



# JEOL developments in structural biology

Oléron 2016: Troisième Ecole Nationale  
RéNaFoBIS

M. Gilles THIBAULT

23/05/16

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- Introduction of JEOL
- JEOL Bio TEM products
- JEOL Bio NMR
- Delta software





Akishima, Tokyo

"Japan Electron Optics Laboratory"

May 30, 1949

Japan : Headquarters Factory,

9 branches /Overseas : 39countries

~ 3000 employees (JEOL Group)

International Product Quality Assurance Standard "**ISO9001**" acquired(in 1995)

Environment Management System Standard "**ISO14001**" acquired(in 2002)



Croissy sur Seine, France

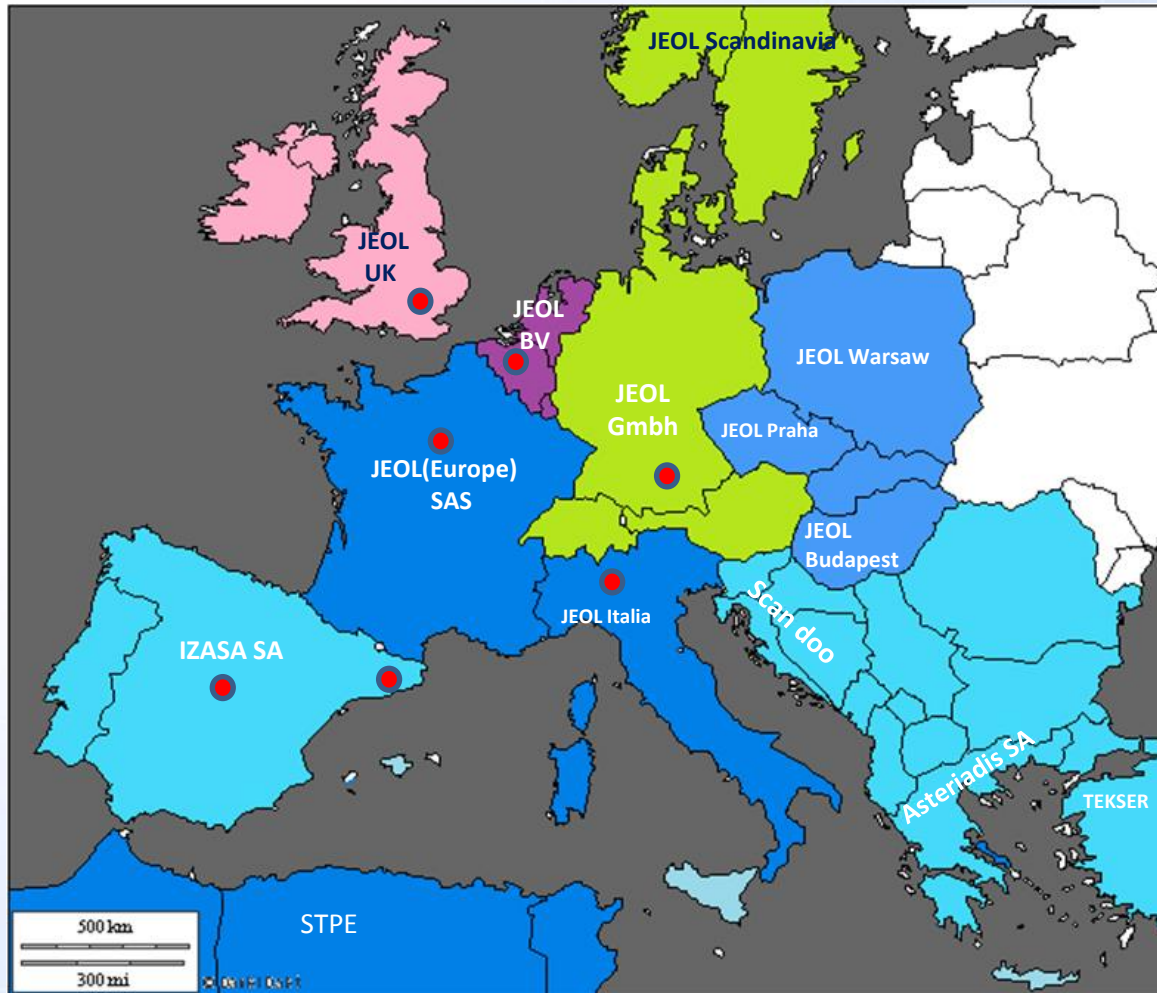
JEOL (EUROPE) SAS

1964

~ 50 employees

+ 800 instruments en  
fonctionnements en France

# JEOL in EUROPE since 1964



**JEOL(Europe)SAS:** 22 European countries – **Turnover ≥ 30 Millions d'EUR**

**Sister Cie:** JEOL Italy - **Branch offices :** Poland-Czech Republic-Slovak Republic-Hungary

**Local Agents:** Spain/Portugal (IZASA.S.A); Greece (Asteriadis.S.A); Turkey (Tekser A.S.)

Slovenia/Croatia/Bosnia(Scan.d.o.o); North Africa (S.T.P.E); Romania / Bulgaria

## ➤ *Electron Optics Products*

- Transmission Electron Microscopes (TEM)
- Scanning Electron Microscopes (SEM)
- Electron Probe Micro Analyzers (EPMA)
- AES, XPS, SPM



## ➤ *Analytical Instrument Products*

- Mass Spectrometers (MS)
- Nuclear Magnetic Resonance (NMR)
- Electron Spin Resonance (ESR)



