Secrets of Structure Elucidation and Spectroscopic Databases

Reinhard Neudert
John Wiley&Sons

- John Wiley&Sons
- Laboratory Automation
- Structure Elucidation
- The SpecInf Internet Database
- Applications
- Links between Databases

John Wiley&Sons - a Global Company

Switzerland

UK

Germany

New York

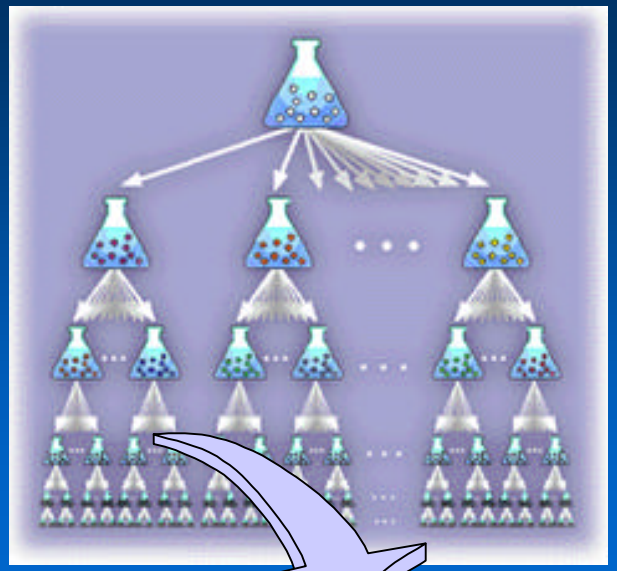
USA

Australia

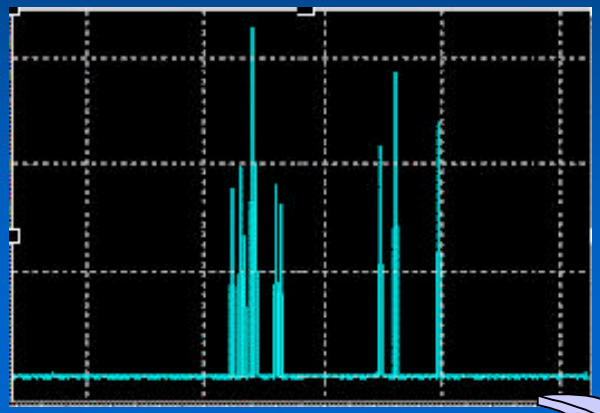
Asia

Canada

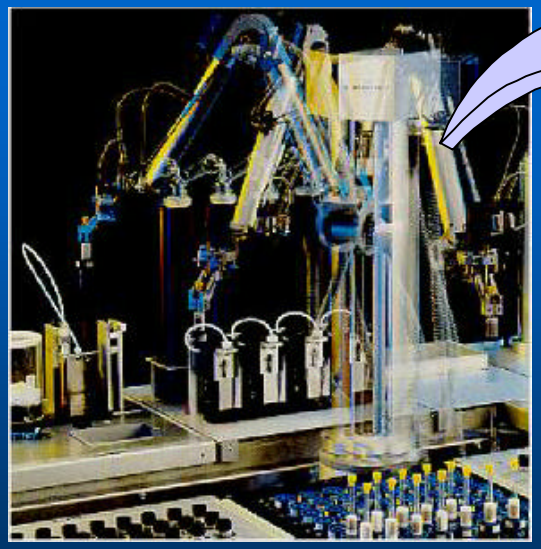
Laboratory Automation



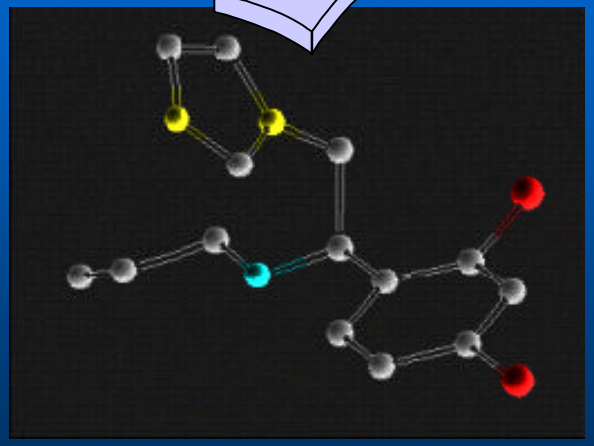
Combinatorial Chemistry



Data Increase



Robots and Sample Changers

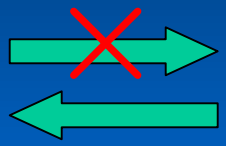


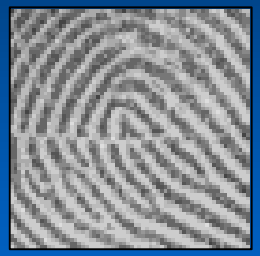
Bottleneck: Data Interpretation

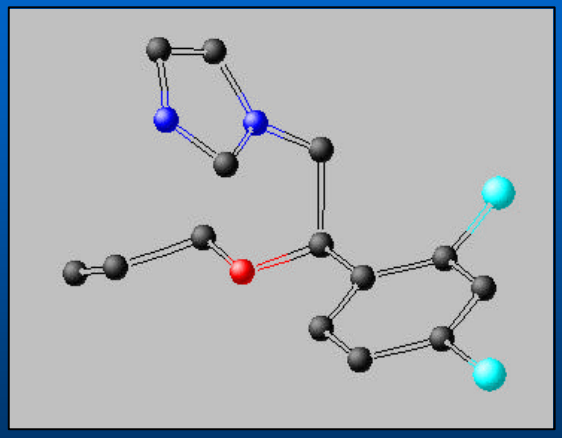
Sensitivity and Structure Elucidation Potential

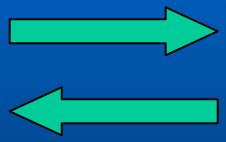
Technique	Required	Structure Elucidation Potential
13CNMR	mg	very high
1HNMR	µg	high
IR	ng	medium
EI/MS	<ng	medium
LC/MS	<ng	low

