Instructions for using BL14B2 XAFS Standard Sample database (Ver. BENTEN)

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1. Connecting to a database site

(1) Open the SPring-8 experimental data transfer system BENTEN (URL: https://benten.spring8.or.jp/). When you open the URL, the top page (Fig. 1) is displayed.

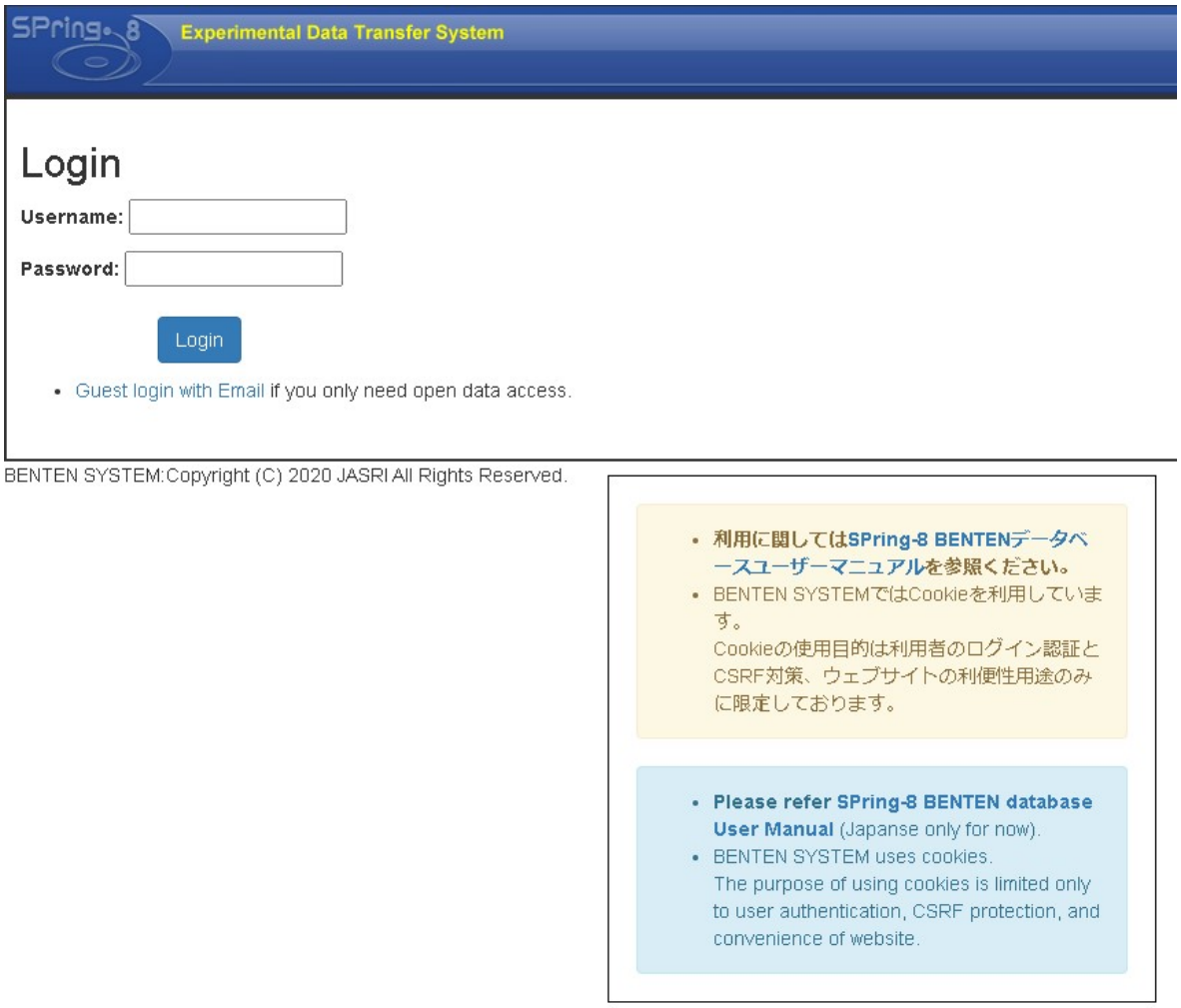


Fig. 1

(2) UI site (SPring-8 / SACLA electronic application system (https://user.spring8.or.jp/)), Enter your user name (SPring-8 ID) and password, and click the Login button. To log in with your personal email address, click "Guest login with Email" under the Login button. The guest login screen shown in Figure 2 will be displayed. Enter your email address and click the Login button. If you log in normally, the screen (Fig. 3) related to email address authentication (two-factor authentication) will be displayed.

*After creating a new UI site account, it will take about a day to log in to this database site.

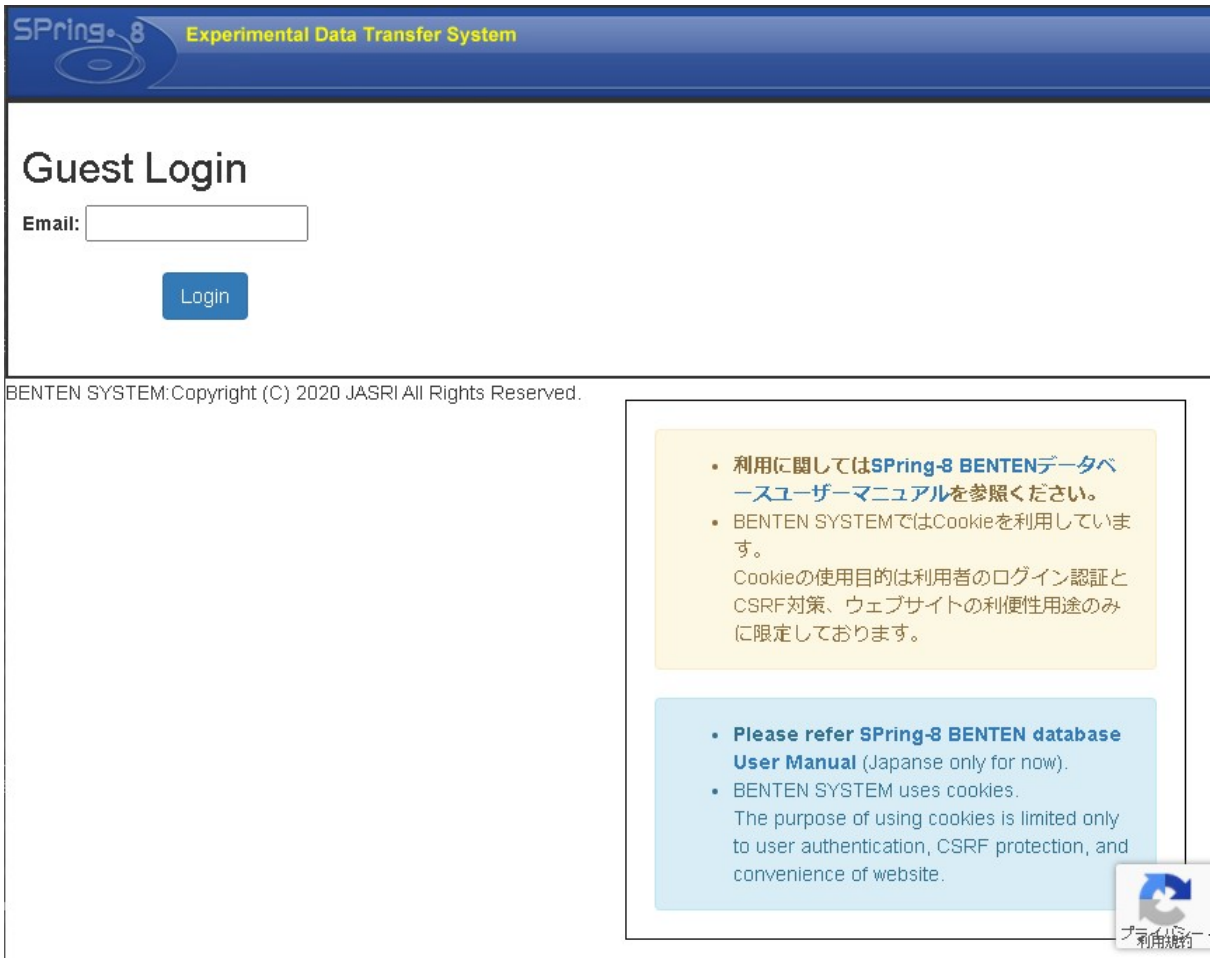


Fig. 2

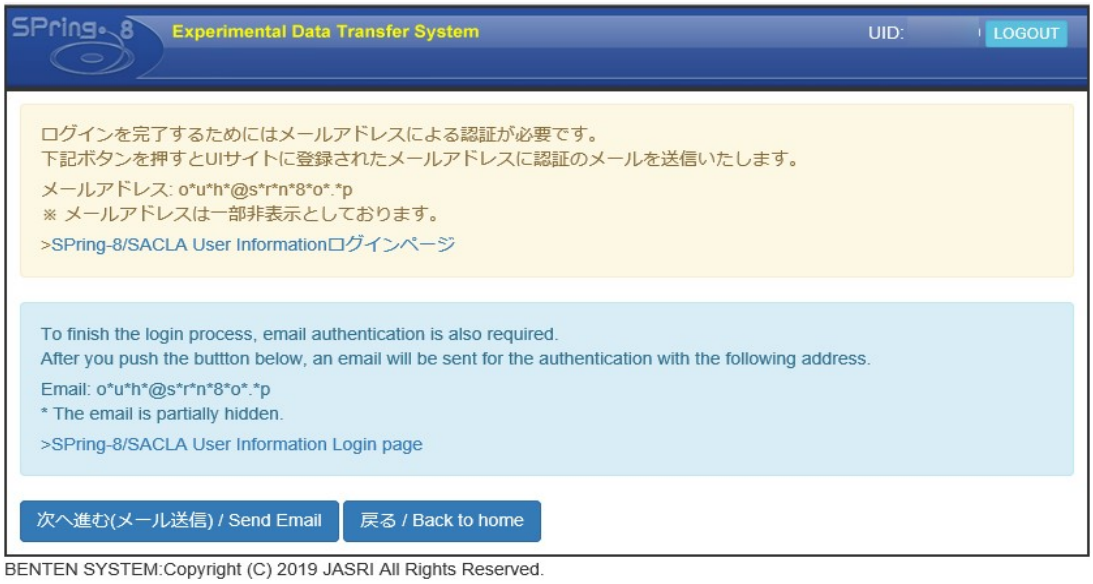


Fig. 3

(3) Click " Send Email" button. Following screen is displayed (Fig. 4).

