IMSERC User Manual for XRF-WDS (Rigaku ZSX Primus IV)

CONTENTS

INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This short set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments, contact a staff member. Please read this user manual and acquaint yourself with the instrument. If during the course of using the system, something happens that you do not understand, please ***stop*** and ***get help***. In any event, be completely prepared to justify your actions. The cost of even minor repairs is considerable.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment
- X-Ray Safety

You need the above certificates in order to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in the hallway near BG75. Protective eyewear is not required in this room, and gloves should be removed when using the computer.

DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied on your group folder on 'imsercdata.northwestern.edu' under 'others/TGA' every few seconds. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

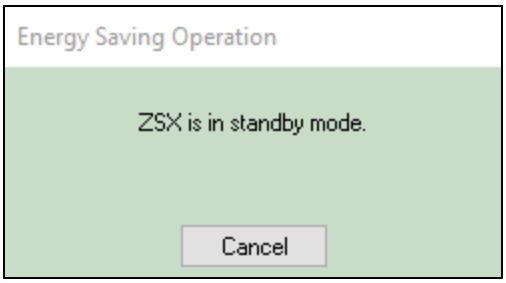
SOFTWARE

Data acquisition, reduction, and analysis can be performed with the "ZSC Guidance" software. Software is installed on the instrument computer. Offline analysis is not available. Please see a staff if you'd like to analyze your data, or utilize the software during your reservation for analysis of your data.

DEFAULT INSTRUMENT STATUS

The default working condition of PCM-XRF-WDS is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUcore in order to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore.
2. The default 'Rigaku' user account should be logged in. In case the computer was restarted, the password for the 'Rigaku' account is (see hardcopy by the instrument)
3. Acquisition software (ZSC Guidance) should be running. Leave the acquisition software open when you are done with the measurement.
4. There should be no error messages on either the front panel of the instrument or the acquisition software. Please check the 'Troubleshooting' session for a potential solution before reporting the error.
5. The X-Ray source may be set to standby mode (after 120 minutes of being idle). If the message "ZSX is in standby mode" is present under the "Energy Saving Operation" tag, simply press the "Cancel" button, and then "Yes" to put the X-Ray source back into normal operating mode.



6. Once the X-Ray source is back to normal operating voltage/amperage, the instrument is ready for data collection

When you are done with your measurement, please remember to:

1. Empty the sample holders that were utilized
2. End your reservation in NUcore
3. Leave lab tables clean and tools/accessories organized

If there is an error or problem with the instrument which is not addressed under the 'Troubleshooting' section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html add place the 'Stop' sign near the instrument computer. 'Stop' signs are located on the shelf above the computers in BG51 and online at the link above
3. Email or talk to a staff member

GENERAL STEPS FOR SAMPLE PREPARATION

1) For Solid Samples, prepare the sample holder(s) for your measurement by gathering the needed materials:
   a) A plastic sample holder and matching ring
   b) Two pieces of pre-cut mylar
   c) An autosampler holder

1c

2) Put your sample(s) into the cup(s) as shown below by placing a layer of the pre-cut mylar on top of the lower cup (2a), depositing sample in the center(2b), placing another layer of pre-cut mylar, placing the lit on top of the second layer of mylar (2c), then finally pressing the lid to mate with the lower cup (2d). Sample can be in any form (fine powder, solid pellet, thin film, liquid, etc.). If you need to prepare standards for quantitative analysis (qualitative measurements do not require standards), make sure that the standards have the same form with your samples (X-ray absorption coefficient must be similar)

2a

2b

2c

2d

3) Next step is to load the sample cup into the autosampler holder

4) **Important** Pass the sample under the “For Tube Above” height check (to ensure that the threads are not cross threaded)

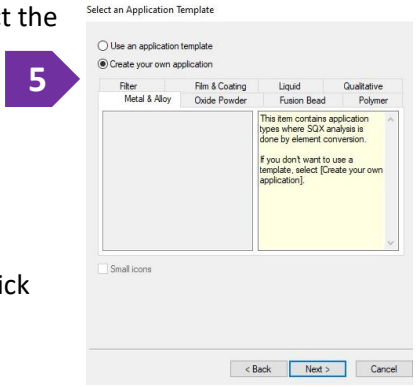
5) Once the sample is loaded into the autosampler holder, it is now time to place the autosampler holder onto the autosampler tray.

