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NMR NEWS June 2016

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* Topics in this issue:

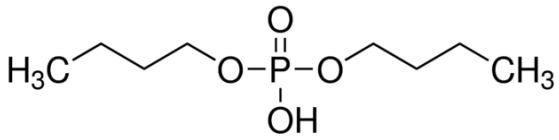
- **^{31}P NMR without ^{1}H decoupling.**
- **Quantitative ^{13}C NMR.**

* ^{31}P single pulse without ^{1}H decoupling.

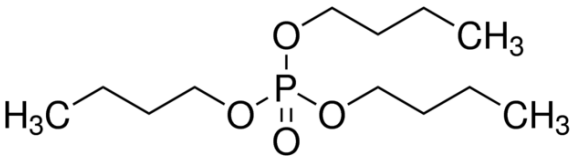
The standard ^{31}P NMR experiment includes ^{1}H decoupling during the acquisition of the FID. The result of this decoupling is to observe a single narrow NMR line for each ^{31}P in the sample.

As an examples, the following ^{31}P NMR spectrum of a mixture of **di-butyl-phosphate** and **tri-butyl-phosphate** shows two ^{31}P lines as expected. One for each compound.

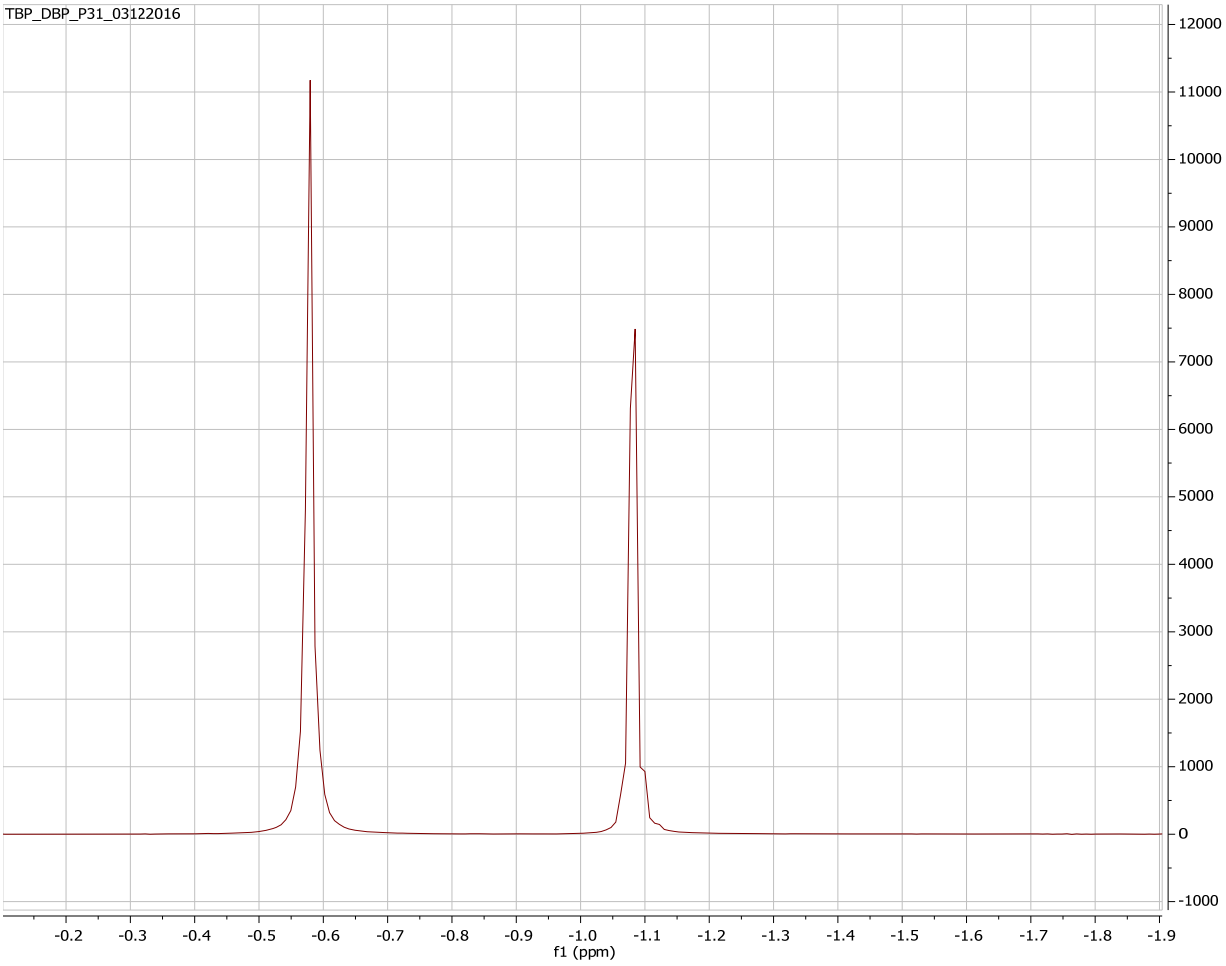
DBP



TBP



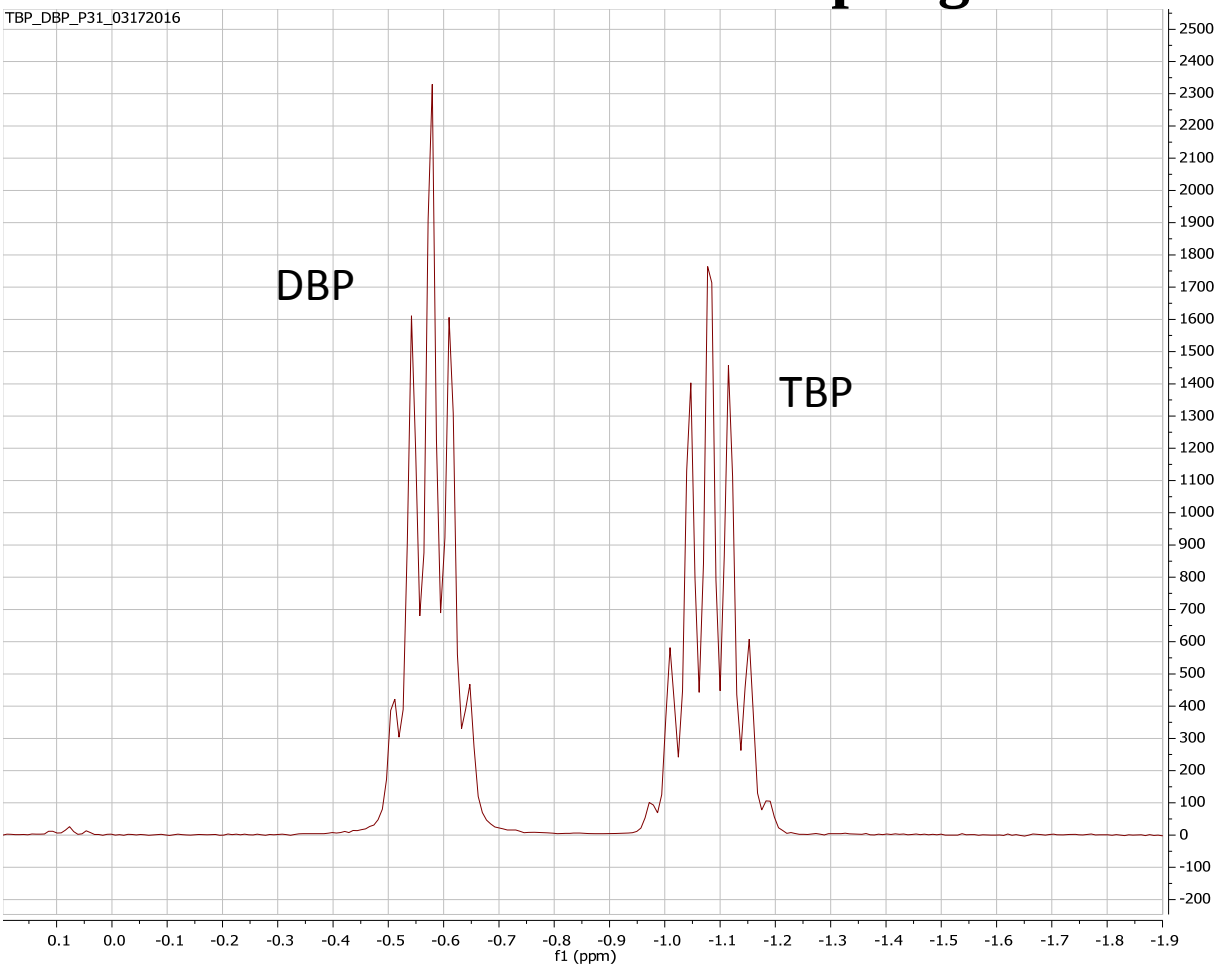
1D ^{31}P NMR with ^{1}H decoupling



Now, the problem is how to identify the signal corresponding to each compound. This problem can be solved by running a ^{31}P

NMR experiment **without** ^{1}H decoupling. The spectrum of this experiment is shown below,

1D ^{31}P NMR without ^{1}H decoupling



The multiplets observed for the two ^{31}P NMR signals are the coupling of ^{31}P with the ^{1}Hs of the (-O-C$\mathbf{H}_2$-) groups.