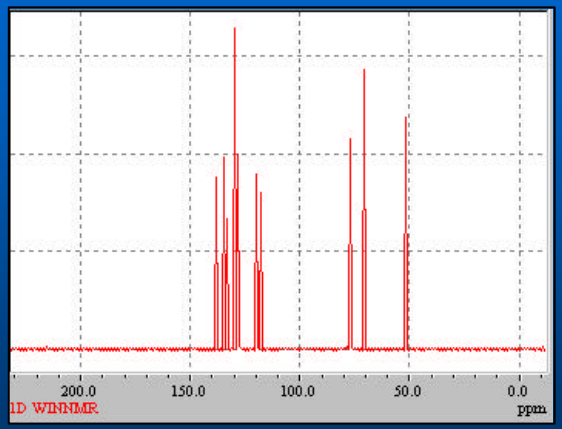
Fingerprints





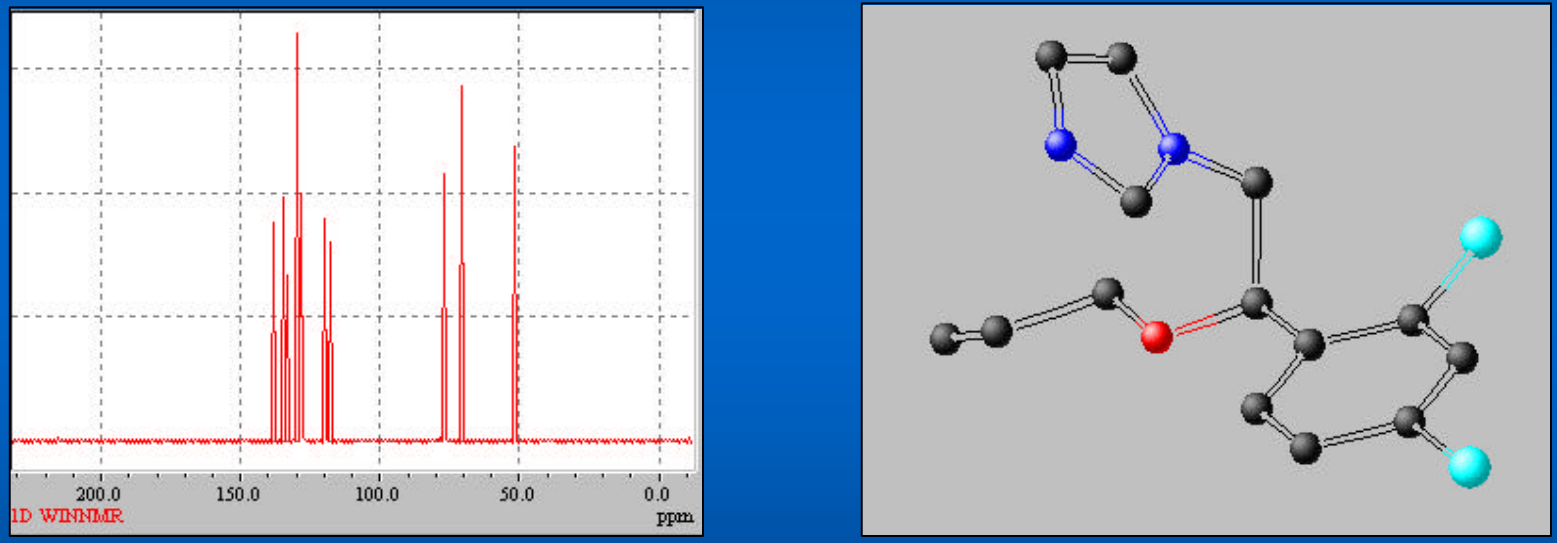






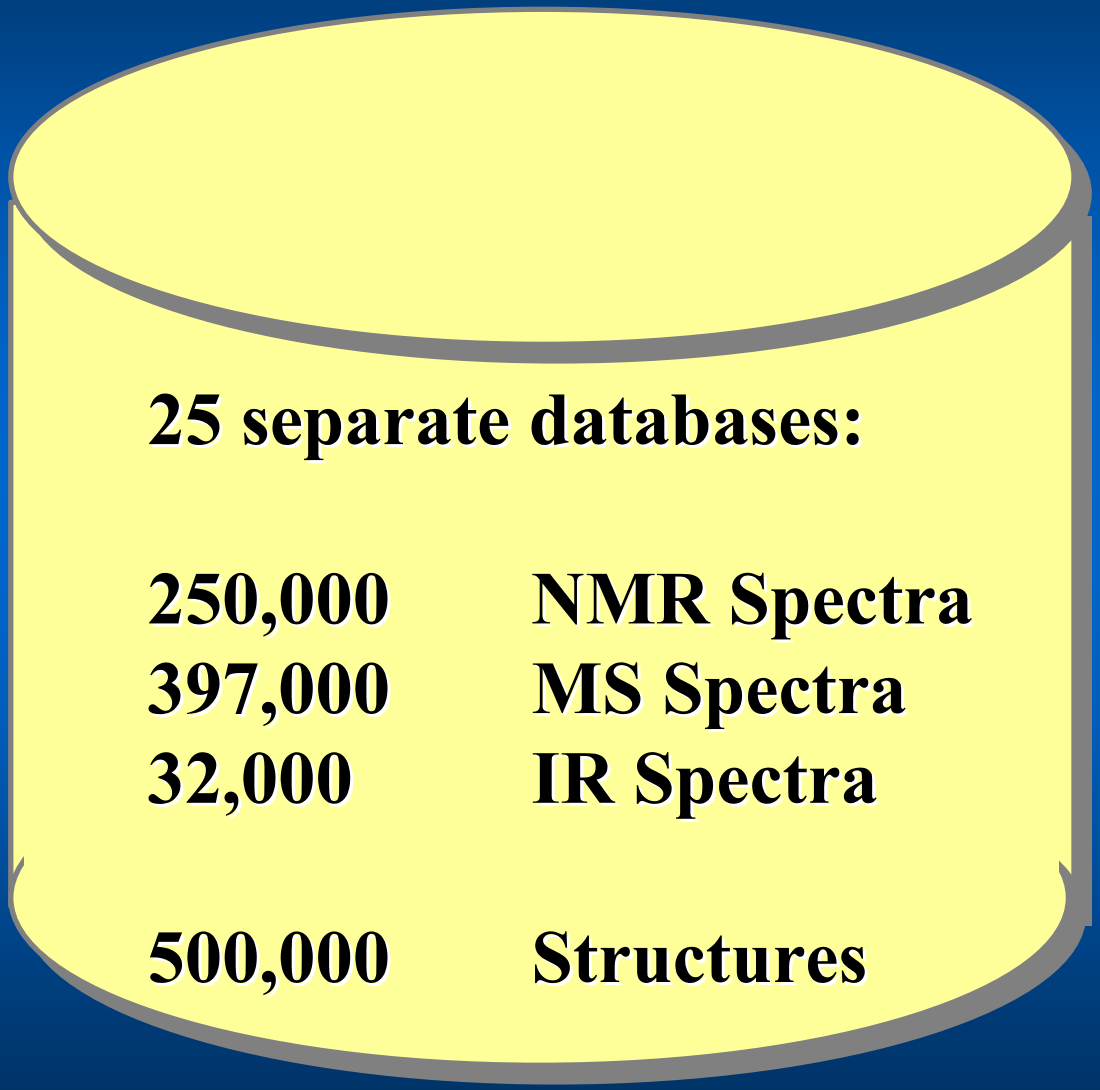
Structure Elucidation

Spectrum Search
Structure Generator

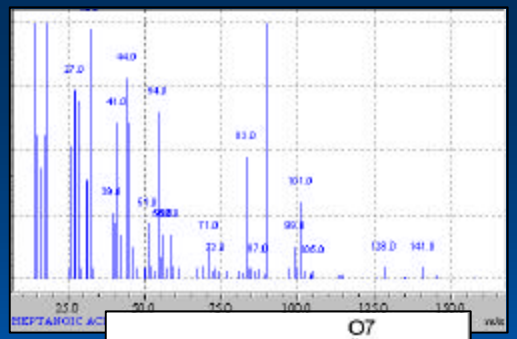


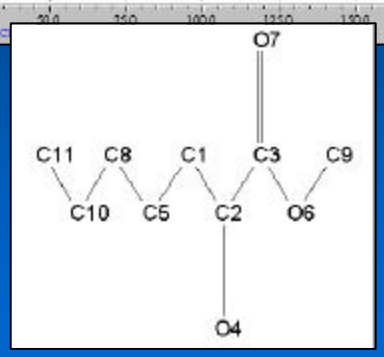
Structure Search
Spectrum Prediction

Spectroscopic Databases: SpecInf



SpecInf : Basic Functions





Spectrum Search

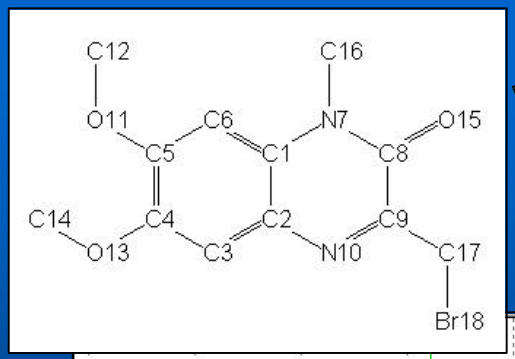
String Search

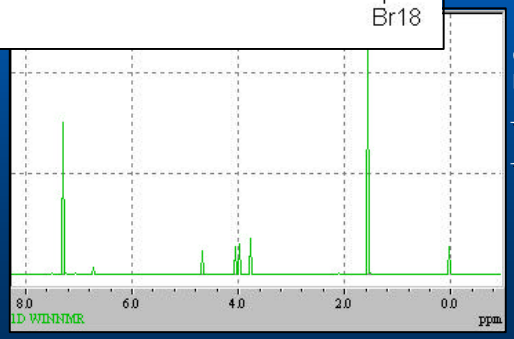
Cephalo%

Cephalosporine

SpecInf

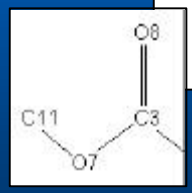
NMR, IR, MS
UV, NIR, Raman
Chemical Structures

