Measuring methods available and examples of their applications

^{13}C NMR (carbon nuclear magnetic resonance)

For most organic compounds, information on the number and character of the carbon is essential for confirmation and proof of their structure. As the most common carbon isotope 12C does not possess magnetic moment, NMR spectroscopy relyes on 13C whose natural abundance is only 1.1%. This, combined with four times lower gyromagnetic ratio compared to 1H, results in much lower measurement sensitivity than in the case ^{1}H NMR and the measurements are therefore more time-consuming. Routine ^{13}C NMR spectra are measured usually with broadband proton decoupling and and sensitivity enhancement (3x) by NOE effect. In our CF, a very sensitive cryoprobe (175 MHz at 16.45 T) is available for the measurement of ^{13}C -NMR, which significantly reduces the measurement times (tens of minutes).

In analogy to proton NMR, ^{13}C NMR allows the identification of carbon atoms in an organic molecule just as proton NMR identifies hydrogen atoms. As such, ^{13}C NMR is an important tool in chemical structure elucidation in organic chemistry.

From a ^{1}H -decoupled carbon (^{13}C) spectrum useful information about ^{13}C chemical shifts is obtained. Alternatively, carbon multiplicity and ^{1}H-^{13}C coupling constants can be determined from the gated decoupled methodology and quantitative measurements can be made from the inverse gated experiments.

Assignment of the ^{13}C spectrum is usually performed with the help of additional NMR methods. As a general strategy:

- Carbon multiplicity is determined from 1D APT or DEPT experiments.
- Correlation with ^{1}H nuclei via ^{1}J(CH) is achieved from HSQC or HMQC-type experiments.
- Correlation with ^{1}H nuclei via ^{n}J(CH) is achieved from HMBC-type experiments.

Examples

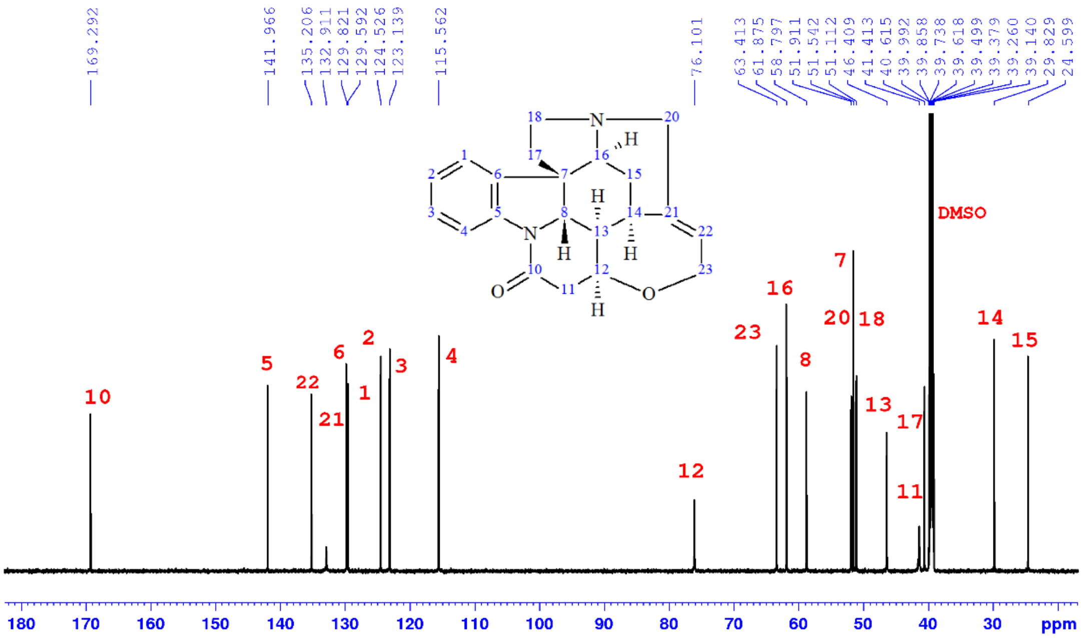

Fig. 1. Strychnine nitrate in DMSO-d6 (approx. 50 mM) ^{1}H -decoupled ^{13}C NMR spectrum with assignment. Spectrometer: AVANCE III HD 700, Probehead: 5 mm CPTXO 13C/15N-1H/D with z gradients, Experiment time: 10 min

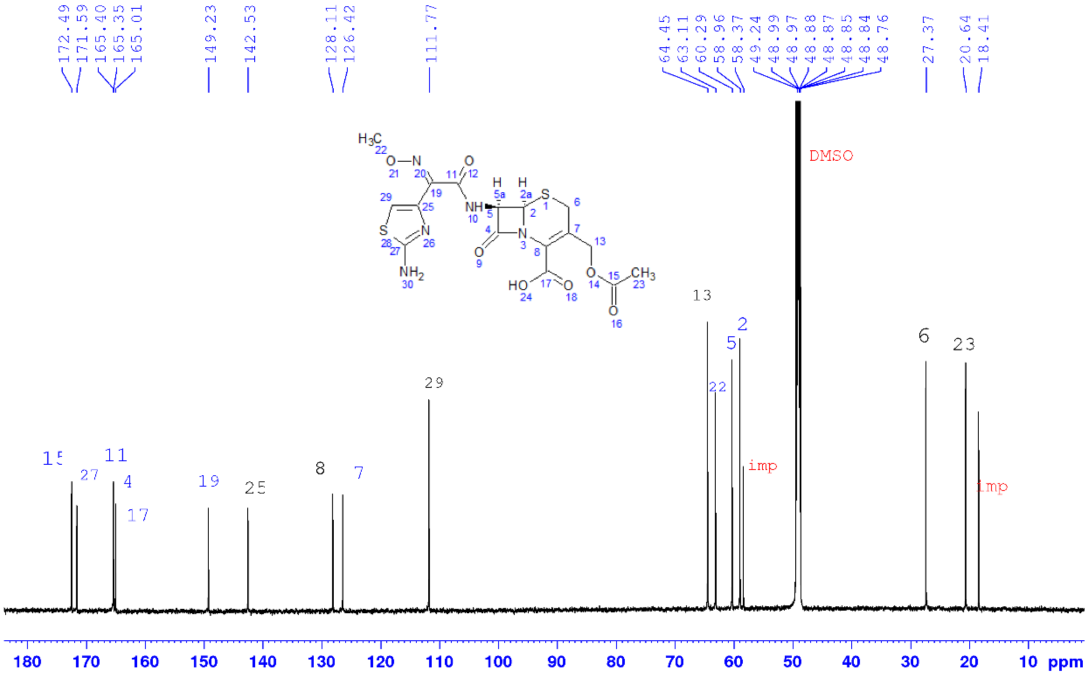

Fig. 2. Cefotaxime Acid (cephalosporin antibiotic), ^{1}H-decoupled ^{13}C NMR spectrum with assignment, Spectrometer: AVANCE III HD 700, Probehead: 5 mm CPTXO 13C/15N-1H/D with z gradients, Experiment time: 10 min

For quantitative applications, it is necessary to obtain reliable integration values. In this case, the ^{13}C spectrum is recorded with a 90º pulse and a long relaxation period (5*T_1(^{13}C)) is used to avoid partial signal saturation. It can be used for example for characterization of synthetic polymers.