

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

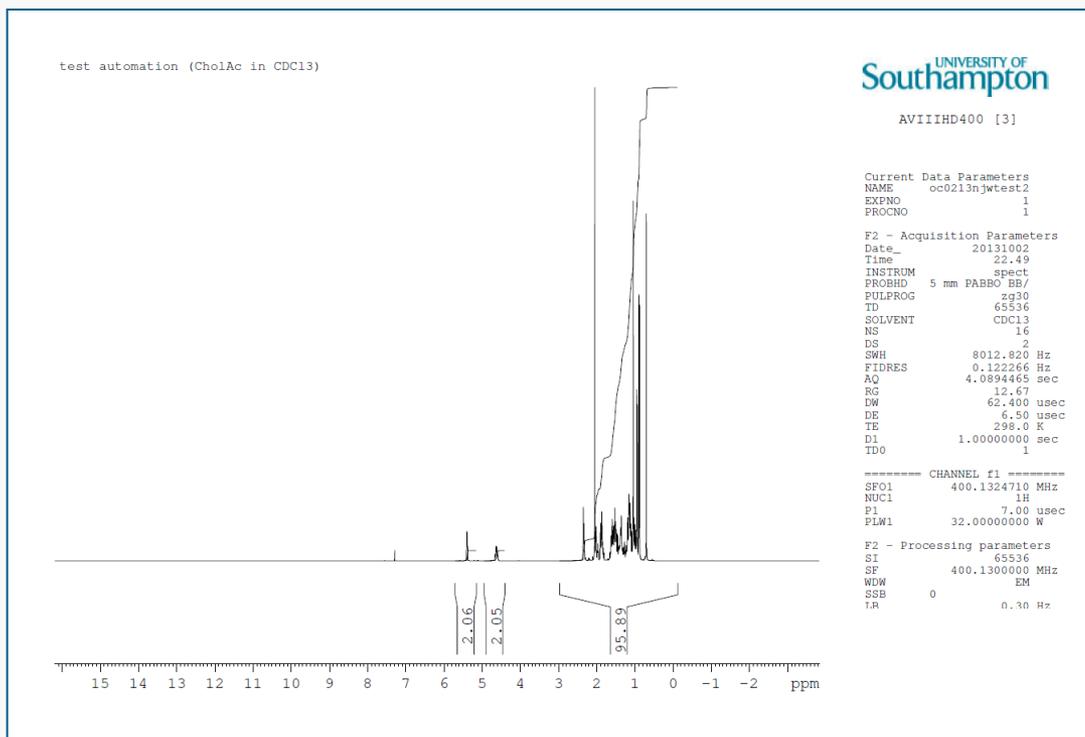
- AVII400 (1) <ftp://152.78.198.53/>
- AVII400 (2) <ftp://152.78.199.41/>
- AVIIIHD400 (3) <ftp://152.78.198.67/>
- AVIIIHD500 <ftp://152.78.199.49/>

- 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)