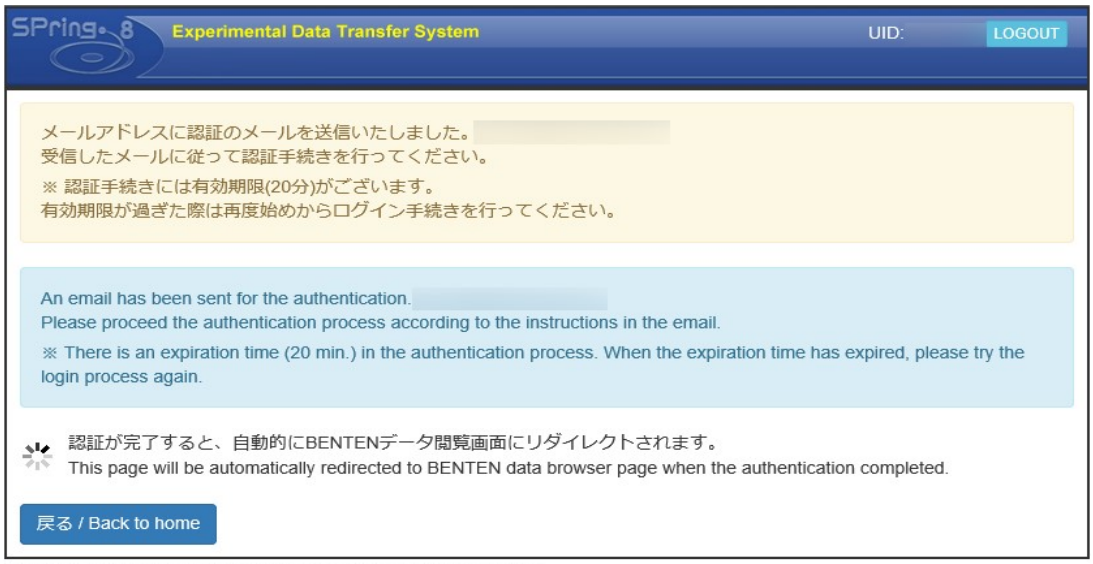


Fig. 4

(4) Check e-mail of subject “Guide for BENTEN authentication” in your e-mail software (Fig. 5)

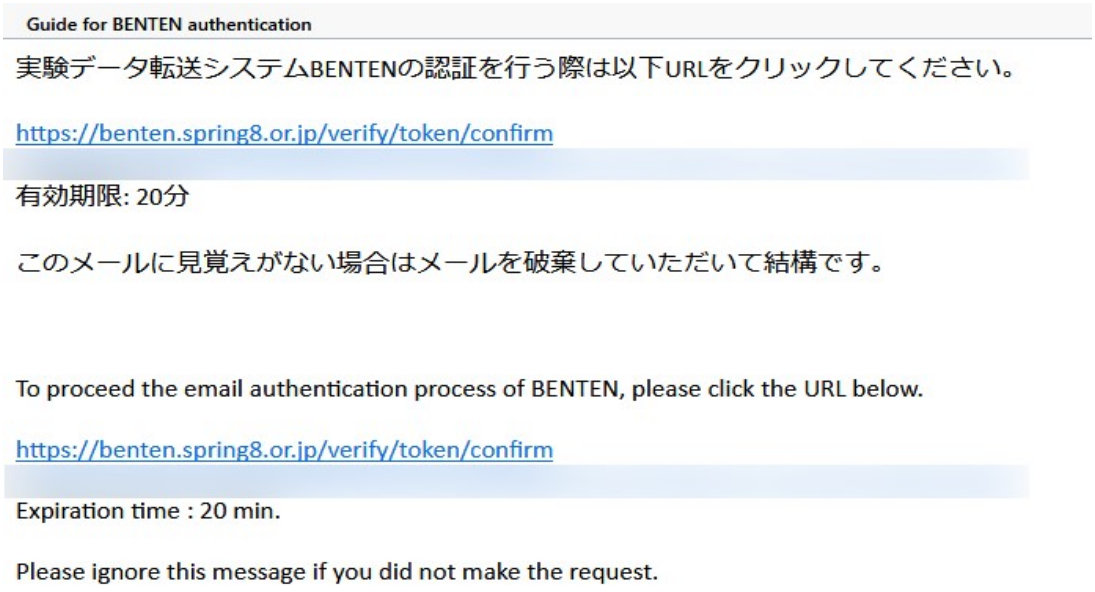


Fig. 5 BENTEN authentication e-mail

(5) When you click the link in the e-mail, a screen will be displayed to prompt for authentication (Fig.6).

Fig.6

(6) Click “Authenticate” button. The authentication completion screen is displayed (Fig. 7)

Fig. 7 Authentication completion screen

(7) The email sending screen in Fig. 4 is redirected to the data viewing screen (Fig. 8).

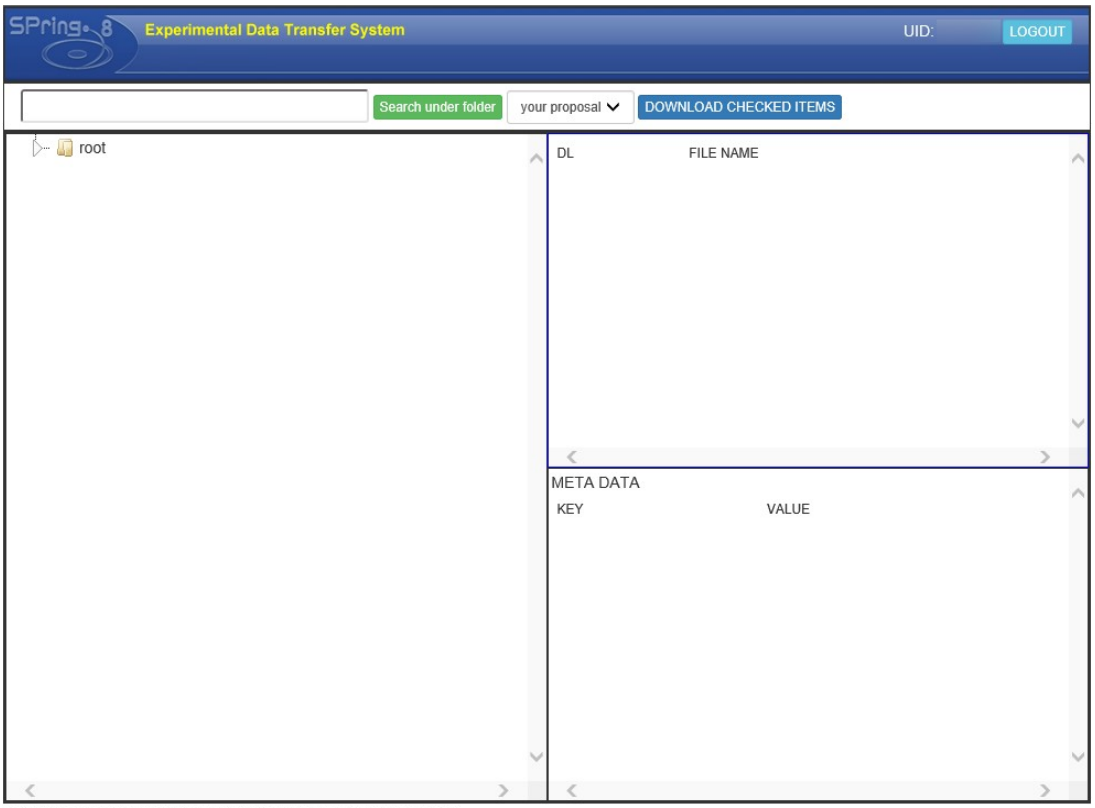


Fig. 8 Data viewing screen

2. Browsing standard sample data

2-1. Viewing data

(1) In the tree display on the left side of the screen, the screen shown in Fig. 9 (a) is displayed. The folder hierarchy of the tree is

・root(Fig. 9 (a))
・SPring-8(Fig. 9 (b))
・BL14B2(Fig. 9 (c))
・XAFS_Standard (Registration folder for XAFS standard sample data、Fig. 9 (d))
・Element(Fig. 9 (e))
・Absorption edge(K,L3,2,1、Fig. 9 (f))
・Net plane(Si(111) or Si(311)、Fig. 9 (g))
・Name of standard sample(Fig. 9 (h))

It is in the order of. In the corresponding data field, the information of the folder one level below the folder selected in the tree display (in the case of the lowest layer, the data file included in the folder) is displayed.