## ➤ *Semiconductor Equipment.*

## ➤ *Thin Film Coating System*

## ➤ *Medical Equipment*

# JEOL Bio TEM Products

300kV



JEM-3200FSC



GRAND ARM

## Special Products



ARM1300



1300NEF

200kV FEG



JEM-2200FS



JEM-2800



F2

LaB<sub>6</sub>

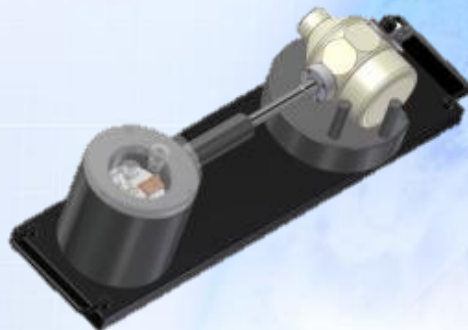
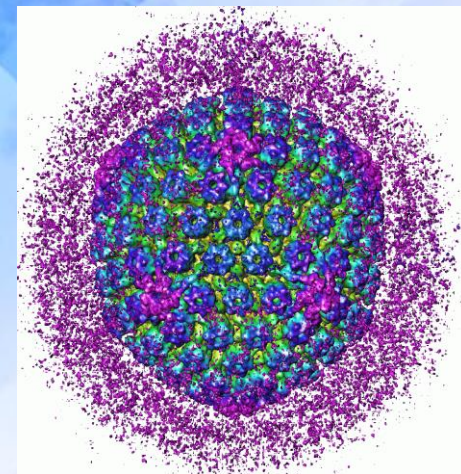
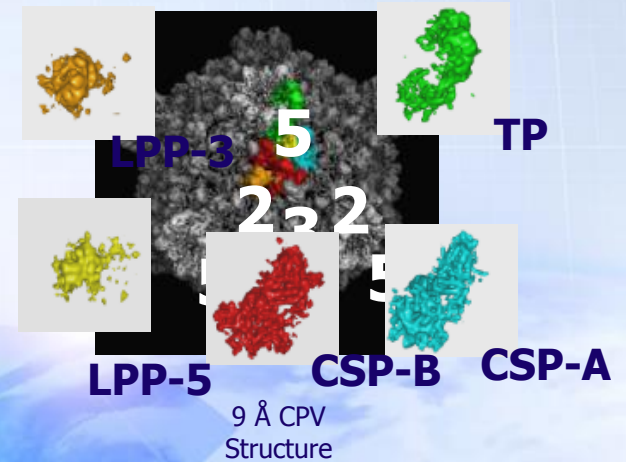
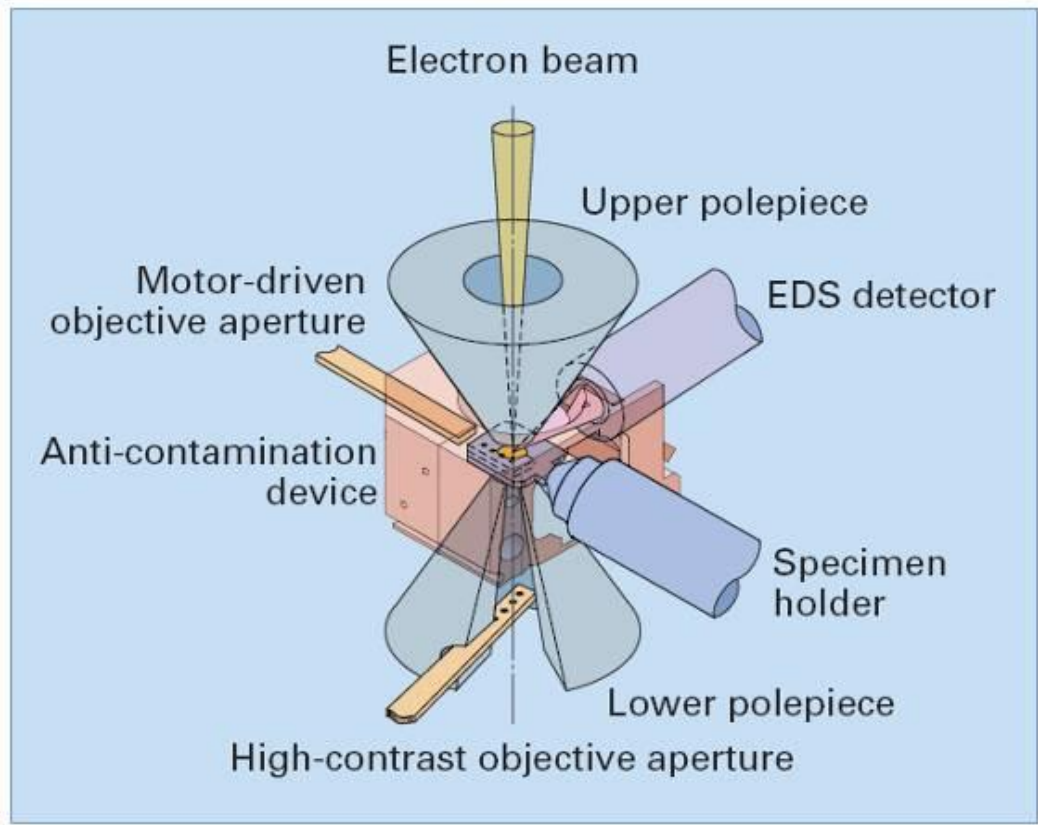


JEM-1400Plus



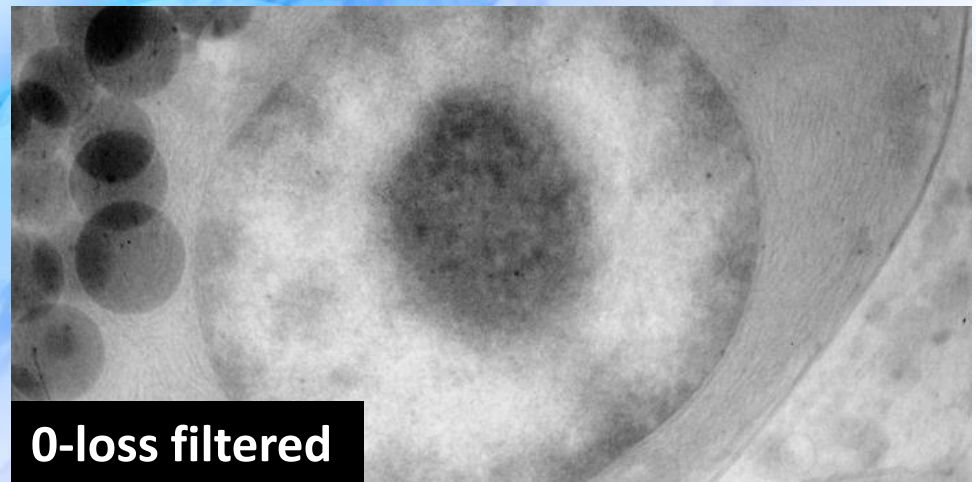
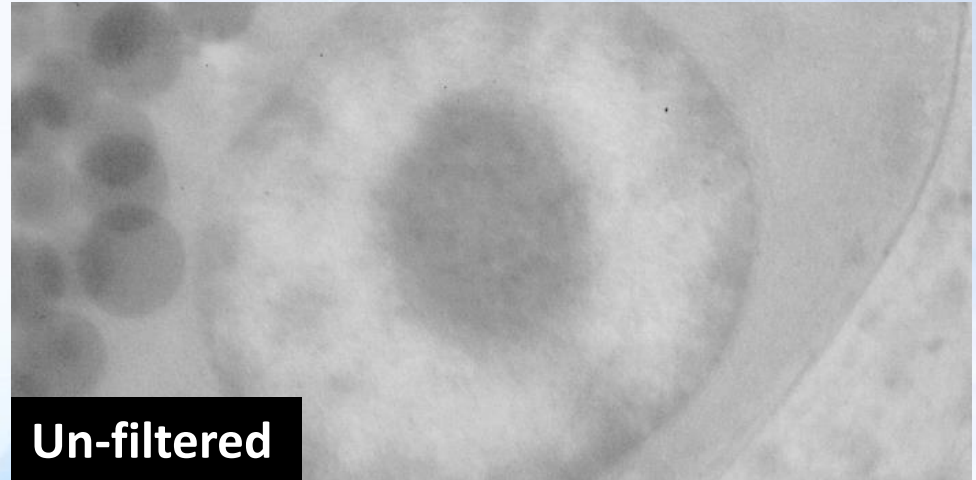
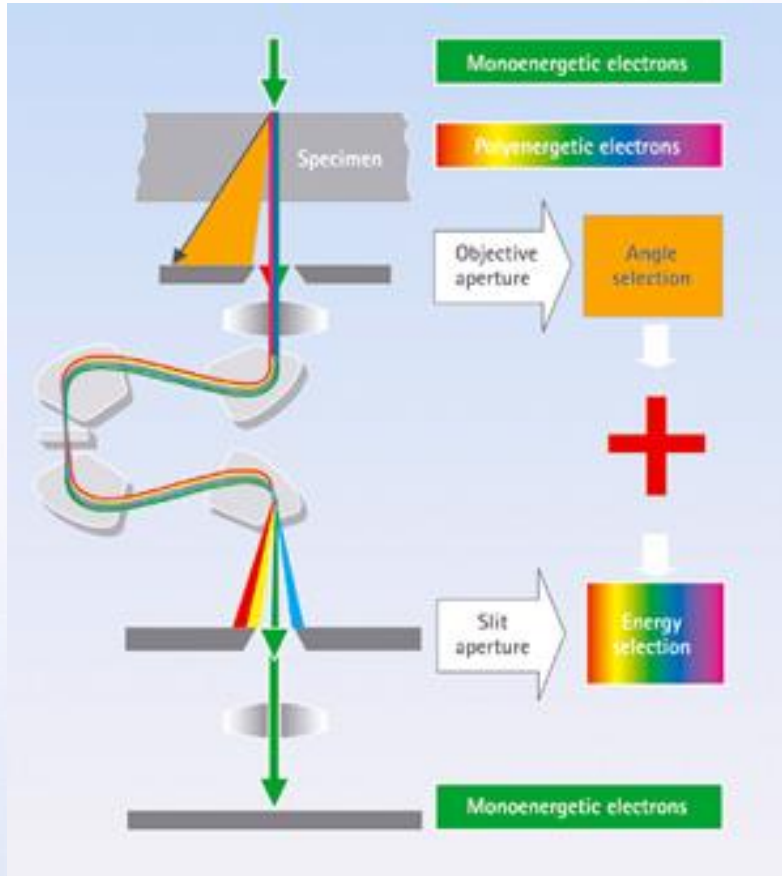
JEM-2100Plus

