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Benchtop NMR in teaching and outreach

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NMR provision at MMU

- One 400 MHz JEOL instrument (located on “research floor”)
- One Oxford Instruments Pulsar benchtop NMR (60 MHz) (located on “teaching” floor) purchased summer 2016

- 3rd year undergraduate students submit NMR samples through the NMR service
- 4th year undergraduate / postgraduate students can either use service or get trained to use spectrometer themselves

Why benchtop NMR?

- Give students experiential learning of NMR without putting pressure on our only high-field instrument (putting theory into practice)
- Map on to our hands-on approach that we want our lab classes to possess
- Increase student exposure to NMR by increasing the representative number of experiments that utilise NMR
- In some cases, to screen samples prior to submission to high-field service

Undergraduate labs that utilise benchtop NMR

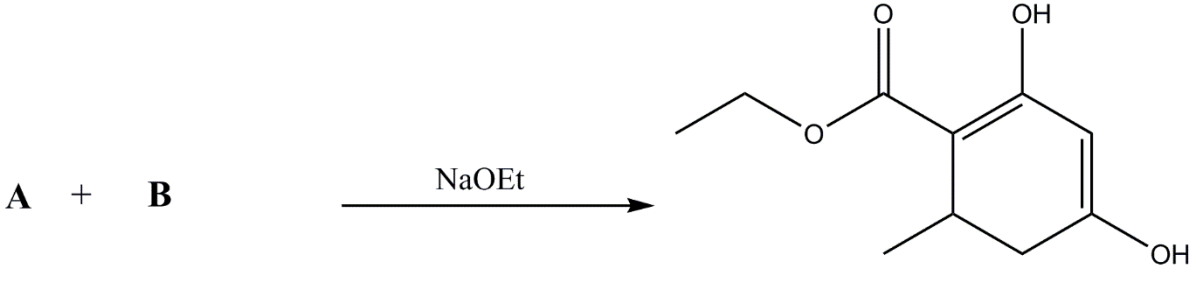
- Primarily at the moment only one second year undergraduate lab class (with many more in development), plus third and fourth year projects. Also rolled out to FdSc course

Example practical experiments

- NMR coursework
- Reduction of 3-nitrobenzaldehyde to 3-nitrobenzylalcohol
- Synthesis of dimedone
- Analysis of transition metal acac complexes

NMR coursework

- Students are asked to elucidate the structures of two unknown materials using NMR data, CHN, IR and mass spec.
- Utilises organic chemistry knowledge being taught at year 2 (enolate chemistry, reaction mechanisms, Michael additions etc.)



ethyl 6-methylhydroresorcylate

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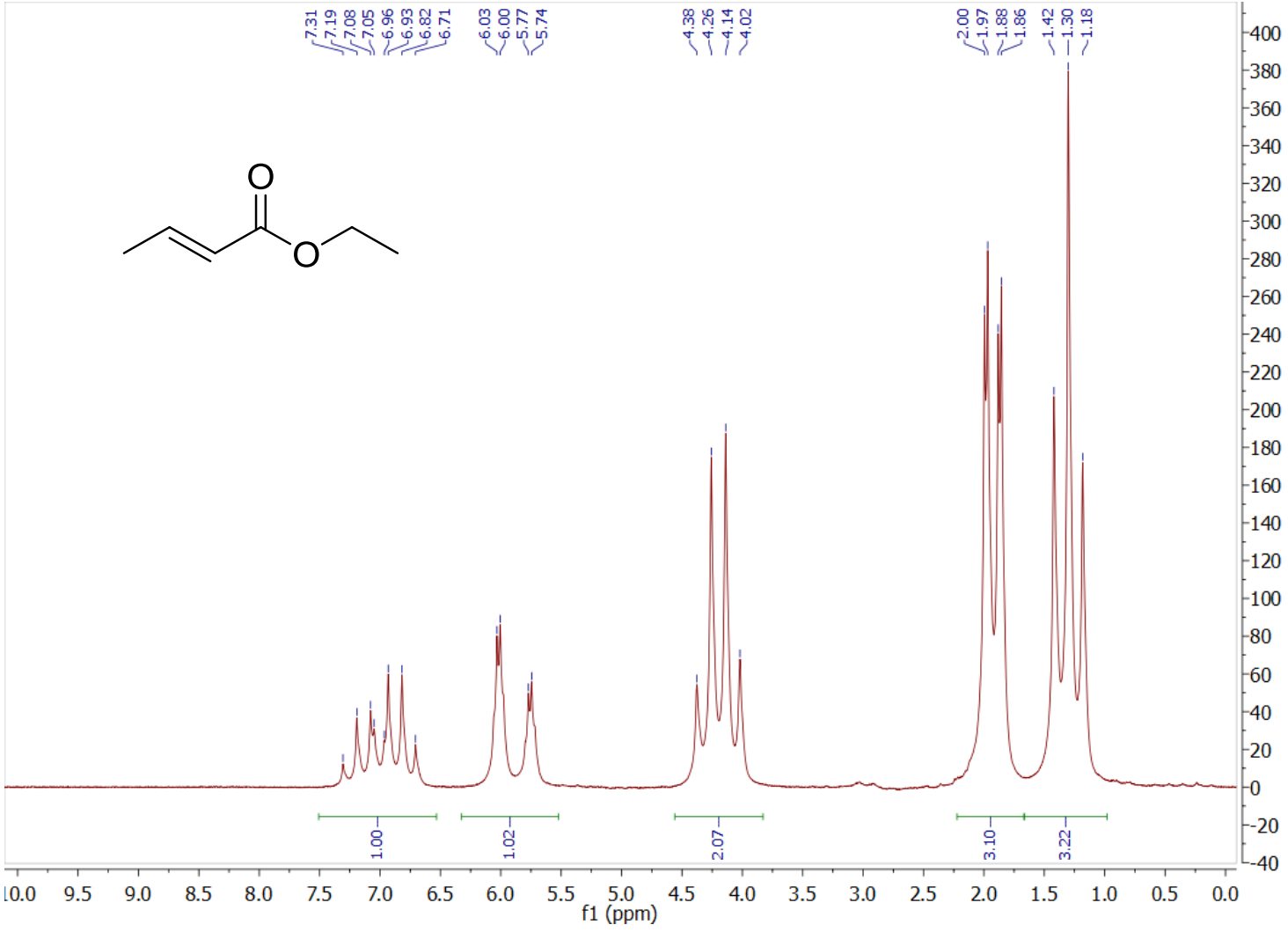