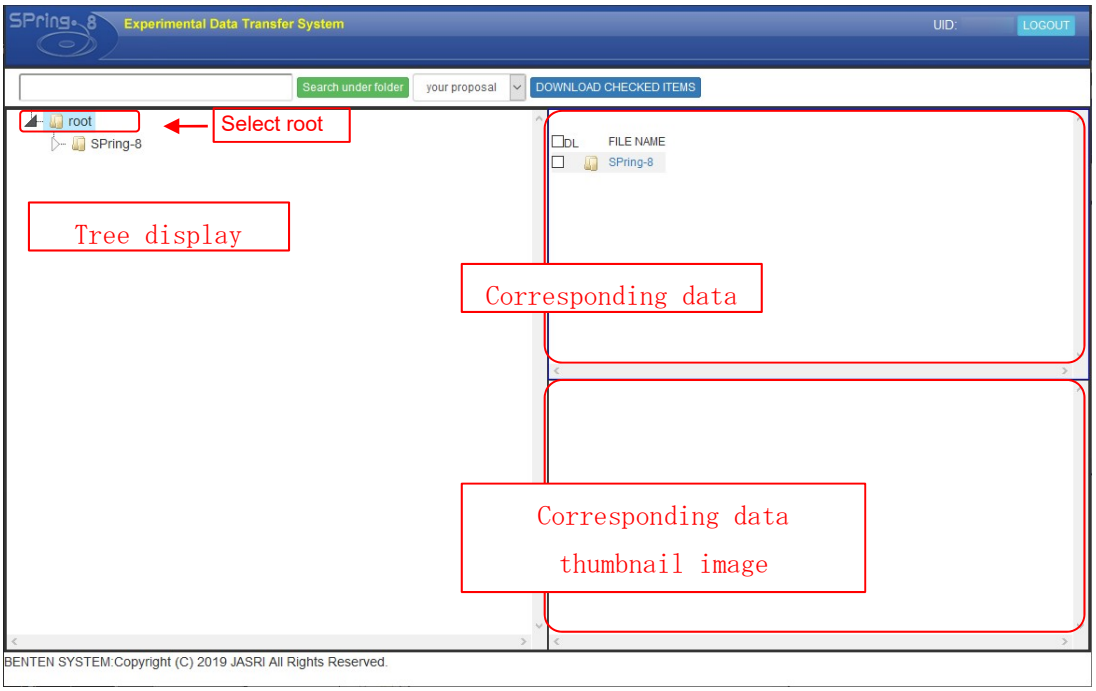
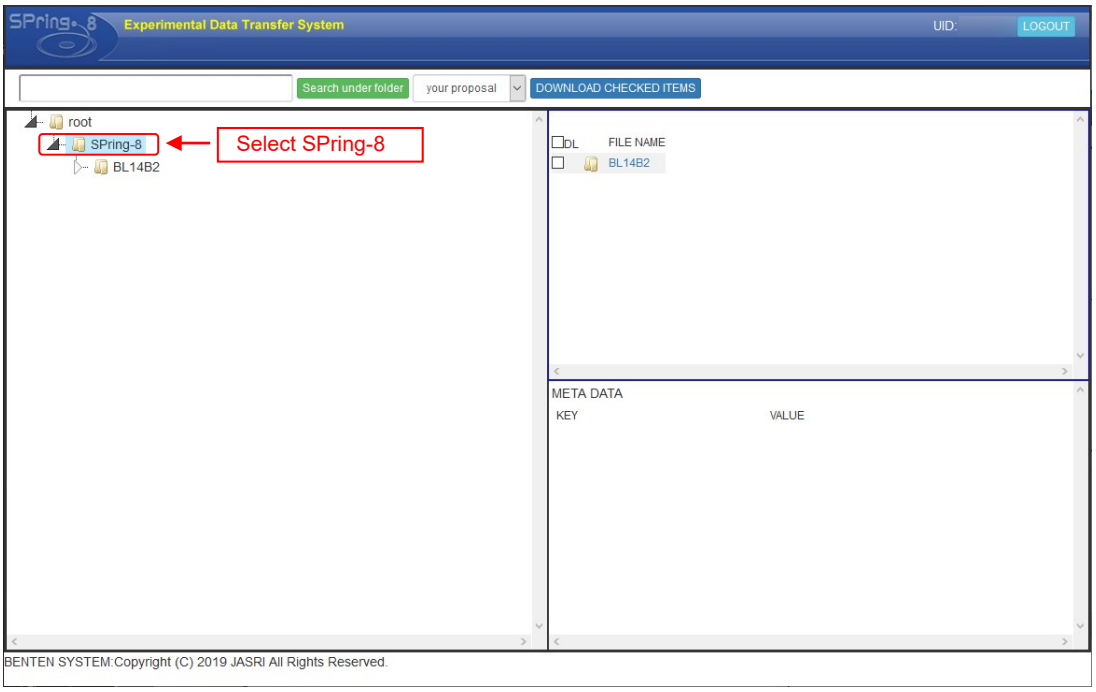
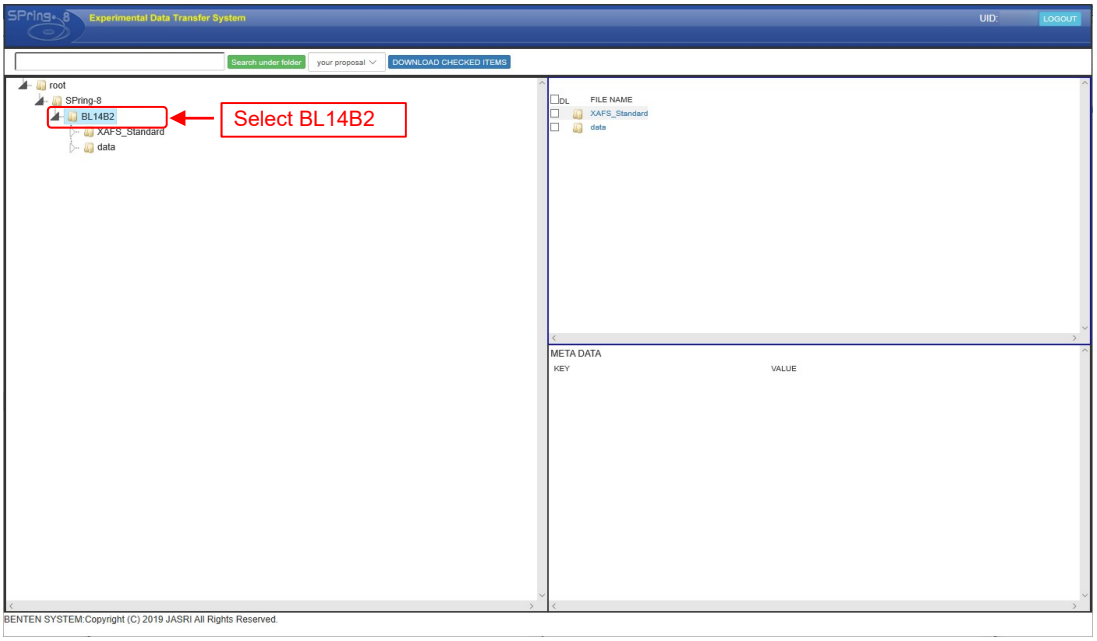


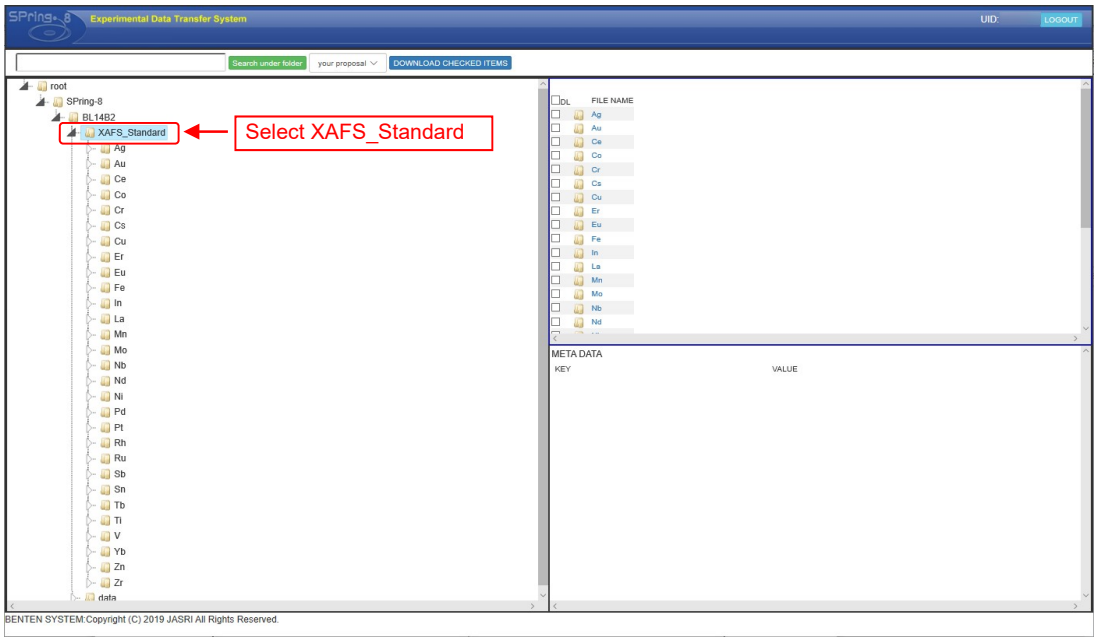
Fig.9 (a) Initial screen for tree display
(select the top (root) of the tree display)