# Cryo TEM



# How to increase contrast in cryo TEM

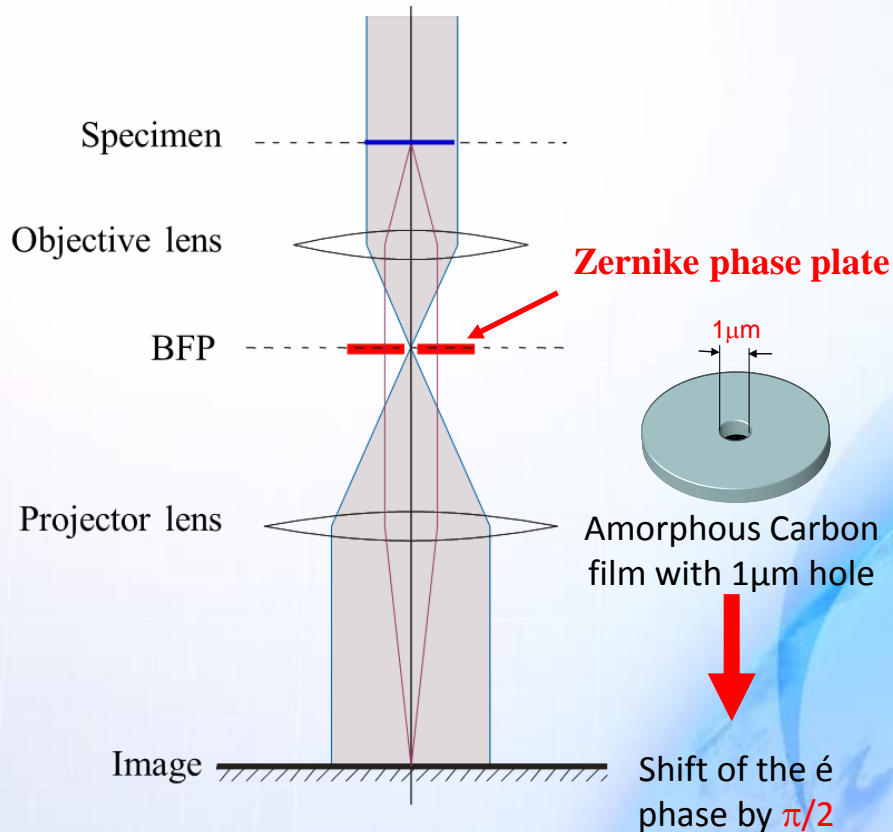
- **Omega filter**



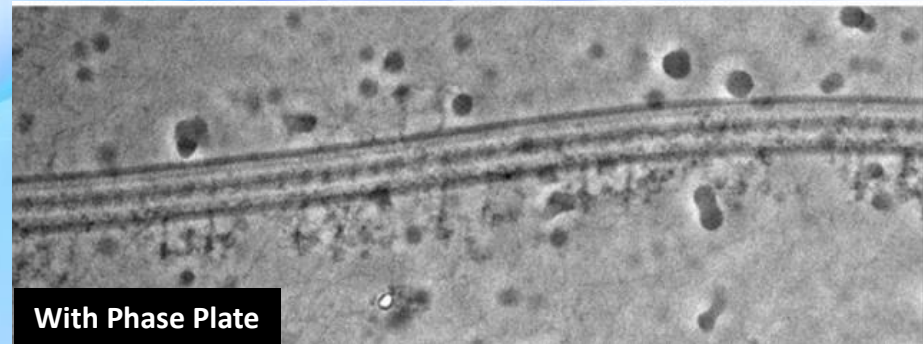
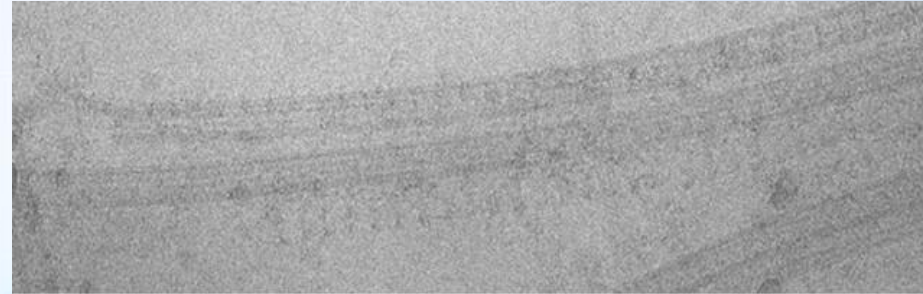