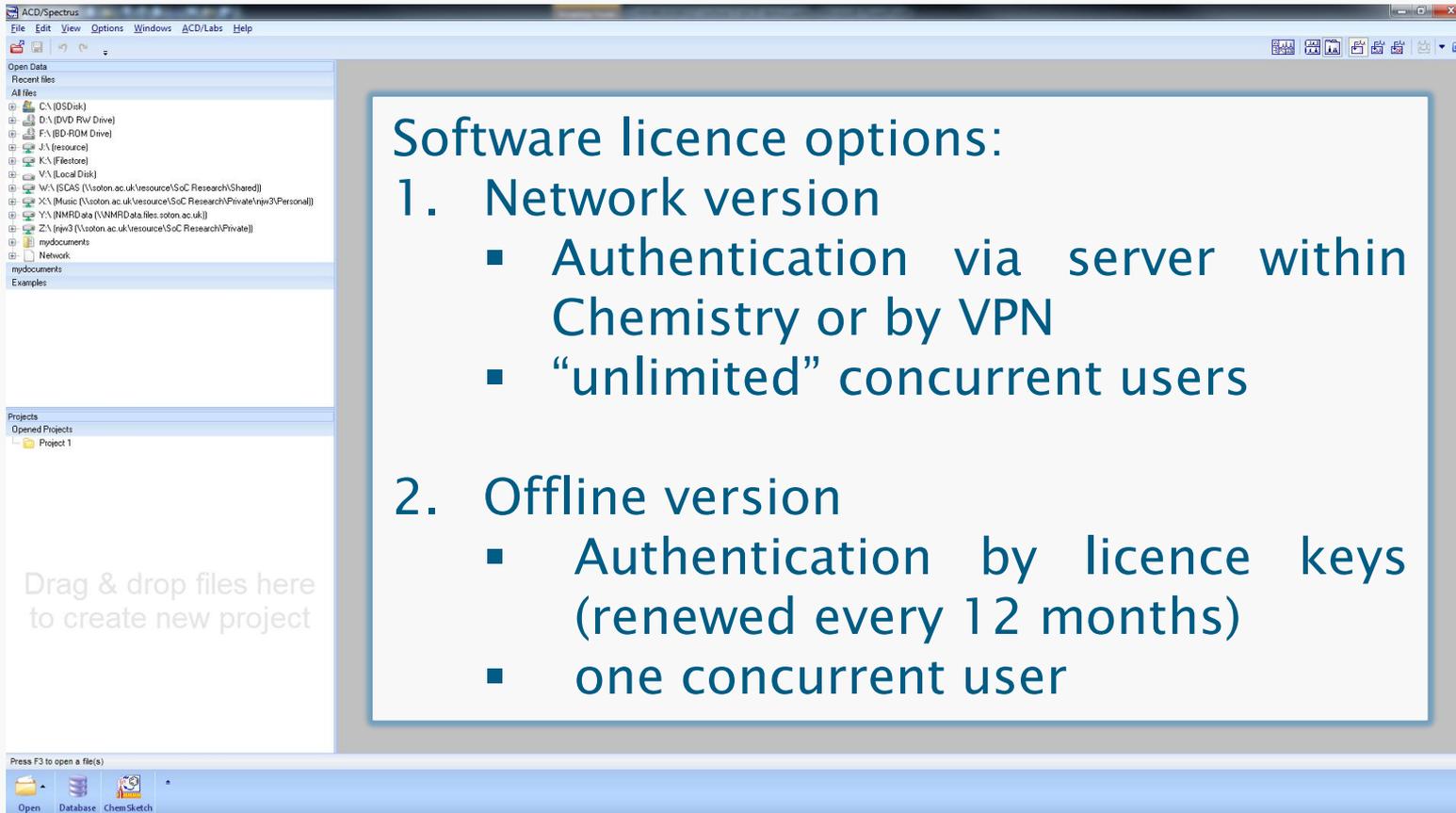
```
Current Data Parameters  
NAME      oc0213njwtest2  
EXPNO      1  
PROCNO     1
```

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/>



The image shows a screenshot of the ACD/Spectrus software interface. The window title is "ACD/Spectrus" and the menu bar includes "File", "Edit", "View", "Options", "Windows", "ACD/Labs", and "Help". The left sidebar shows a file explorer with "Open Data" and "Recent files" sections. The "Open Data" section lists various drives and network locations. The "Recent files" section is empty. The "Projects" section shows "Opened Projects" with a sub-entry for "Project 1". A large text box is overlaid on the right side of the interface, containing the following text:

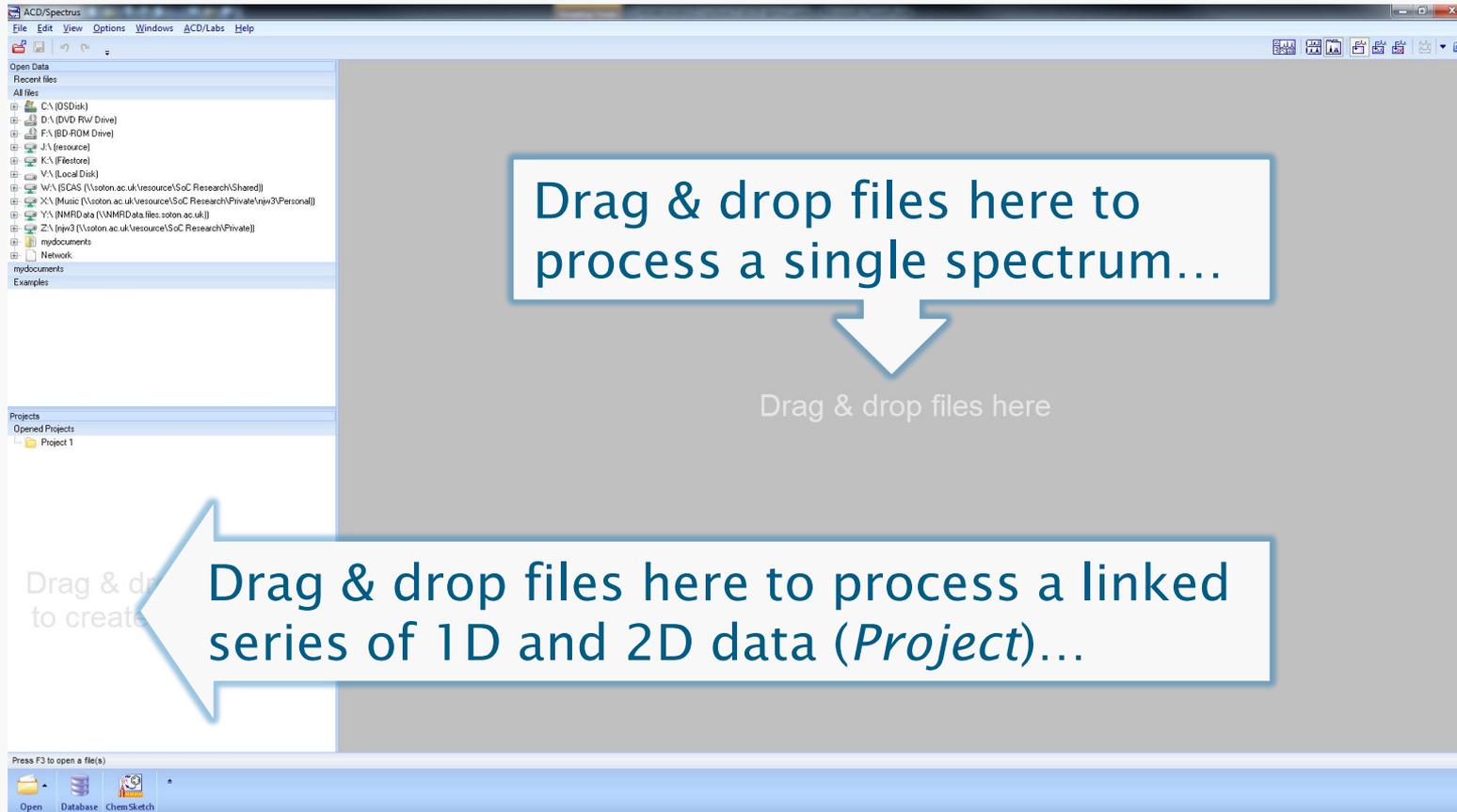
Software licence options:

1. Network version
 - Authentication via server within Chemistry or by VPN
 - “unlimited” concurrent users
2. Offline version
 - Authentication by licence keys (renewed every 12 months)
 - one concurrent user

