromatics (excl CO)	f_a^*
Fraction Aliphatics	f_{al}
Fraction Aliphatic C's bonded to Oxygen	f_{al}^O
Fraction CO	f_a^{CO}
Fraction Phenolics	f_a^P
Fraction Alkylated Aromatics	f_a^S
Fraction Non-Protonated C's in aromatic region	f_a^N
Fraction Protonated C's in aromatic region	f_a^H
Fraction Bridgehead C's	f_a^B
Fraction Non-Protonated C's+Methyl groups in Aliphatic region	f_{al}^{N*}
Aliphatic CH+CH2	f_{al}^H
Mole fraction of Aromatic Bridgehead C's	X_b
Average # of Aromatic C's per cluster	C
	#Clusters/100
#of attachments per cluster	σ + 1

Table 1: structural parameters used for the characterization of coal samples

These results, measurement parameters and conditions as well as explanations regarding the methodological approach and the determination of the parameters are summarized in a report. We will provide you with the raw and xy data as well the plots of the integrated spectra as shown in figure 1.
Since the determination of these parameters is based on semi-quantitative SS NMR methods (see paragraph “methodological approach”) the analysis is best applied, when trying to compare a series of samples, where trends are considered rather than absolute values.

Costs and requirements

- Sample amount required for analysis: **0.5 g**
- coals must be **non magnetic!**
- preferably demineralized samples. (the presence of paramagnetic compounds in non-demineralized samples cause usually technical problems restricting the results of the analysis
- Please provide us with the information of the elemental composition, if available.

The costs for a standard coal analysis are summarized in the table below:

Prices*	academics	commercial	description of services
First sample	R 2 800	R 4 700	- Sample preparation and CP optimization, - CP MAS without and with DD - processing and integration of the spectra - calculation of structural parameters (table 1), - report
Any further similar sample	R 1 800	R 3 000	- Sample preparation, - CP MAS without and with DD, - processing and integration of the spectra, - calculation of structural parameters (table 1), - update of the report

***** prices are valid for carbon-rich, demineralized coals

- pitch samples can be analyzed in a similar way, if they are not too liquid! Since the sample preparation and cleaning of the sample holders is more time-consuming an additional fee of R 200 (academics)/ R 300 (commercial) will be added.

Methodological approach:

Carbon-rich coals are analyzed by ^{13}C cross polarization magic angle spinning (CP MAS) and CP MAS with dipolar dephasing (DD, often also referred as “interrupted decoupling”) NMR experiments at higher spinning frequencies. The spectra are integrated according to typical chemical shift ranges of the different carbon species[1] as illustrated by figure 1. The structural parameters listed in table 1 are calculated using the integrals of both experiments.

Due to practical considerations (= reduction of measurement time / analysis costs) cross polarization methods are commonly used, although they are not strictly quantitative. Since the magnetization of protons is transferred to carbons, the ^{13}C CP MAS experiment underestimates aromatic and other weakly or non-protonated carbon species. Therefore, the DD experiment is additionally applied. This method is based on a CP MAS experiment, but in the DD experiment the ^{1}H decoupling is interrupted for a certain time, before the ^{13}C magnetization is acquired. In the absence of ^{1}H decoupling, the ^{13}C magnetization will diphase rapidly as a result of ^{1}H-^{13}C dipole-dipole interaction. As a consequence the DD MAS spectrum is dominated by weakly proton coupled carbons like bridgehead aromatics and can be used to determine f_a^B and thus X_b and C parameters. Based on these parameters the coal lattice parameters can be calculated.

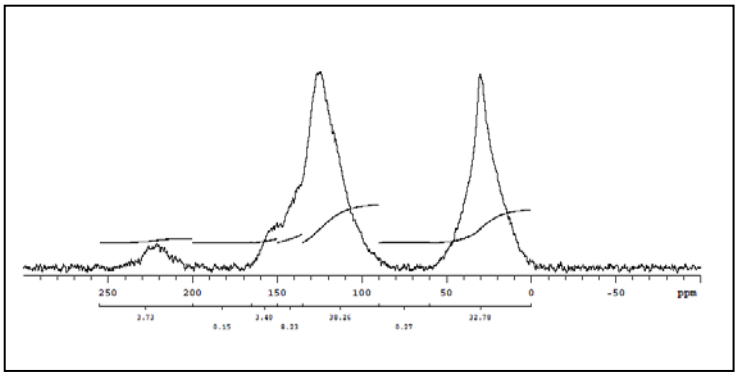

Figure 1: CP MAS spectrum of a coal sample and illustration of its integration

[1] Mark S. Solum, Ronald J. Pugmire, David M. Grant, ***Energy*** & ***Fuels*** 1989, ***3,*** 187-193.