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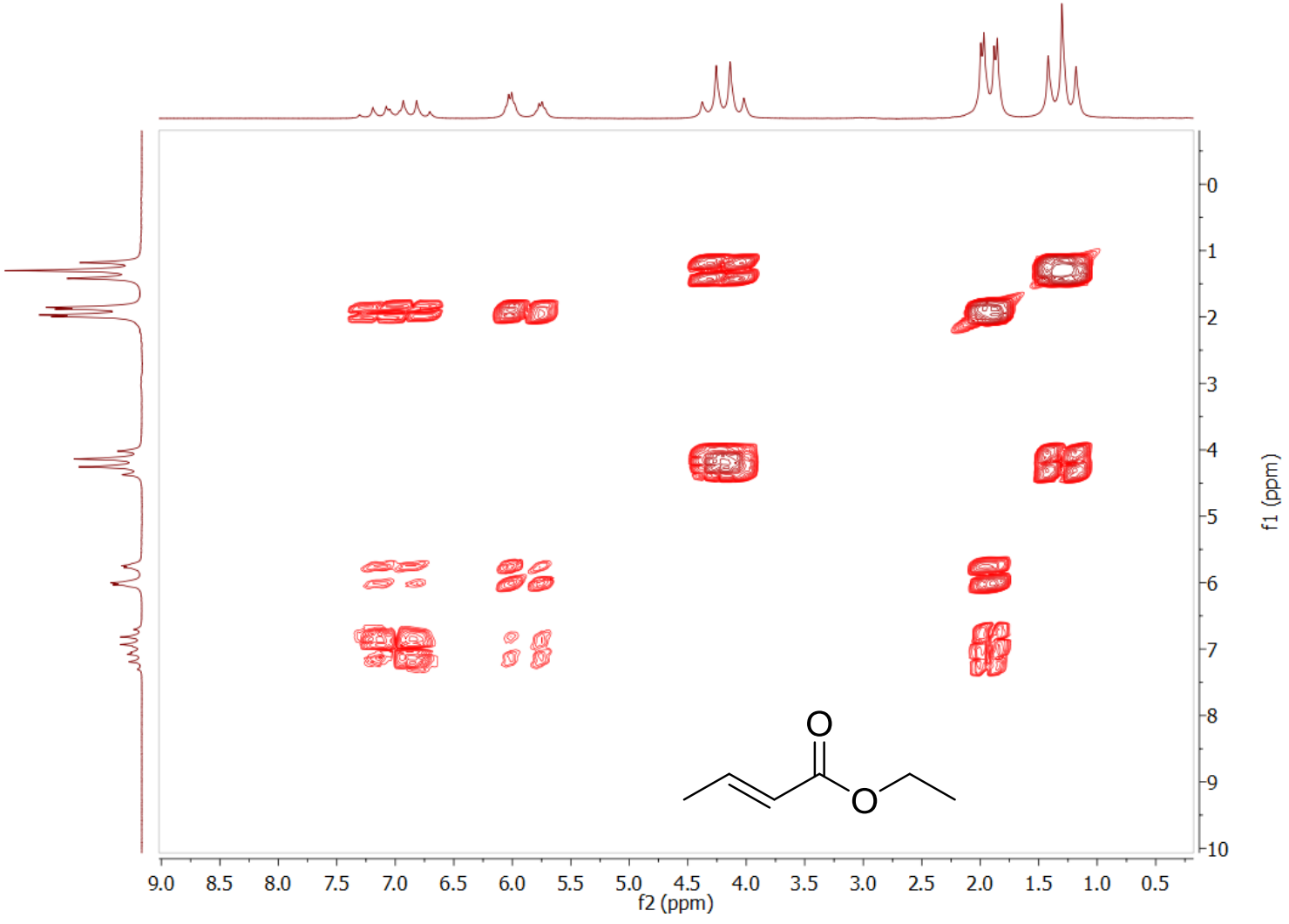

