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Quantitativeness of CP and DP spectra

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Cross polarization (CP) magic angle spinning (MAS) 13C-NMR spectroscopy is a solid state NMR technique widely used to study the chemical composition of soil organic materials as they are mostly unsoluble in the common deuterated solvents used to run liquid state NMR experiments. Due to its enhanced signal intensity and reduced relaxation delays, 13C-CPMAS NMR spectroscopy is often applied in environmental chemistry to obtain quantitative information on the chemical composition of natural organic matter (NOM) (Conte et al., 2004). However, its quantitative assessment is still matter of heavy debates. In fact, many authors (Baldock et al., 1997; Conte et al., 1997; 2002; Dria et al., 2002; Johnson et al., 2005; Kiem et al., 2000; Knicker et al., 2005; Kögel-Knabner, 2000), reported that the application of appropriate instrument setup as well as the use of special pulse sequences and correct spectra elaboration may provide signal intensities that are directly proportional to the amount of nuclei creating an NMR signal. However, many other papers dealt with the quantitative unsuitability of 13C-CPMAS NMR spectroscopy. Among those, Mao et al. (2000; 2002), Smernik and Oades (2000), Preston (1996; 2001), and Smernik et al. (2002a; 2002b) reported that cross-polarized NMR techniques may fail in a complete excitation of the 13C nuclei.

Often direct polarization (DP), also called Bloch decay experiment, is recommended to acquire quantitative spectra. However, this technique requires very long recycle delays and unreasonably long measurement times might result. Furthermore, with natural organic samples one is never completely sure if the chosen recycle delay is sufficient to allow for complete relaxation of all regions.

Mao et al. (2000) already presented a way to assess if the DP spectra are completely relaxed and if not how much of which regions is missing in the spectra. The authors achieved this through the application of a CP/T1C sequence developed by Torchia (1978). However their elegant procedure is not widely applied in the field of environmental NMR.

We will present a series of standard substances and natural organic samples measured with CP, DP and CP/T1C and try to assess the fastest way to quantitative results of unknown organic samples. Furthermore, we will try to determine conditions for which quantitative results for unknown samples can be expected from CP spectra.