NMR

Sample preparation

and Analysis

Preparing the NMR Sample

- The NMR solvent is Chloroform-*d* ($CDCl_3$)
- The solvent is stored in the fridge, please return after use
- The 5mm NMR tubes and caps are at a designated point in the Lab

NMR tubes are made of high precision glass - treat with care

Preparing the NMR Sample

- To prepare a solid sample
  - carefully place approximately 5mg of the compound in the NMR tube
  - using a **NEW** pasteur pipette, put about 1ml of $CDCl_3$ into the NMR tube
  - the solution in the NMR tube should be about 5cm in height

  DO NOT reuse the pasteur pipette with the $CDCl_3$

- To prepare a liquid sample
  - using a **NEW** pasteur pipette, put about 1ml of $CDCl_3$ into the NMR tube
  - the solvent in the NMR tube should be about 5cm in height
  - now add One **VERY SMALL** drop of the liquid sample to the NMR tube

- Place the samples in the designated point in the Lab. The samples will be run for you. You can process the NMR data in the computer area with-in the Cocker lab or on your own computer

Labeling NMR samples

- Use the pink labels which are provided
- *DO NOT* use sticky labels
- Name as follows :

Have your full name on the sample label
Put the experiment number (1, …6) on the label
Put the name of the product on the label

The following code is used to retrieve the data :

JS_*xxxx*_01 for product from experiment 1
JS_*xxxx*_02A for first product from experiment 2
JS_*xxxx*_02B for second product from experiment 2 *etc.*

Where xxxx is the code assigned to your name

NMR data analysis

The Bruker *TOPSPIN* package is used to analyse and print the NMR data

Data can be processed in two ways :

(i) on 4 networked PCs in the Cocker laboratory
(ii) on your computer, if you download the Topspin software

Downloading the Bruker Topspin package for use on your computer

- The Topspin program can be downloaded from the Bruker website for free use to academic users at *Bruker.com*

- goto *service , support –software downloads, nuclear magnetic resonance*

  - see area of the page highlighting free Topspin for academic users

  - follow the link and instructions for **TOPSPIN 3.5**

Proton (^{1}H) spectra

- Submit sample as instructed in the Laboratory Manual
- Analysis is carried out using the *TOPSPIN* program
- Open the TOPSPIN program
- The NMR data is found under JS_2017

Analysis of Proton NMR

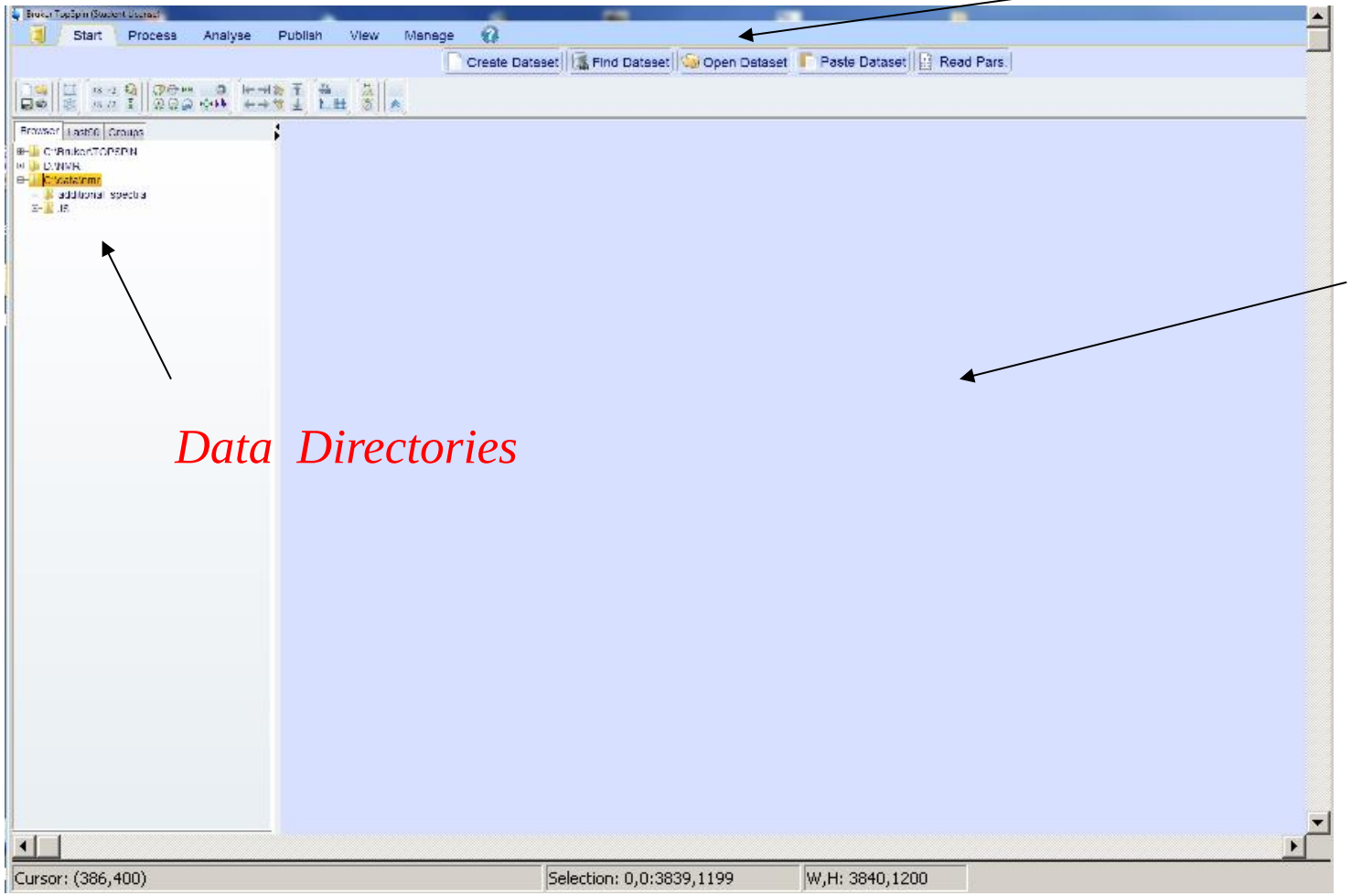
- You must submit printout of proton NMR(s) as indicated in the laboratory manual for each experiment