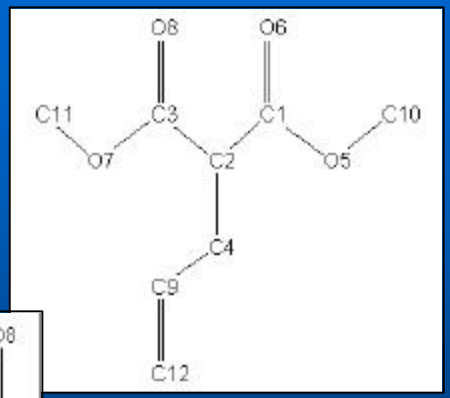




Spectrum Prediction

Structure Search

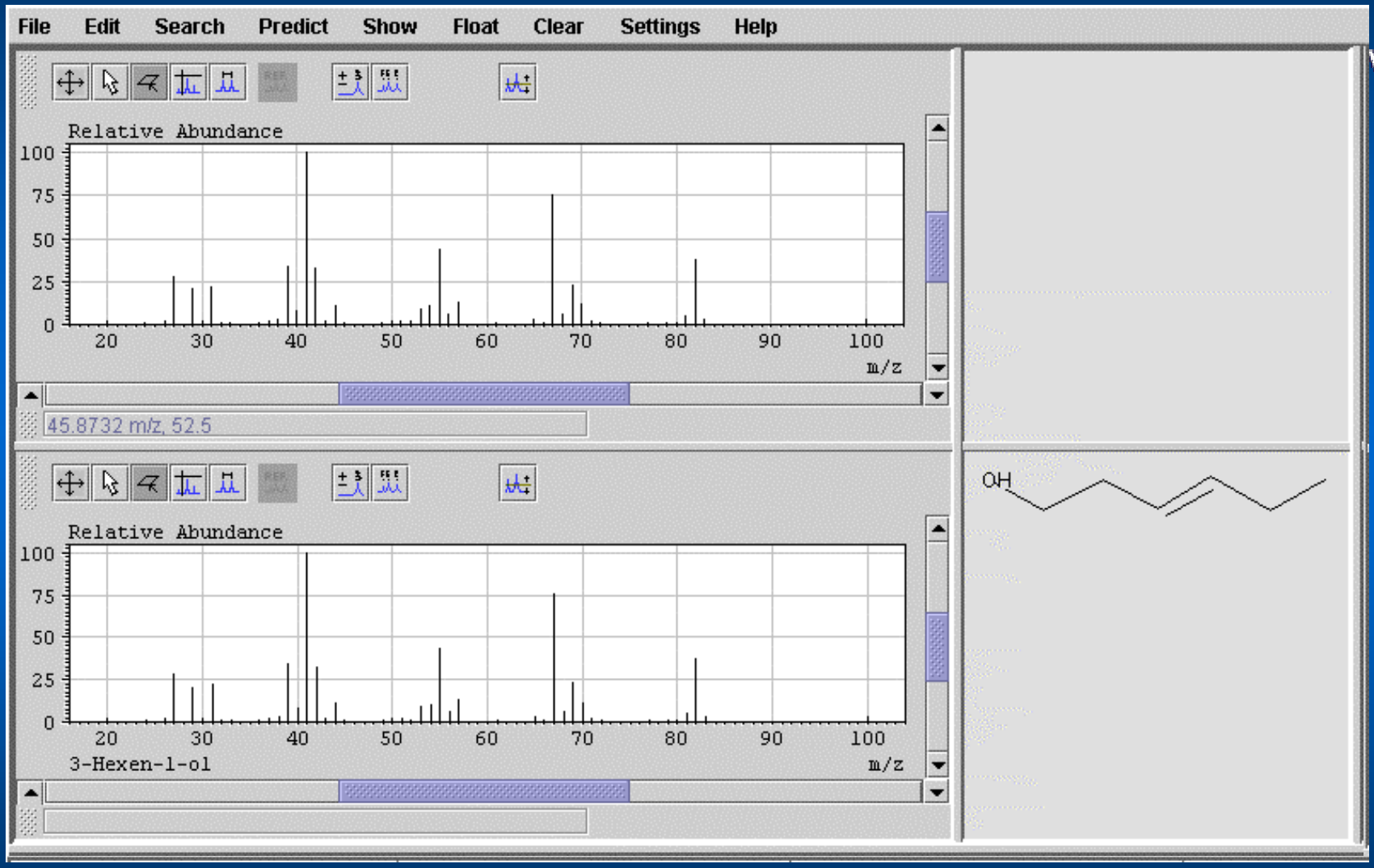




Spectroscopic Databases: SpecInfo

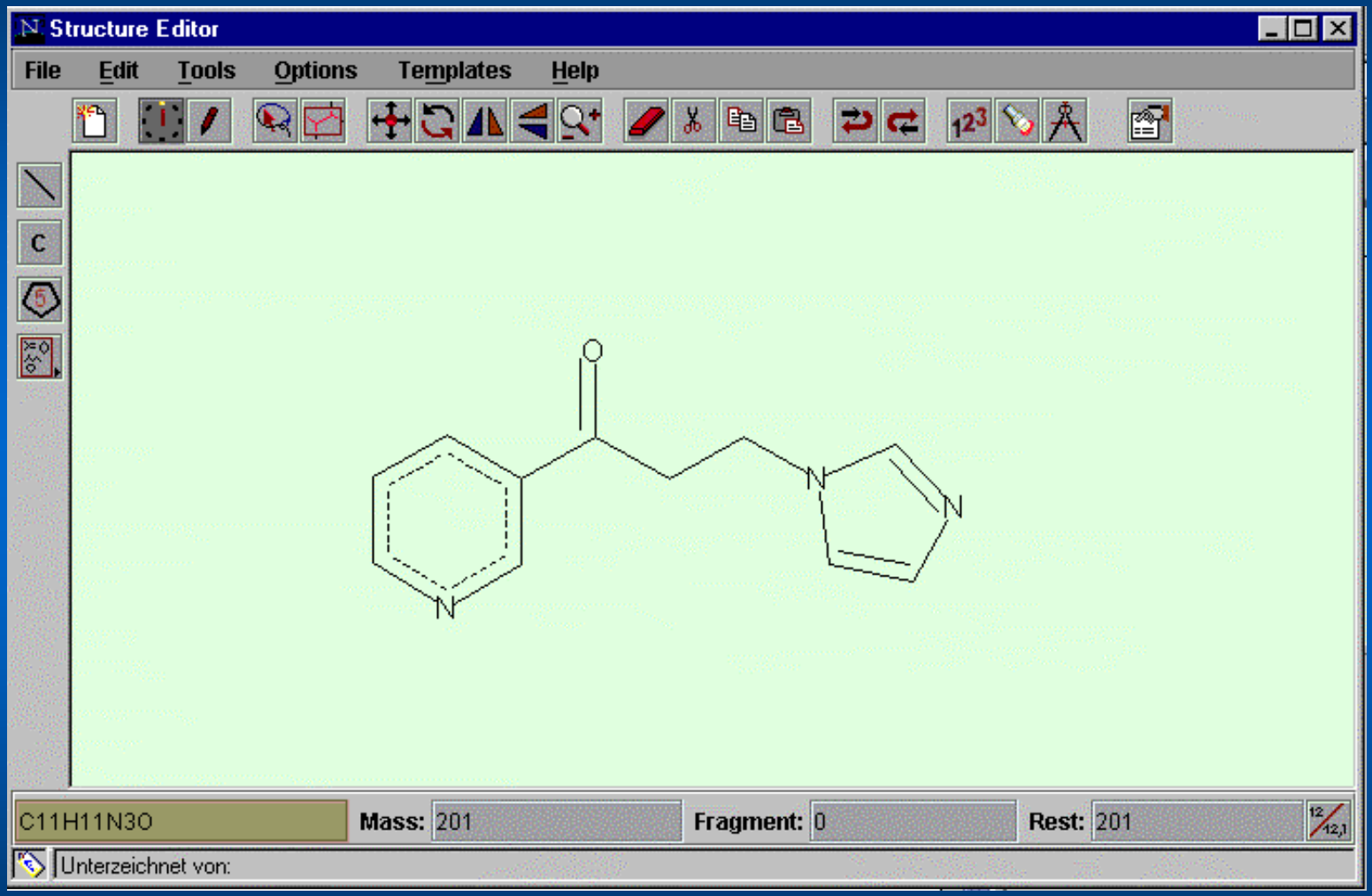
Access through Web Browsers

The Web Client SpecSurf



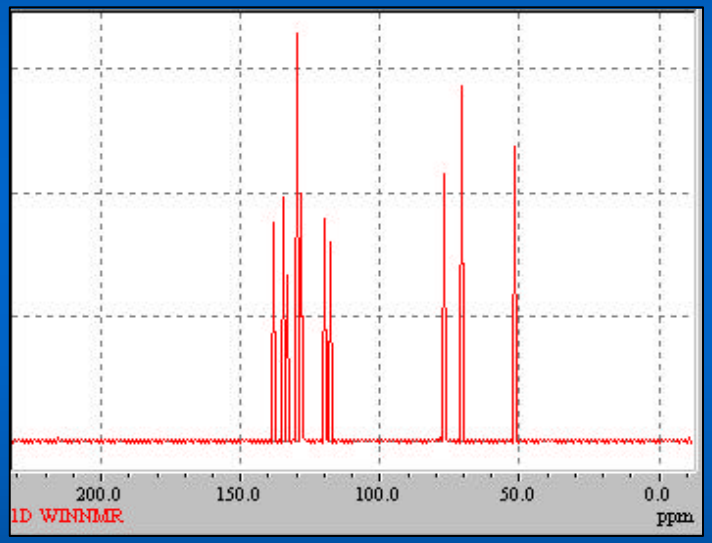
Spectroscopic Databases: SpecInfo

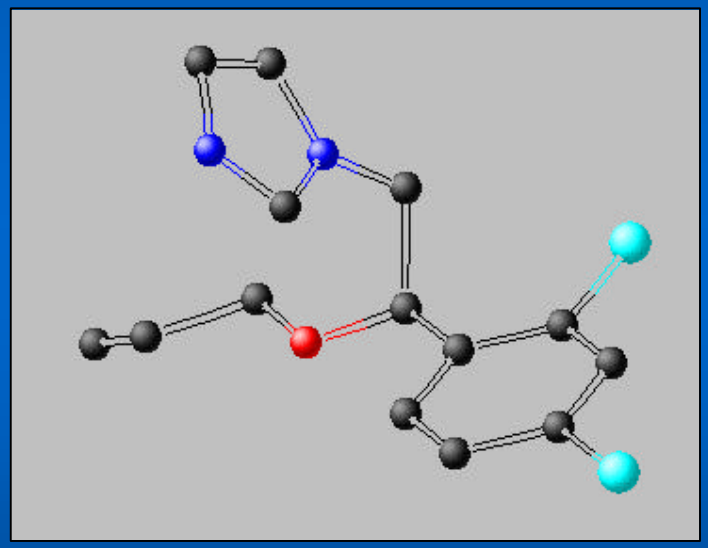
Structure Drawing



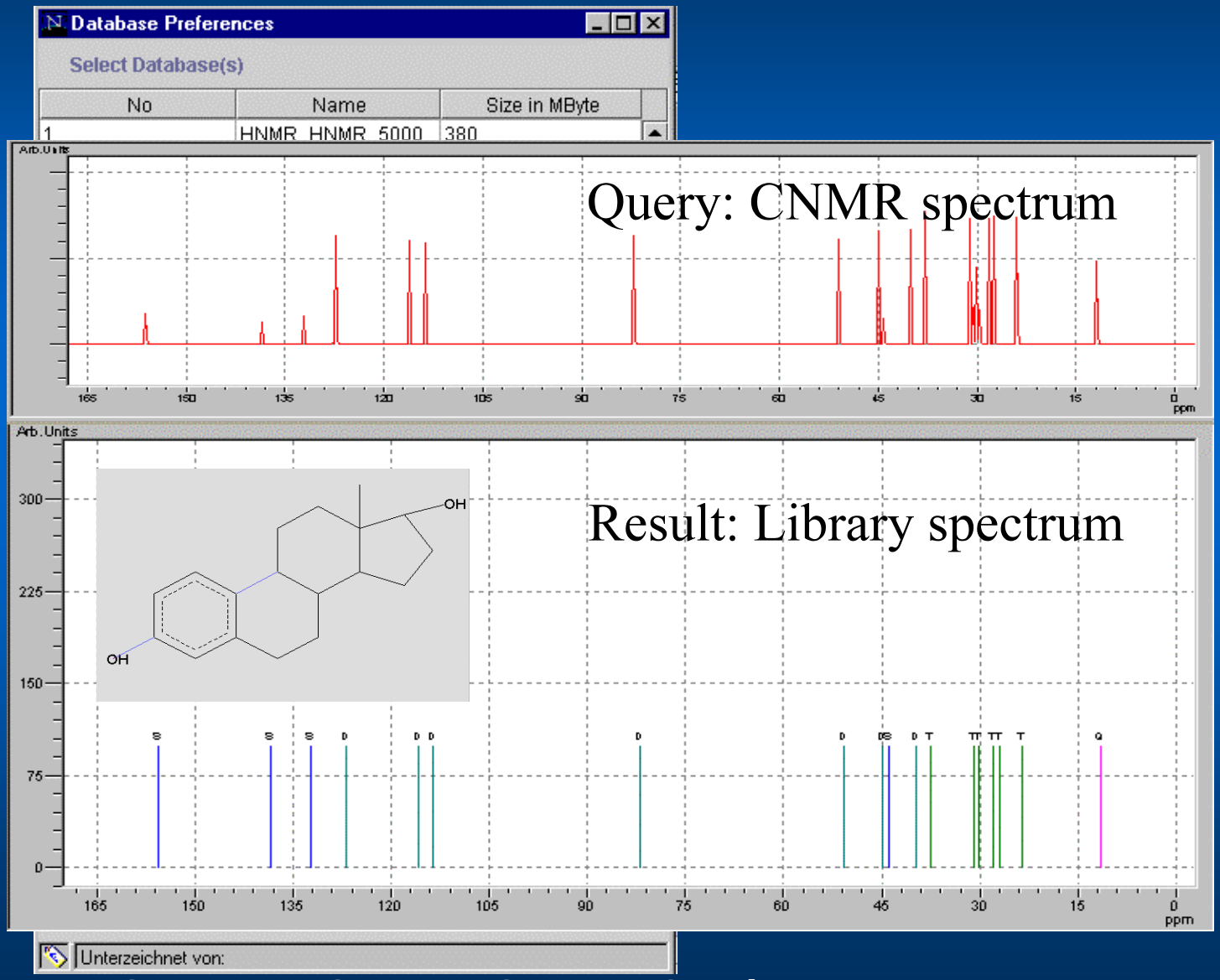
Applications

Spectrum Search



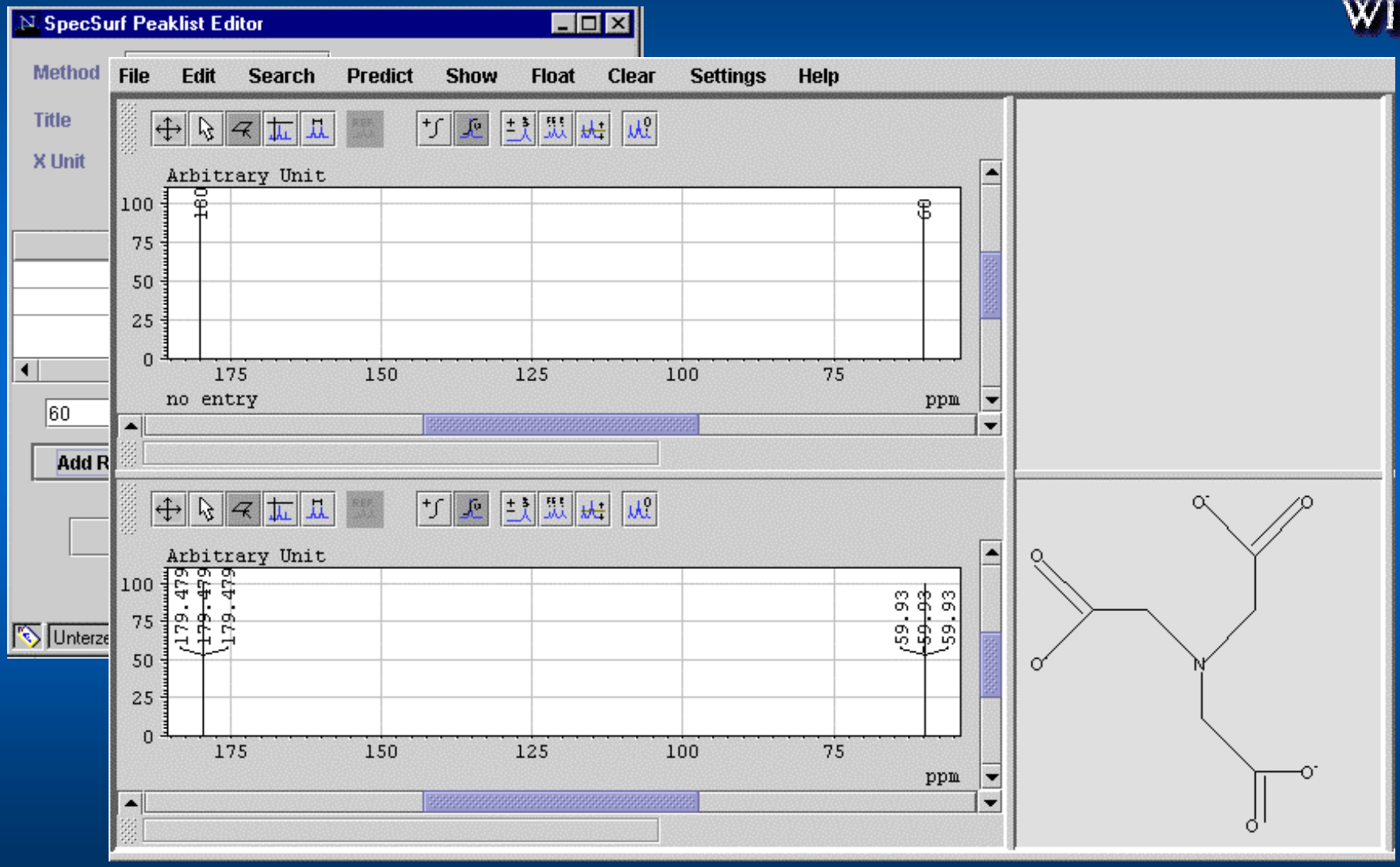


From Spectrum to Structure: Full Spectrum Search

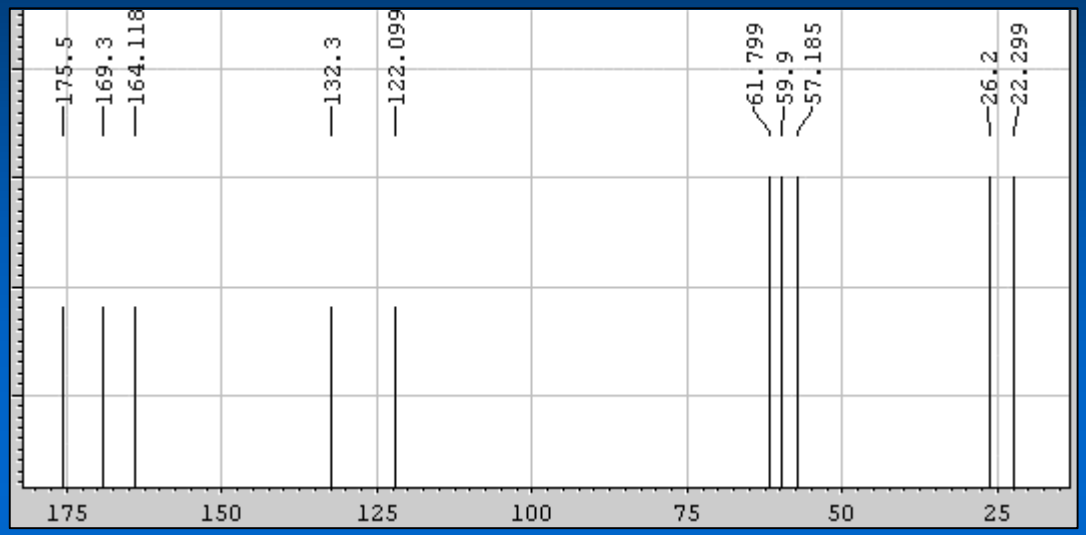


Case 1: Query contained in the Library

From Spectrum to Structure: Peak List Search

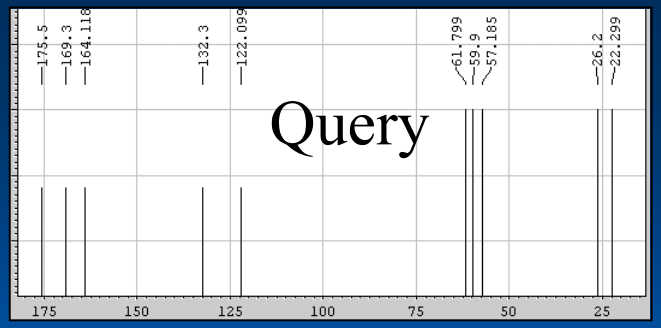


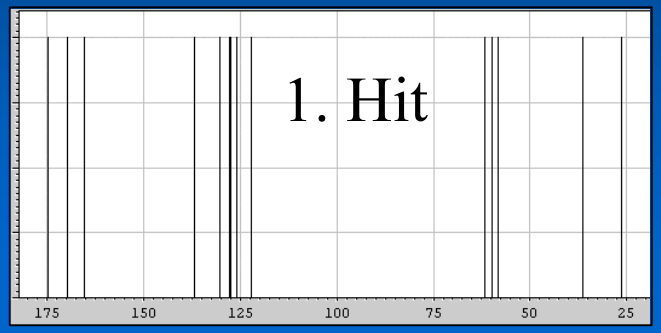
Spectral and Structural Similarity