Di-butyl-phosphate → 4 equivalent ^{1}Hs → **5 lines**, intensity ratios →1,4,6,4,1

Tri-butyl-phosphate → 6 equivalent ^{1}Hs → **7 lines**, intensity ratios → 1,6,15,20,15,6,1

The example shows how ^{31}P NMR (without ^{1}H decoupling) can be used to identify or verify the environment of a ^{31}P nucleus.

* Quantitative ^{13}C NMR.

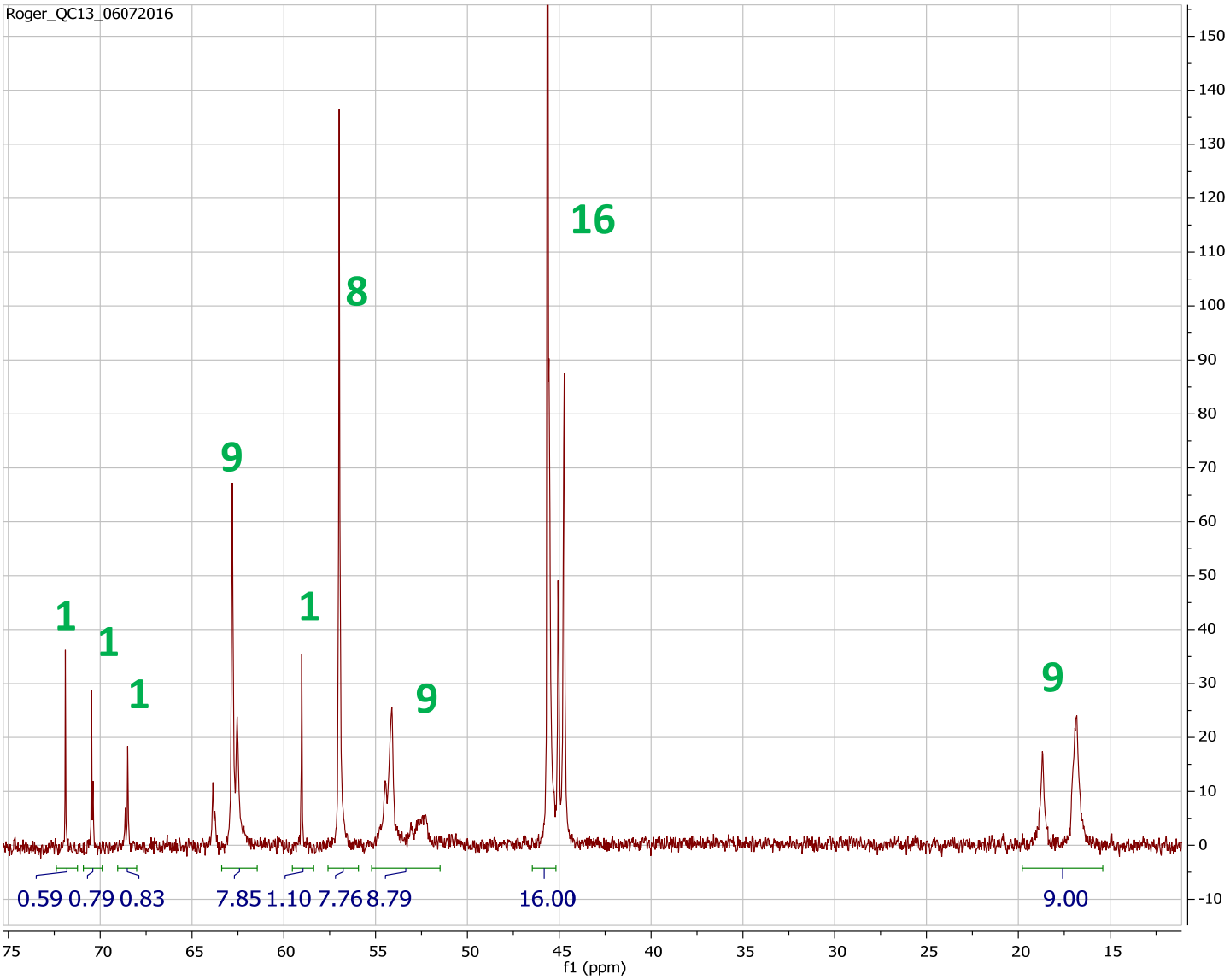
As pointed out in NMR Newsletters 17, quantitative NMR is not a standard experiment. A 90^{o} pulse must be used and the delay between scans must be adjusted according to the relaxation times of the nuclei in the sample.

For the case of quantitative ^{1}H NMR, the standard ^{1}H experiment with a 45^{o} degree pulse works for most samples. See Newsletter 17 for an exception.