- Use the TOPSPIN program to analyse you NMR data

  - Load your data *JS_your name 0***
  - Peak pick the signals
  - Integrate the signals
  - Adjust the spectral window as appropriate
  - Print the screen

TOPSPIN NMR program

Command Lines and Pull down Menues



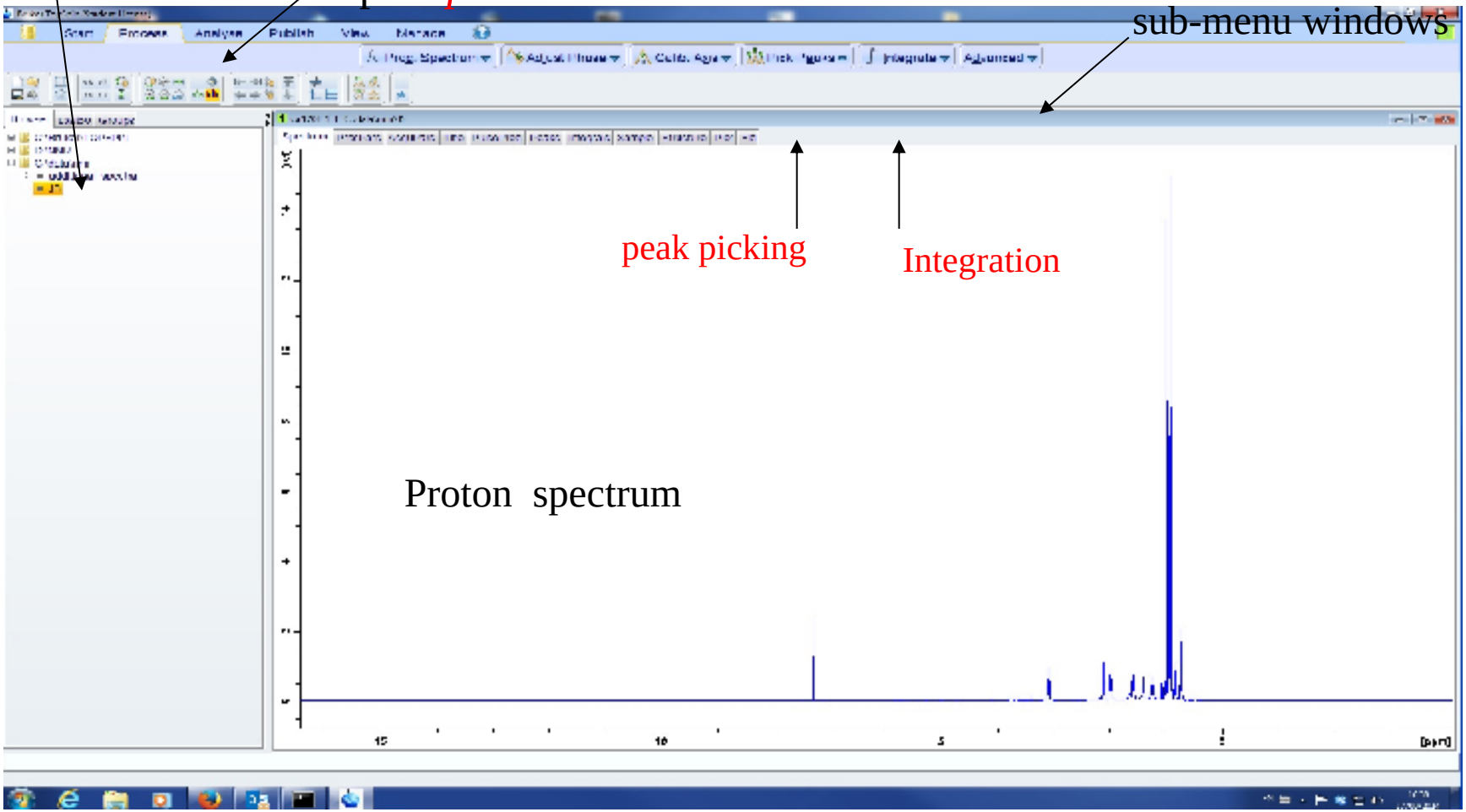
Spectral window

Data Directories