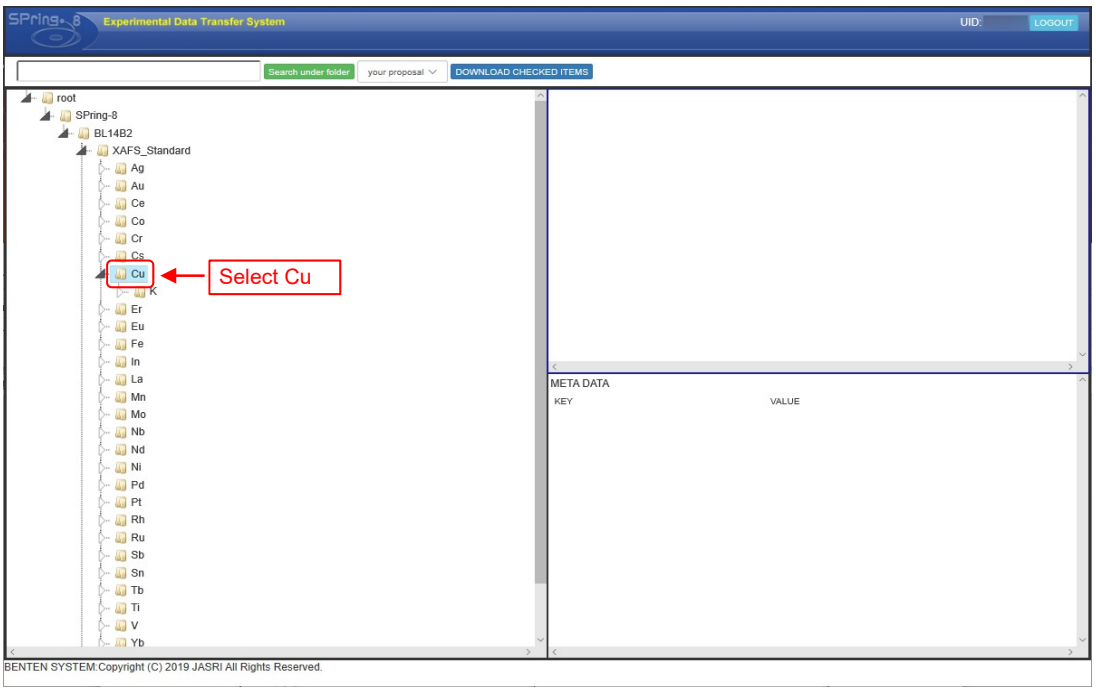
(b) Selecting SPring-8 folder



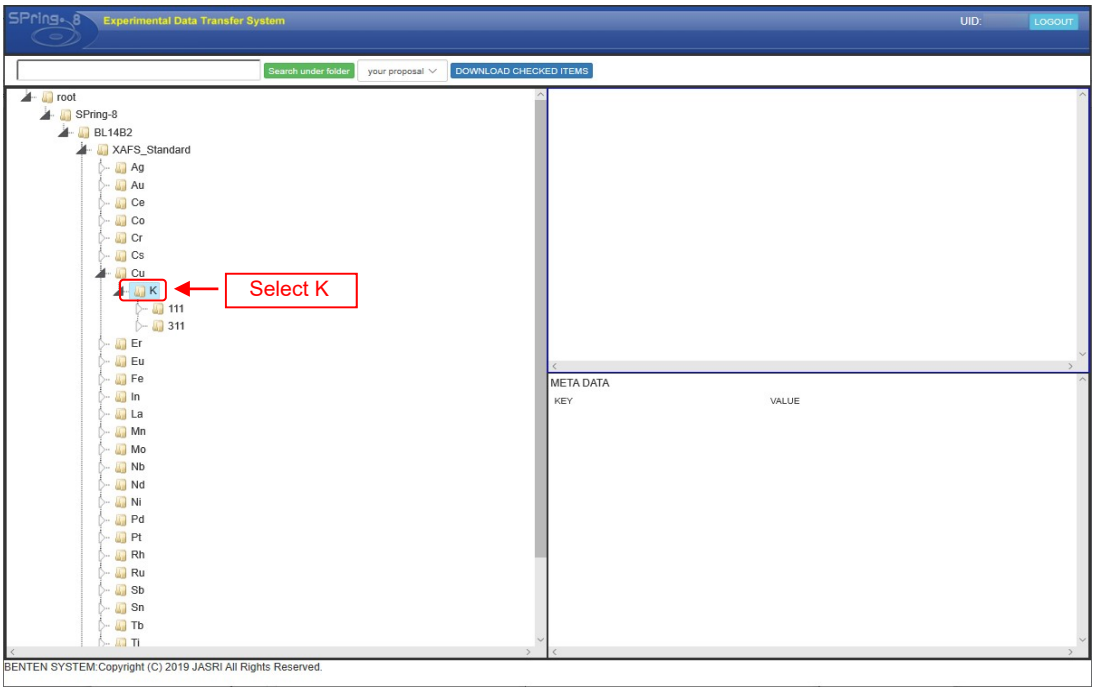
(c) Selecting BL14B2folder



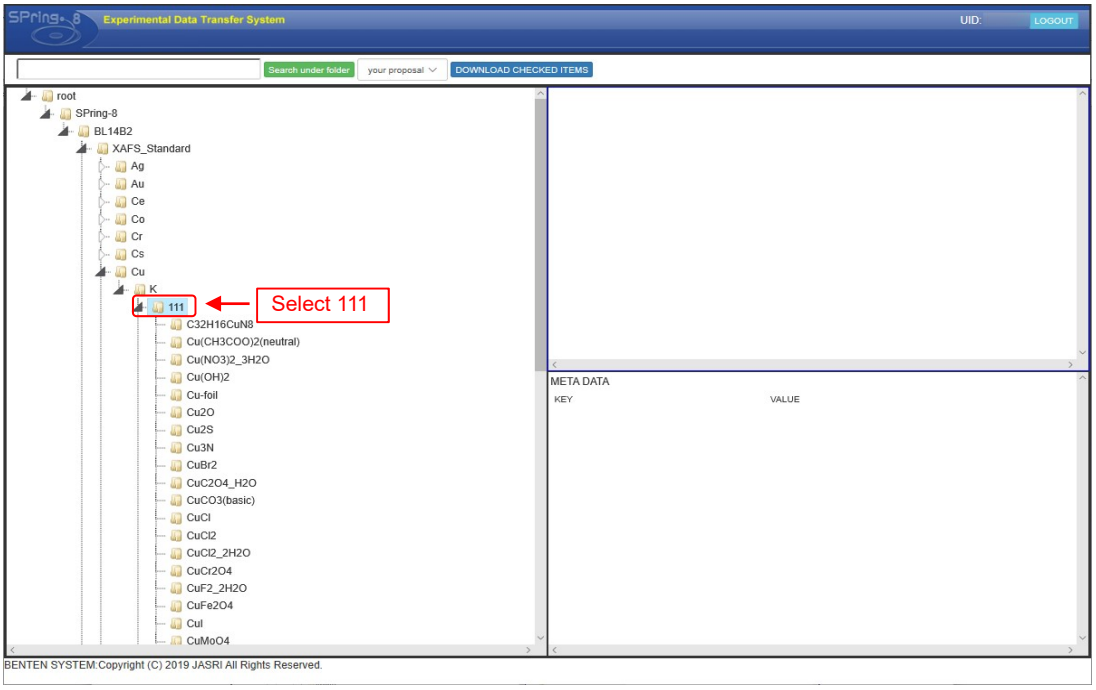
(d) Selecting XAFS_Standard folder



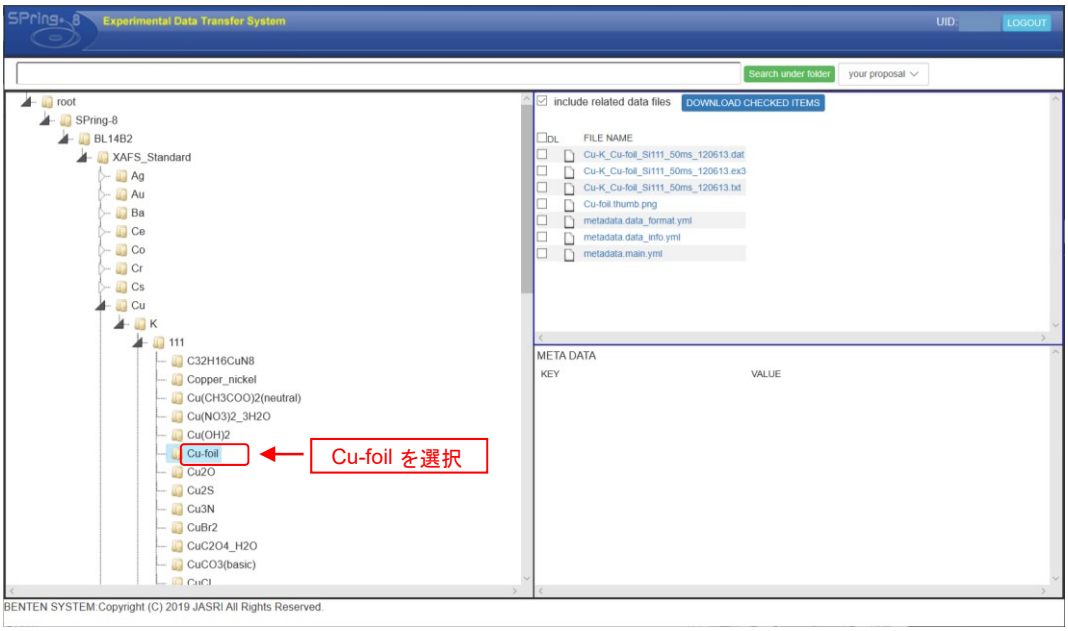
(e) Selecting element folder (ex. Cu folder)



(f) Selecting absorption edge folder (ex. K)



(g) Selecting net plane folder (ex. 111)



(h) Selecting sample name folder (ex. Cu-foil)

2-2. Browsing experimental data information

(1) Select the folder of the sample you want to browse from the tree display and display the corresponding data screen (Fig. 10).