# How to increase contrast in cryo TEM

- Phase Plate developed by JEOL



Cryo-TEM of microtubule doublets from *Chlamydomonas* flagella



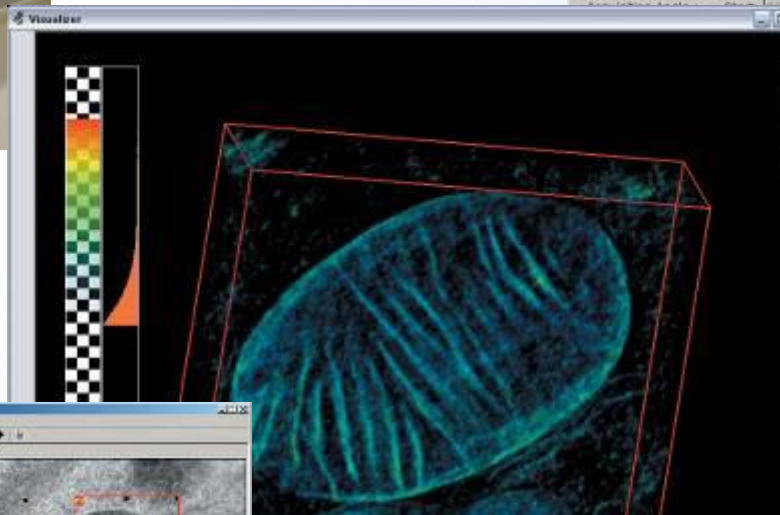
Increase of 3 to 5 times the specimen contrast

# 3D Tomography (option) +/-70°

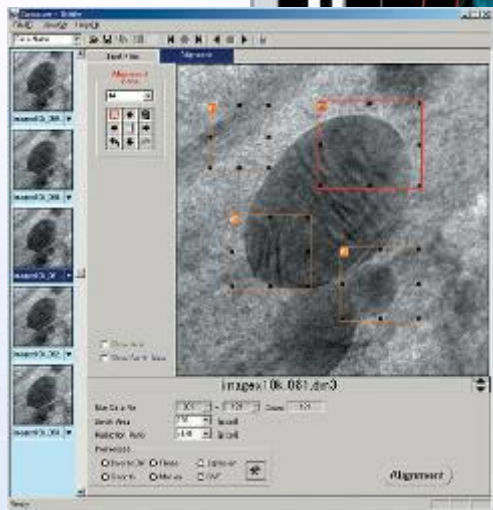
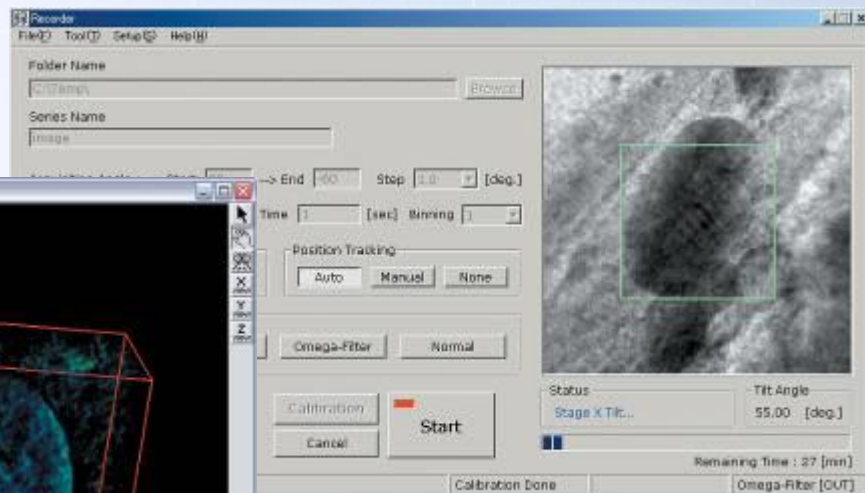


High Tilt Retainer

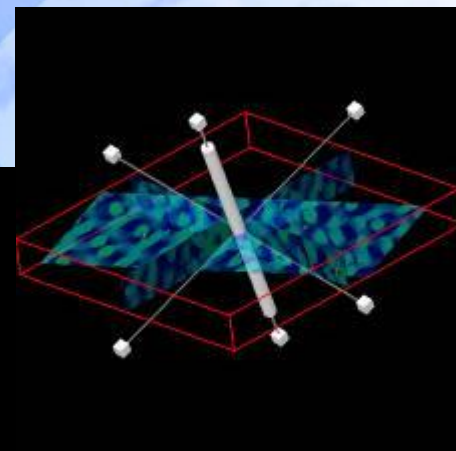
Visualizer



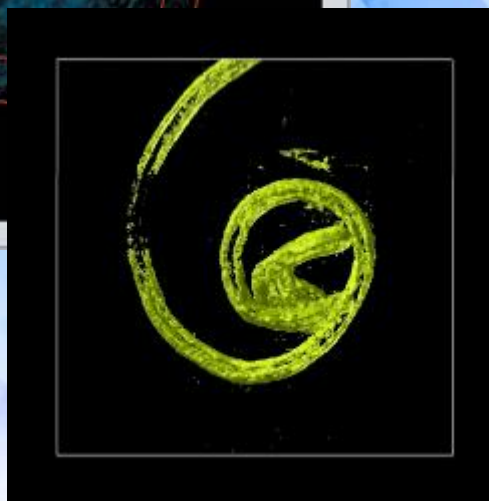
Recorder



Composer



Slicer



Movie

# 3D Structure in CRYO

Courtesy of CBS de Montpellier

*Team « Biologie structurale multi-échelles »*

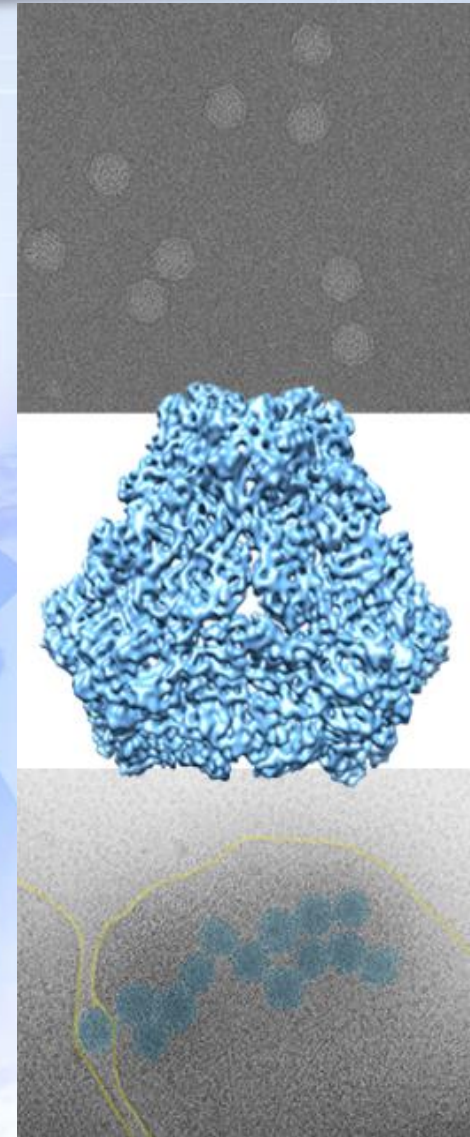
*Dr P. Bron*



JEM-2200FS – Omega filter

Virus ArMV  
Observed in  
cryo-MET  
with a JEOL 2200FS

Reconstruction of  
Virus ArMV  
a 5.5 Å of  
résolution



# New NMR Developments

- The latest digital and high frequency technologies
- Compact size chassis with even greater expandability
- High Speed & Most Precise RF switching time : “5ns”
- Improved usability to accessories