Look for NMR data in the C:\ data\nmr\js_2017 directory

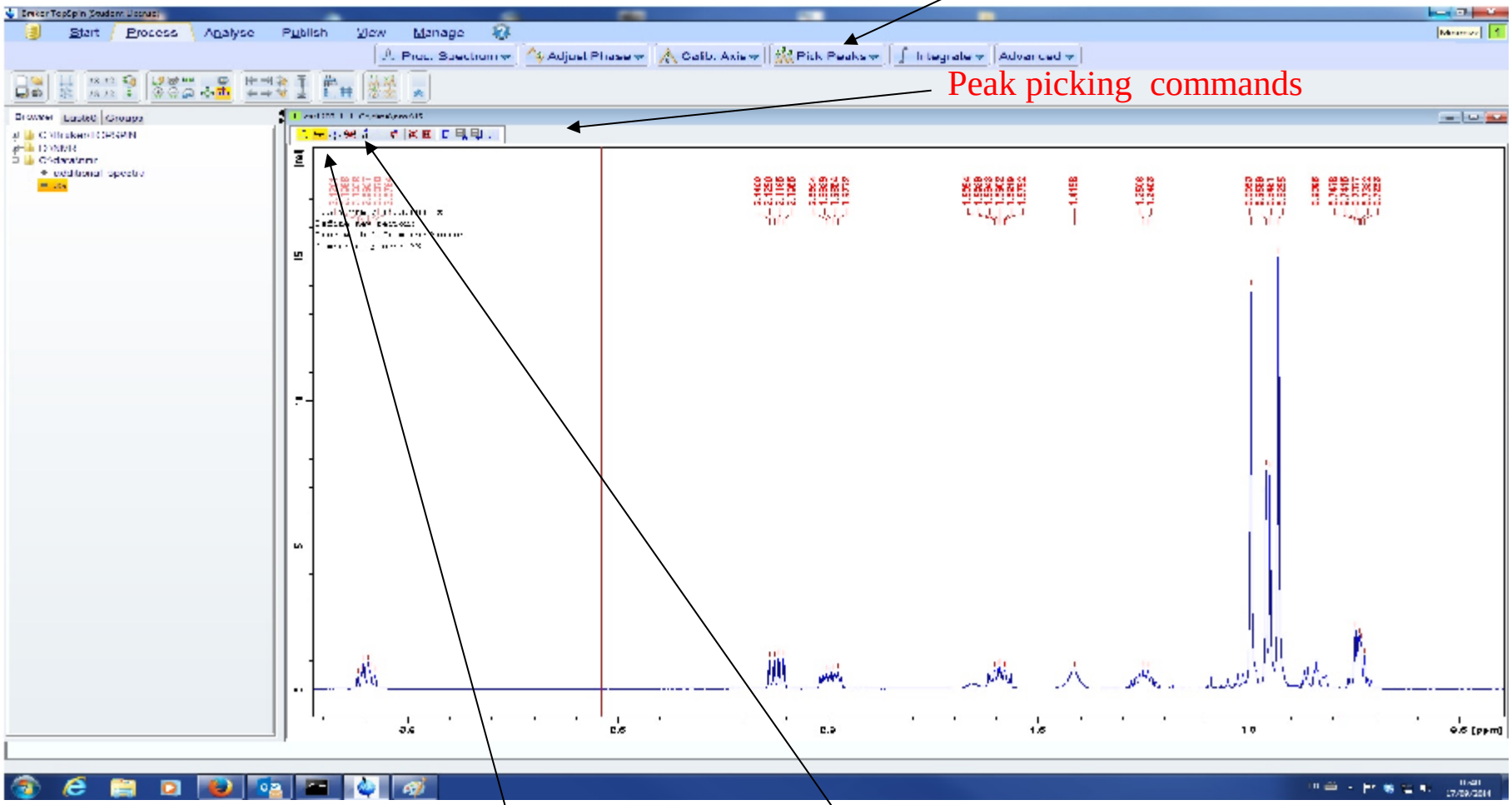
Processing NMR data - Proton

Load your file *JSxxx0?* 1 1 from C:\data\nmr\js_2017 into the spectral window



Peak Picking

Open the pick picking routine by highlighting the peak picking icon

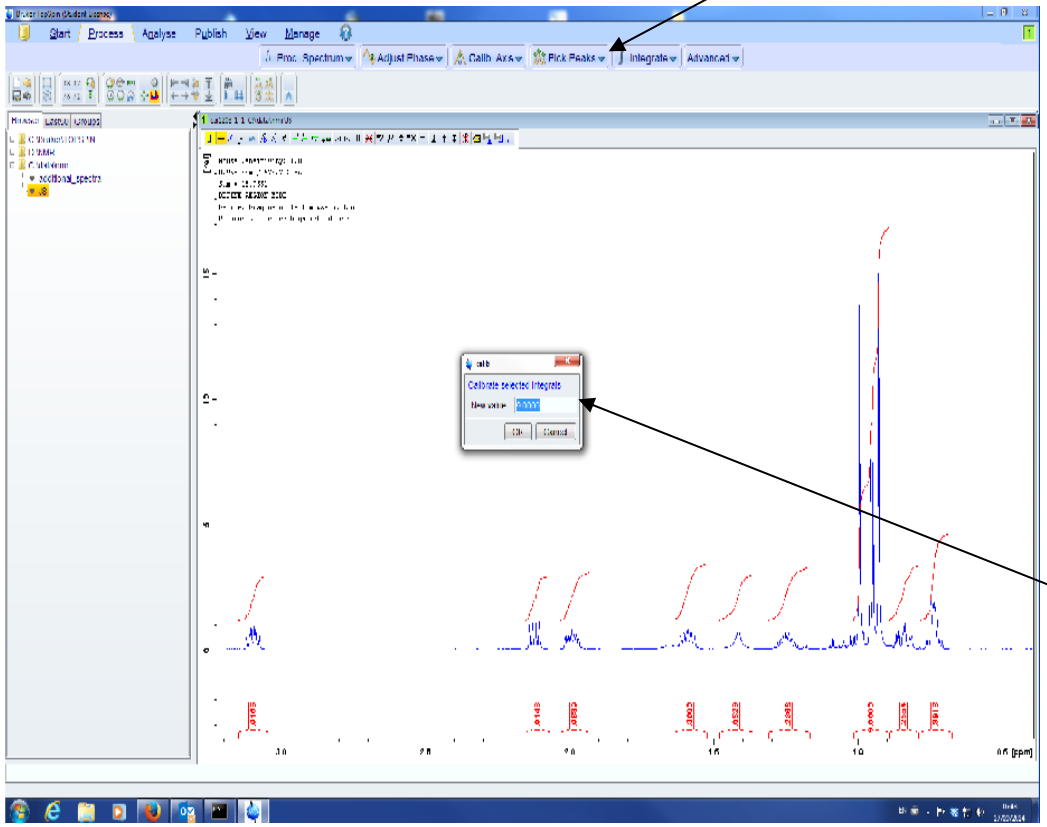