~ 30 min
collection time
2 M sample

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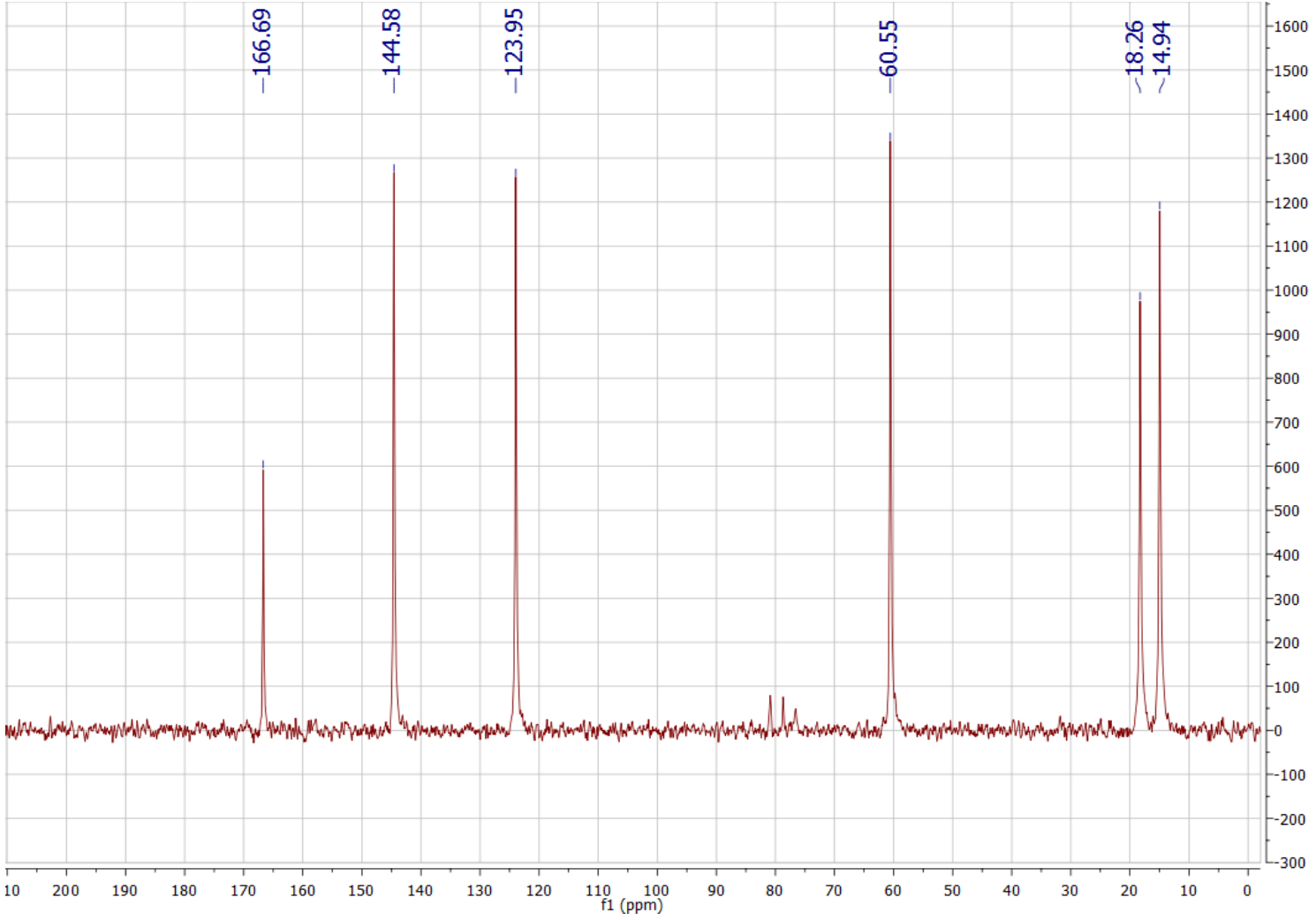
$^{13}C\{^{1}H\}$ spectrum of ethyl crotonate



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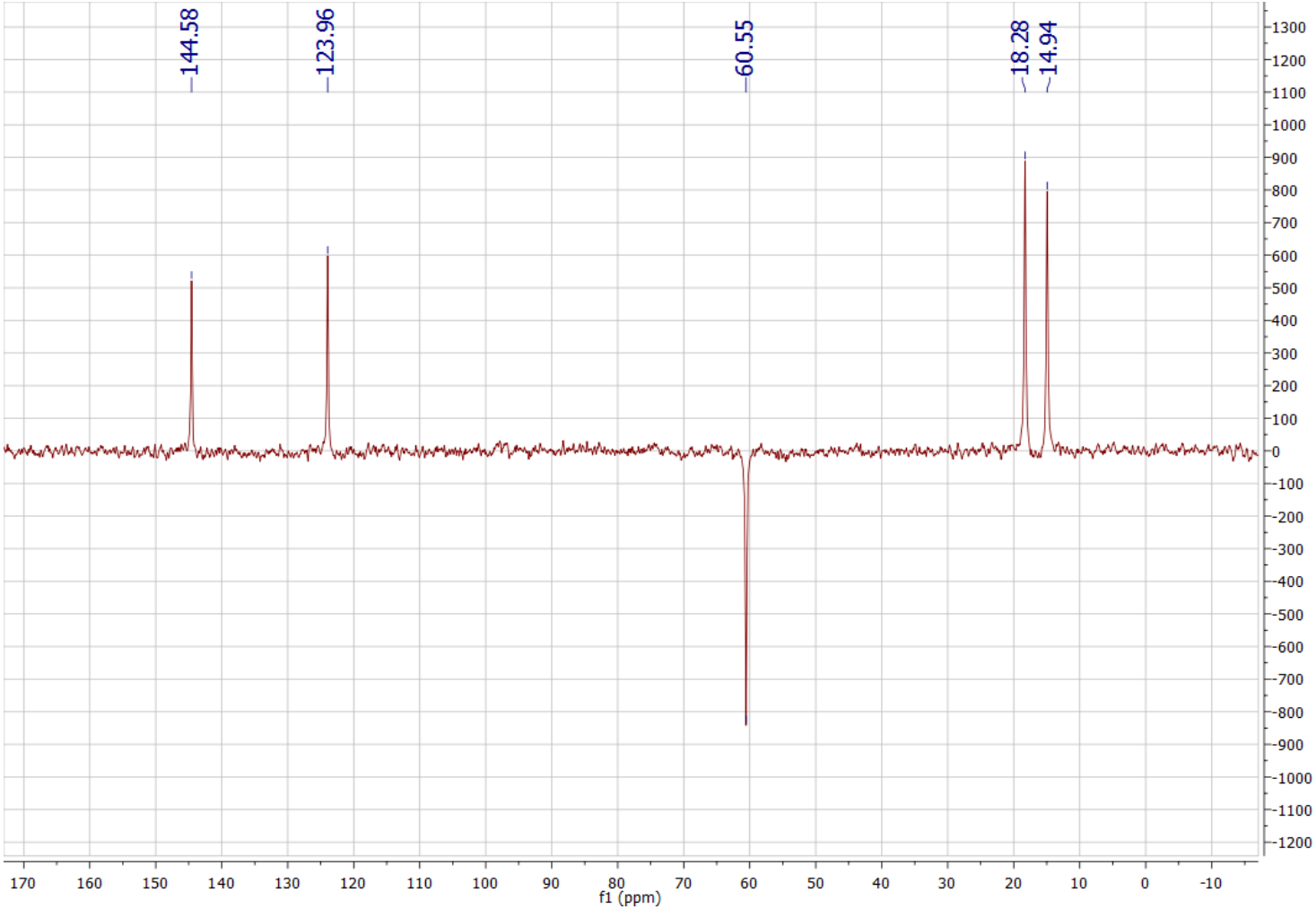
DEPT-135 of ethyl crotonate



