UNIVERSITY OF Southampton

CHEM6026(8032)

NMR Spectroscopy & Mass Spectrometry of Organic Compounds

Lecture 3 – ACD/Labs NMR Processing Software

Dr Neil Wells (njw3@soton.ac.uk)
Building 30 Room 1065

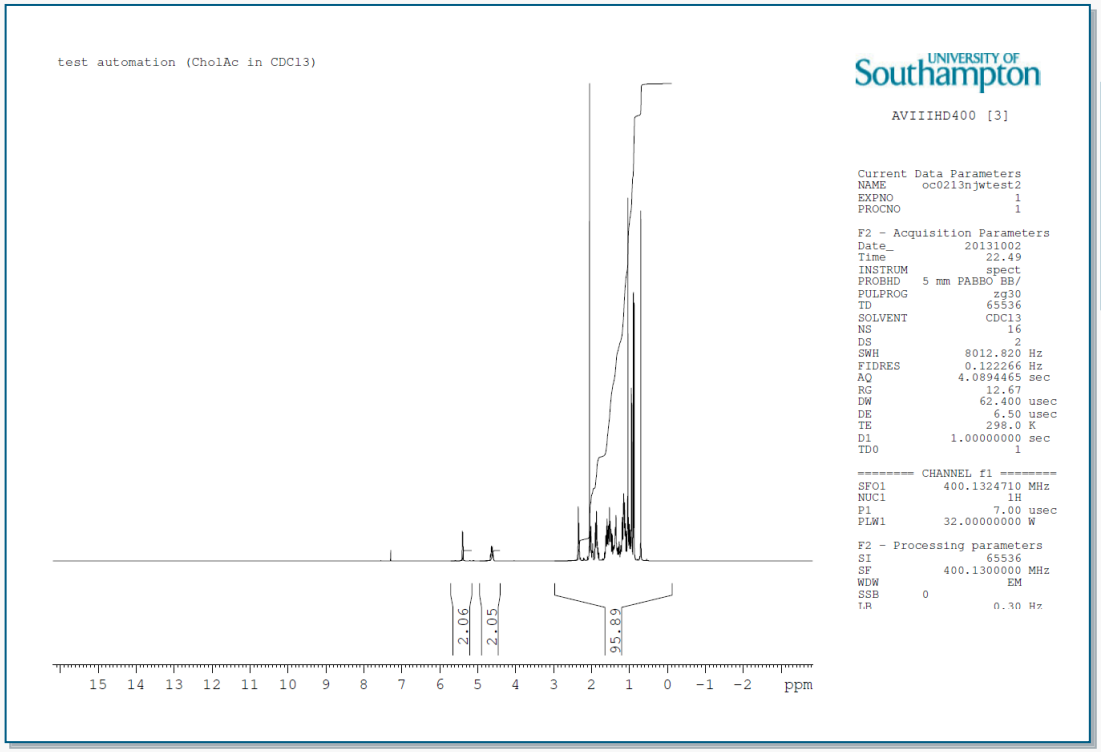
File Locations

- Current instrumentation
  - AVII400 (1) ftp://152.78.198.53/
  - AVII400 (2) ftp://152.78.199.41/
  - AVIIIHD400 (3) ftp://152.78.198.67/
  - AVIIIHD500 ftp://152.78.199.49/

- Legacy instrumentation
  - AV300/1, AV300/2, DPX400/1, DPX400/2
  - Legacy data stored on the following FTP server: ftp://152.78.196.44/

Data Format

- Processed data available automatically:
  - pdf (400s & 500)