Peak pick all signals by region (default) or individually, then save to exit

Integration

Open the Integration routine by highlighting the Integrate icon

Integral Icon



Further commands are activated with the right-hand mouse key

Place cursor over an integral

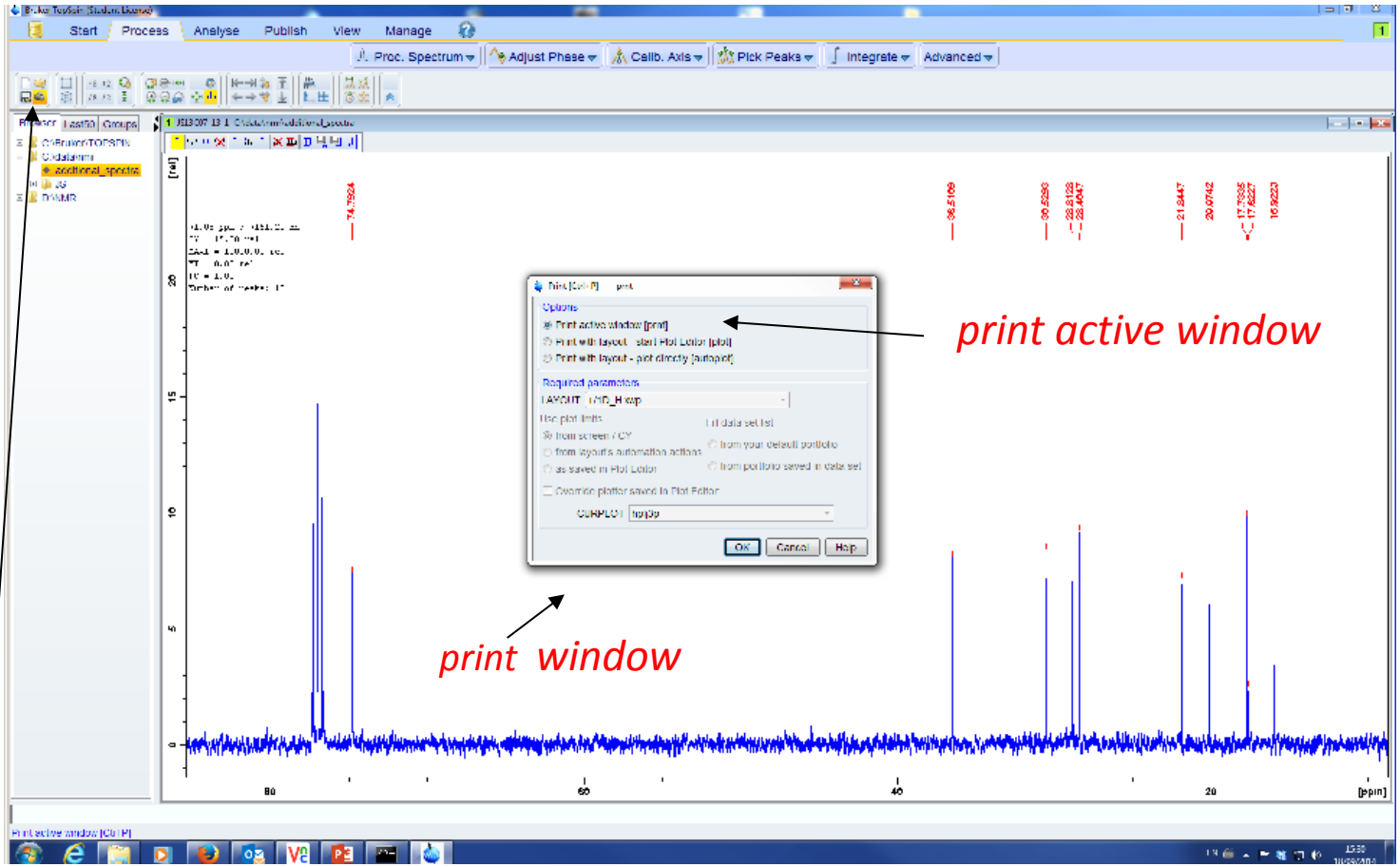
Use the right-hand mouse key

Highlight with Select/deselect function