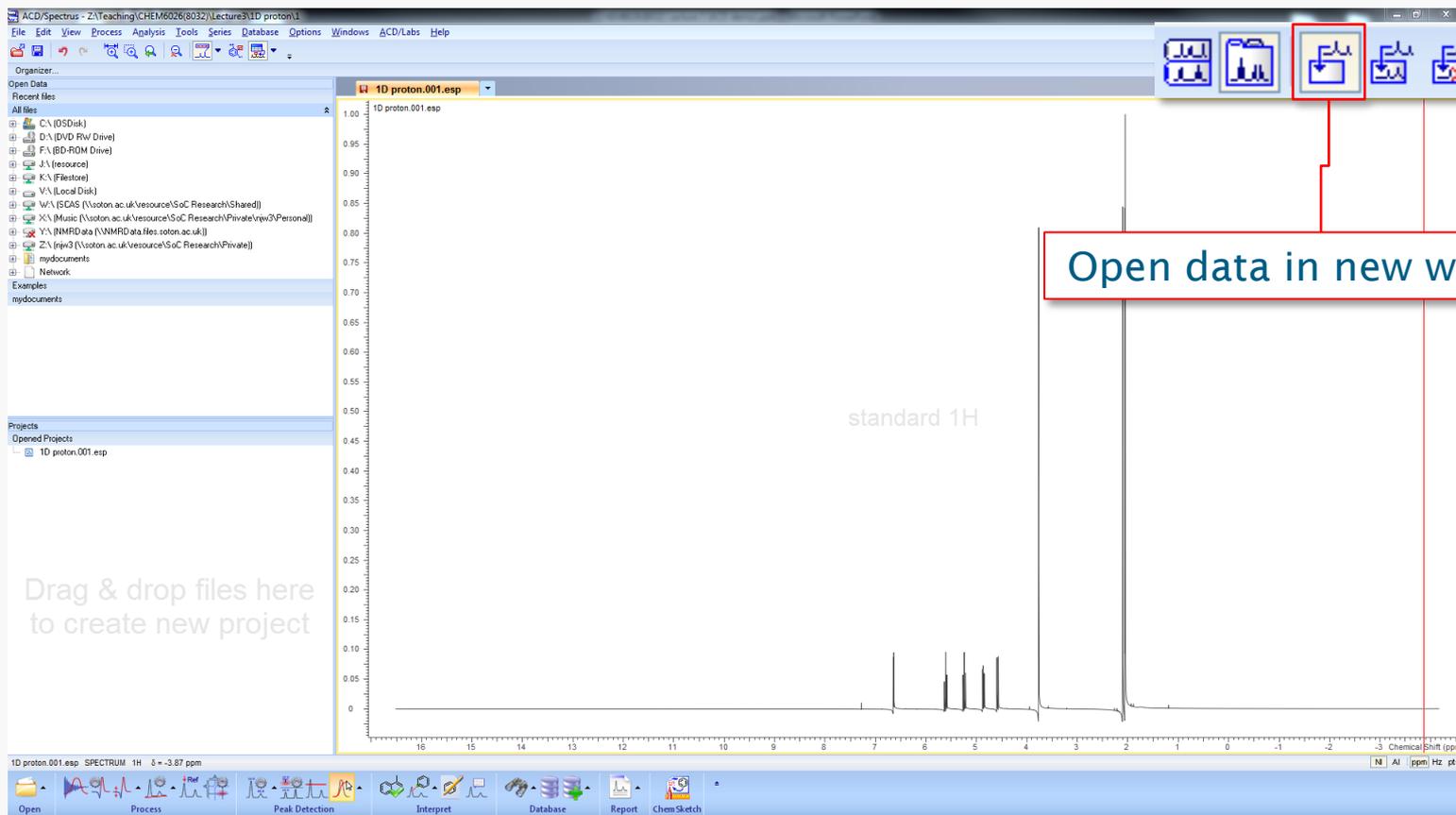
At the bottom of the interface, there is a status bar with the text "Press F3 to open a file(s)" and a toolbar with icons for "Open", "Database", and "Chem Sketch".