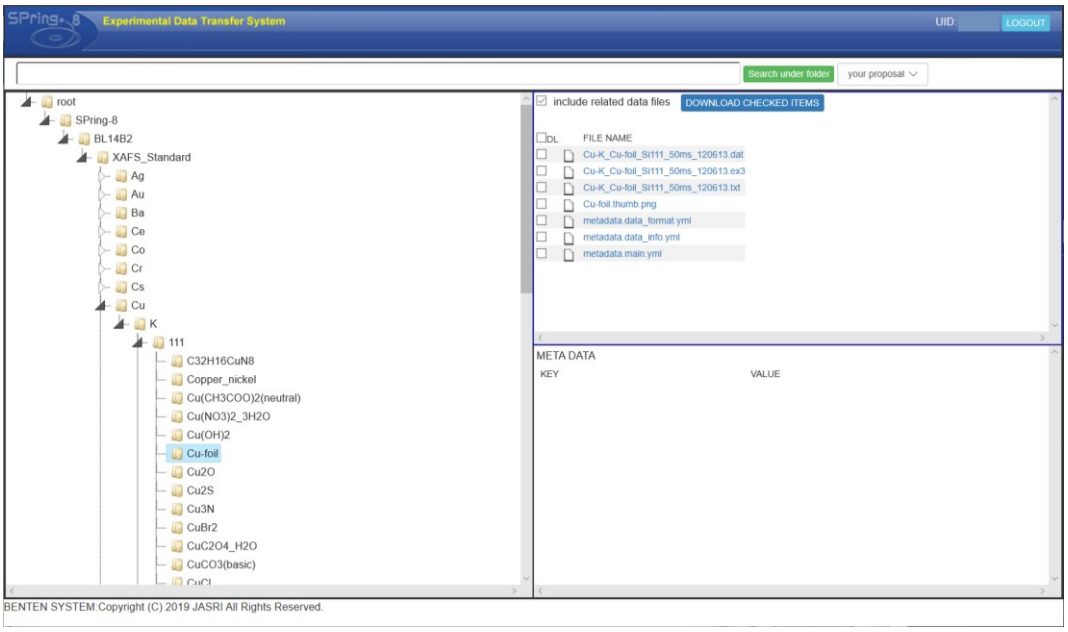
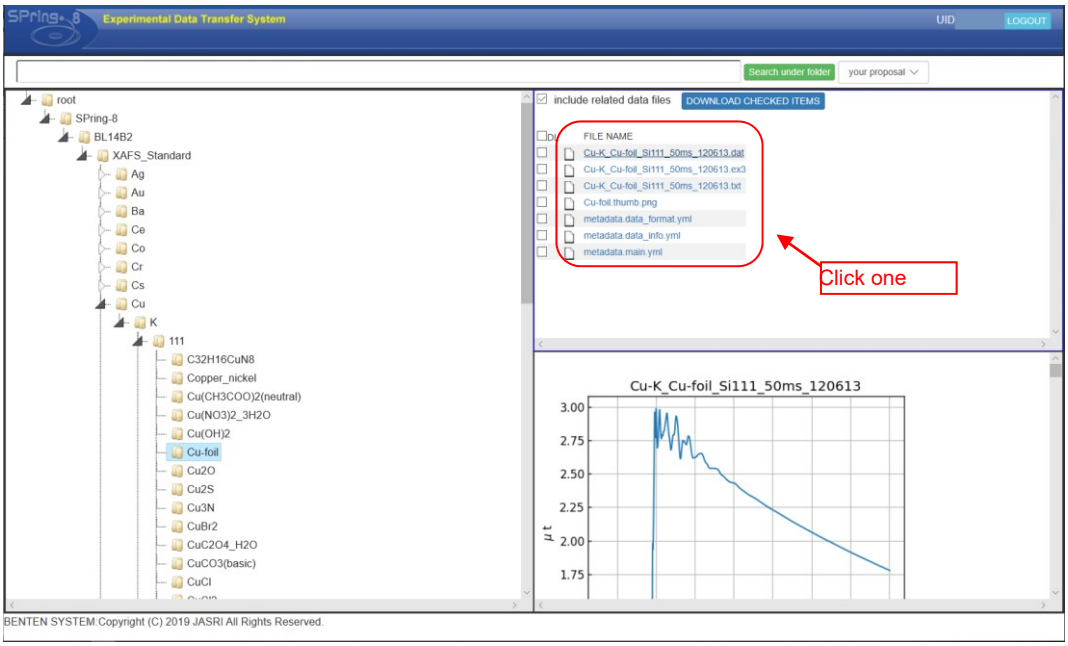
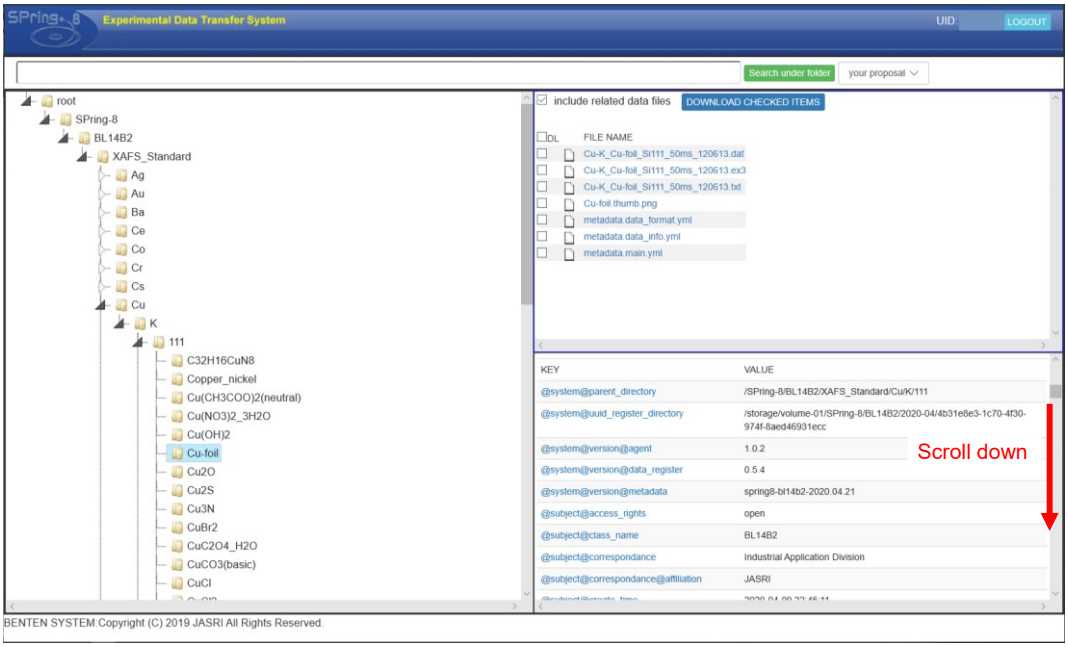


Fig.10 Display of applicable data screen (selecting Cu-foil folder)

(2) Click any of the data in the corresponding data screen on the upper right. Click to display experimental data information (Fig. 11 (a) thumbnail image, (b) simple information).



(a)



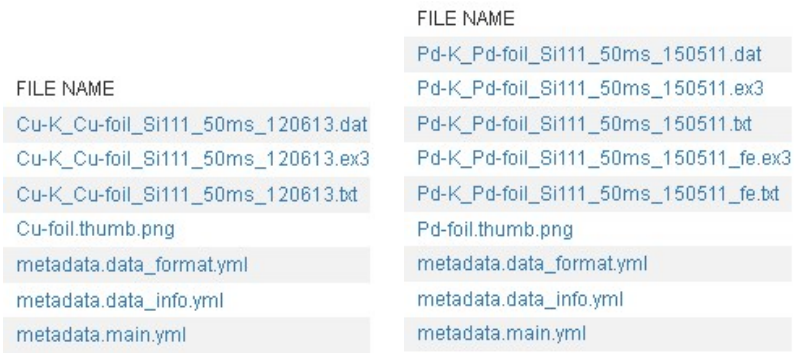
(b)

Fig.11 Experiment data information screen

(a) Thumbnail image (b) Simple information(Scroll down the lower right screen)

2-3. Types of experimental data

(1) In the BL14B2 XAFS standard sample database, 3 types of experimental data (extensions dat, ex3, txt ((5 types if fitted encoder processing is available)) for one registration number, and detailed information of the experimental data. (metadata.main.yml, metadata.data_format.yml, metadata.data_info.yml) is registered.



FILE NAME
Cu-K_Cu-foil_Si111_50ms_120613.dat
Cu-K_Cu-foil_Si111_50ms_120613.ex3
Cu-K_Cu-foil_Si111_50ms_120613.txt
Cu-foil.thumb.png
metadata.data_format.yml
metadata.data_info.yml
metadata.main.yml

FILE NAME
Pd-K_Pd-foil_Si111_50ms_150511.dat
Pd-K_Pd-foil_Si111_50ms_150511.ex3
Pd-K_Pd-foil_Si111_50ms_150511.txt
Pd-K_Pd-foil_Si111_50ms_150511_fe.ex3
Pd-K_Pd-foil_Si111_50ms_150511_fe.txt
Pd-foil.thumb.png
metadata.data_format.yml
metadata.data_info.yml
metadata.main.yml

(a) (b)

Figure 12 Database registration data ((a) Cu-foil, (b) Pd-foil)

The measurement information is described in the file name of the experimental data in the following order.

Element－Absorption edge－Sample name－Net plane－Dwell time－Measurement date－(fe).Extension

An example of the measurement information shown in the file name of each registered data in FIG. 12 is shown below.

(Example 1) Cu-K_Cu-foil_Si111_50ms_120613.dat

Element: Cu
Absorption edge: K-edge
Sample name: Cu-foil
Net plane: Si (111)
Dwell time: 50msec per point
Measurement date: June 13, 2012
Extension: dat (9809 format)

(Example 2) Pd-K_Pd-foil_Si111_50ms_150511_fe.txt Element:

Pd
Absorption edge: K-edge
Sample name: Pd-foil
Net plane: Si (111)
Dwell time: 50msec per point
Measurement date: May 11, 2015
fe: With fitted encoder processing (*)
Extension: Readable by XAFS analysis software Athena
((*) Refer to Appendix I about fitted encoder processing)

Table 1 shows the file formats of the experimental data extensions.