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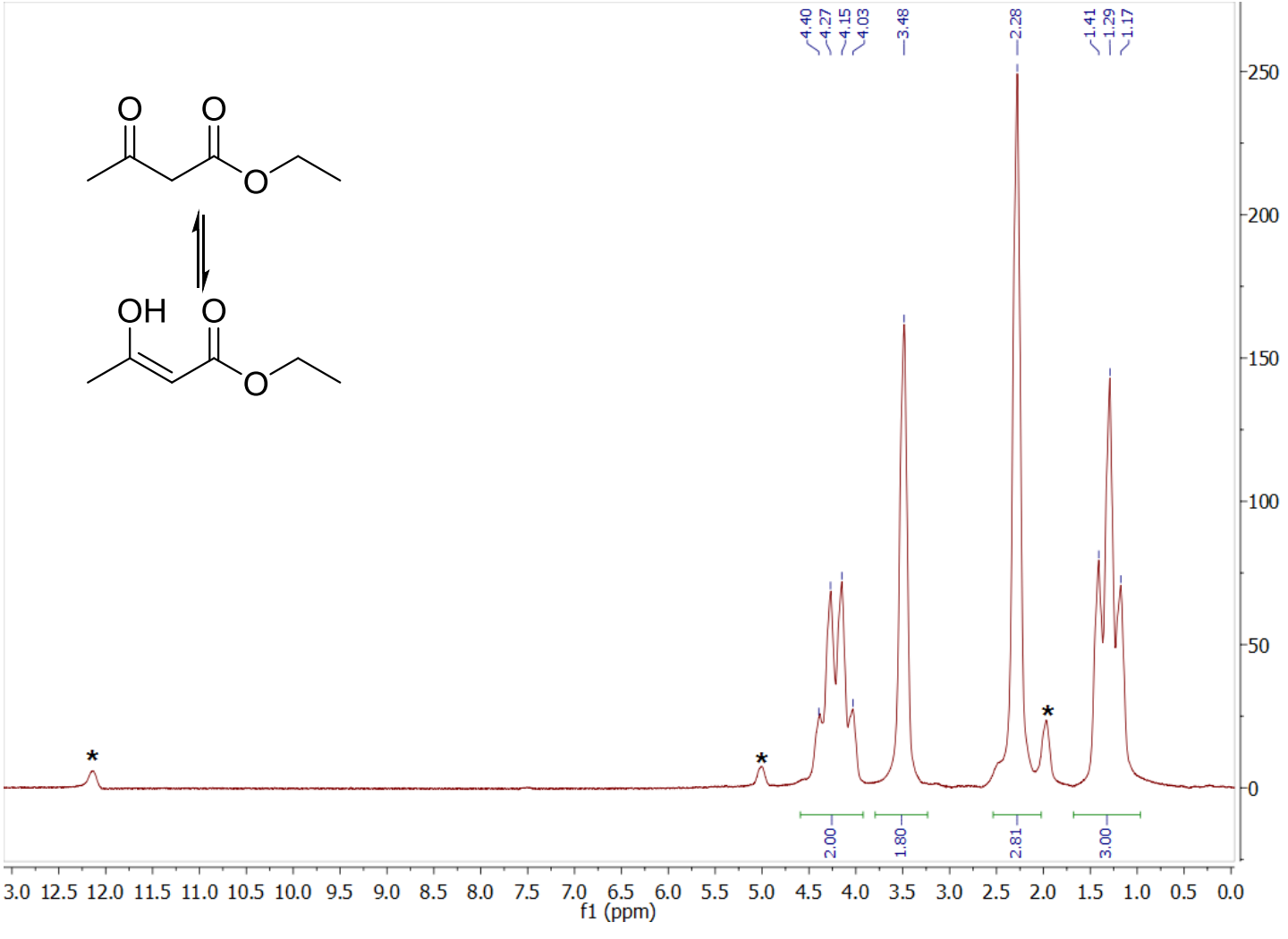