ACD/Spectrus

- Download software from:
<ftp://152.78.196.44/>

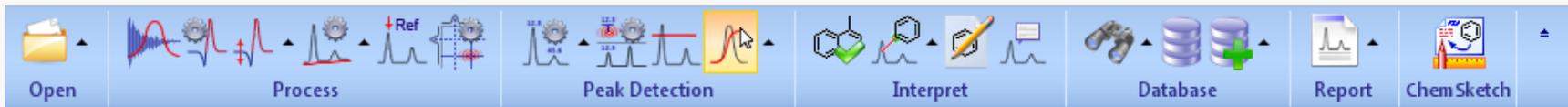


Processing 1D Data



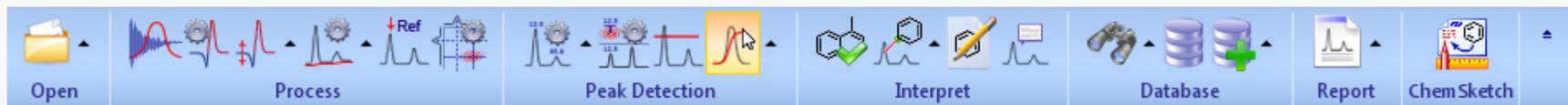
Drag & drop files here
to create new project

Open data in new window



Processing 1D Data: Workflow

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



1. Interactive FT

Exponential (EM) Window Function

^1H LB = 0.1–0.3 Hz

^{13}C LB = 2.0 Hz

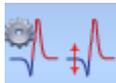


Gaussian (GM) Window Function

^1H 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

ACD/Spectrus - Z:\Teaching\CHEM6026(6032)\Lecture3\1D proton1

File Edit View Process Analysis Tools Series Database Options Windows ACD/Labs Help

Organizer...
Open Data
Recent files
All files
C:\ (OSDisk)
D:\ (DVD RW Drive)
F:\ (BD-ROM Drive)
J:\ (resource)
K:\ (Filestore)
V:\ (Local Disk)
W:\ (SCAS (\\soton.ac.uk\resource\SoC Research\Shared))
X:\ (Music (\\soton.ac.uk\resource\SoC Research\Private\rijw3\Personal))
Y:\ (NMRData (\\NMRData.files.soton.ac.uk))
Z:\ (rijw3 (\\soton.ac.uk\resource\SoC Research\Private))
mydocuments
Network
Examples
mydocuments

projects
Opened Projects
1D proton.001.esp

1D proton.001.esp

standard 1H

Drag with mouse to set left/right limit for integrals and height for peak-picking

M01: 6.63 ppm; d; J=4; 1H

Chemical Structure

396.005587 ID: 1 1/1

NI AI ppm Hz pts

Open Process Peak Detection Interpret Database Report ChemSketch

W&A (\\soton.ac.uk\resource\SoC Research\Private\rijw3\Personal)

Drag & drop files here to create new project

Chemical Structure

COC(=O)C1=CC(=C(C=C1)OC(=O)C)OC(=O)C

Peak Assignment

The screenshot displays the ACD/Spectrus software interface. The main window shows a 1D proton NMR spectrum with chemical shift (ppm) on the x-axis (2.25 to 1.95) and intensity on the y-axis (0 to 1.45). Two overlapping peaks are visible, with a context menu open over them. The menu options are: Edit Multiplet, Apply Peak Fitting, Split Multiplet, Delete Multiplet, Convert to Dark Region, and Add New Assignment. The text 'standard 1H' is overlaid on the spectrum. The software title bar indicates the file path: 'ACD/Spectrus - Z:\Teaching\CHEM6026(8032)\Lecture3\1D proton.1'. The bottom status bar shows '1D proton.001.esp SPECTRUM 1H δ = 2.03 ppm' and various tool icons.