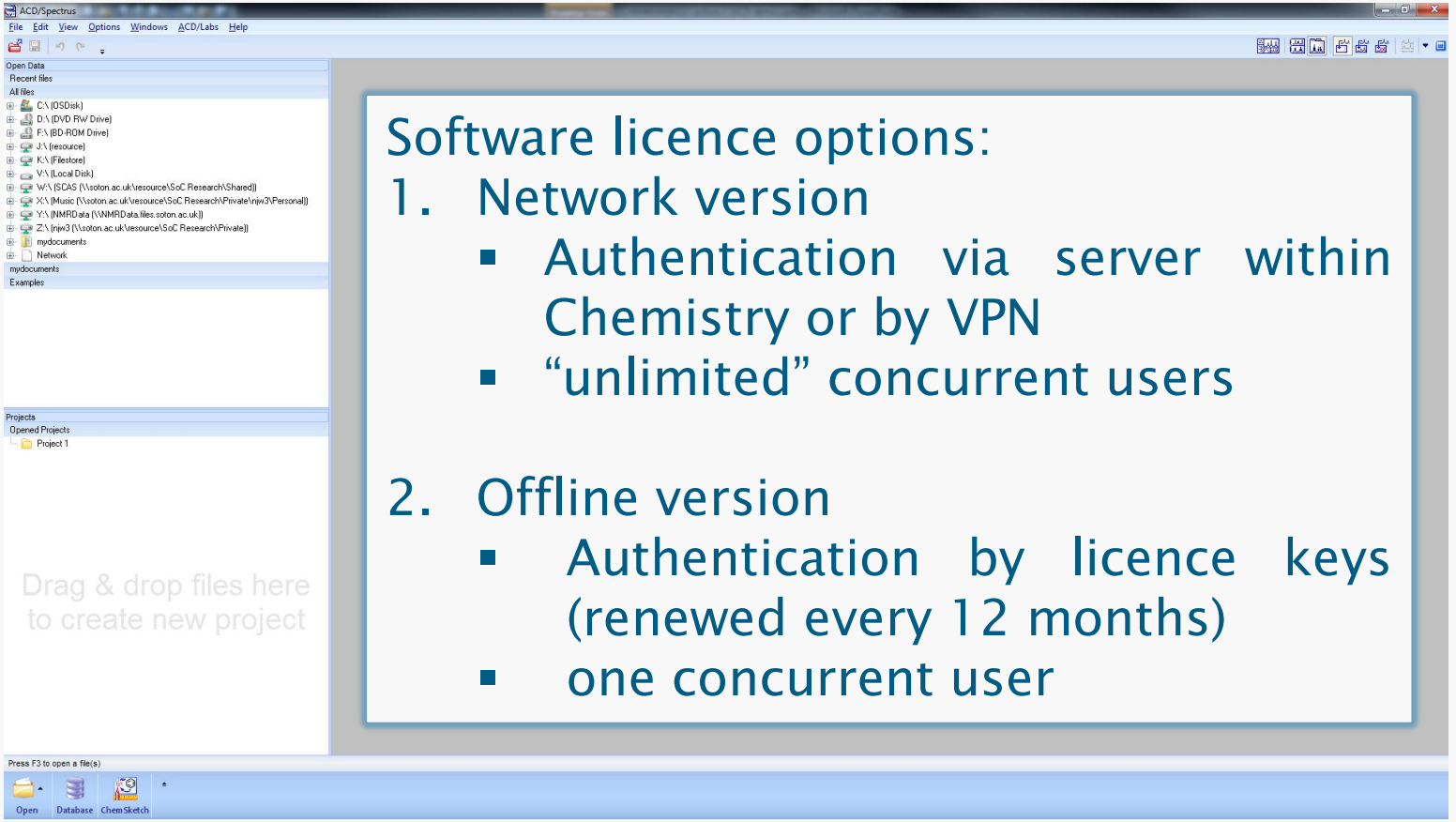

```
Current Data Parameters
NAME      oc0213njwtest2
EXPNO                  1
PROCNO                 1
```

Raw data stored in EXPNO: fid (1D); ser (2D)