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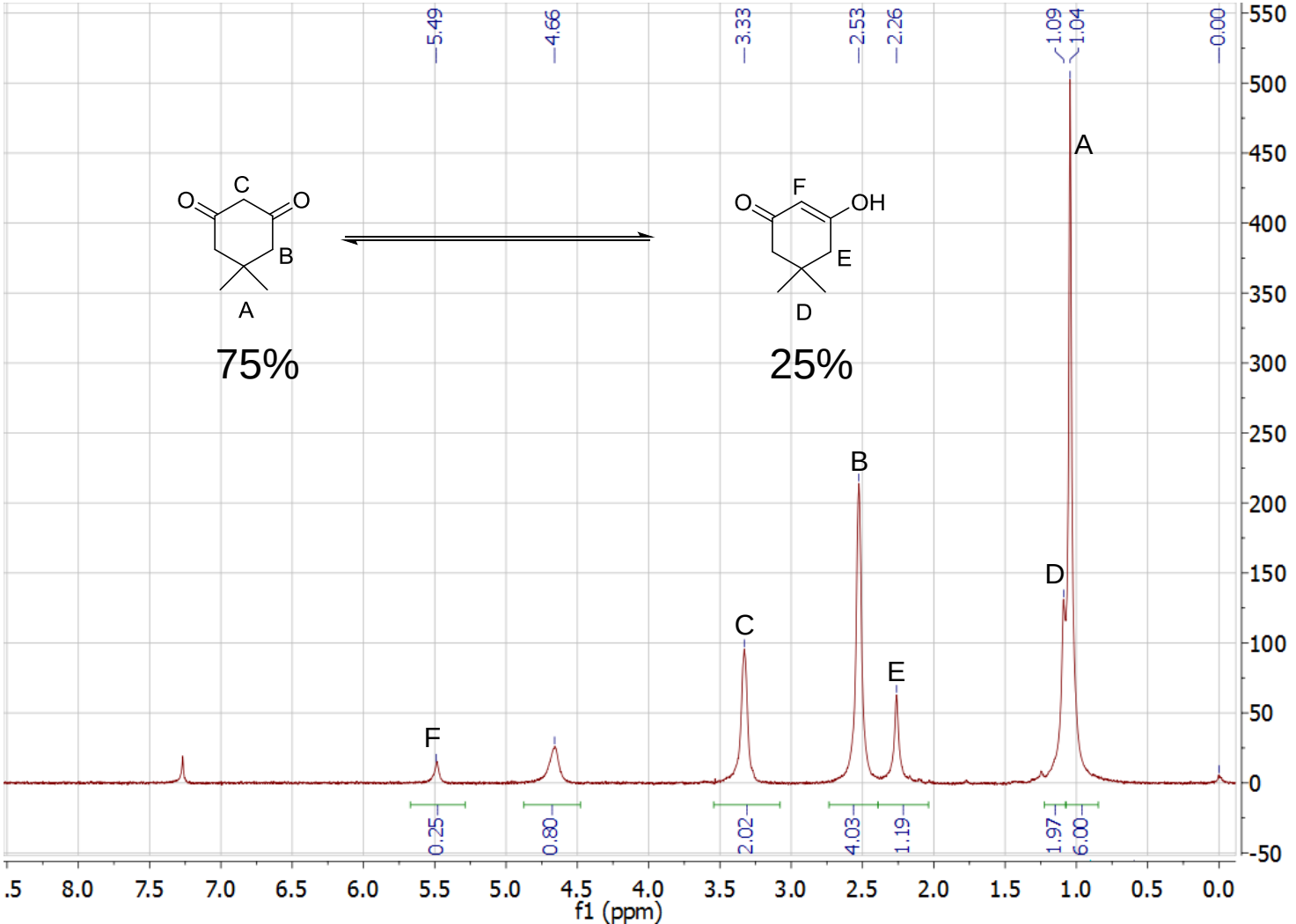
Analysis of dimedone



Acquisition time less than 1 min (sample concentrated)