Drag & drop files here to create new project

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

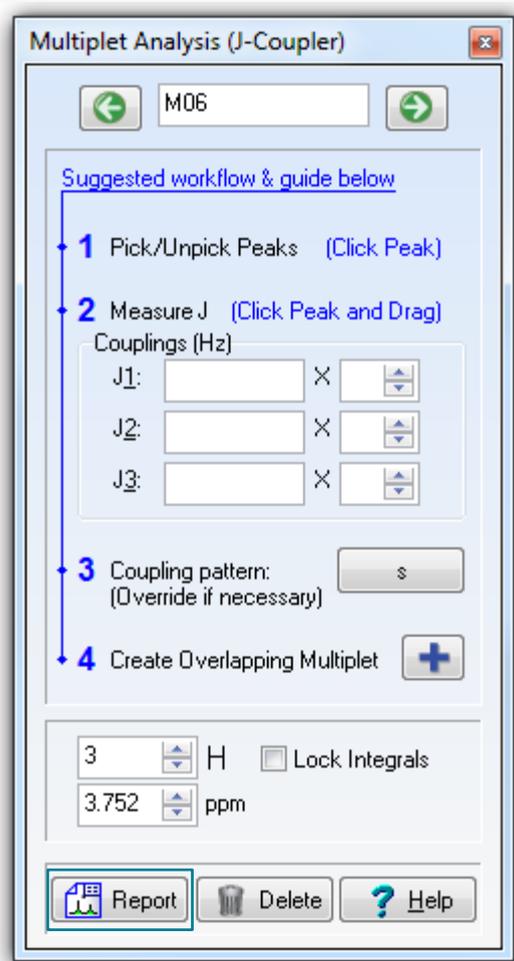
The screenshot displays the ACD/Spectrus software interface. The main window shows a 1D proton NMR spectrum for the file "1D proton.001.esp". The x-axis represents the chemical shift in ppm, ranging from approximately 7.0 to 1.5. The y-axis represents intensity. Several peaks are identified and assigned with multiplicity and integration values:

- M01 (d, 1-eq): Integration 1.00, peaks at 6.64 and 6.63 ppm.
- M02 (m, 3-ax): Integration 1.01, peaks at 5.62 and 5.57 ppm.
- M03 (dd, 4-ax): Integration 1.01, peaks at 5.26 and 5.23 ppm.
- M04 (dd, 2-ax): Integration 1.06, peaks at 4.88 and 4.83 ppm.
- M05 (m, d, J=10.1, 1H): Integration 1.00, peak at 4.57 ppm.
- M06 (s): Integration 3.16, peak at 3.76 ppm.
- M07 (s): Integration 1.14 and 1.21, peaks at 2.09 and 2.04 ppm.

The chemical structure of the compound is shown on the right side of the interface. It is a substituted cyclohexane ring with two ester groups and a methyl group. The protons are numbered 1 through 10. A context menu is open over the structure, with the option "Assign to 5-ax" selected.

At the bottom of the window, the software title bar reads "1D proton.001.esp SPECTRUM 1H $\delta = 4.57$ ppm". The bottom toolbar includes buttons for "Open", "Process", "Peak Detection", "Interpret", "Database", "Report", and "ChemSketch".

Multiplet Reports



^1H 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\MULTIPLEREPORTS

Report Templates

The screenshot displays the ChemSketch software interface with a report template for 1D NMR data. The template is contained within a dashed border and is divided into three main sections:

- Top Left:** A box labeled "1D NMR Multiplets in Journal Format".
- Top Right:** A box labeled "1D NMR Chemical Structure".
- Bottom:** A large box labeled "1D NMR Spectrum".