However, things are different for quantitative ^{13}C NMR experiments. A 90^{o} pulse is needed because the signals are very weak. This is because NOE enhancement cannot be used. Also, T_1s for ^{13}C nuclei must be measured and the delay between scans calculated accordingly.

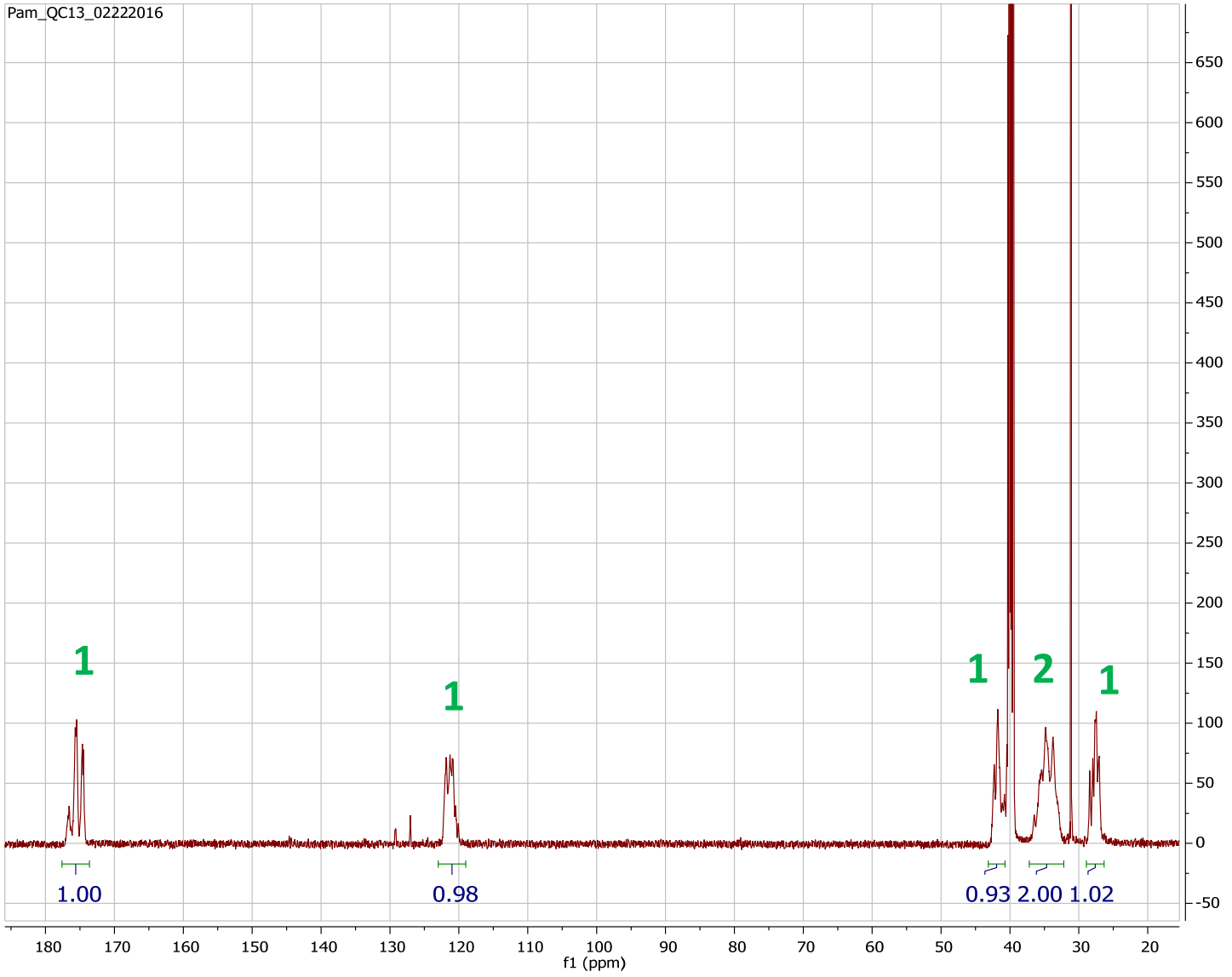
Here we present results obtained with the Quantitative ^{13}C experiment for two polymer samples from Dr. Zhao's group.

Expected values for the integrals are shown in green. Signal that integrates to 16 was used as the reference value for the integrals.



The sample is a copolymer. The ratio is 8:1 as calculated from the ^{1}H spectrum. The same result was obtained from the Quantitative ^{13}C spectrum.

The second example is a Copolymer with a ratio of 1:1. The ^{1}H spectrum was not suitable for quantitative measurements due to the width of the signals and overlap between them.



Contact us for a tutorial on how to set up a Quantitative ^{13}C experiment.