## NMR spectrometer **Z** **ECZR** and **ECZS**

# How to increase resolution

## Use of high temperature super conductor

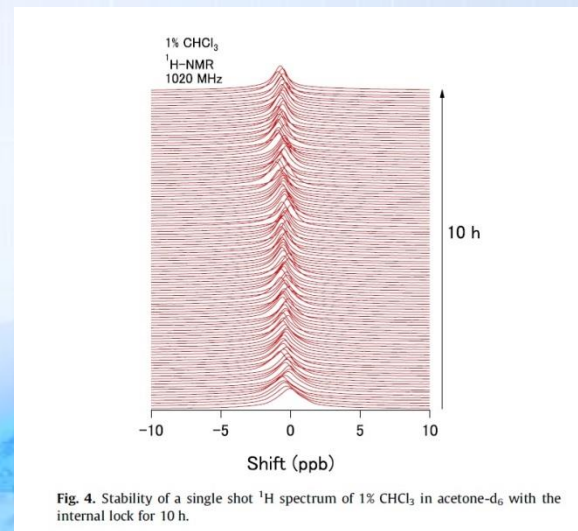


Fig. 4. Stability of a single shot <sup>1</sup>H spectrum of 1% CHCl<sub>3</sub> in acetone-d<sub>6</sub> with the internal lock for 10 h.

- 1) Very high magnetic field > 1.0 GHz
- 2) Stable and homogeneous B<sub>0</sub> field
- 3) Long time operation
- 4) Low energy consumption

Efforts behind the LTS/HTS system are

- 1) Materials for HTS
- 2) Stable room temperature
- 3) External lock, etc...

**It is the same as LTS systems!**

Japan's National Institute for Materials  
Science (NIMS)

K. Hashi et al., J. Magn. Reson. 256 (2015) 30-33.

# How to increase resolution

## Use of Cryogenic Probe

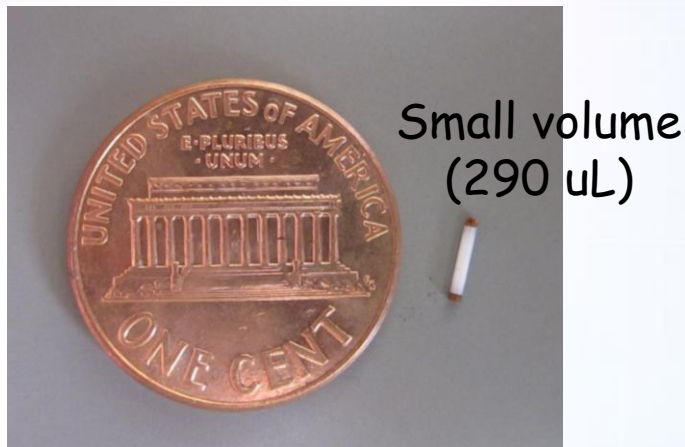
- **≥ 3-fold\* higher sensitivity**  
\* compared with R.T. tuneable probe
- **V.T. range: -40° to +150°C**
- **Power consumption ¼ vs Helium probe**
- **Observations:  $^{13}\text{C}$ ,  $^{31}\text{P}$ ,  $^{15}\text{N}$ ,  $^{29}\text{Si}$ ,  $^1\text{H}$ ,  $^{19}\text{F}$**
- **No water chiller required**

### **2 different configurations available**

- Circulating refrigerator type (closed configuration)
- Liquid N<sub>2</sub> cooling type (open configuration)

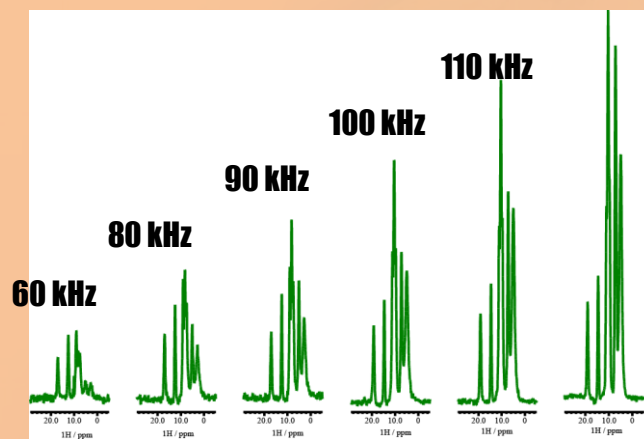


# BioNMR : Use of ultrafast MAS



Resolution & sensitivity improvement in  $^1\text{H}$  NMR

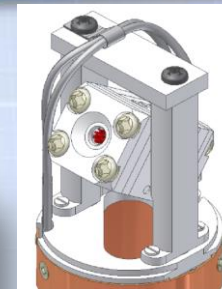
**120 kHz**



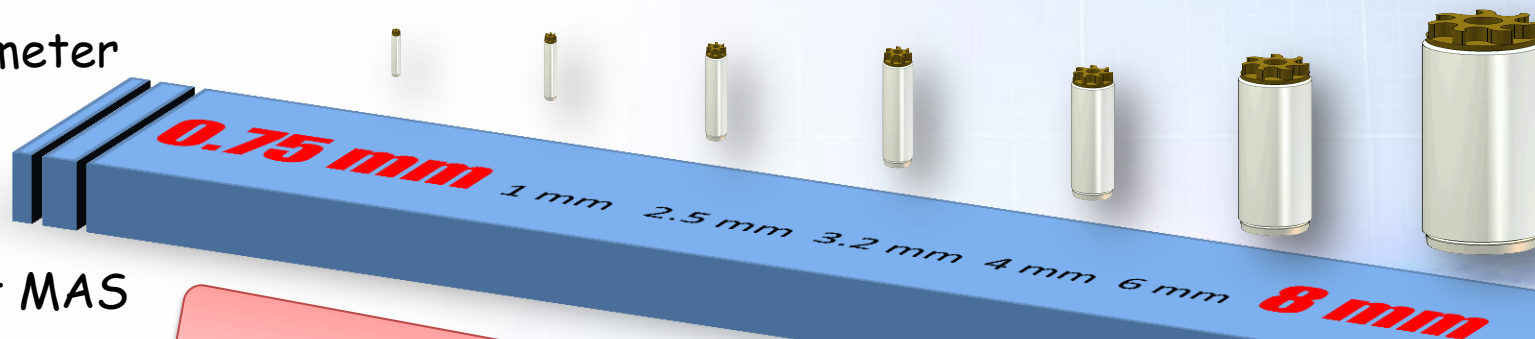
$^1\text{H}$  INADEQUATE

# MAS rates and diameters

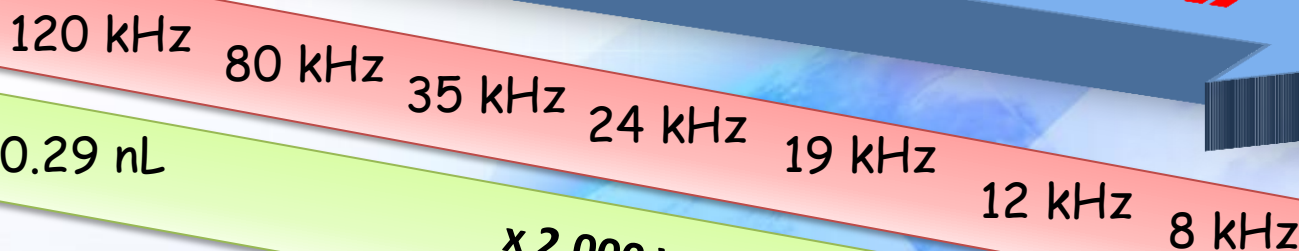
The maximum spinning frequency is limited due to material (Zirconia).