The software interface includes a menu bar (File, Edit, Pages, Tools, Object, Templates, Options, Documents, Add-Ons, ACD/Labs, Help), a toolbar with various drawing and editing tools, and a status bar at the bottom showing "NONAME01.SK2 Modified Page 1/1". The status bar also includes navigation icons and the text "1-ChemSketch 2-Processor 3-Add Structure".

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)

The screenshot displays the ACD/Spectrus software interface. The main window shows an HSQC NMR spectrum with the following peak assignments:

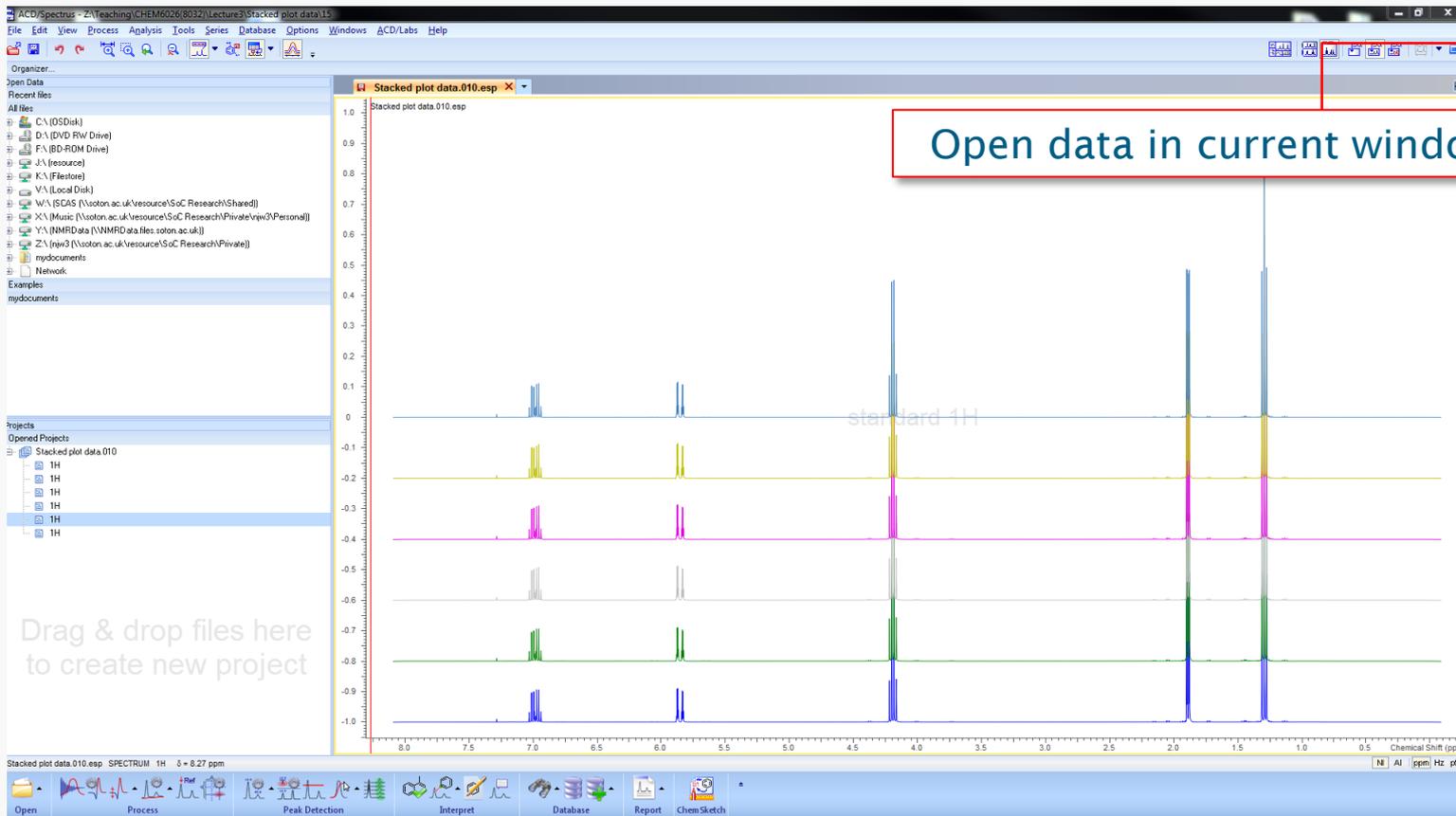
- $6.64, 85.77$ (1<eq>, 1)
- $5.61, 69.69$ (3<ax>, 3)
- $5.24, 68.88$ (4<ax>, 4)
- $4.86, 70.70$ (2<ax>, 2)
- $4.58, 72.44$ (5<ax>, 5)
- $3.77, 53.50$ (23, 23)