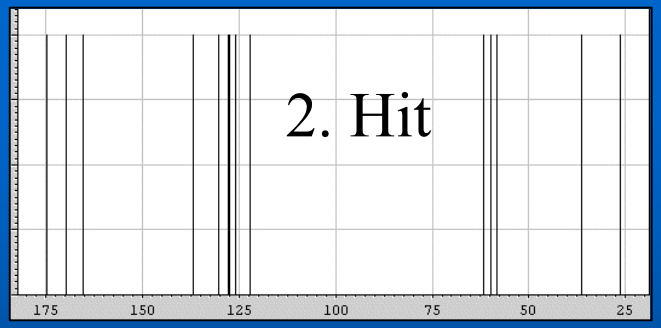


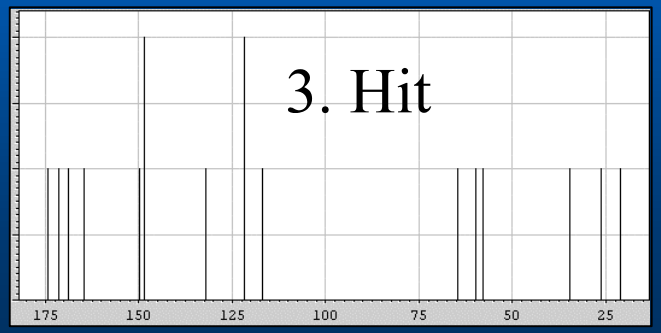
Case 2:
Query
not contained
in the
Library

Rank	Quality	Folder ID	Property
1/3	505	18:FO0000036681	7-Acetamido-cephalosporanic acid, anion
2/3	495	18:FO0000036689	7-(2-[2]Thienyl-acetamido)-desacetyl-cephalosporanic acid,...
3/3	408	18:FO0000029698	7-(Phenyl-acetamino)-cephalosporanic anion

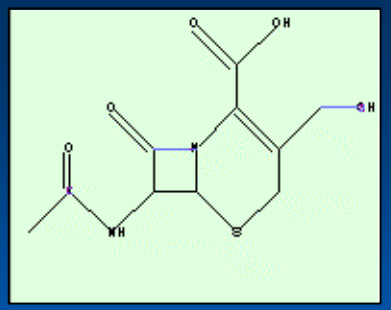


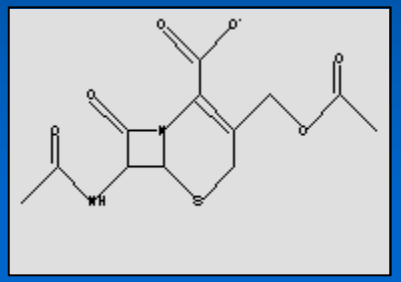


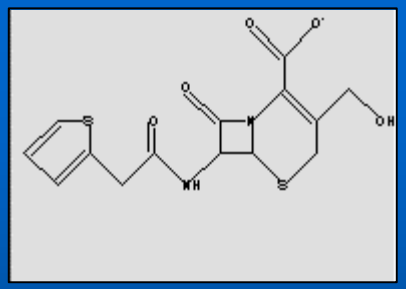


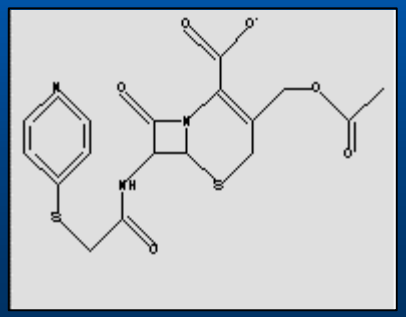


Similar Spectra correspond to Similar Structures

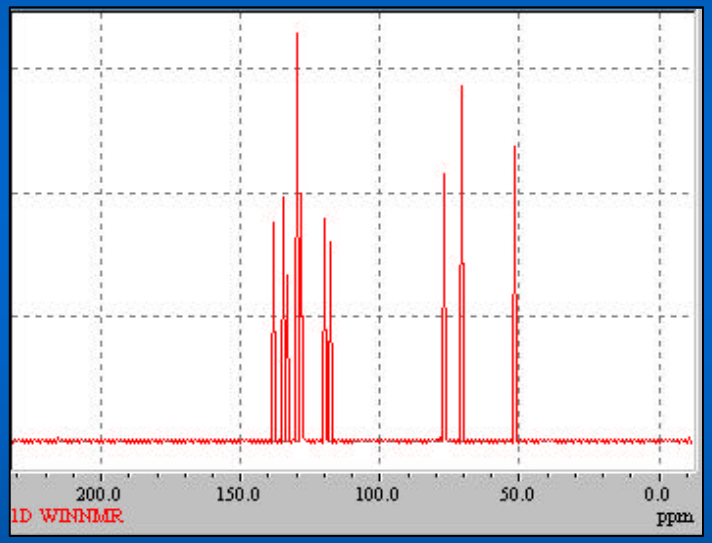


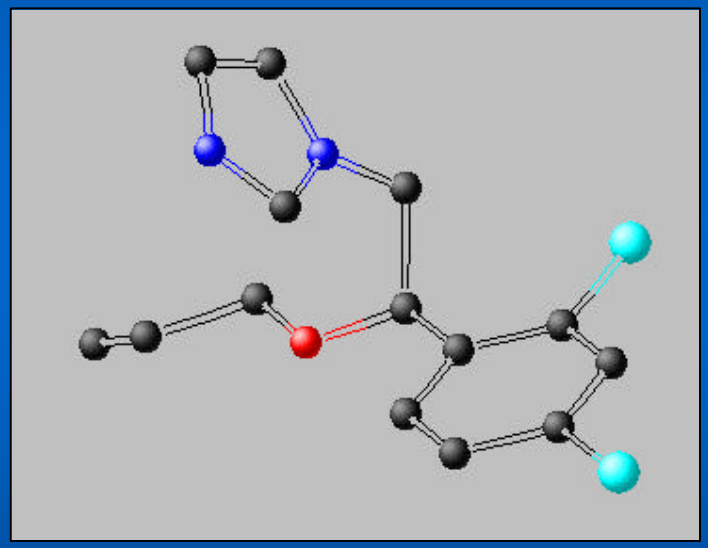






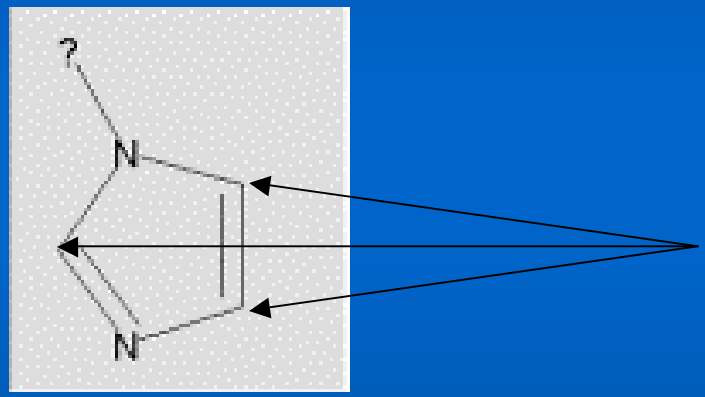
From Spectrum to Structure: Spectrum Search