Processed data stored in ..\pdata\1\

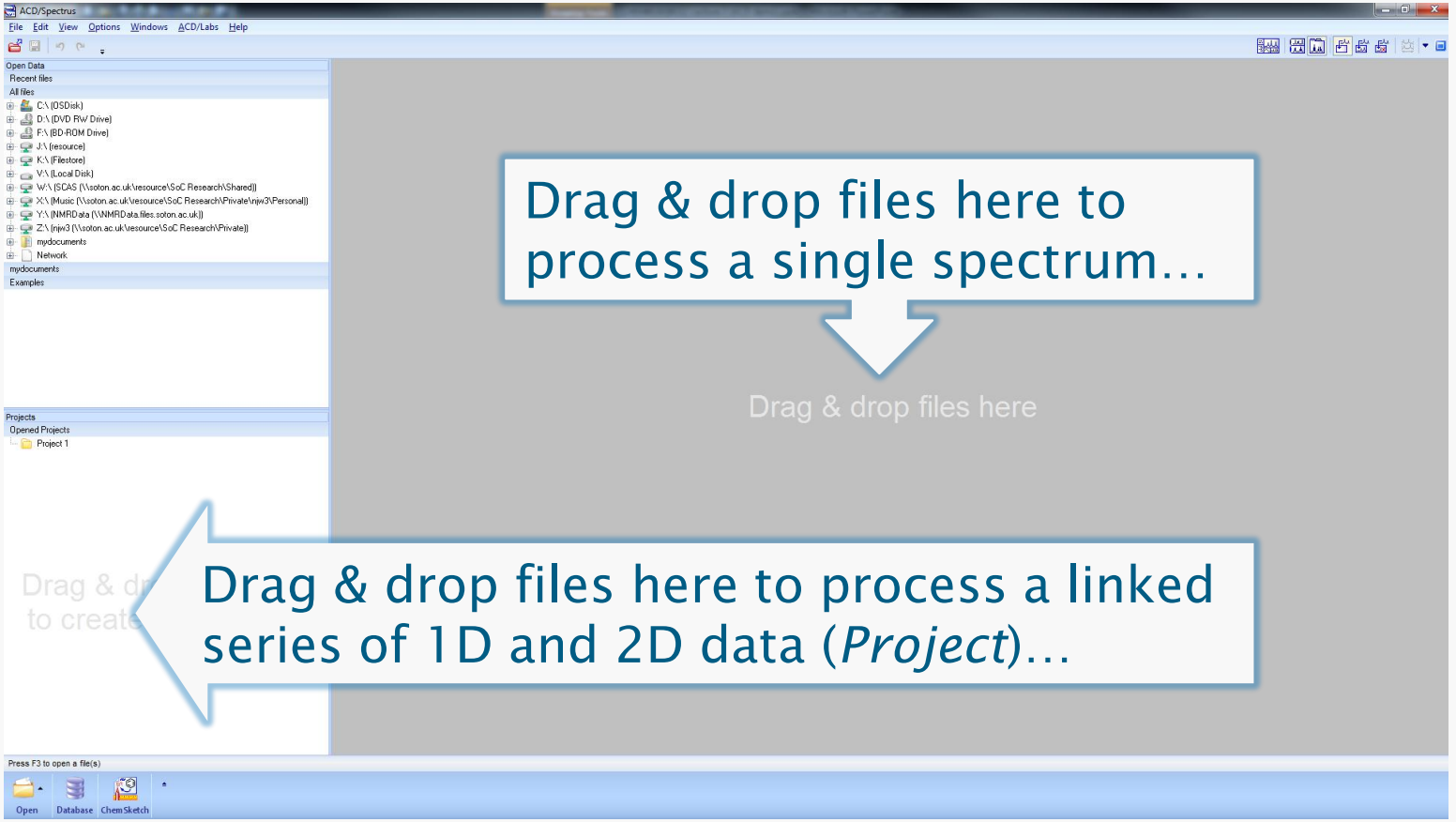
ACD/Spectrus

- Download software from:
  ftp://152.78.196.44/

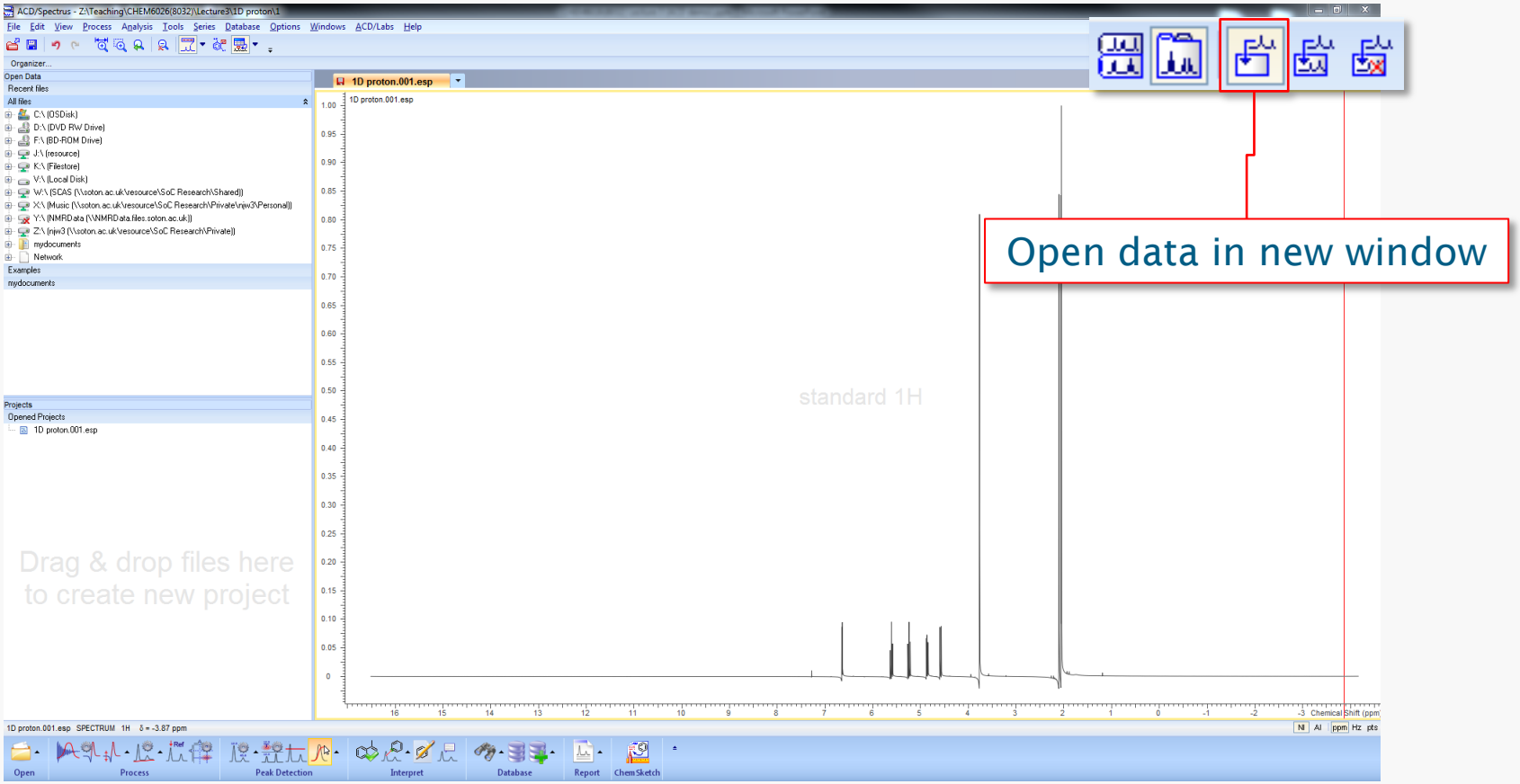


ACD/Spectrus

- Download software from:
  ftp://152.78.196.44/

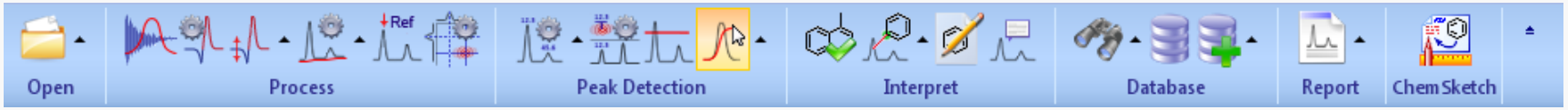


Processing 1D Data



Processing 1D Data: Workflow

- The workflow bar is context sensitive (different for 1D & 2D data).