Table 1 Extension file format

Extension	File type
dat	Measurement data (9809 format)
ex3	Readable by XAFS analysis software REX2000
txt	Readable by XAFS analysis software Athena

The file with the extension yml contains information such as standard samples, measurement conditions, measurement data, and main items. These are YAML-formatted text files that can be viewed with a text editor or browser (Fig. 12). Currently, three types of metadata.main.yml, metadata.data_format.yml, and metadata.data_info.yml are registered. Table 2 shows the items in each yml file.

```yaml
sample:
- chemical_formula: PbO
  lot_number: '5018791'
  model_number: PBO11PB
  name: Lead(II) oxide
  supplier: KOJUNDO CHEMICAL LABORATORY
  tag: main
- chemical_formula: Pb-foil
  model_number: PB000100
  name: Lead
  supplier: Goodfellow
  tag: reference
measurement:
  method:
    category: spectroscopy
    sub_category: XAFS
    absorption_edge: Pb L3-edge
instrument:
  slit:
  - name: 4Dslit
    vertical_size[mm]: 1.0
    horizontal_size[mm]: 5.0
  xafs:
    I0:
      element_number: 1
      element:
      - type: Ion Chamber
        gas: N2 75% + Ar 25%
        HV[V]: 1000.0
        amp_gain[V/A]: 10000000.0
    I1:
      element_number: 1
      element:
      - type: Ion Chamber
        gas: Ar 100%
        HV[V]: 1000.0
        amp_gain[V/A]: 10000000.0
    I2:
      element_number: 1
      element:
      - type: Ion Chamber
        gas: Ar 100%
        HV[V]: 1000.0
        amp_gain[V/A]: 100000000.0
local:
  measurement_time[sec]: 235.0
  xafs_filename_list:
  - Pb-L3_PbO_Si111_50ms_210209.dat
```

Fig.12

Table 2 Items listed in the yml file (a) metadata.main.yml

sample (Category of sample)	
chemical_formula	Chemical formula of the sample
lot_number	Lot number of the sample
model_number	Model number of the sample
name	Substance name of the sample
supplier	Supplier name of sample
tag	The sample (main, measured by I0-I1 detector) or reference sample (reference, measured by I1-I2 detector)
measurement (Category of measurement parameters)	
category	Measurement method category
sub_category	Measurement method subcategory
absorption_edge	Measuring element and absorption edge
instrument (Category of measuring instrument)	
vertical_size [mm]	4D slit size ((W) width)
horizontal_size [mm]	4D slit size ((H) height)
type	Types of I0, I1 and I2 detectors
gas	Type of gas used for I0, I1 and I2 detectors
HV [V]	Applicable voltage value to I0, I1 and I2 detectors
amp_gain [V / A]	Gain value of I0, I1 and I2 detectors
local	
measurement_time [sec]	Time taken from the start to the end of measurement
xafs_filename_list	File name of experiment data (9809 format)

(b) metadata.data_format.yml

xafs_raw_data_format (Category of measurement data)	
extension	extension
name	item name
unit	unit
additional_metadata	Additional information for each item
xafs_processed_data_format (Category of converted data)	

extension	extension
name	item name
unit	unit
formula	Conversion formula from measurement data
additional_metadata	Additional information for each item

(c) metadata.data_info.yml

proposal_number	proposal number
access_rights	Access rights
data_depositor (Category of data writer)	
name	Name of the data writer
organization	Data writer's organization
contact_name (Category of data responsible)	
name	Name of the data manager
organization	Data responsible organization
role	The role of the data manager

4. Download experimental data

4-1 How to download data

(1) Check the experimental data of the sample you want to download (Fig. 14).

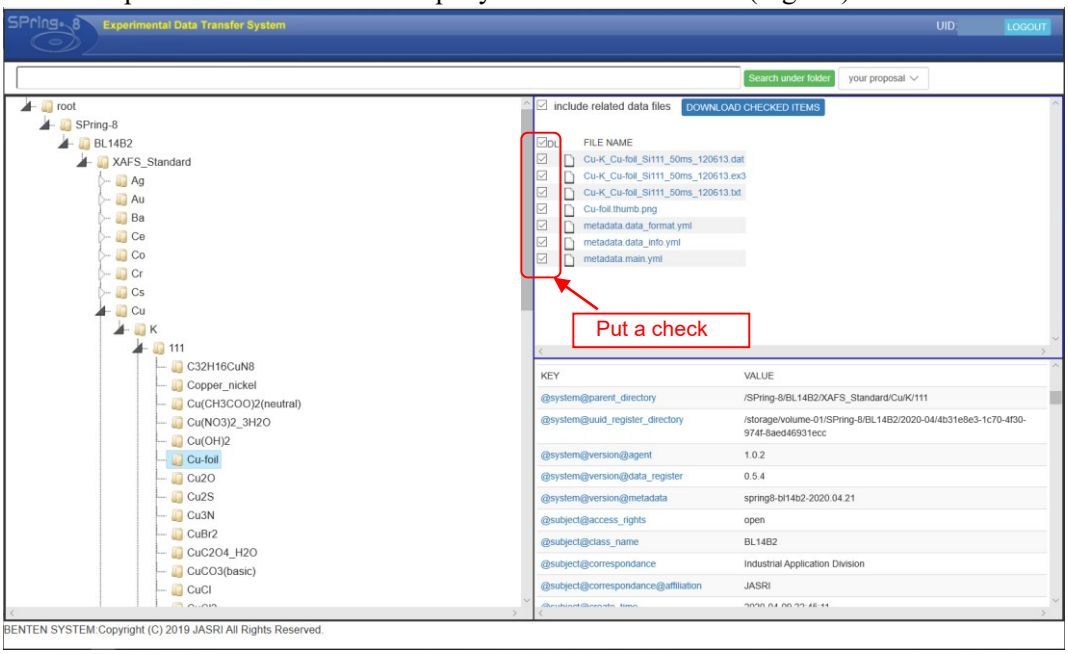


Fig. 14 Experimental data information screen (Cu-foil)

(2) Click the "DOWNLOAD CHECKED ITEMS" button on the screen. A zip file save message will be displayed, so save it in any folder.

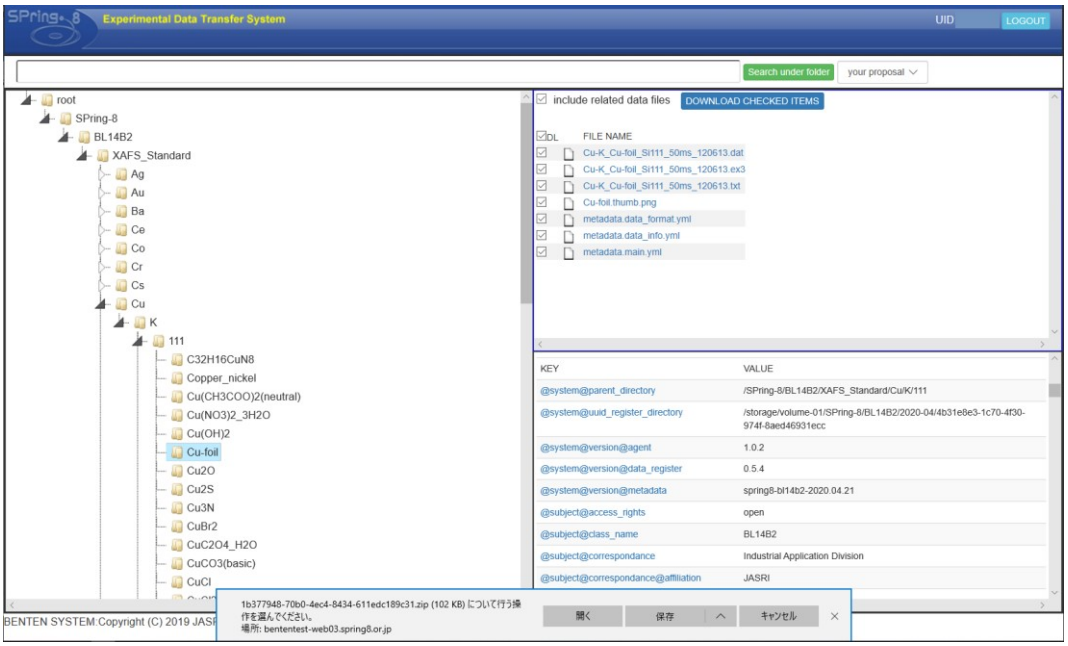


Fig. 15 Experiment data save screen (for Microsoft Edge)

4-3 How to download multiple data

(1) If you want to download multiple data at once, check the check box of the data you want to download and click the "DOWNLOAD CHECKED ITEMS" button (Fig. 16 (a)-(c)).