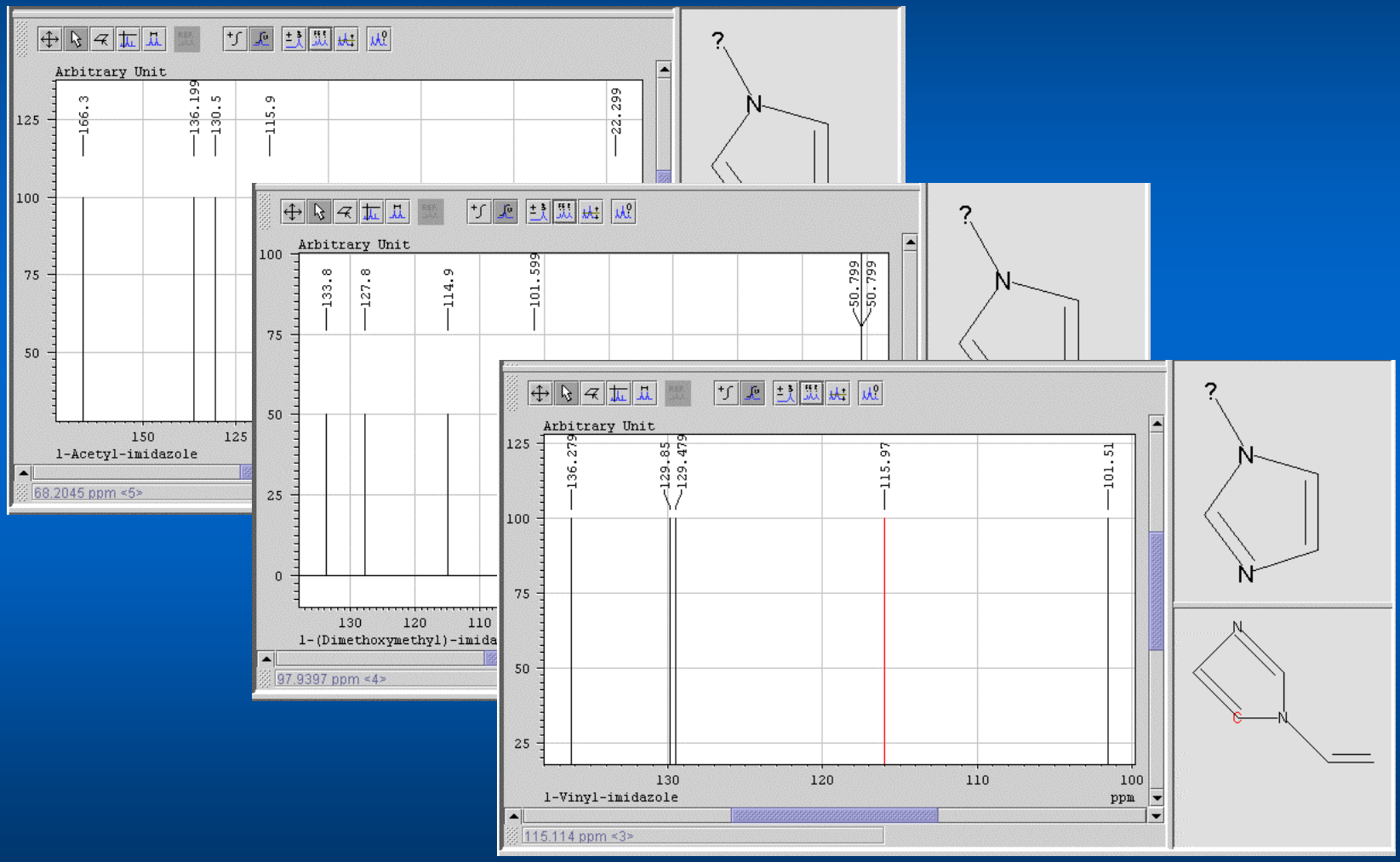
Structure Search

From Structure to Spectrum : Structure Search

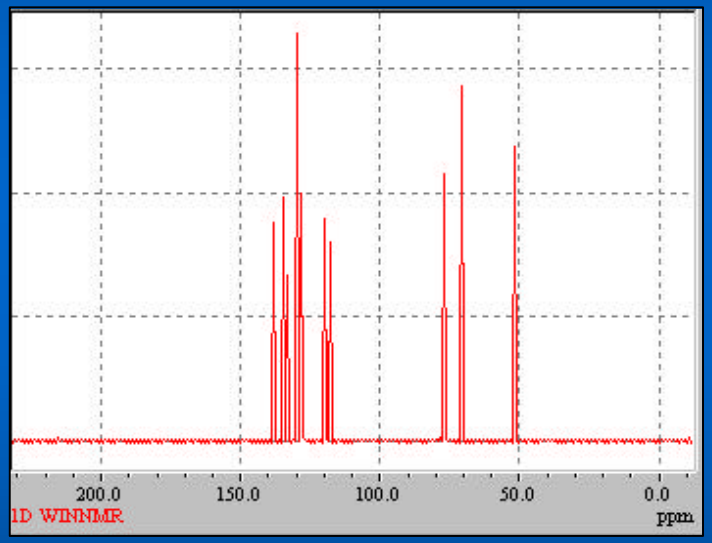


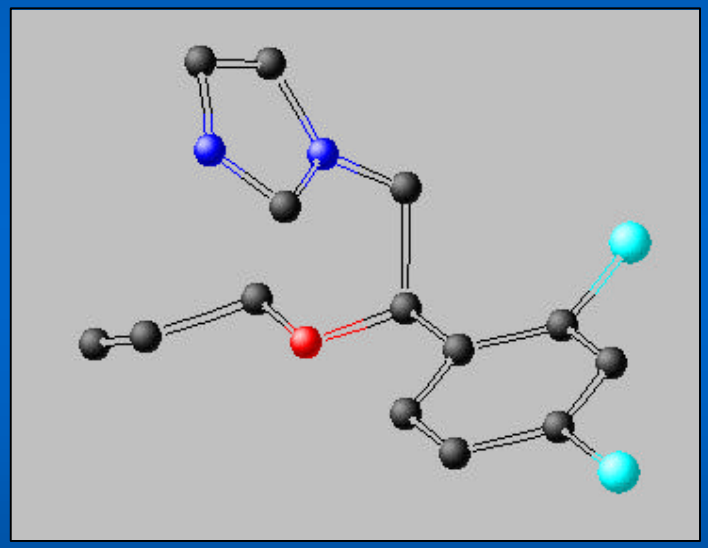
Inf
varying substituents
on the C shif
Imidazole

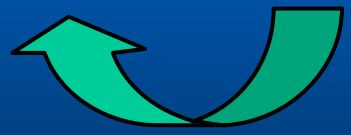
Substructure Search Result



From Structure to Spectrum : Structure Search







Spectrum Prediction

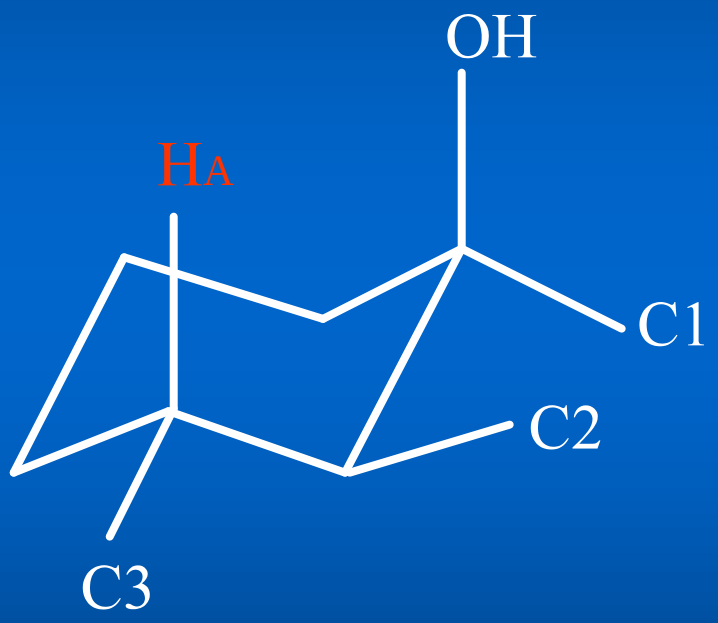
From Structure to Spectrum : Spectrum Prediction

Strategies

- Rule Based Systems
- Database Based Systems

➢ Rule Based Systems

H_A Chemical Shif



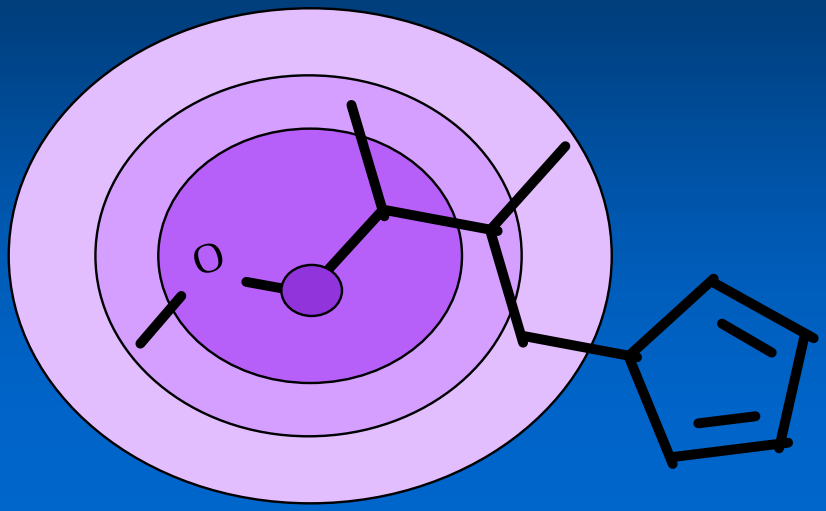
1.44 ppm f

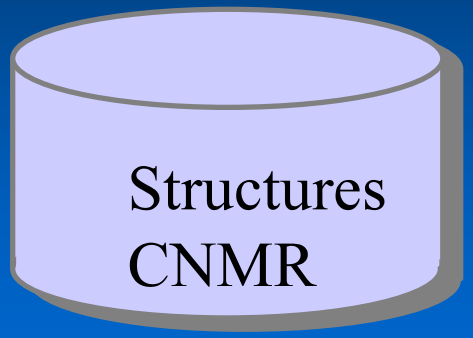
+0.15 ppm f	C3 eq
- 0.31 ppm f	C2 eq
+0.03 ppm f	C1 eq
+0.46 ppm f	

