Non-destructive Chemical Analysis - Selected Chapters

Ivan Gržetić, Konstantin Ilijević, Jovana Orlić
Guide for WDXRF quantitative standardless analysis
Within this lecture, you will:

- Learn about principles of WD-XRF as analytical technique
- Get familiar with the basic steps performed in WD-XRF analysis
- Perform WD-XRF measurements of previously prepared samples with the help of Oxas software
- Acquire concentrations by using standardless UniQuant software
Background:

If atoms with 2 or more electron levels are irradiated with X-rays of sufficient energy, electrons in the inner levels will be ejected from the atom.

Electrons from the higher levels descend to the lower levels, emitting in the process X-rays with lesser energy (X-ray fluorescence).

Emitted radiation is characteristic for each element and dependable on number of atoms in the analyzed sample.
WD-XRF Principles:

- Continuum of X-rays is produced in X-ray tube

- Sample is exposed to the X-rays which leads to X-ray fluorescence

- A wavelength dispersive detection system physically separates emitted X-Rays according to their wavelengths.

- The x-rays are directed to a crystal, which diffracts (according to Bragg’s Law) the X-Rays in different directions according to their wavelengths (energies).

- Collimator system leads X-rays to the one of two available detectors
UniQuant is software which enables **quantitative** elemental analysis of samples without use of standards. UniQuant is software for use with a **ARL™ PERFORM'X Sequential X-Ray Fluorescence Spectrometer**.

Using Oxsas software we give the task to instrument in order to perform analysis, while UniQuant is latter implemented whithin Oxsas software.

More informations about UniQuant software can be found on: [http://www.uniquant.com/](http://www.uniquant.com/)
Start up UniQuant software

To start up UniQuant software it is necessary to find OXSAS icon first:
Before performing analysis, it is necessary to define few parameters. Click on \textit{Quantitative Analysis} option.
When Quantitative Analysis window is open it is necessary to open tab for new sample by click on New Sample button.
When tab for new sample is open, it is necessary to define some parameters.

1. Task should be defined. When standardless analysis is planned, option UniQuant should be chosen from drop-down menu.

2. When task is defined, from drop-down menu select the desired method.

Enter **Sample Name** and **Sample N°** (serial number of analysis). If repeated measurements are required in section **Runs Requested** enter desired number of repetitions. Also, **Sample Position** in instrument should be defined.
When all the parameters are set and sample is on defined position in instrument, start analysis by clicking on the **SID OK + Start** button.
On this window it is possible to monitor the progress of the analysis.
When the analysis is completed click on **UniQuant** button.
When UniQuant window is open, select the analysis from the list with double-click.
This is how the UniQuant analysis window looks like.
### General Data

<table>
<thead>
<tr>
<th><strong>Sample Identification</strong></th>
<th>Soil - 3288</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Creation Date</strong></td>
<td>24.9.2018 13:20:55</td>
</tr>
<tr>
<td><strong>Remark</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Chemistry</strong></td>
<td>Element</td>
</tr>
<tr>
<td><strong>Shape &amp; Impurities</strong></td>
<td>Teflon</td>
</tr>
<tr>
<td><strong>Case Number</strong></td>
<td>0 = All Known</td>
</tr>
<tr>
<td><strong>Kappa List</strong></td>
<td>AnySample</td>
</tr>
<tr>
<td><strong>Method</strong></td>
<td>Test jul 2014</td>
</tr>
<tr>
<td><strong>Environment</strong></td>
<td>Vacuum</td>
</tr>
<tr>
<td><strong>Film</strong></td>
<td>None</td>
</tr>
<tr>
<td><strong>Sector [degr]</strong></td>
<td>360</td>
</tr>
<tr>
<td><strong>Viewed Area [mm^2]</strong></td>
<td>490.63</td>
</tr>
<tr>
<td><strong>Viewed Diameter [mm]</strong></td>
<td>25.00</td>
</tr>
<tr>
<td><strong>Sample Diameter [mm]</strong></td>
<td>40.00</td>
</tr>
<tr>
<td><strong>Viewed Mass [mg]</strong></td>
<td>8593.750</td>
</tr>
<tr>
<td><strong>Sample Mass [mg]</strong></td>
<td>22000.000</td>
</tr>
<tr>
<td><strong>Rho [g/cm^3 = mg/mm^3]</strong></td>
<td>4.38</td>
</tr>
<tr>
<td><strong>Sample Height [mm]</strong></td>
<td>4.00</td>
</tr>
<tr>
<td><strong>Shadow int. Loss [%]</strong></td>
<td>0</td>
</tr>
<tr>
<td><strong>Known Concentration [%]</strong></td>
<td></td>
</tr>
</tbody>
</table>

**Sample identification** is automatically entered and contain Sample Name and Sample N°.

**Creation date** is information about date and time when analysis is performed.

**Remark** is the field in which you can enter the necessary comments.

With **Chemistry** option you define in which chemical form elements in the sample are present (element for alloys, oxides for samples were you expect oxide form of elements, etc.).
For **Viewed Diameter** it is necessary to enter 24.6 mm. This value is always required when the pellet of 32 mm diameter is analyzed.

For **Sample Diameter** it is necessary to enter diameter of analyzed sample (usually 32 mm).

In section **Sample Mass** enter the mass of sample in mg.

Sections **Viewed Area** and **Viewed Mass** are automatically recalculated based on the entered parameters.
### For Sample Height

It is necessary to enter the height of analyzed sample in mm.