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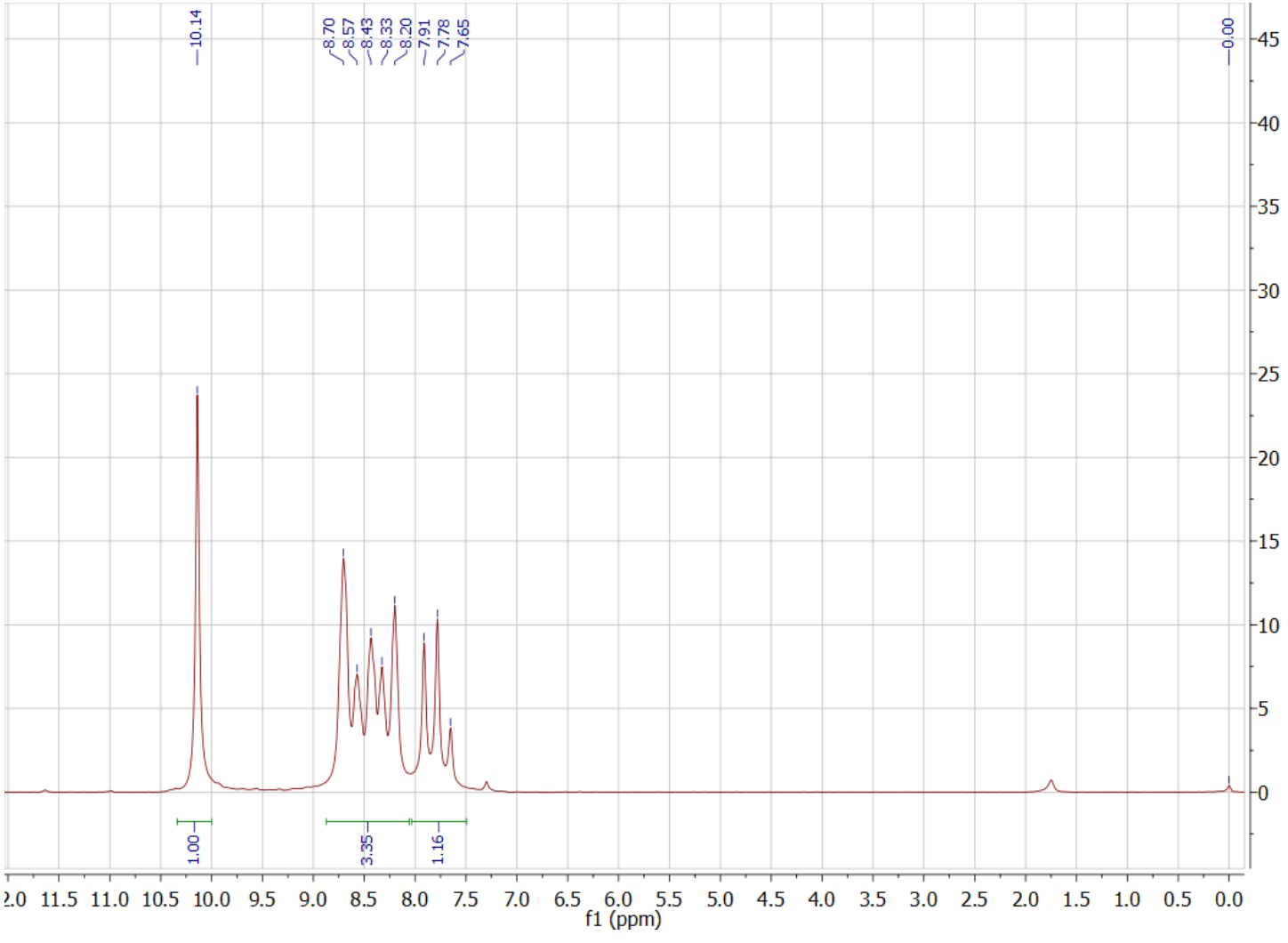

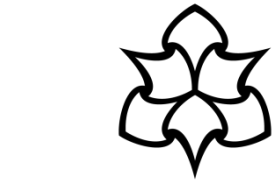

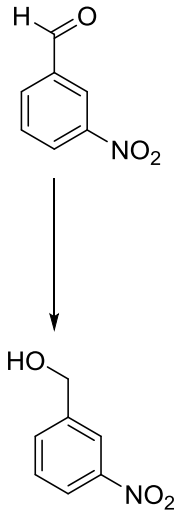

This spectrum was collected by FdSc students

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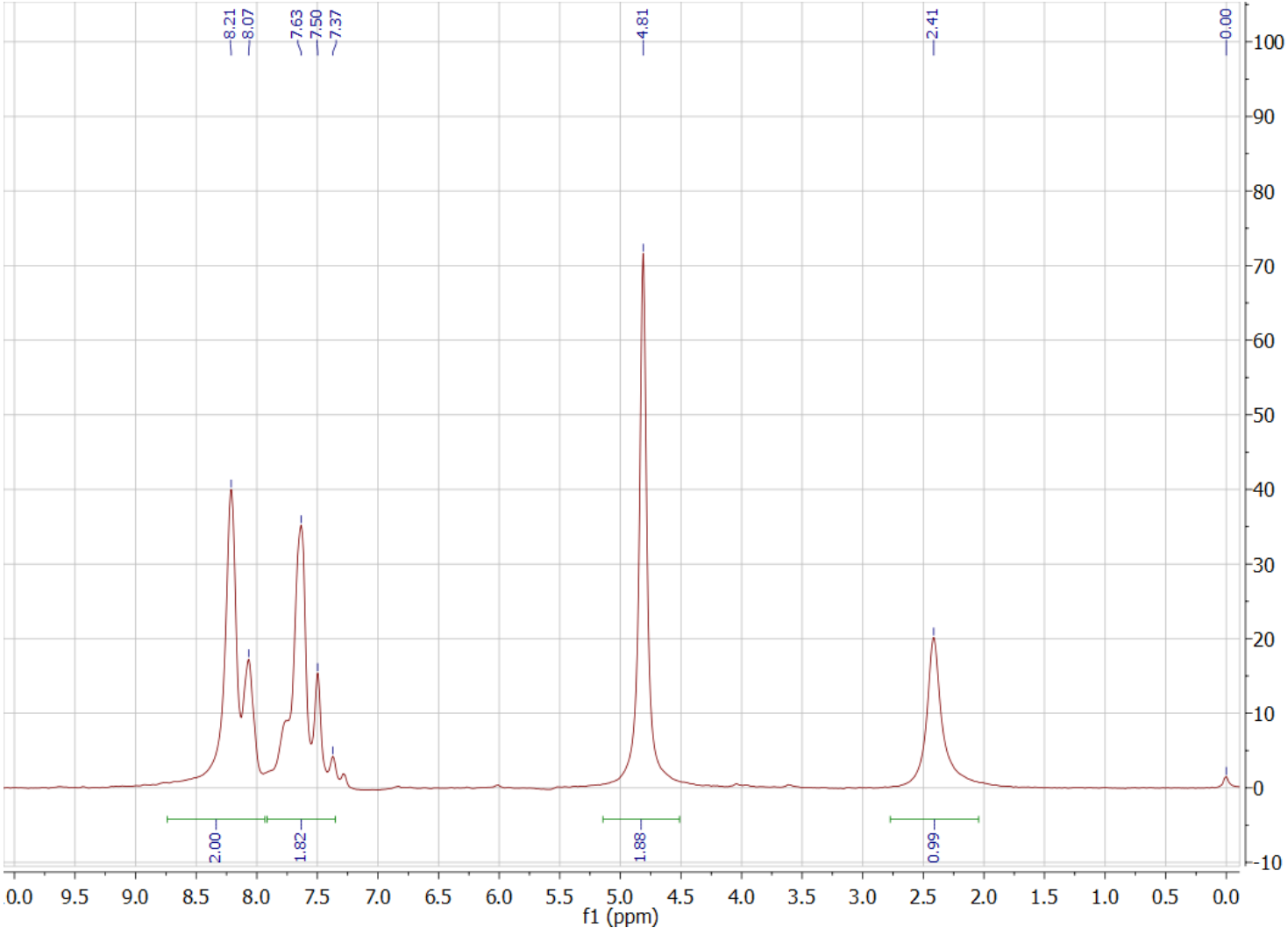