Use Calibrate to normalise the integral by setting an appropriate value

Peak all areas to be integrated by region, then save to exit

Preparation for printing



To access items for printing - click on printer icon

a *print window* will appear on screen

ensure that the *print active window* is active

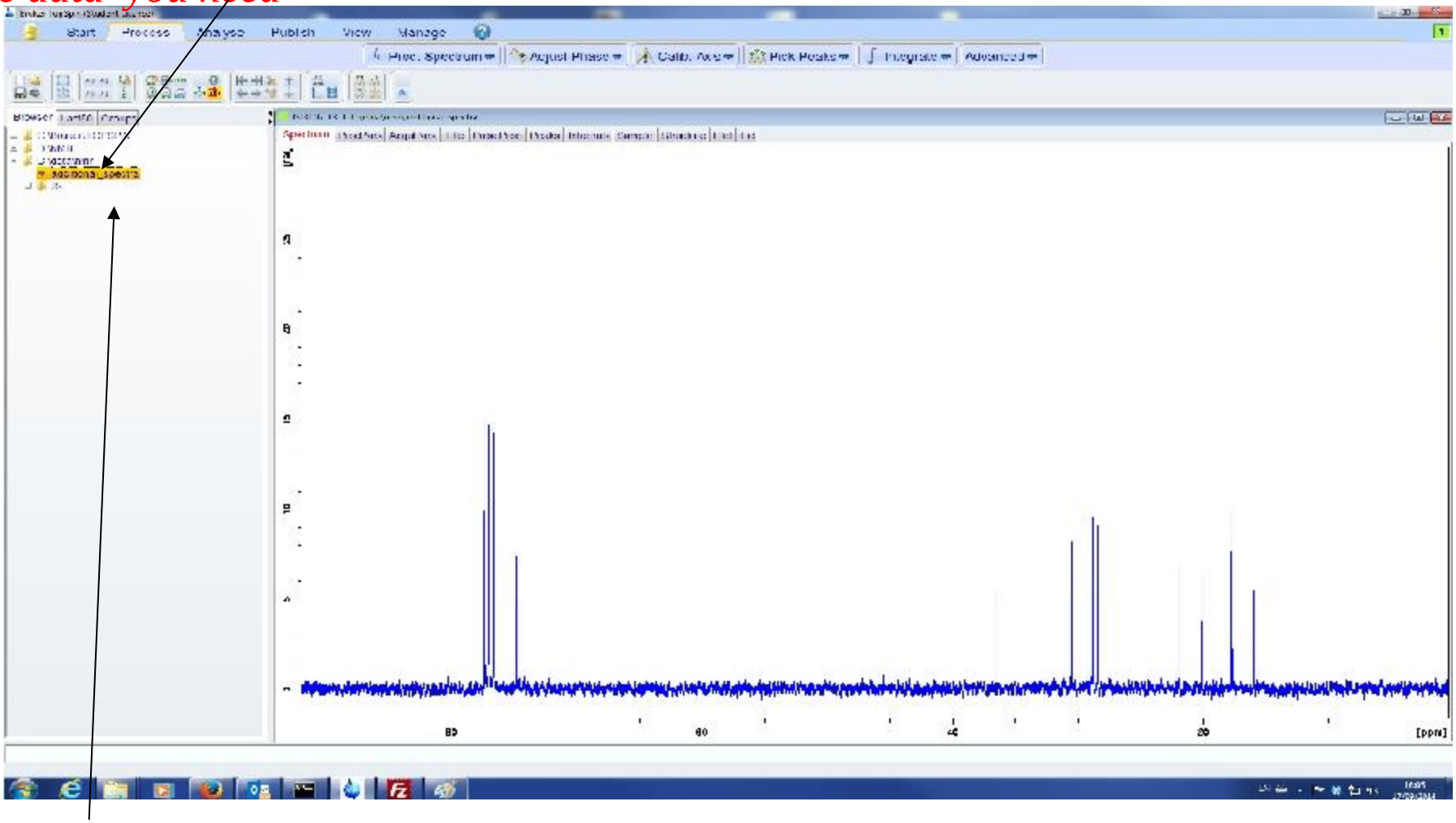
click on OK to print

Carbon-13 spectra

- Carbon-13 spectra have been prepared for *all* samples where this NMR information is required
- Carbon DEPT 135° and 90° spectra are also provided
- The data is to be found using TOPSPIN in the C:\data\nmr***Additional Spectra*** directory as *JS13C01_carbene, JS13C02A_benzyl_phosponate, etc.*