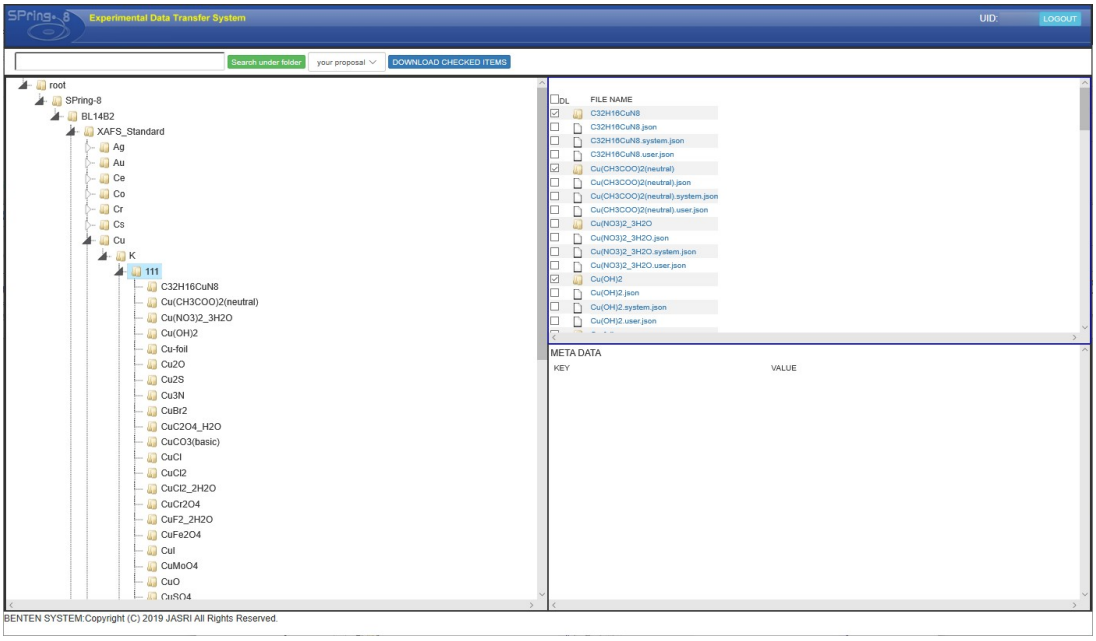
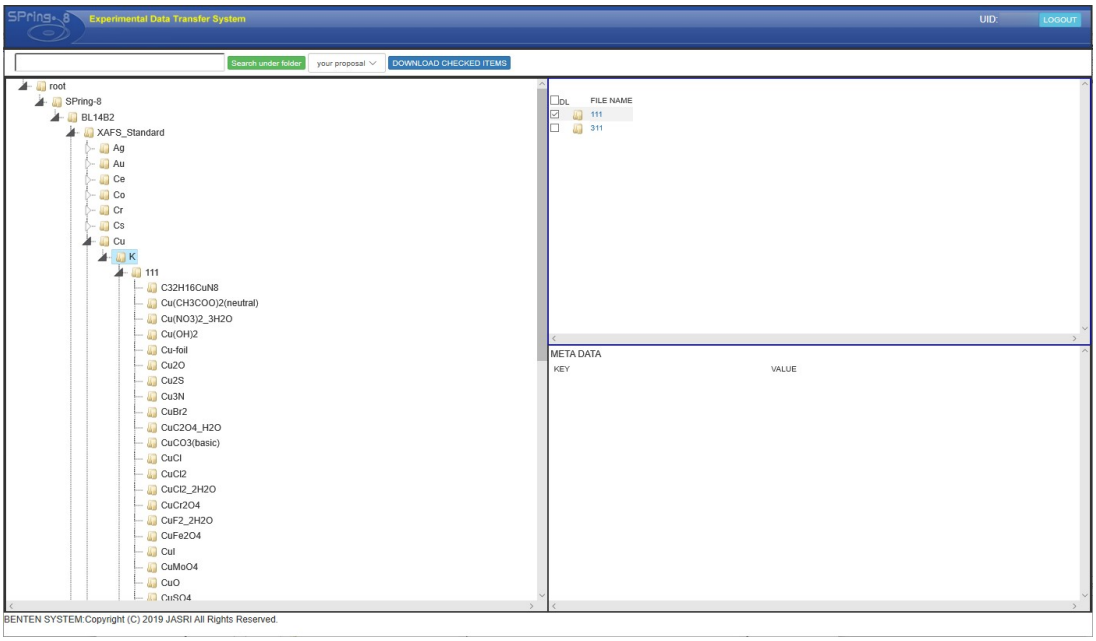
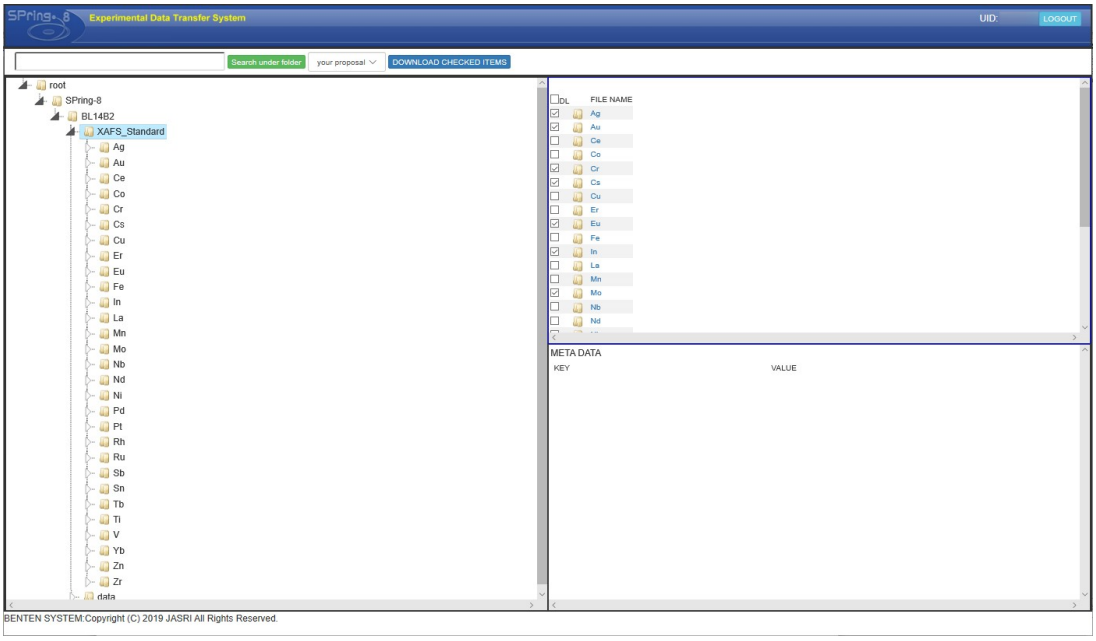


Fig. 16 (a) Downloading for each sample folder



(b)Downloading for each net plane folder



(d) Downloading for each element folder

Appendix

I. Fitted encoder processing for Angle selection

In the beamline BL14B2, short-period vibrational components different from EXAFS vibrations sometimes overlap on the high-energy side of XAFS spectra measured in the high-energy region. As example, the normalized EXAFS of $Pd(NH_3)_4Cl_2 \cdot H_2O$ measured at the Pd-K edge and Si(111) net plane is shown in Fig.17. As the wavenumber increases, the short-period vibrational component becomes more pronounced. This short-period vibrational component is not due to the sample, but to the encoder of the crystal monochromator.

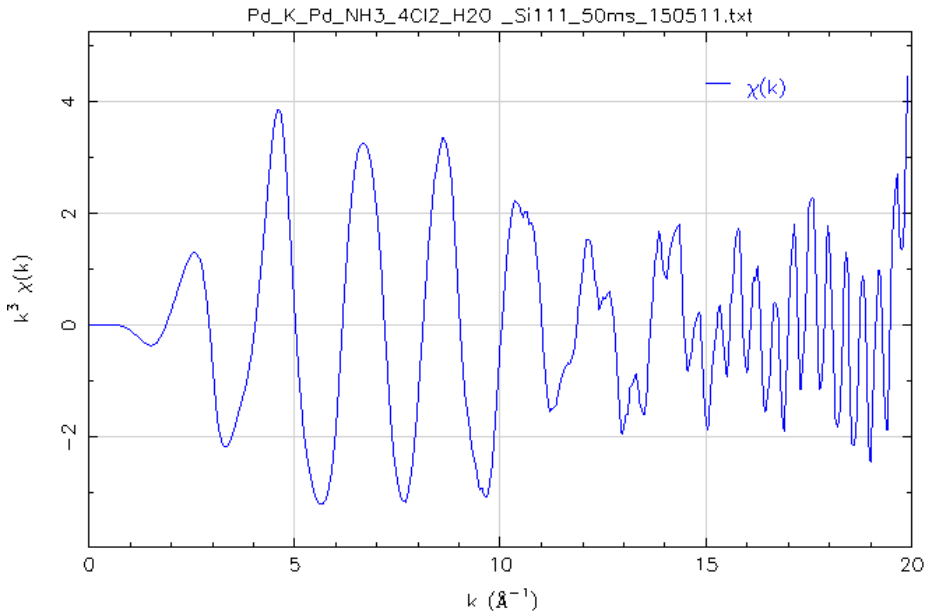


Fig.17 Normalized EXAFS of $Pd(NH_3)4Cl_2 \cdot H_2O$ (Pd-K, Si(111) net plane)
(Angle(o) is specified for Angle selection when converting data with XAFS_DataConverter.)

Figure 18 shows the angles of the crystal monochromator (PC and encoder values) when the XAFS spectrum in Fig. 17 was measured, and it can be seen that there is a periodic shift in the encoder values with respect to the PC angle. Since the sample-derived vibrational component is larger at the low wavenumber side of the normalized EXAFS, the effect of this periodic vibration is negligible. However, at the high wavenumber side, where the amplitude of the vibration component becomes smaller, the effect cannot be ignored, and a short periodic vibration component appears as shown in Fig.17. This periodic vibration of encoder values is not a problem specific to the beamline BL14B2, but is also seen in other beamlines. (This has been confirmed at the beamline BL19B2 and BL46XU. However, the degree of periodic deviation and the energy region where the deviation occurs vary from beamline to beamline. In most cases, the periodicity is much different compared to the EXAFS oscillation component, so the measured data are not affected).