Diameter



Fast MAS rate



Small Sample volume

0.29 nL

x 2,000 Volume

616 uL

slow

large

$$(\text{maximum MAS rates / kHz}) \times (\text{rotor diameter / mm}) \sim 80$$

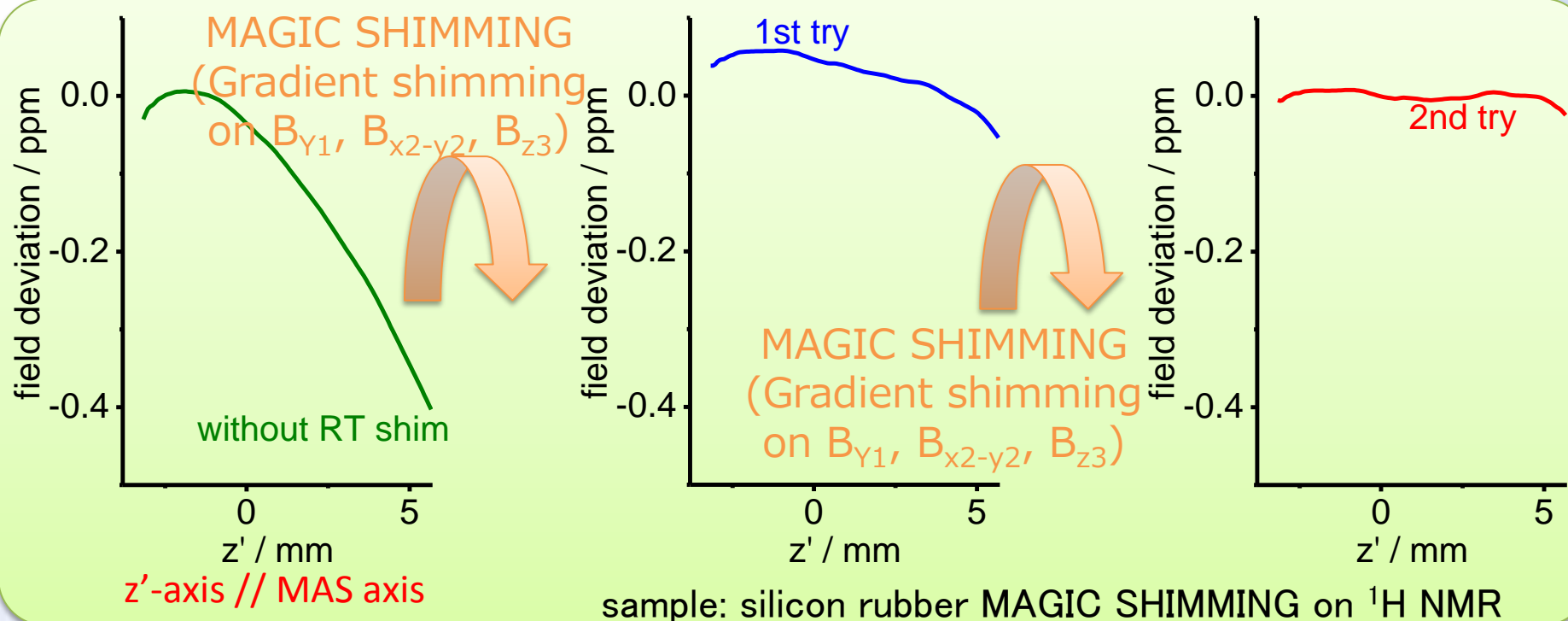
$$(\text{sample volume / uL}) \sim 0.8 (\text{rotor diameter / mm})^3$$

$$(\text{sensitivity}) \propto (\text{rotor diameter})^{3/2}$$



# MAGIC SHIMMING

Gradient shimming at magic angle (MAGIC SHIMMING)  
works well less than few minutes.



standard shim coil  
(homospoil field gradient)

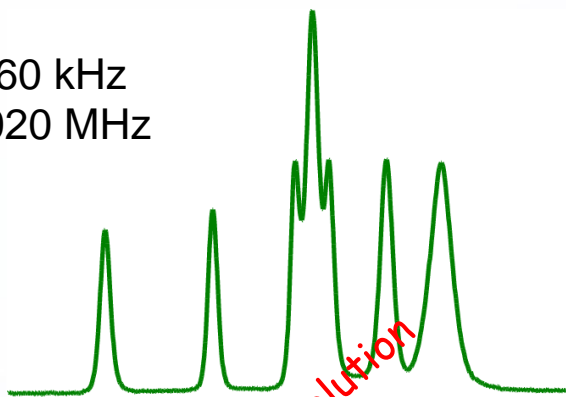


standard MAS probe

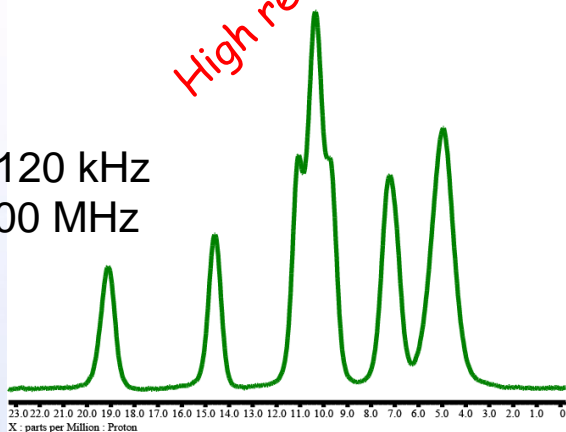
# High magnetic field

The resolution and sensitivities are highly improved at high magnetic field with fast MAS.