To get the 13C data : goto the ***Additional_Spectra*** *directory*

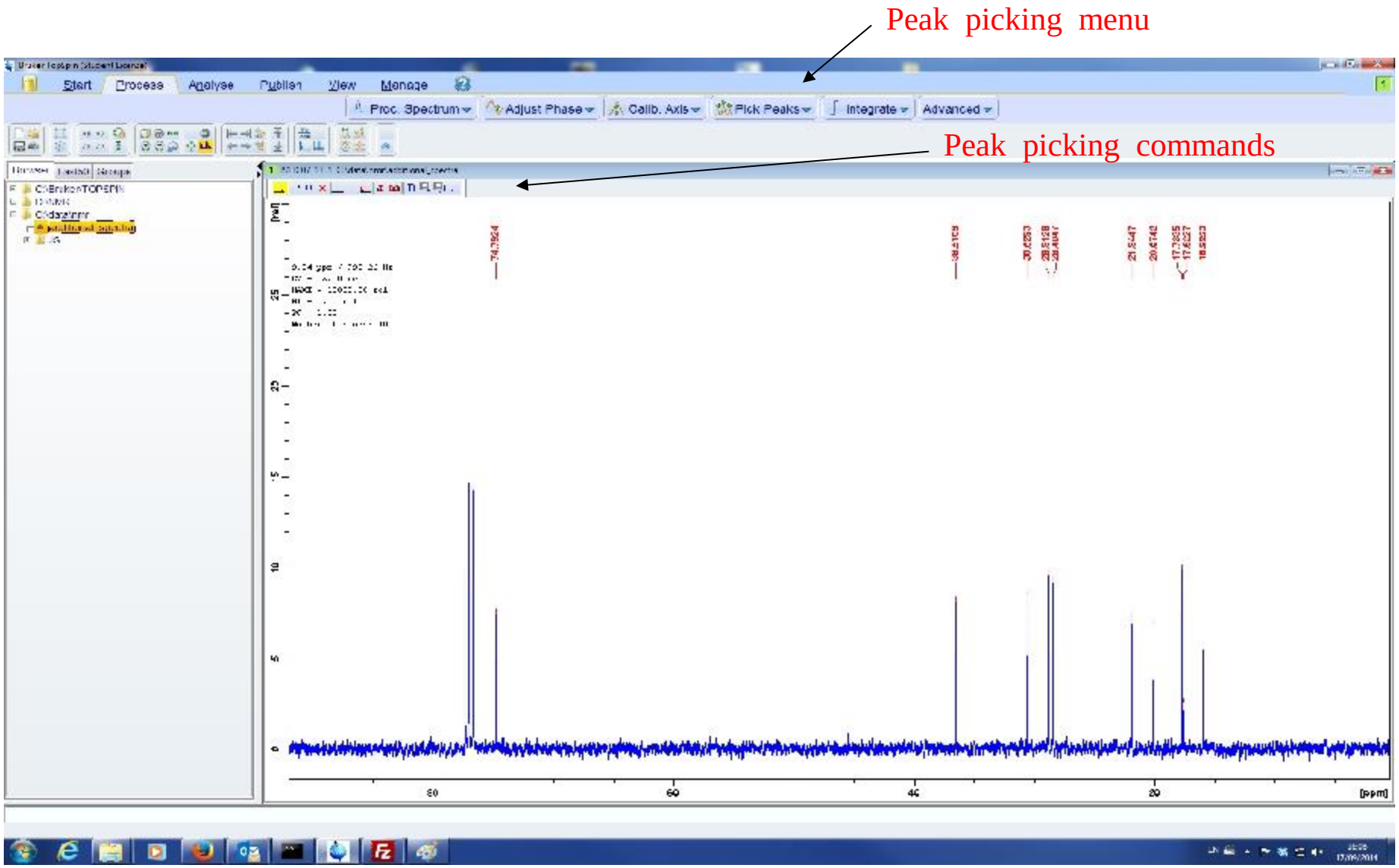
look for the JS13C01_carbene to JS13C07_caranol directories and highlight the data you need