The chemical structure on the right is a substituted benzothiazine derivative with atoms labeled 10 through 23. The spectrum shows correlations between ^1H and ^{13}C signals, with integration values (1.00, 1.05, 1.00, 3.13) shown above the peaks. The x-axis is labeled 'F2 Chemical Shift (ppm)' and the y-axis is labeled 'Zoom Area'.

Drag & drop files here to create new project

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

Stacked Plots



Open data in current window (as series)

Drag & drop files here to create new project

Preparing Papers & Reports

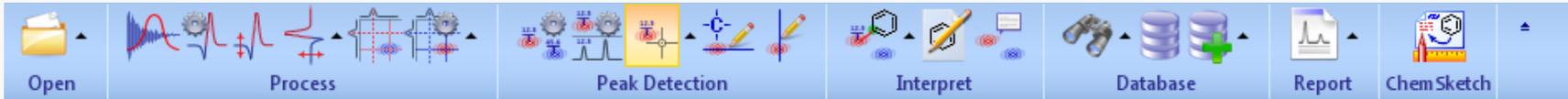
1. Templates in ChemSketch:

- 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



ACD/SpecDB: Database Window - (Z:\Teaching\CHEM6026\8032)\Lecture3\example database.nd9

Database Edit View Record Search Lists Plates Options ACD/Labs Help

LOCAL REMOTE

File Table Default Plates Chromatography Epi&Calc Spectra

User Data
Structures
 Pur: 294.3908; Formula: C19H22N2O
 User Data
Documents
 Document 7 (13C)
 User Data
 Spectrum Parameters
 User Notes
 Tables
 Table of Assignments
 Table of Peaks
 Table of Annotations
 Table of Integrals
 Table of Multiplets
 Document 8 (13C)
 User Data
 Spectrum Parameters
 User Notes
 Tables
 Table of Assignments
 Table of Peaks
 Table of Annotations
 Table of Integrals
 Table of Multiplets
 Document 9 (13C)
 User Data
 Spectrum Parameters
 User Notes

No. Atom Exp. Shift (p... / Calc. Shift (ppm) Difference (ppm)

Document User Data
Number of Nuclei: 15 C's / 19 C's (spectrum / structure)
Multiplets Integrals Sum: 0.00

Structure User Data
[M+H]⁻: 293.165937
[M+H]⁺: 295.180490
M⁻: 294.173762
M⁺: 294.172665
Monoisotopic Mass: 294.173213
Nominal Mass: 294
FW: 294.3908
Formula: C₁₉H₂₂N₂O

Record User Data
Project GUID: {0BD8523A-53DB-4981-9C29-ED52CC6FFCE}
Project File Name: "Z:\Teaching\CHEM6026\8032)\Lecture3\Lecture 3 example data
Spectral Data Table: "Z:\Teaching\CHEM6026\8032)\Lecture3\Lecture 3 example da

C=CCN1CC[C@H](C2=CN=CC=C2)[C@@H]1O

ID: 2 A: 2/2 B: 2 13C Single DB 11/4/2013 8:58:47 AM

1-ChemSketch 2-Processor 3-Database