1. Interactive FT
Exponential (EM) Window Function
^{1}H LB = 0.1–0.3 Hz
^{13}C LB = 2.0 Hz

Gaussian (GM) Window Function
^{1}H LB = –1; GB = 0.4 Hz

2. Phasing
Automatic or manual

3. Reference
Button (or right-click on a peak)

4. Add structure (if known)
Draw in ChemSketch (or paste from ChemDraw)

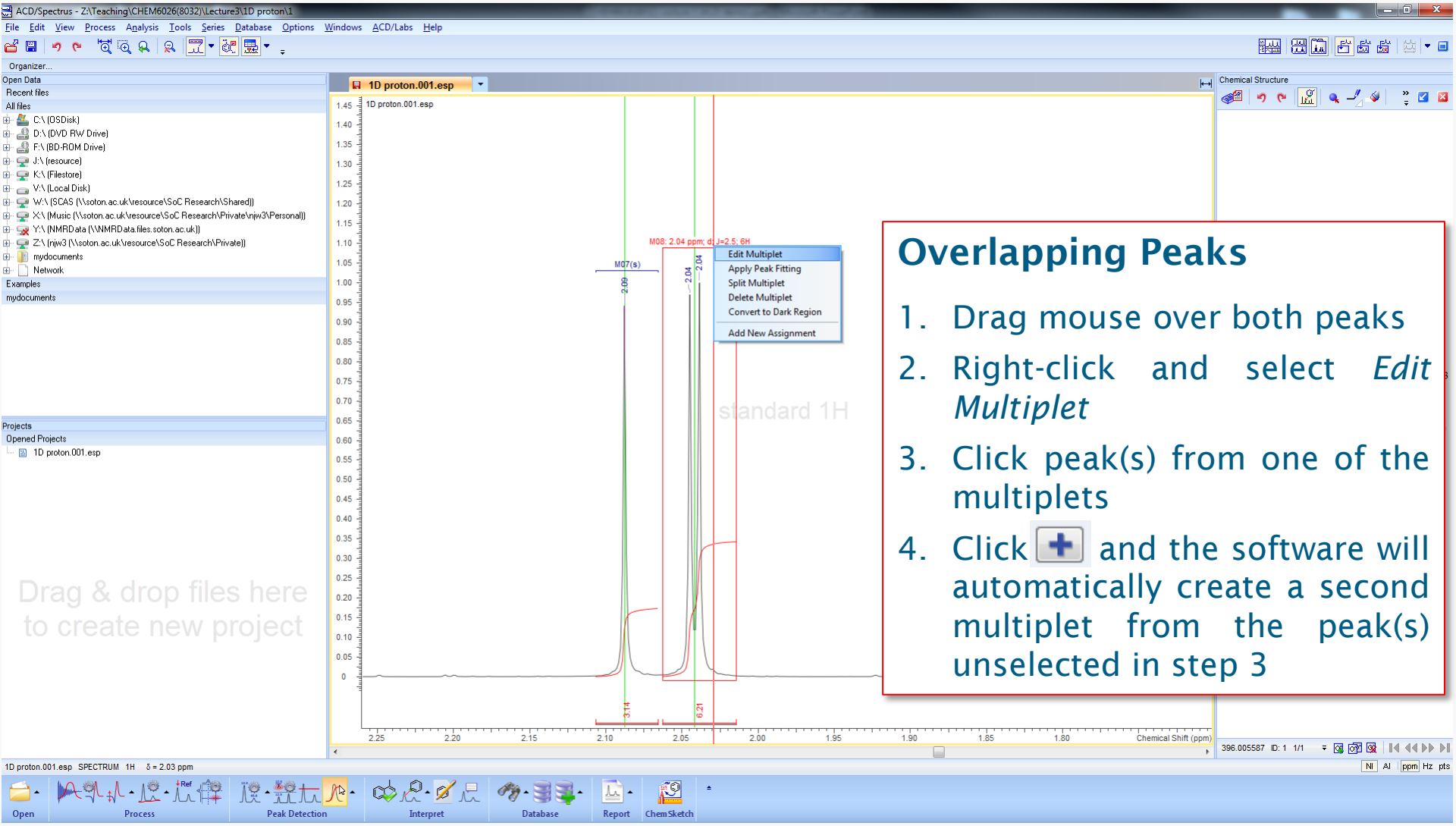
5. Peak Assignment
Automatic or peak-by-peak

Resolution Enhancement (GM)