_____

1.77 ppm total

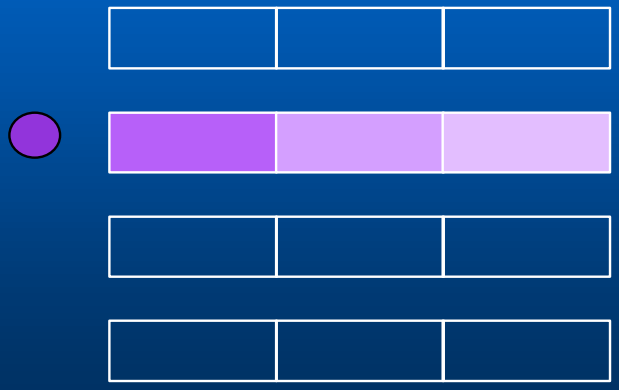
➢ Database Based Systems





Substructure codes
(three spheres)

13CNMR chemical shif



74.20 ppm

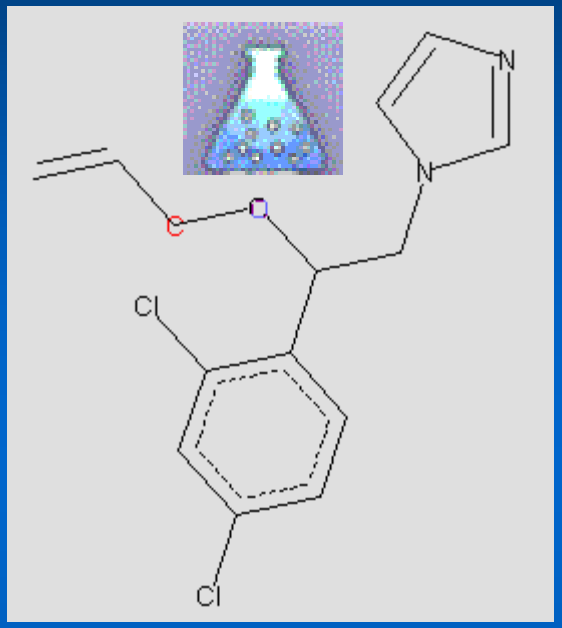
72.20 ppm

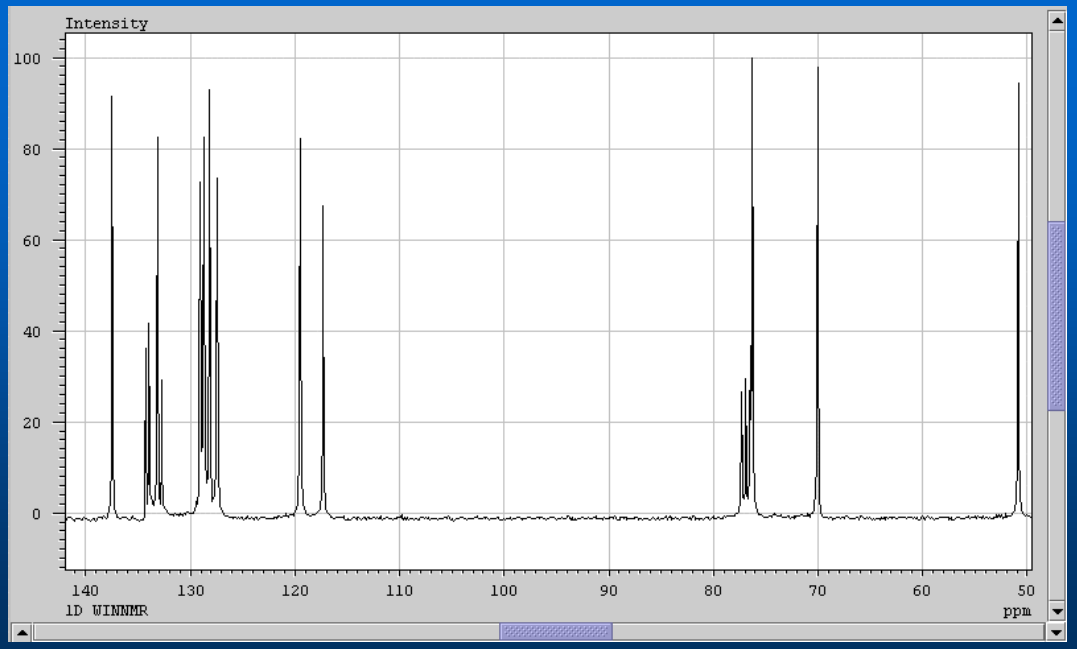
71.80 ppm

71.50 ppm

From Structure to Spectrum : Spectrum Prediction

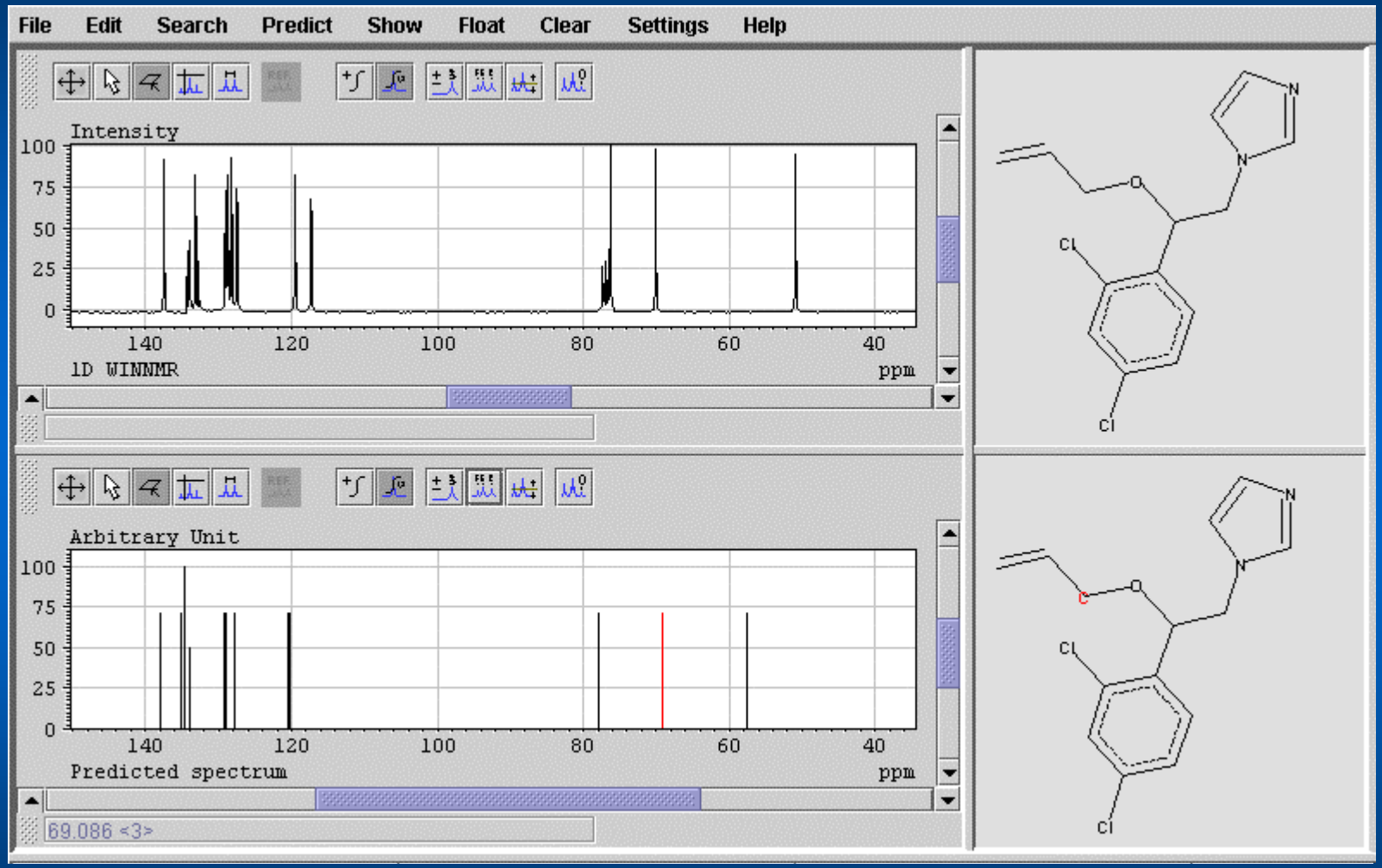
Application 1: CNMR Conf





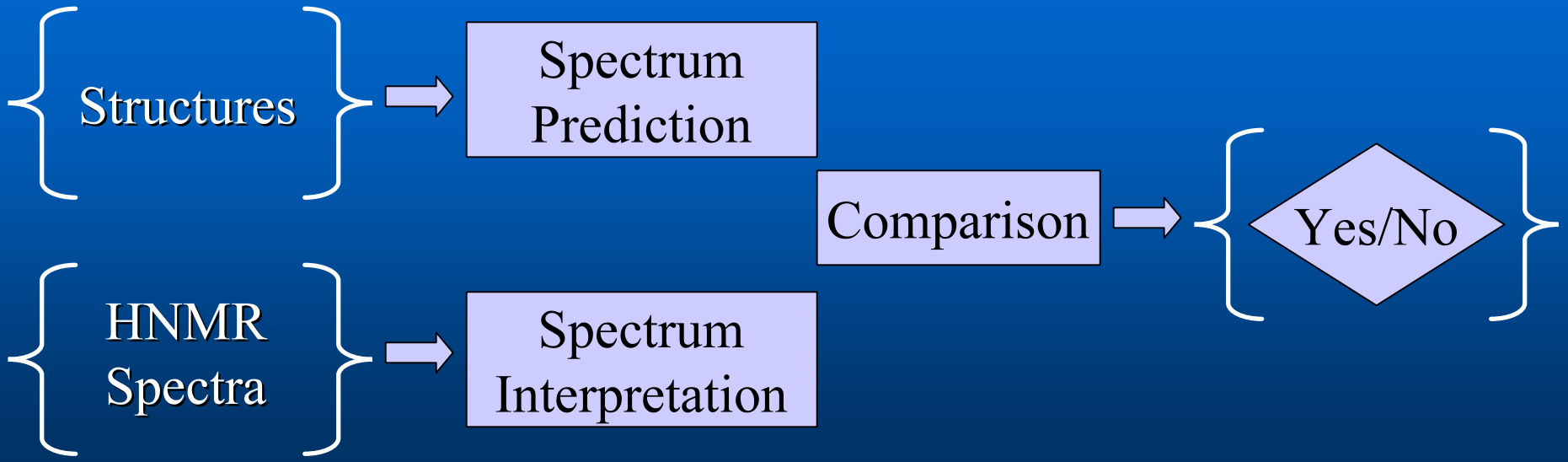
From Structure to Spectrum : Spectrum Prediction

CNMR Conf



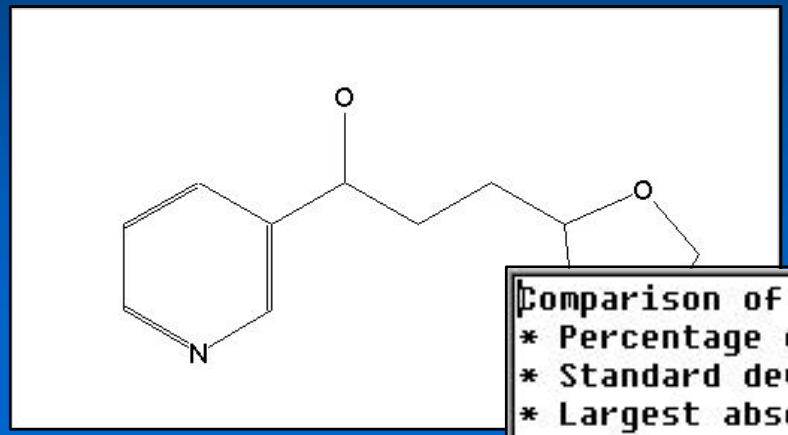
From Structure to Spectrum : Spectrum Prediction

