Some Practical Considerations to Obtain Reliable ^{13}C-NMR Spectra of Highly Aromatic Carbon Materials.

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Nuclear magnetic resonance (NMR) spectroscopy has been used to study several types of solid carbonaceous materials and their transformations under chemical and/or physical processes. The application of special techniques such as cross-polarization (CP), high-power dipolar decoupling (DD), and magic-angle spinning (MAS) has allowed the attainment of high-resolution spectra with the identification of the chemical environments of ^{13}C, ^{1}H, and other nuclei existing in the material.[1] Then, solid state ^{13}C CP MAS NMR spectroscopy is one of the most powerful methods for characterising carbonaceous solid materials. However, it is sometimes preferable to use the more time-consuming but more quantitatively reliable experiment of direct polarization (DP) of ^{13}C nuclei. That is the case of high-rank carbons and charcoals, where a low percent of hydrogen and a high percent of graphitic carbon happen together. But even so, the spectra so obtained show an extremely great band broadening, a very poor resolution and very low sensitivity. Several authors have associated these features to physical characteristics of the sample related to the graphite content, as conductivity[2] or magnetic susceptibility.[3]

During our work on the characterisation of charcoals through solid state ^{13}C DP MAS NMR, the quantitative analysis of the band intensities could be achieved by introduction of an electronic reference signal (the so called ERETIC ™). This technique made possible to quantitatively compare the sensitivity attained for the same sample under different measurement conditions. As a relevant result it has been clearly established that he total signal intensity of the DP MAS ^{13}C-NMR spectrum can be greatly (more than twice) increased by dilution of the charcoal sample with silicagel instead of measuring the neat charcoal. This effect and other practical considerations to obtain reliable 13C NMR spectra of charcoals are discussed.[4]

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