- Distortions around the baseline are not unusual and are perfectly acceptable.
- Resolution enhanced spectra should never be integrated as the results will be meaningless.
- Check the spectrum against a non-resolution enhanced copy to guard against spurious splitting artifacts generated resulting from a split-field.
- **Always check that a signal that must be a singlet remains one following Gaussian multiplication.**

Peak Assignment

Peak Assignment

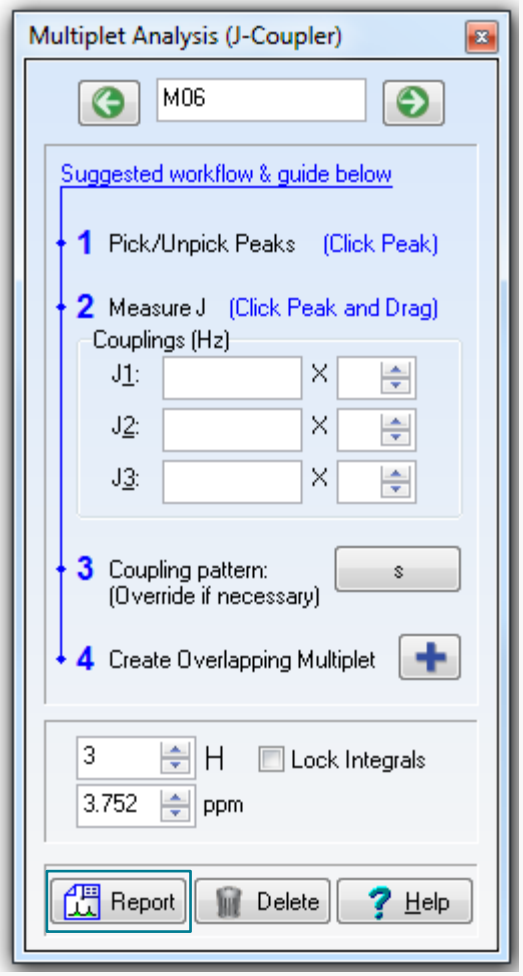


Overlapping Peaks

1. Drag mouse over both peaks
2. Right-click and select *Edit Multiplet*
3. Click peak(s) from one of the multiplets
4. Click + and the software will automatically create a second multiplet from the peak(s) unselected in step 3

