

IMSERC User Manual for Karl-Fischer Coulometer

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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 set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at <http://imserc.northwestern.edu/pcm-instruments.html>. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and available under the corresponding instrument section at <http://imserc.northwestern.edu/pcm-instruments.html> by pressing on the 'User manual' button. If while 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 could be 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
- Compressed Gas Cylinders

You need the above certificates 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 outside of room B172 at the north side. Protective eyewear is required in this room, and gloves should be removed when using the computer.

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DATA MANAGEMENT

The default operation of our Karl-Fischer coulometer is performed directly from the touchscreen of the coulometer. Data (water content) is printed on the touchscreen, therefore no files are created and uploaded on our fileserver.

SOFTWARE

No additional software is needed for data analysis. Coulometer directly prints the water content and files are created when using the touchscreen interface.

DEFAULT INSTRUMENT STATUS

The default measurement mode of the Karl-Fischer coulometer is ***water quantification in liquid samples using the standard measurement cell***. Please notify the right staff member well in advance if you would like to run an experiment in a different mode than the one listed above, and you are not trained to perform the require mode-switch. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either [contact a staff member](#) or check the corresponding capabilities section at <http://imserc.northwestern.edu/pcm-instruments.html>.

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. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. [Contact a staff member](#) for instructions

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GENERAL STEPS FOR DATA ACQUISITION USING THE TOUCHSCREEN INTERFACE

The general steps for data acquisition described in this section illustrate the standard workflow of the coulometer. The various sections listed in this user manual provide more details about each specific mode and its settings. Please follow these general steps as discussed during your training session.

There are different ways of measuring the water content in your sample depending on its form and solubility:

- If sample is in liquid form, you could directly inject it into the measuring cell
- If sample is solid and soluble in methanol, you could drop your solid directly into the measuring cell
- If sample is solid, not soluble in methanol, and can release moisture when heated up to 300C, then connect and use the drying oven accessory

Regardless of the form and solubility of your sample, the general workflow for measuring the water content of your sample using the touch-screen interface is the same, and as follows:

1. Start your reservation through NUcore, otherwise the device will not turn on
2. Verify that instrument is idle by checking that the magnetic stirring bar is not spinning
3. If coulometer is completely off, press on the powder button (circled in figure 3) and wait for a few seconds until the system is online
4. Login with the 'Administrator' account by
 - a. Pressing on the 'User Name' field
 - b. Selecting 'Administrator' from the list of users
 - c. Pressing on the 'Login' button at the bottom right corner of the screen
5. On the home screen, press on the 'Methods' button at the top right corner of the screen
6. From the list of methods, select the one with ID 'M314' (should be on the top of the list) that has the title of 'Water standard 1.0 mg/g'
7. Press on the 'Start' button at the bottom right corner of the screen
8. Leave the settings as they are and press on the 'Start' button again
9. The 'Pretitration' procedure will start, and the magnetic bar will start spinning. Depending on how long the coulometer has been idle for, the pretitration might take from a few minutes up to an hour



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10. Once the drift ($\mu\text{g}/\text{min}$) is below the threshold ($25 \mu\text{g}/\text{min}$), the system will switch from 'Pretitration' to 'Standby' which means you are ready to start your measurements
11. Press on the 'Start sample' button at the bottom right corner of the screen and introduce your sample. See sections below for the different forms of introducing your sample into the cell. System will ask you about the mass of the sample that was introduced into the cell
12. Titration will start and after a few minutes (depends on the release rate of water from your sample) the water content will be displayed on the screen
13. Keep repeating steps 10-11 to measure all your samples
14. When done with all measurements, to turn off the coulometer
 - a. Press on the 'More' button
 - b. Select the 'Stop method' button
 - c. Acknowledge stoppage by pressing on the 'Yes' button
 - d. On the home screen, press on the 'Log out' button
 - e. On the login screen, press on the 'Shut down' button
 - f. System will shut down within a few seconds
15. Remove all your samples, syringes, needles, etc. and leave the area near the coulometer clean

A. MEASURE LIQUID SAMPLES

Follow the general steps to have the coulometer equilibrated and in the 'Standby' state.

1. After equilibration of the coulometer, the 'Start sample' button will be enabled
2. Load the liquid to be analyzed into a syringe with a needle. Measure the weight of the syringe loaded with liquid using the balance
3. Press on the 'Start sample' button, and the system will prompt for sample insertion
4. Inject the liquid into the measurement cell by injecting through the septum on the top of the stopper
5. Measure the weight of the syringe with any leftover liquid. Calculate the difference which is equal to the mass of liquid that was injected into the cell
6. Coulometer will prompt for the sample mass once some water has been detected. Provide the mass of the liquid and press 'OK'. The titration process will keep running until a stable drift has been achieved
7. The water concentration will be displayed on the screen

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B. MEASURE SOLID SAMPLES SOLUBLE IN METHANOL

Follow the general steps to have the coulometer equilibrated and in the 'Standby' state. The electrolyte in the measurement cell contains methanol that will dissolve your solid. If your solid sample is not soluble in methanol, please follow section 'C. Measure solid samples using the drying oven accessory'.

1. After equilibration of the coulometer, the 'Start sample' button will be enabled
2. Measure the weight of your solid and place it on a weighing paper
3. Press on the 'Start sample' button, and the system will prompt for sample insertion
4. Remove the stopper on the measurement cell and load your solid into the cell
5. Coulometer will prompt for the sample mass once some water has been detected. Provide the mass of the liquid and press 'OK'. The titration process will keep running until a stable drift has been achieved
6. The water concentration will be displayed on the screen

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PUBLICATION

A. EXPERIMENTAL SECTION

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

“Coulometric Karl-Fischer titration was performed at room temperature using a Mettler-Toledo C30 titrator. **Solid / liquid** sample was injected into the coulometric cell which was filled with a methanol-based electrolyte (Aquastar CombiCoulomat fritless) and equipped with a double Pt sensor.”

B. ACKNOWLEDGEMENT

“This work made use of the IMSERC facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University.”

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TROUBLESHOOTING

A. THE INSTRUMENT SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

B. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT LISTED IN THIS SECTION

If there is an error or problem with the instrument that 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. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
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REVISIONS

v1.02 2021/09/28	<ul style="list-style-type: none">• Release of original version of the user manual for the coulometer
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