MAS 60 kHz  
@ 1020 MHz

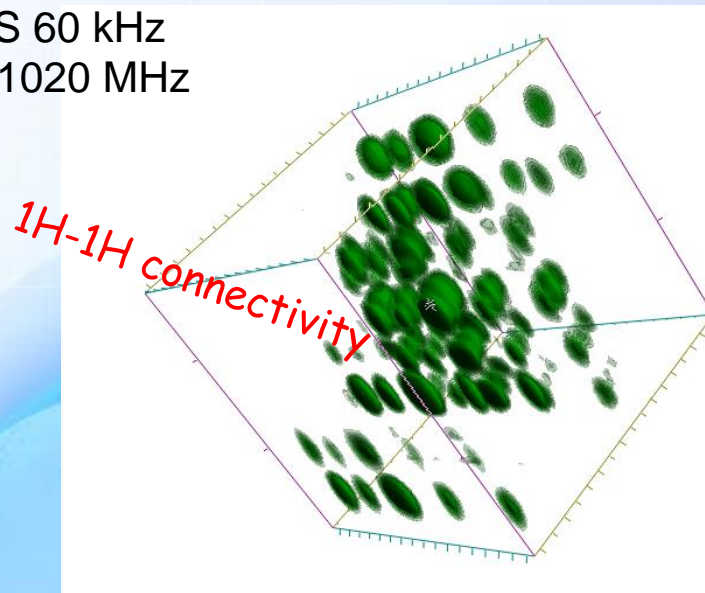


MAS 120 kHz  
@ 600 MHz



$^1\text{H}$  NMR of L-histidine HCl H<sub>2</sub>O

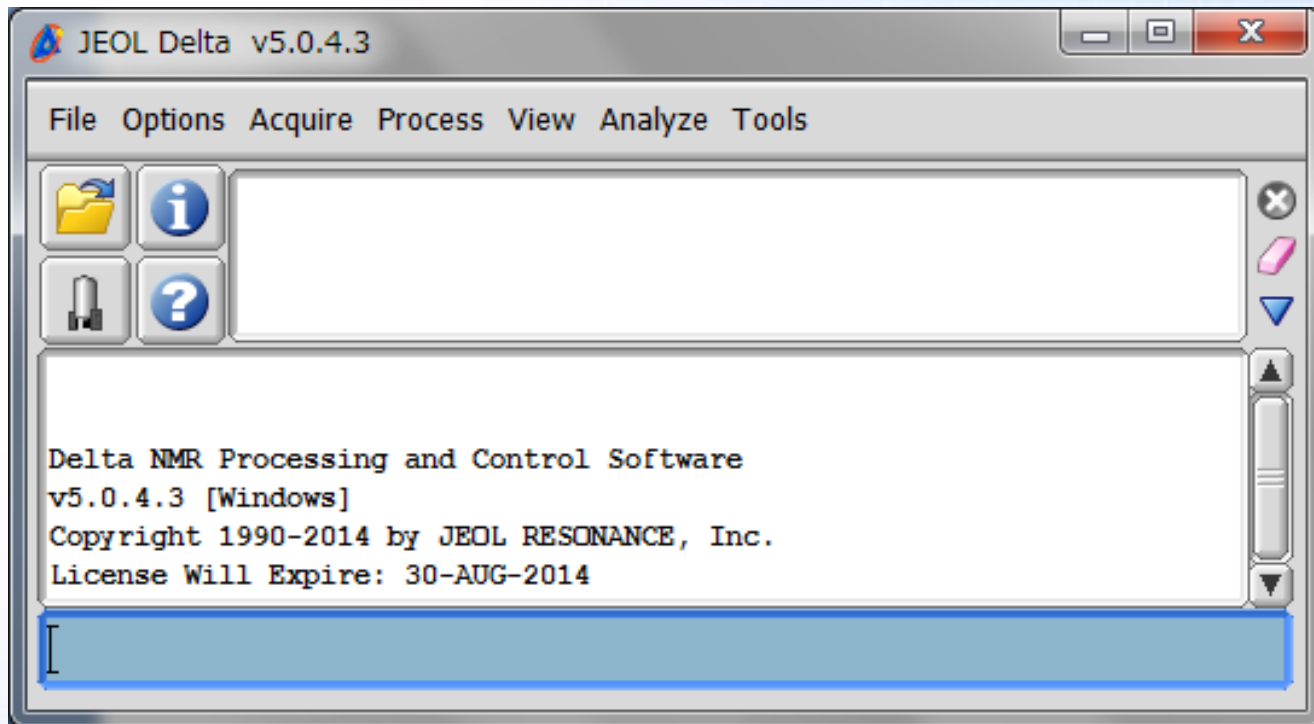
MAS 60 kHz  
@ 1020 MHz



$^1\text{H}$  3D SQ/DQ/SQ NMR of L-histidine HCl H<sub>2</sub>O

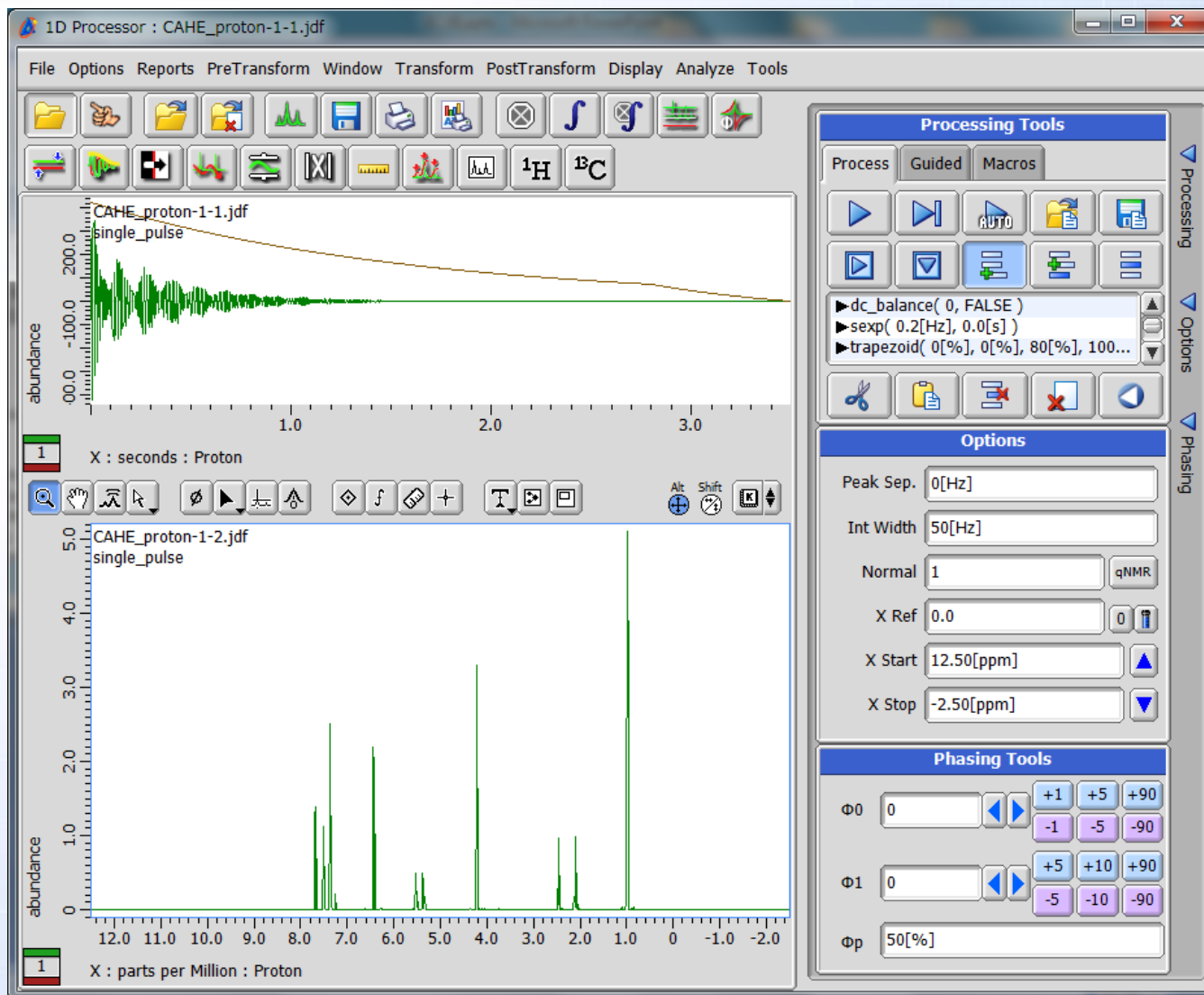
Collaboration with NIMS, COBELCO, RIKEN, U of Michigan