### Sections Rho

Rho is sample density and it is automatically recalculated based on the entered parameters.

### For Sample Height

It is necessary to enter the height of analyzed sample in mm.

### Sections Known Concentration and Rest %

Sections Known Concentration and Rest % are optional and are used when we know the exact content of a some material in the sample. Example: The exact moisture content is known.
During the sample preparation procedure for the XRF analysis by pelleting, a particular amount of the binder is usually added to the sample. In section **Diluent/Sample** the ratio of the diluent mass and sample mass is entered.

The material of the added binder is selected in the **Material** section drop-down menu.
When all parameters are entered click on Calculate button. By selecting Normalized option, the sum of all concentrations will be 100% in total.
When the results are normalized, by clicking the **Report** button it is possible to export the results of the analysis in the form of a report.
In **Report** section drop-down menu select desired type of the report (do you want results in element or oxide form, etc.)
The Report View window will open. Click on icon.
In the **Export Report** window select a folder in which the report will be saved.
Enter the desired report name (**File name**). Select the format in which you want the report to be saved (**Save as type** drop-down menu).
By click on **Save** button save the report in the specified folder.
With **Save** button confirm that the export of the report has been successfully completed.
When report has been successfully saved, by click on the **Cancel** button return to the list of analyzed samples.
## DESCRIPTION OF REMOTE ACCESS

### 1. NETCHEM COMMUNICATION SIDES

**(NOTE: NETCHEM Communication is defined as event that involves all kinds of internet interactions (in real time and not in real time) between participants via devices (PCs, laptops, tablets and mobilephones))**

<table>
<thead>
<tr>
<th>Host side</th>
<th>Guest side</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>participant’s PC in classroom</strong></td>
<td><strong>participant’s PC in laboratory</strong></td>
</tr>
</tbody>
</table>

**(NOTE: Host side of NETCHEM Communication is defined as PC who invites other users to join the session)**

**(NOTE: Guest side of NETCHEM Communication is defined as PC who joins the invitation to session)**

### 2. COMMUNICATION SOFTWARE

| Team Viewer | Meeting: No  
Remote control: No  
Meeting and Remote control simultaneously: No |
|-------------|-------------|
| Skype       | Call 1:1: No  
Conference Call: Yes |

### 3. COMMUNICATION HARDWARE

<table>
<thead>
<tr>
<th>on host side</th>
<th>1 PC for each participant</th>
</tr>
</thead>
<tbody>
<tr>
<td>on guest side</td>
<td>1 PC, 1 headsets with microphone, camera</td>
</tr>
</tbody>
</table>

### 4. INFORMATION EXCHANGE TYPE

| Educational  
(one side is dominantly receptive) | Yes  
Place of Educator participant: guest side  
Number of educator(s): 1  
Place of student participant: **host side**  
Number of student participant(s): **15** |
|-----------------|-------------------|
| Consultative  
(two sides are equal in giving-receiving information) | Number of host side participant(s): **No**  
Number of guest side participant(s): **No** |
Remote Access Connection Instructions

What makes these labs different and unique from other classroom experiments is that we have incorporated a section in each activity to remotely characterize your samples from your classroom.

Request a remote lab session specifying information such as: the day, the time, and the instrument you are interested in using by visiting our web site:

http://netchem.ac.rs/remote-access

You will see the list of partners with the instruments provided to chose from.
You will be contacted by a Remote Access staff member to set up a test run to ensure you are set up properly and have the required infrastructure.
Send samples or verify the in-house sample you would like us to prepare and load for characterization.
Send your samples to the Remote Access center that you chose on your request.
There are two communications soft-ware packages, that will allow us to communicate instructions and answer questions during the session.

- TeamViewer: You can obtain a free download at:
- Skype
Remote Access Connection Instructions

You will need:

a) Computer with administrator access to install plug-ins and software
b) An internet connection
c) Speakers
d) Microphone
e) Projector connected to the same computer
f) Web browser (Firefox preferred)

During the test run you can refer to this guide to perform the following steps, but it’s very important that you only proceed with these steps during your scheduled times. You may interfere with other remote sessions and potentially damage equipment if you log in at other times.

a) Open and logon to your Zoom/Team-viewer account. You will be given the access code to enter at the time of your test and then again during the remote session.
   - If you are using the Zoom software, Remote Access staff will give you the access code.
   - If you are using the Team-viewer software, Remote Access staff will give you the ID & password.
b) You should soon see the Remote Access desktop and at this point you can interact with the icons on the screen as if it were your desktop.
c) Switch to full screen mode by selecting the maximize screen option in the top right corner of the screen.
d) Upon completion of the session, move your mouse to the top right corner of the screen, and click on the X to disconnect the remote session. It will ask if you want to end the remote session. Click Yes.
This remote access laboratory was created thanks to work done primarily at University of Belgrade.

Contributors to this material were: Ivan Gržetić, Konstantin Ilijević, Jovana Orlić

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Refereeing of this material was done by: _______________________

Editing into NETCHEM Format and onto NETCHEM platform was completed by: _______________________

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References and Supplemental Material

The NETCHEM platform was established at the University of Nis in 2016-2019 through the Erasmus Programme.

Please contact a NETCHEM representatives at your institution or visit our website for an expanded contact list.

The work included had been led by the NETCHEM staff at your institution.