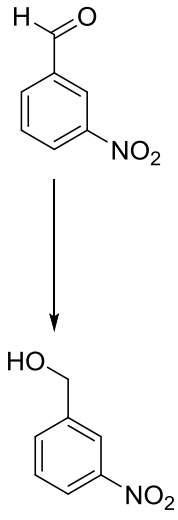

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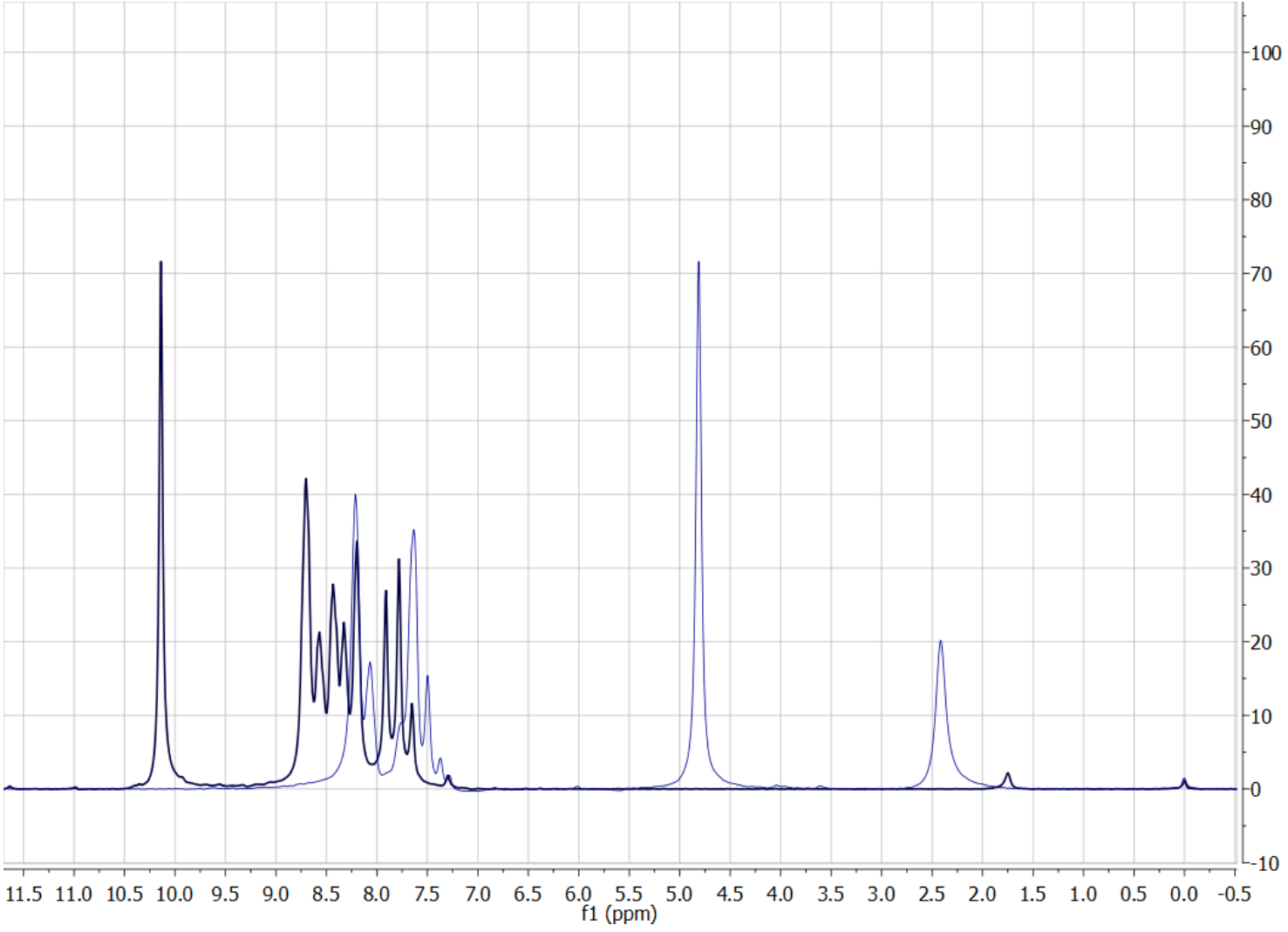
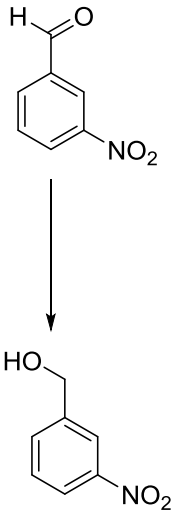
Overlay of the two products