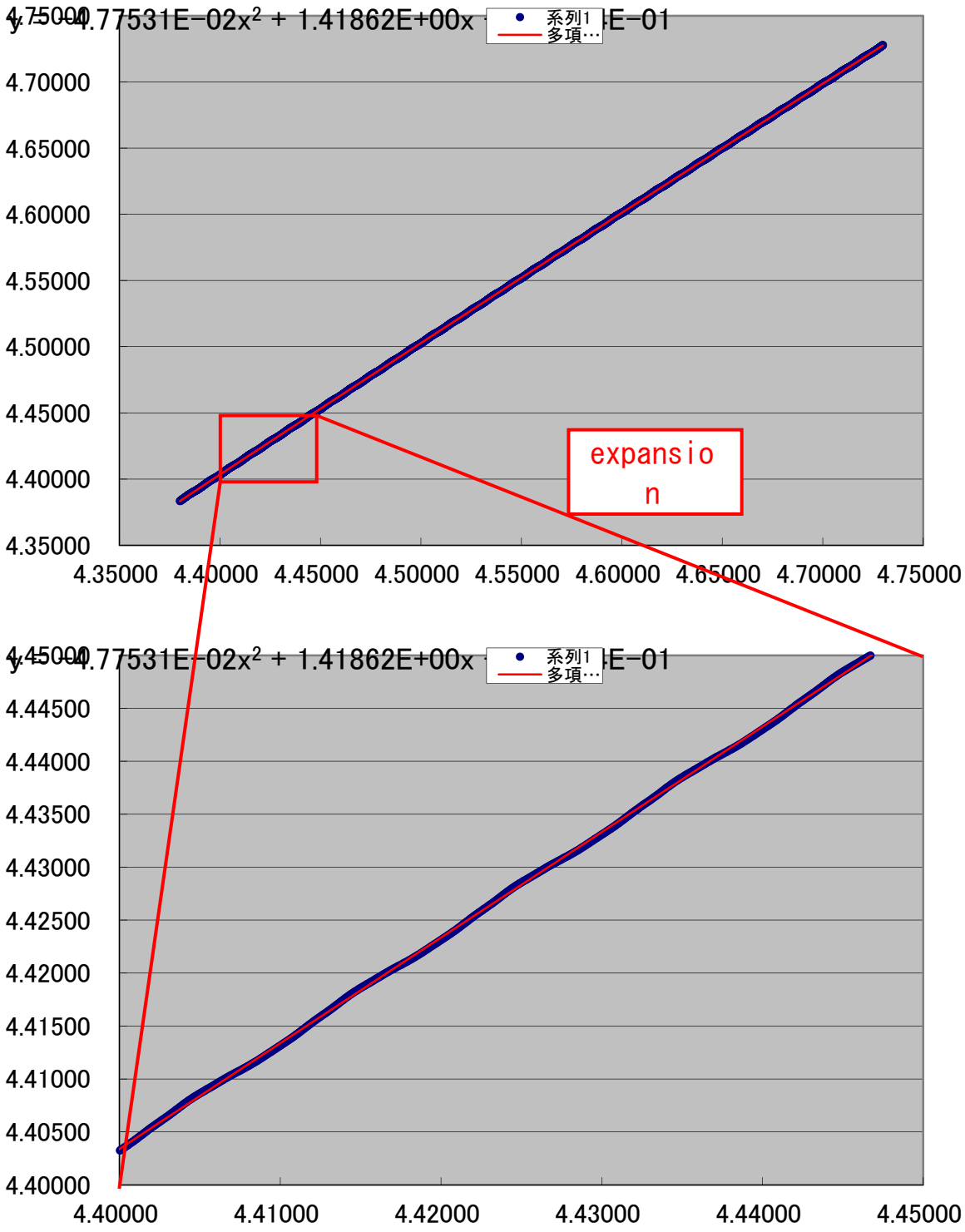


Fig.18 Angles of Spectroscopic Crystals

(From the file created by "Output Excel file (for angle correction confirmation)" in XAFS_DataConverter)

The XAFS data conversion program corrects for this periodic shift using a polynomial approximation (red line in Fig. 18) by processing the fitted encoder. Figure 19 shows a comparison of the normalized EXAFS spectral shape with and without the fitted encoder process, and it can be seen that the fitted encoder process eliminates the short-period vibration component on the high wavenumber side.

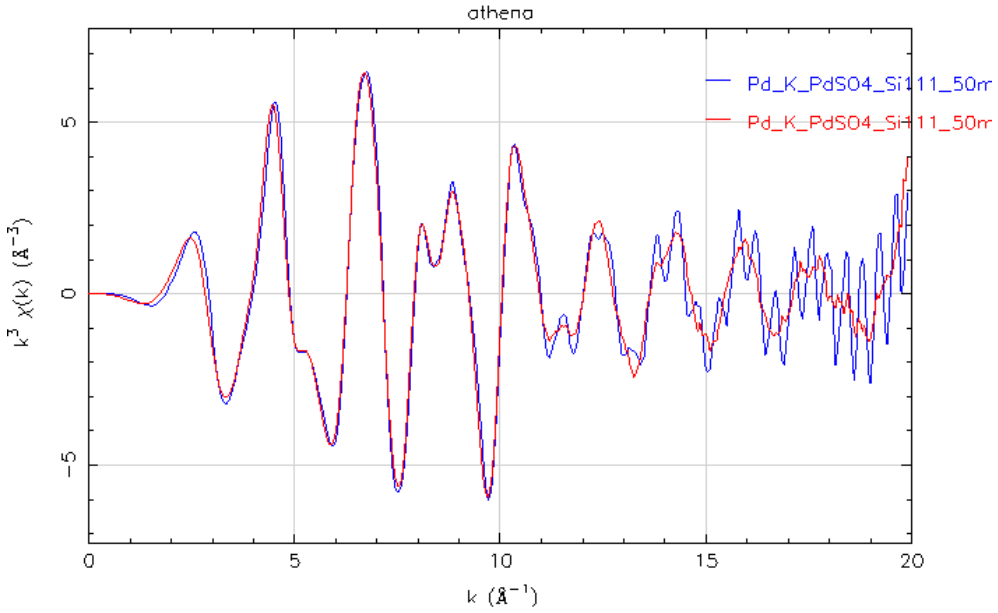


Fig.19 Normalized EXAFS of $Pd(NH_3)_4Cl_2 \cdot H_2O$ (Pd-K edge, Si(111) net plane)
(Blue line: without fitted encoder processing, red line: with fitted encoder processing)

When fitted encoder processing is performed, the XANES spectrum is modulated from the original data as shown in Fig.20. Therefore, when discussing XANES spectra, care should be taken not to mix fitted encoder processed data with non-fitted encoder processed data.

In addition, not all encoder-derived vibration components appear on the high wavenumber side of all EXAFS spectra in the high-energy region. Figure 21 shows the normalized EXAFS of $Pd(NH_3)_4Cl_2 \cdot H_2O$ measured on the Si(311) net plane, and unlike the measurement on the Si(111) net plane, no short-period vibration component is observed on the high wavenumber side, and the spectrum shape is almost unchanged after fitted encoder processing. Therefore, it is possible to determine whether the short-period vibration on the high wavenumber side originates from the encoder or not by comparing the spectrum without fitted encoder processing (Angle(o)) and with fitted encoder processing (Angle(fitted encoder)).

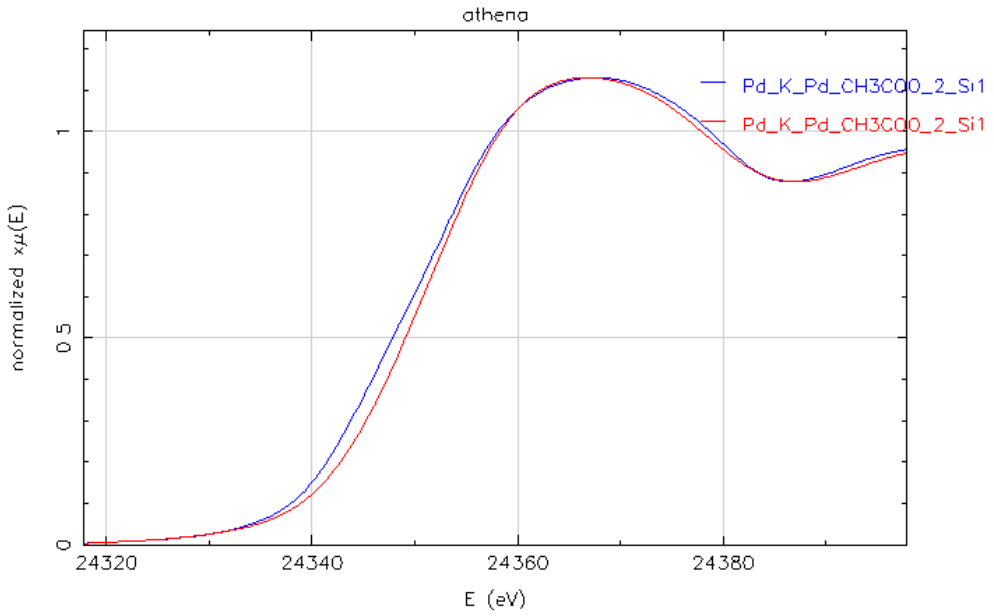


Fig.20 XANES spectra of $Pd(NH_3)_4Cl_2 \cdot H_2O$ (Pd-K edge, Si(111) net plane)
(Blue line: without fitted encoder processing, red line: with fitted encoder processing)

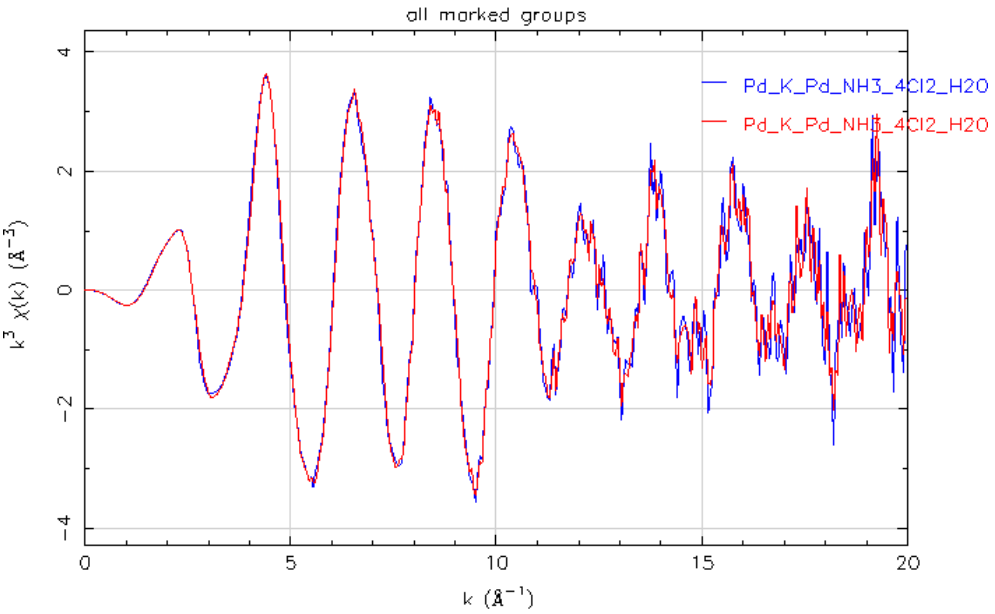


Fig.21 XANES spectra of $Pd(NH_3)_4Cl_2 \cdot H_2O$ (Pd-K edge, Si(311) net plane)
(Blue line: without fitted encoder processing, red line: with fitted encoder processing)

Revision history

Revision date	Reviser
3.31.2022	Hironori Ofuchi
11.24.2022	Hironori Ofuchi