Peak Assignment

Multiplet Reports

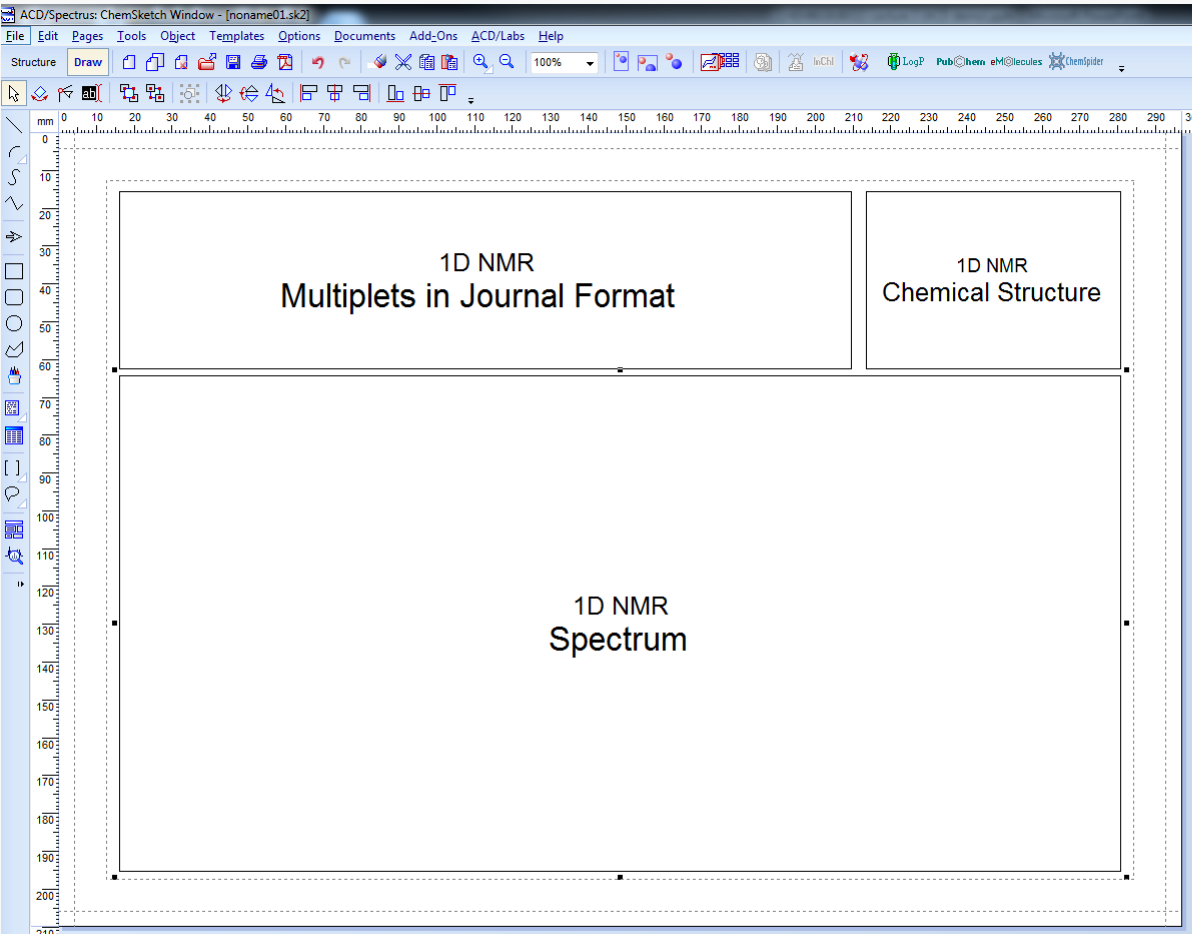


^{1}H NMR ($CDCl_3$, 400 MHz) δ = 6.63 (1H, d, *J* = 4.0 Hz, H-1<eq>), 5.60 (1H, t, *J* = 10.1 Hz, H-3<ax>), 5.23 (1H, dd, *J* = 10.4, 9.3 Hz, H-4<ax>), 4.85 (1H, dd, *J* = 9.6, 4.0 Hz, H-2<ax>), 4.57 (1H, d, *J* = 10.1 Hz, H-5<ax>), 3.75 (3H, s, H-23), 2.09 (3H, s, H-8), 2.04 (3H, s, H-10), 2.04 (3H, s, H-12) ppm.

Report templates saved in:

C:\ACD2012LSM\SCRIPTS\MULTIPLETREPORTS

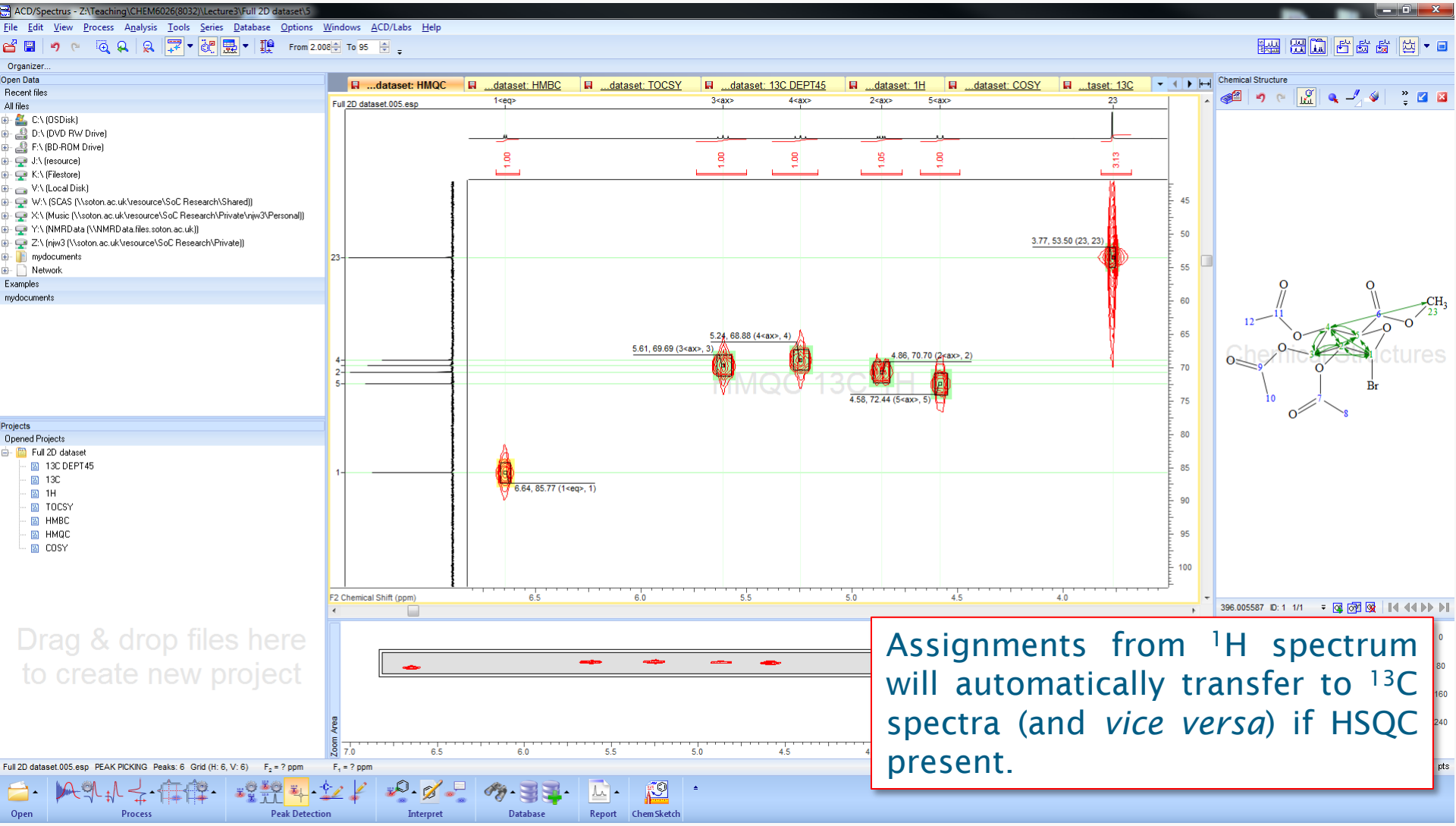
Report Templates



General 1D Hints and Tips

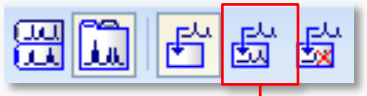
- Add a structure at the start (if known!).
- **Integration**: for greatest accuracy, the leading and trailing edges of the integral should be parallel to the baseline; errors can be corrected with *Bias Corr*.
- **Peak Picking**: hold shift-key for free selection of peaks (otherwise apex will be selected).