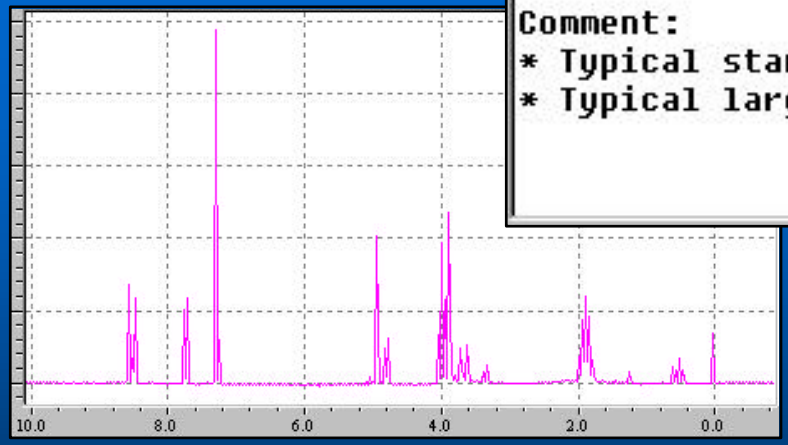
Application 2: Automated HNMR Compatibility Check



From Structure to Spectrum : Spectrum Prediction

Yes/No

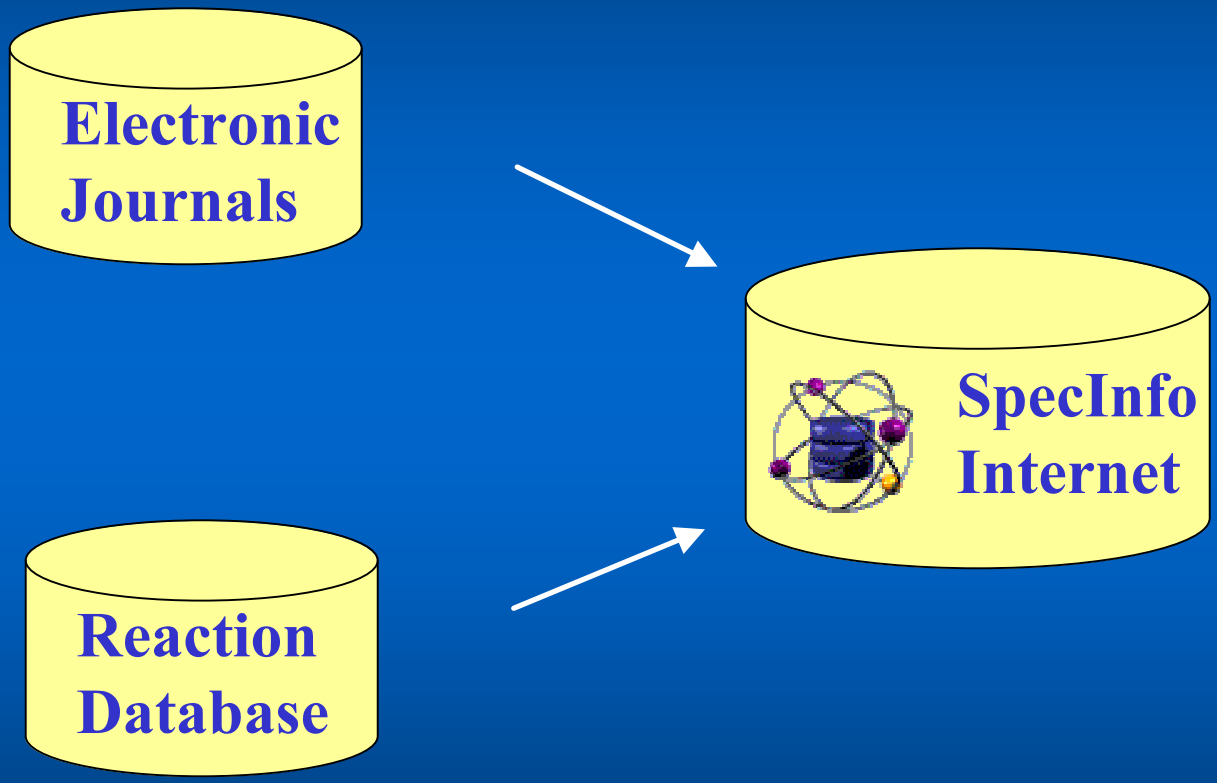




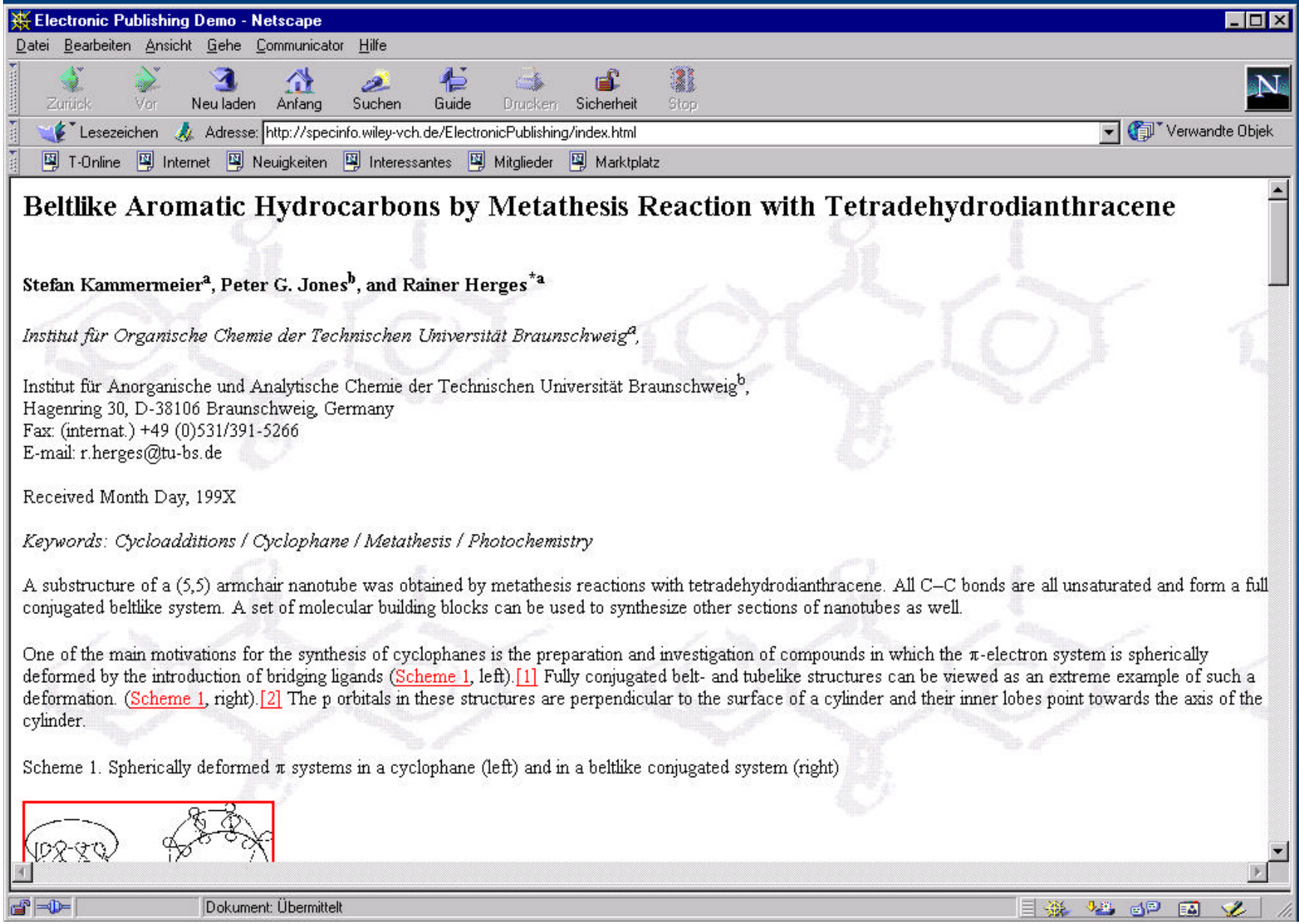

```
Comparison of 1H-NMR shifts from spectrum and structure:
* Percentage of unexplained H atoms in spectrum =  0.00 %
* Standard deviation = 0.15 ppm
* Largest absolute deviation = 0.28 ppm

Comment:
* Typical standard deviations : 0.2 - 0.3 ppm
* Typical largest absolute deviations : up to 1 ppm
```

