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Use of SSFP ^{13}C NMR to monitor *in situ* electrochemical reaction in spectroelectrochemical cell

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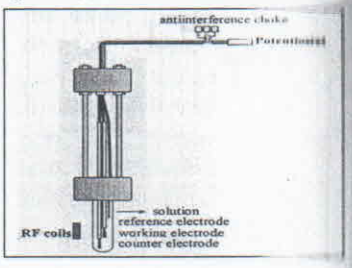
The main advantage *in situ* measurements, which couple, electrochemistry techniques (EC) and nuclear magnetic resonance spectroscopy (NMR) is obtain information in real time about electrogenerated species, in solution. Most EC-NMR studies uses the ^{1}H NMR detection to monitor the electrochemical processes due to ^{1}H high sensitivity and fast data acquisition.[1,2] To obtain ^{13}C spectra faster spectrum than conventional ^{13}C NMR sequence to monitor *in situ* the electrolysis's reaction (organochloride reduction) we examined the application of ^{13}C Steady State Free Precession sequence (SSFP). Figure 1 shows the diagram of EC-NMR cell assembled in a 10 mm NMR tube. The spectroelectrochemical cell contains the three electrodes, the reference, working and counter electrodes. The *in situ* electrochemical reaction was performed with potentiostat coupled in the cell placed inside the high-field NMR spectrometer. The ^{13}C SSFP measurements were performed for 10 minutes during the electrochemical reaction. The signal to noise enhanced provided by SSFP sequence demonstrates by first time the possibility of *in situ* monitoring of ^{13}C NMR in spectroeletrochemical study.



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Assignment of the Proton and Carbon-13 Resonances of an unsymmetrical beta-Cyclodextrin Derivative

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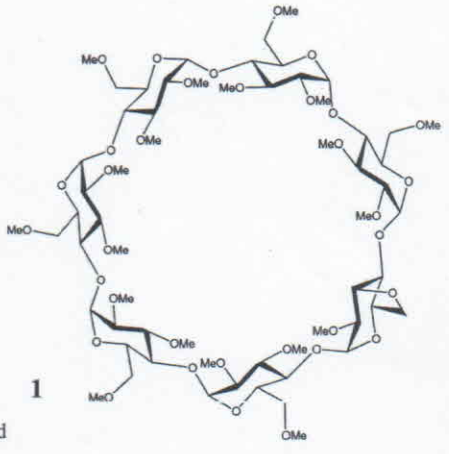
Compound **1** is a starting material in the synthesis of chiral stationary phases for gas chromatography.[1] The assignment of its ^{1}H and ^{13}C sugar resonances was achieved by means of new and conventional pulse sequences.

The sequential assignment of the sugar units was obtained using a F_1 decoupled F_1 band-selective 2D TOCSY – ROESY experiment. The ^{1}H and ^{13}C resonances in each sugar unit were assigned by means of sensitivity optimized 3D TOCSY – DQFCOSY and TOCSY – HSQC spectra, of F_1 band-selective 2D HSQC – RELAY and of aliased 2D HSQC – TOCSY[2] spectra.



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