USING THE SOFTWARE FOR A QUALITATIVE MEASUREMENT

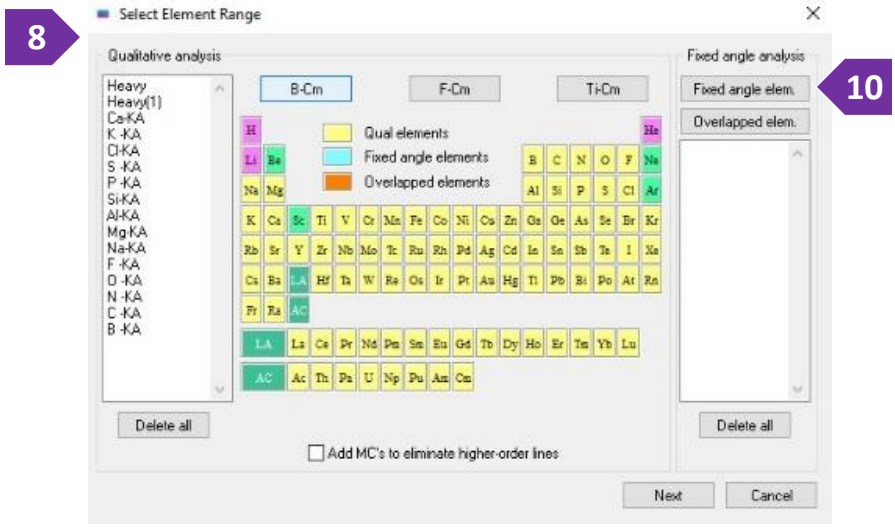
1. Go to the top taskbar of ZSX Guidance, and click on the “Qual 

Application” button.

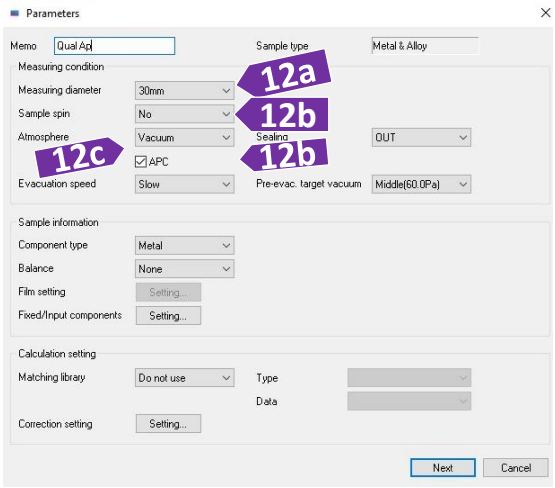
2. The “Qual Flow Bar” will appear on the left had side of the screen. This will act as the flow chart to create a qualitative application.
3. Click on “Application File” to start making a new application
4. Select “Create a new application” unless you would like to complete one of the other actions listed.
5. Here we are going to first select “Create your own application”, then select the most appropriate form your sample is (does not have to be exact)
6. Input the name you would like for the application, and provide a description (optional)
7. In the “Folder” field, go to your personal folder under the Methods/YourPI/Yourfolder to save your application in your folder, and click on the “next” button.