# Delta Software V5.0.X

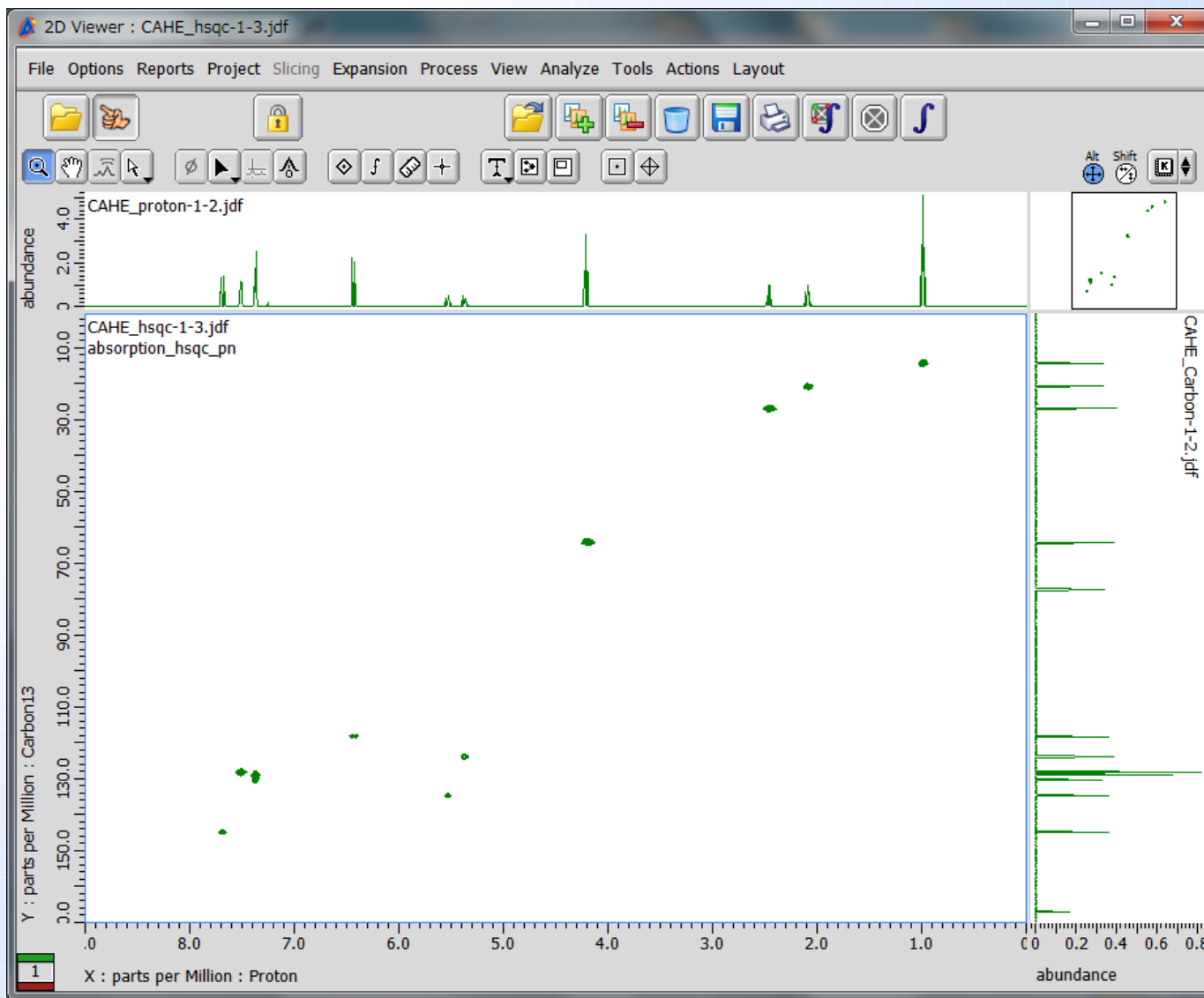


- Completely new, optimized design
- More efficient and easy to operate
  - Fully integrated automation
- All data files are primarily saved on data server

# Data Processing - 1D Processor



# Data Processing - 2D Processor



nmrsupport.jeol.com

JEOLUSA.com JEOL Resonance

JEOL Solutions for Innovation

Search NMR Support

## Welcome to JEOL Delta / NMR Support

The JEOL Delta / NMR support web site provides support for users interested in the Delta NMR Software package and JEOL's NMR Spectrometers. The web site includes Delta NMR software kits for NMR data processing only and kits for JEOL NMR Spectrometer control with data processing. Areas for Delta License Key generation, discussion forums, FAQ's, and documentation downloads are also available on this web site.

**To get started, simply:**

- 1. Open a User Account**  
Registration to this site is moderated - to gain full access, be sure to **also submit your spectrometer information** so that we can assign you the proper authorization for software downloads and/or license key generation.
- 2. Login to Your Existing Account**  
You will then see content and features available for your account type.
- 3. Migrate Your Account from AI-SUP, the old Delta support web site**  
If you are a user migrating from [ai-sup.jeol.com](http://ai-sup.jeol.com) please **click to login and update your account and spectrometer information in order to gain access to the appropriate area(s) of the site.**

**Once your account registration and spectrometer information is reviewed, you will be granted the appropriate access to other areas of the site.**

### Read Me 日本語

Title
NMR Support Read Me (English)
NMR Support Read Me 日本語 (Japanese)

Get ADOBE READER

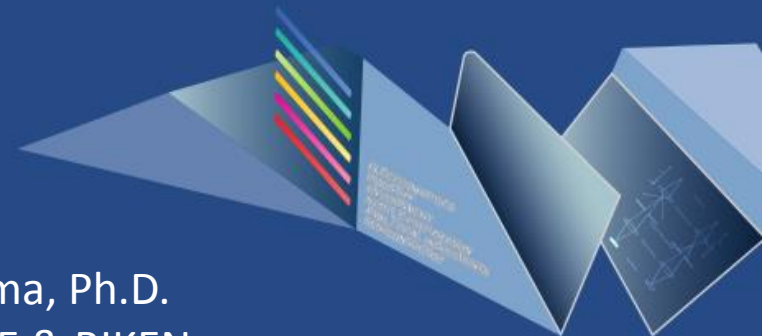
If you have comments or questions regarding this web site, please contact the site **administrator**.  
DO NOT send questions about Delta operation or installation. DO NOT request Delta License Keys from the administrator.

JEOL USA, Inc.  
11 Dearborn Road, Peabody, MA 01960

Please direct questions or comments about Delta or JEOL NMR spectrometers to the discussion boards.



Yusuke Nishiyama, Ph.D.  
JEOL RESONANCE & RIKEN  
Specialist Solid-State NMR



# Thank you for your attention

QUESTION ?



[thibault@jeol.fr](mailto:thibault@jeol.fr)