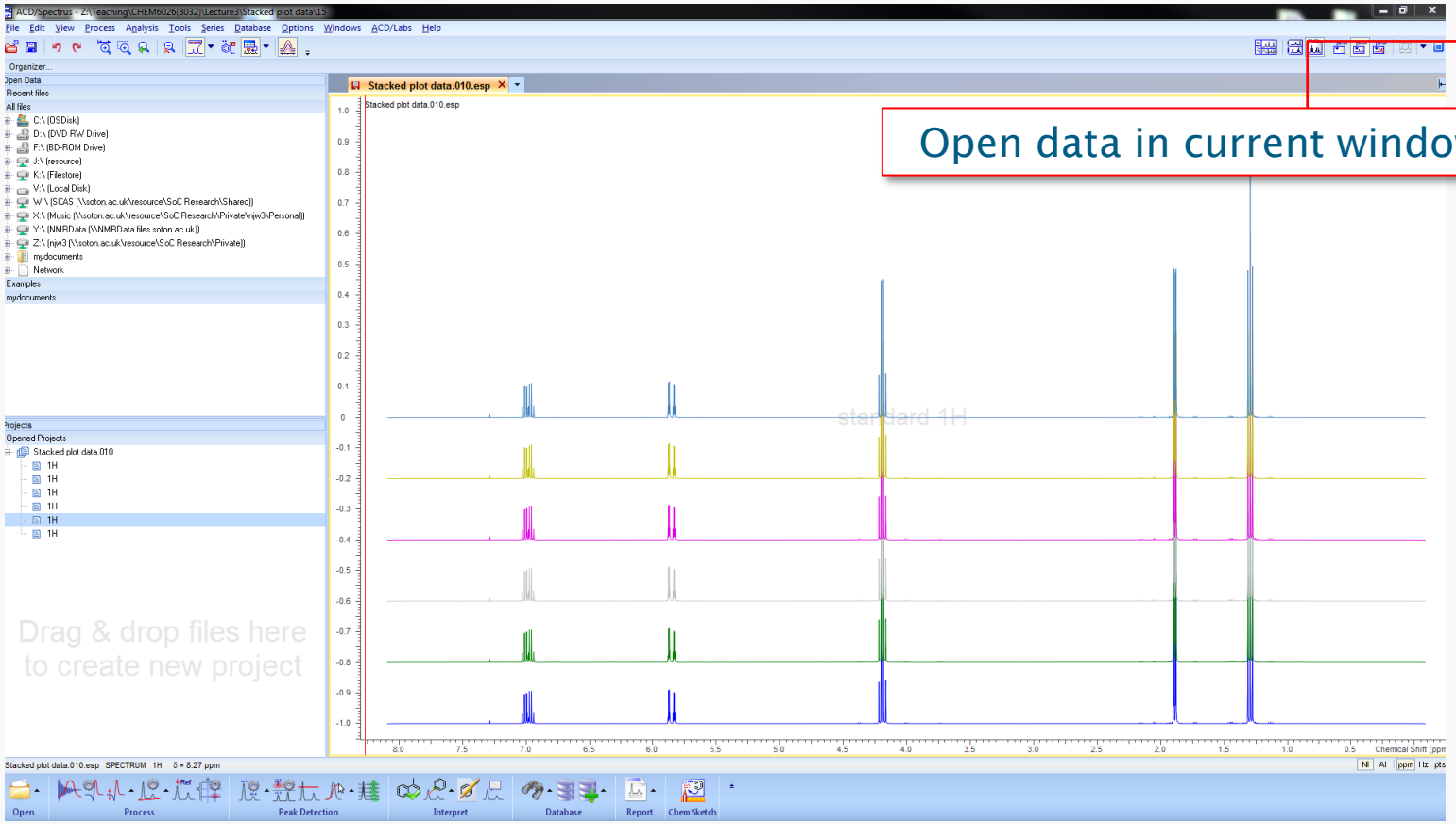
Projects (assigning 1D & 2D data)



Assignments from ^{1}H spectrum will automatically transfer to ^{13}C spectra (and *vice versa*) if HSQC present.