From Structure to Spectrum : Spectrum Prediction

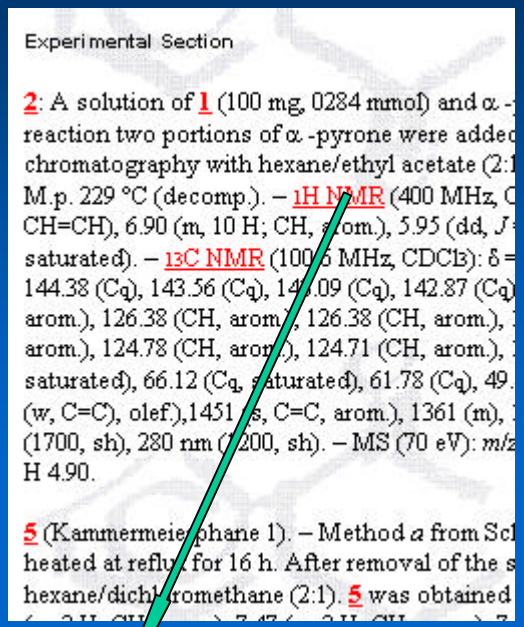
Hyperlinks between Databases



From the Electronic Journal to Spectra



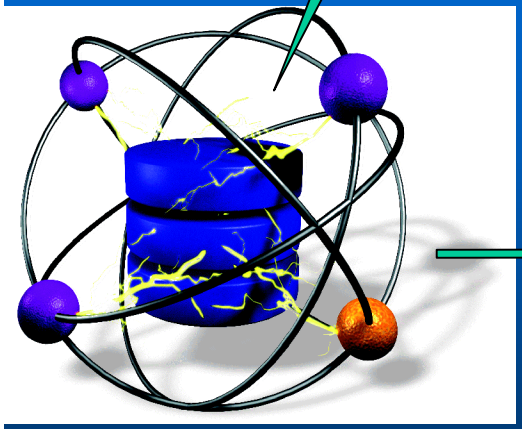
Experimental Section



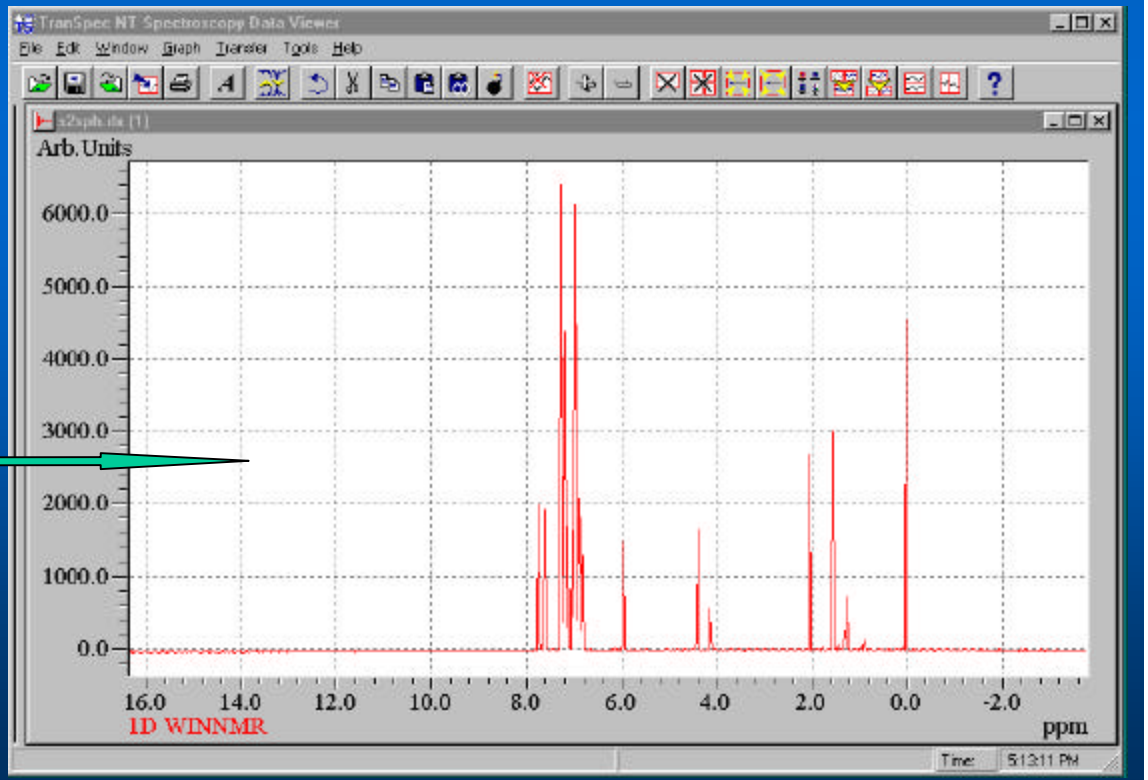
Experimental Section

2: A solution of 1 (100 mg, 0284 mmol) and α -
reaction two portions of α -pyrone were added
chromatography with hexane/ethyl acetate (2:1
M.p. 229 °C (decomp.). – 1H NMR (400 MHz, C
CH=CH), 6.90 (m, 10 H; CH, arom.), 5.95 (dd, J =
saturated). – 13C NMR (100.6 MHz, CDCl3): δ =
144.38 (Cq), 143.56 (Cq), 142.09 (Cq), 142.87 (Cq)
arom.), 126.38 (CH, arom.), 126.38 (CH, arom.),
arom.), 124.78 (CH, arom.), 124.71 (CH, arom.),
saturated), 66.12 (Cq, saturated), 61.78 (Cq), 49.
(w, C=C), olef.),1451 (s, C=C, arom.), 1361 (m),
(1700, sh), 280 nm (4200, sh). – MS (70 eV): m/z
H 4.90.

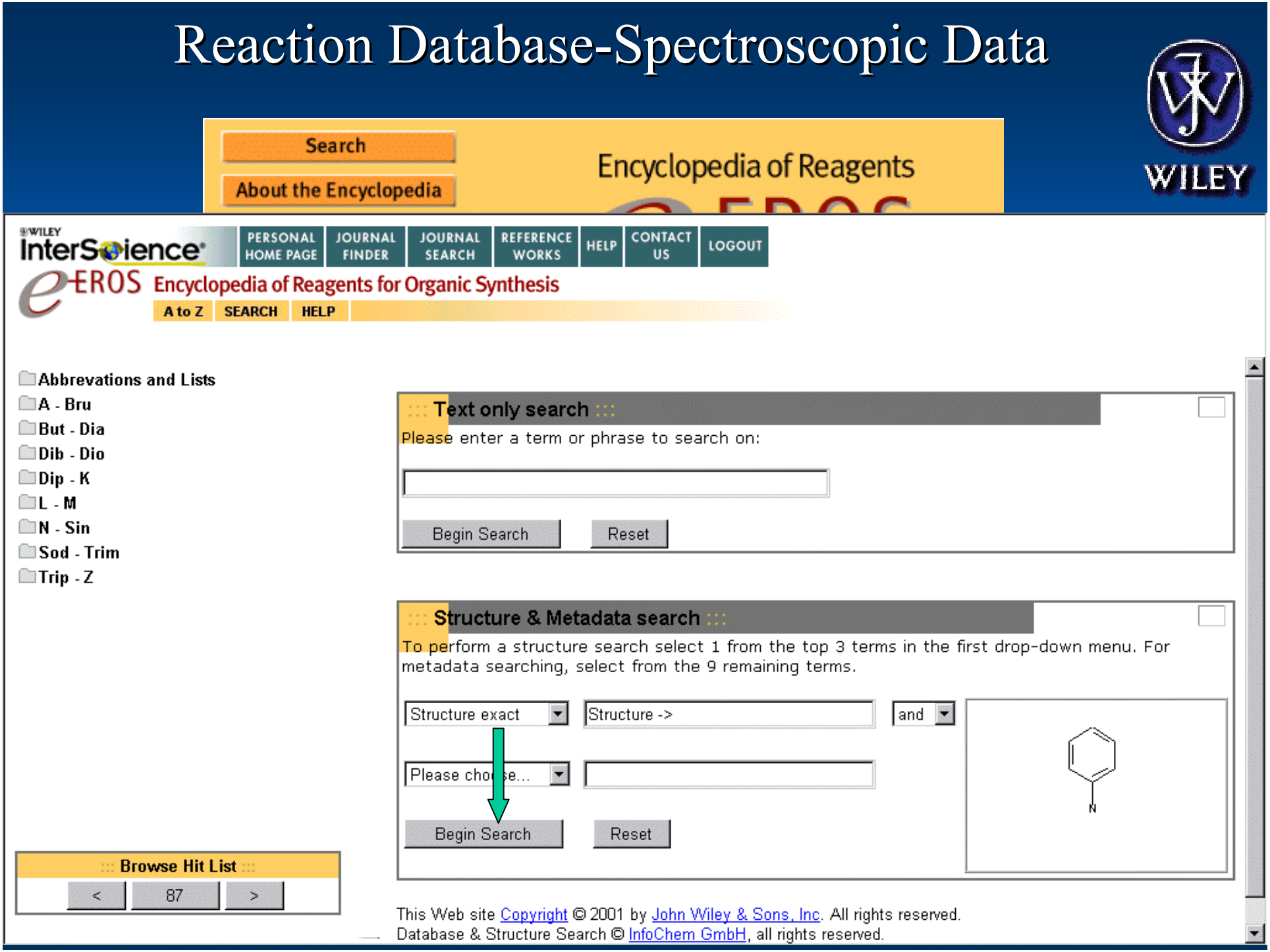
5 (Kammermeier-phane 1). – Method a from Scl
heated at reflux for 16 h. After removal of the s
hexane/dichloromethane (2:1). 5 was obtained



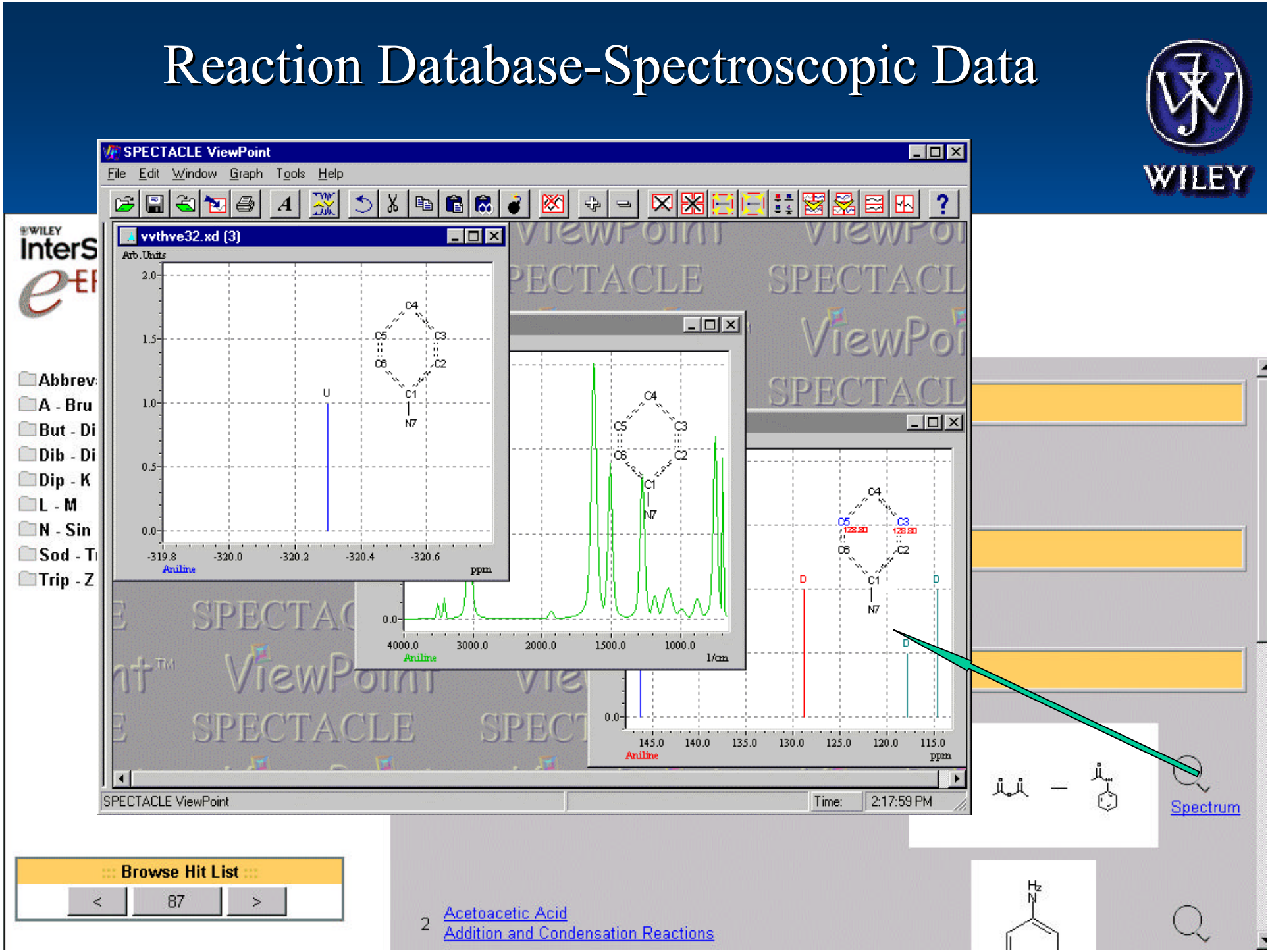
SpecInf Internet



Reaction Database-Spectroscopic Data



Reaction Database-Spectroscopic Data



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