8. Now a “Select Element Range” window will appear.

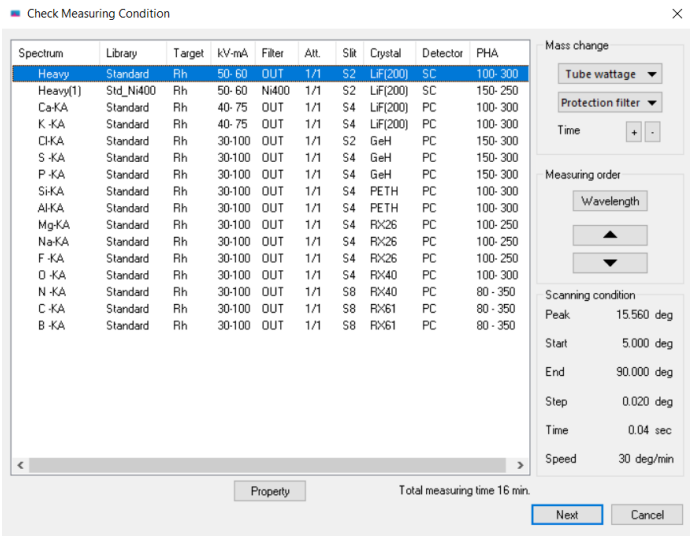


9. For a Qualitative application, If you click on the “B-Cm”, “F-Cm”, or the “Ti-Cm” that range of elements will be selected. Feel free to delete undesired elements. Also, you can left click the specific elements of interest. Note: the Heavy scan will be collecting everything from Ti to Cm, and the Heavy(1) scan will be collecting for Rh in your sample (subtracting out the Rh peaks from the source via a filter)
10. (Optional) you may select the “Fixed Angle Elem.” Option. This will do an additional scan (sperate from the heavy scan) in the 2theta region for that specific element.
11. Next the “Sample Preparation” tab will appear, press “next”
12. The “Parameters” Tab allows you to select specific parameters for your application. Key points to decided on: 12(a) select the size of the sample holder that you plan to use, 12(b) enable or disable the sample spinning, 12(c) Select the atmosphere for the sample to be in during the collection (Vacuum for **only** solid samples, Helium for solid or liquid samples), 12(d) APC (Automatic Pressure Controls) enabling this feature will place the sample into the antechamber and reach a desired vacuum before introduction to the turret assembly.

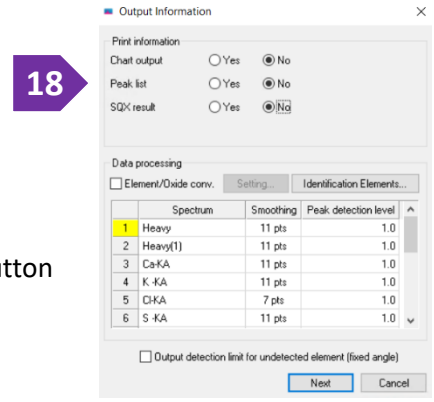


13. Press on the “Next” button

14. The “Check Measuring Condition” window will appear. This stated the specific Spectrum that that you requested to collect, as well as the KV and ma settings default for the KA line of that element. It also contains if a filter is going to be used, if the attenuator is being used (only available on the 30mm sample holders), which crystal will be utilized, as well as which detector/PHA setting. If you would like to change from the default settings, just double click on the spectrum of interest, and changes can be made. Note: This window will also show the amount of time per measurement.



15. Press on the “Next” button
16. The “Output Information” window will appear
17. Select “no” for all of the Print information fields
18. Press on the “Next” button
19. Now this application is saved in the desired folder, press on the “exit” button to close the Qual Flow Bar



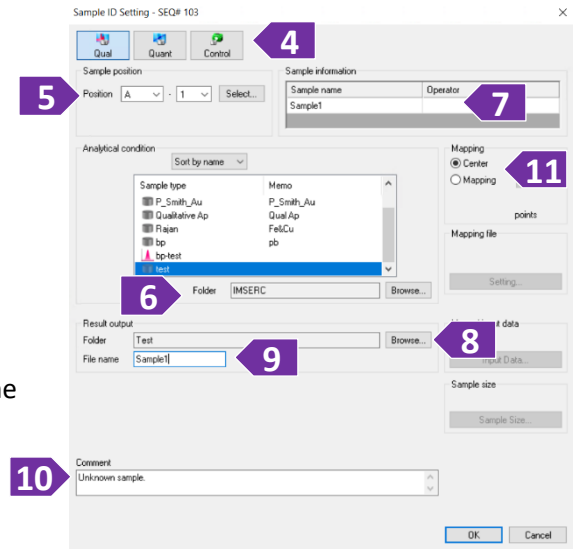
USING THE SOFTWARE FOR A QUANTITATIVE APPLICATION

STARTING A DATA COLLECTION (QUAL, QUANT, OR MAPPING)

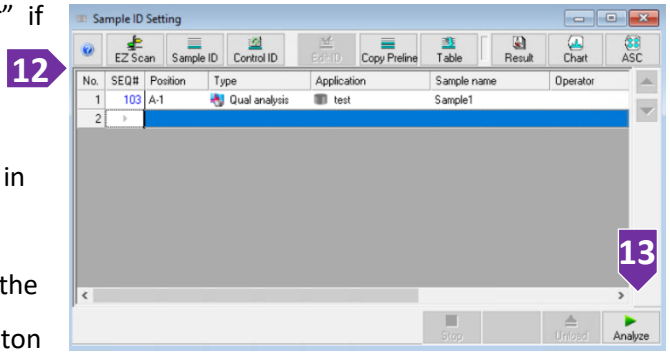
Once an application is created, that application needs to be selected and queued into the auto sampler.

1. Press on the “Analysis” 

button to open the appropriate windows
2. A series of windows will open, focus on the “Sample ID Setting” window, double click on the blue rectangle
3. A new window named “Sample ID Setting – SEQ# XXX” will open.