^{13}C is 13, *DEPT 135° is 14, DEPT 90° is 15*

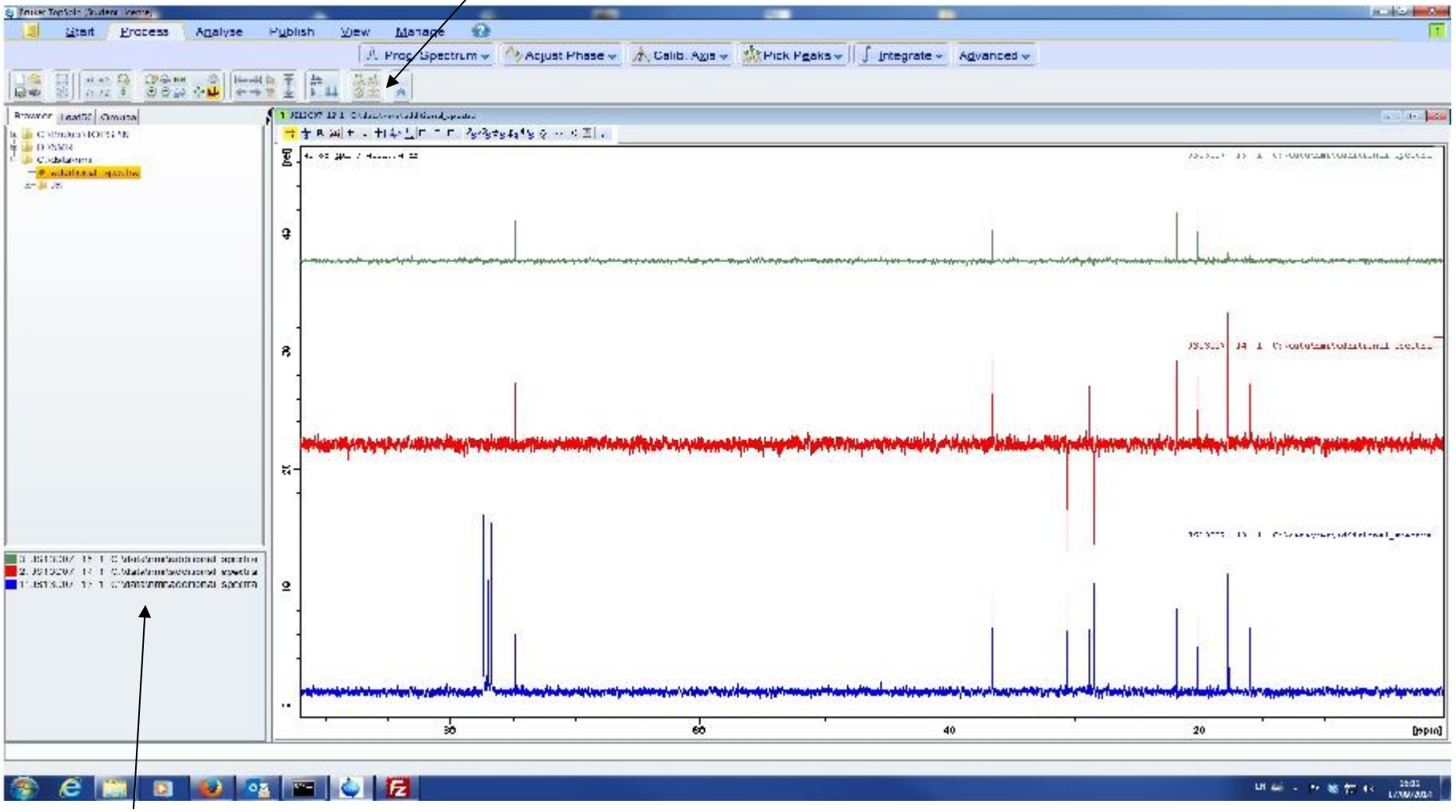
e.g. JS13C06_caranol 13 1 will load the 13C data for sample 6

Peak Pick the Carbon-13 signals



Peak pick all signals by region (default) or individually, then save to exit

Multiple display function



This function allows multiple spectra to be compared
In this case the CARBON-13 and DEPT 135° , DEPT90°
Open the relevant directory and load the 13, 14 and 15 sub-directories
To print - Open File and use the print command

Phosphorous -31 NMR

One Phosphorous-31 NMR has been prepared

This spectrum is found in the C:\data\nmr***Additional Spectra*** directory as ***JS31P02a_phosphorous_NMR***

Load this spectrum, peak pick and print

The procedure is the same as for Carbon-13 NMRs