Stacked Plots



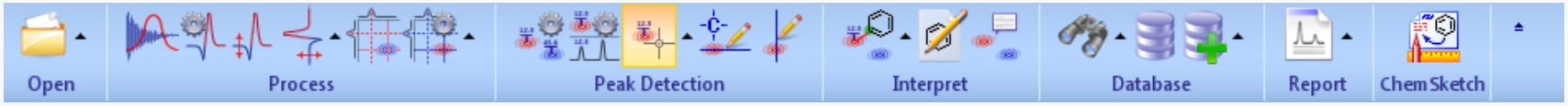


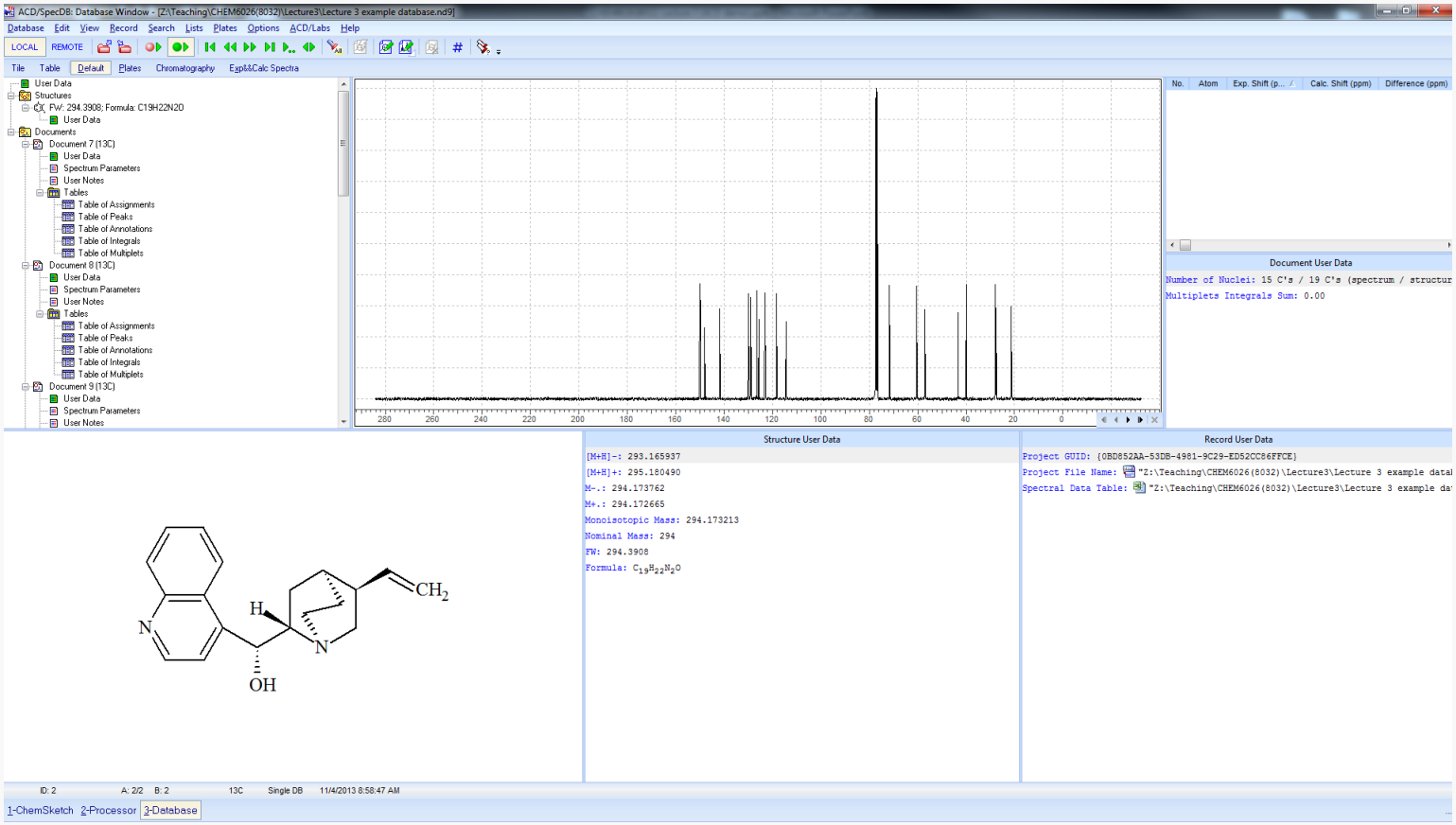
Preparing Papers & Reports

1. Templates in ChemSketch:
   - Export as wmf, bmp, gif, tif, png from ChemSketch report templates.

2. Windows clipboard:
   - Copy to clipboard
   - Paste as ChemSketch object
   - Paste Special (emf, wmf)

Databases


Open
Process
Peak Detection
Interpret
Database
Report
ChemSketch


ACD/SpecDB: Database Window - [Z:\Teaching\CHEM6026(8032)\Lecture3\Lecture 3 example database.nd9]
Database Edit View Record Search Lists Plates Options ACD/Labs Help
LOCAL REMOTE
Tile Table Default Plates Chromatography Expt&Calc Spectra
User Data
Structures
FW: 294.3908; Formula: C19H22N2O
Documents
Document 7 (13C)
Spectrum Parameters
User Notes
Tables
Table of Assignments
Table of Peaks
Table of Annotations
Table of Integrals
Table of Multiplets
Document 8 (13C)
Document 9 (13C)
No. Atom Exp. Shift (p... Calc. Shift (ppm) Difference (ppm)
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Nominal Mass: 294
FW: 294.3908
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1-ChemSketch 2-Processor 3-Database