4. Select the type of measurement you would like to collect (Qual, or Quant)
5. Select the Sample Position you placed your sample
6. Click on the “Browse” button, and select your folder, then select the application you previously created
7. Provide a Sample name
8. Click on the “browse” button, and select your folder for the result output.
9. Provide a file name.
10. (Optional) Provide a comment for this data collection (the Sample name and File name fields have a character limit, so feel free to add additional information in the comment field.
11. (Optional) If doing a normal collection, select “center” if doing a Mapping collection, Select “Mapping”
12. Once selecting “OK” the follow “Sample ID Setting” window will appear, with the data collection defined in steps previous populated in the No.1 position.
13. Once all of the data collections desired are added to the “Sample ID Setting” window, press the “Analyze” button to start the data collection

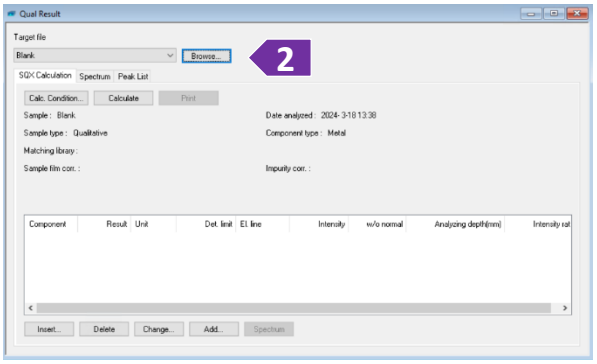


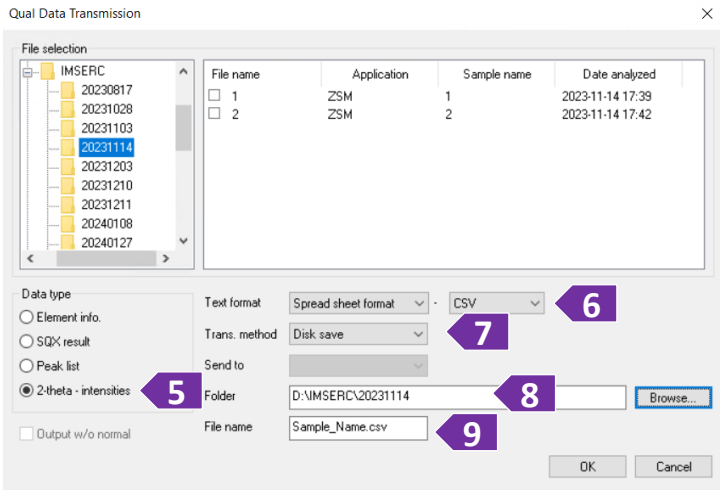
EXPORTING/VIEWING DATA

To View/Export your collected spectra to CSV, please follow these steps:

1. Click on the “Data Processing” tab 

and select the “Qual Result” option.
2. Click on the “Browse” button, and select your folder, then the specific data file.
3. Here you can see three (3) different ways of viewing the data collected. The “SQX Calculation” will give a semi-quantitative result based off of the elements the software automatically identified in combination with internal standards. The “Spectrum” Tab will allow you to plot the qualitative measurements in 2theta versus intensity. Finally, the “Peak List” will give you a list of the peaks the software found.
4. To Export your Qualitative data: While the Qual result window is open, press on File->Transfer Qual Data… then the “Qual Data Transmission” window will open





5. Here you will select “2-theta-intensities” Data type (or which every data type you desire)
6. Select “text Format” as “Spreadsheet format – CSV”
7. Select “Disk Save”
8. Click on Browse and select the appropriate folder to save the data
9. Provide a file name for your data

PUBLICATION

A. EXPERIMENTAL SECTION

Modify the text below according to the setup and conditions you used during the measurement:

"X-ray fluorescence spectroscopy was performed in a Rigaku ZSX Primus IV spectrometer equipped with an Rh X-ray tube and a XXXX detector. Sample was placed in a special plastic cup and powder was supported on a 3 µm Mylar film. Data collection was performed at room temperature ***under vacuum | air | Helium*** with a ***X-filter***. X-ray tube was operated at ***XX*** keV and ***YY*** µA, and fluorescence spectra were collected for ***ZZ*** s."

B. ACKNOWLEDGEMENT

TROUBLESHOOTING

A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

B. THERE IS AN ERROR/PROBLEM WITH THE INSTRUMENT THAT IS NOT ADDRESSED UNDER THE TROUBLESHOOTING SECTION

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html add place the 'Stop' sign near the instrument computer. 'Stop' signs are located on the shelf above the computers in BG51 and online at the link above. Email or talk to a staff member
3. Email or talk to a staff member

APPENDICES

REVISIONS

v1.00 2024/03/31	• Release of original version of the user manual