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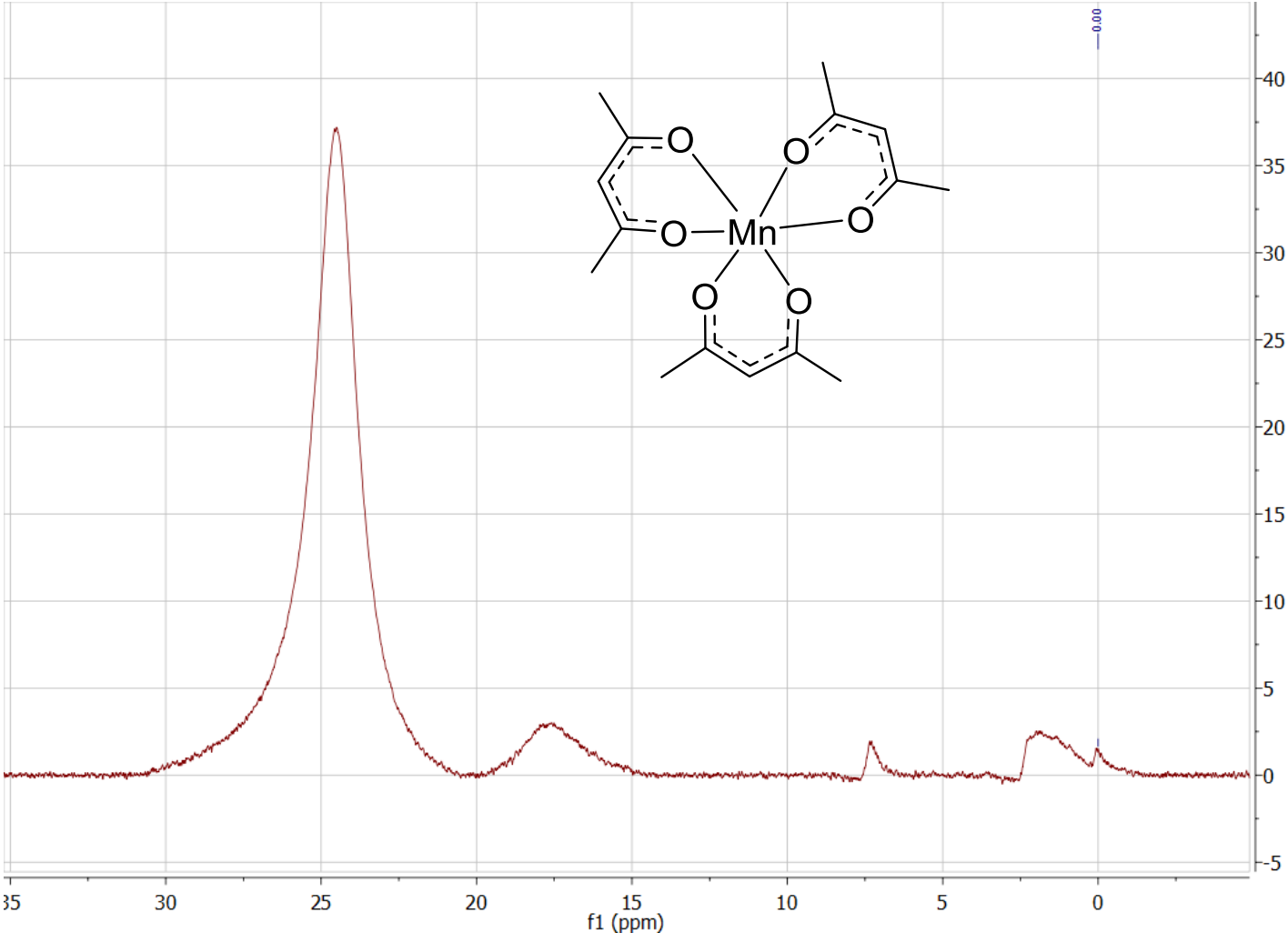
An inorganic example



Mn(III) – d^4 – high spin

Magnetic susceptibility measurements are also collected to compare to spectra collected (enable no. of d e^-s to be calculated)

Student comments on benchtop NMR and outputs

- Student responses collected as part of a PGCAP unit w.r.t. to coursework experiment

- “I am the type of person where I learn more when I am able to apply it, rather than theoretically doing stuff”
- “doing a practical as part of the coursework was good and gave me a better understanding”
- “a lot more fun to understand a new process”
- “knowledge [of NMR] is useful for future job prospects”

Where to next?

- Currently working on a ferrocene experiment for second year students
- Also considering Evan's method experiments (utilised by a member of staff for projects)

Student outputs


Using Pulsar to measure spin-lattice relaxation data to determine the longevity of polarised spin-states produced by a parahydrogen based hyperpolarisation technique

David Ashworth, Lisa Clayton and Dr Ryan Mewis, Manchester Metropolitan University.

Technical note prepared by two second year students (2015)