

Quantulus GCT 6220 SOP

Physics Department, TTU

CEES, ESB I 153

Scope: The instrument operation and tritium content determination of electrolyte sample containing LiOH or LiOD using Quantulus GCT is described.

Materials:

PET vial +66021-735 VWR

Ultima Gold uLLT scintillation cocktail, PN 6013681

50 ml beakers

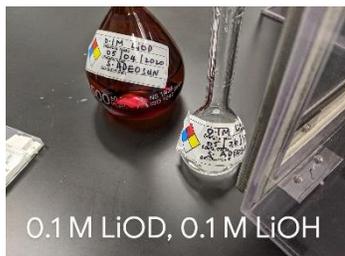
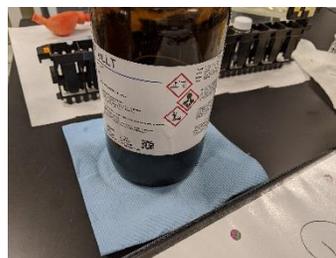
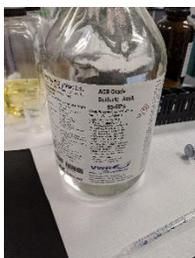
Pipette 5 ml and 1 ml size pipette tips

Background solution, 0.1 M LiOD solution

Calibration solution kit

pH paper

PP filter when necessary, 10 micron pore size, Chemglass OP-6602-12 or similar



Sample Preparation:

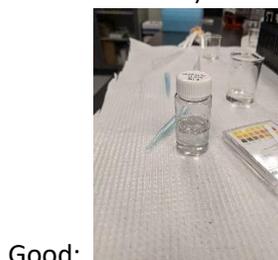
This section describes the LiOD electrolyte sample preparation for CPM analysis. The final appearance of the sample in the vial should be clear and void of particles and color.

Pipette 9 ml of ULTIMA GOLD uLLT scintillation cocktail into one of the vials

(Use a small beaker to pour cocktail into from the bottle to avoid contamination of the bottle. Never pipette directly from the bottle. Pour as little excess as possible into the beaker to reduce waste. Pour the excess and the rinse water from the beaker into the waste bottle)



1. Measure about 10 ml of electrolyte solution to be tested into a beaker. (There should not be any particulates in the solution. Filter the solution if necessary)
2. Add 2 to 3 drops of sulfuric acid solution. Check pH to make certain it is not basic anymore. Pipette 2 ml of the electrolyte sample from step 2 into the PET vial containing the sample. (The appearance of the final solution should be clear)



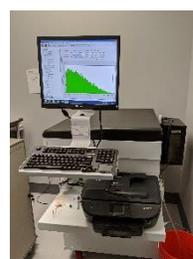
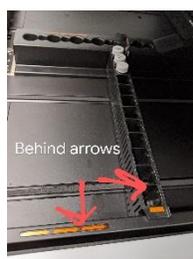
3. Place the sample in a dark cabinet to allow it to dark adapt and for any bubbles to dissipate for 1 hour prior testing. (This step can be completed inside the instrument)
4. Prepare the **background solution vial** following the steps 1-5 but using unused LiOD solution instead of electrolyte solution.

Waste disposal containers:



Loading samples into instrument:

1. Place the sample vial in the cartridge in one of the positions.
2. Place the protocol flag whose number corresponds to the assay to be used on the cartridge that contains the sample.
3. Move the slider on the flag to the left.
4. Open cover of the instrument. Place the cartridge on the right-hand slide towards the back.



Defining Count Per Minute (CPM) assay:

This section describes the procedure for setting up a new counting process for single nuclide containing samples. For routine electrolyte checks use “electrolyte check” assay.

1. Start a new assay by choosing “New assay” option in the main menu under file.
2. Choose CPM in “select assay type” window that will open. The seven tabs will need to be defined for the new assay. Select the OK button once the selections are made. The following settings are used in the “electrolyte check” assay that has been prepared for routine LiOD analyte checks.
 - Count Conditions
 - Nuclide Group-Name: 3H, Count Mode: Normal, Quench Indicator: tSIE
 - Pre-count Delay: 0 min
 - Assay Count Cycles¹: 1, Repeat Sample Count²: 1
 - Background Subtraction: No background subtraction
 - Number of vials per sample³:1 for single sample
 - Count Time (min): 10
 - 2sigma % - Uncheck the box next to “stop counting”
 - Count Corrections

- Static controller: Checked
- Luminescence correction: Checked
- GCT (Guard Compensation Technology): High
- Report Definitions – A new report, report name can be defined or a report can be previewed in this tab. Any items in “Use” column can be chosen as the items to be included in the report. (Note: yellow color appears in the list of items at the bottom of this tab when all the items selected cannot be fit in one line of the report.) Use “format” column for defining the number of digits for the results to be reported.

Information reported in “electrolyte check” test: S#, Count Time, CPMA, SIS, tSIE, Date, time, LUM, Messages

- Report output: “Print” option is checked for printing the report at the conclusion of the protocol.
- Special Files – Individual Spectra File can be generated if the box is checked next to it. (File will be saved according to Data Paths setting in the assay definition)
- Worklist – Description of a sample can be entered here
- Assay Details – User and/or additional details can be added here. “Electrolyte check” test is locked and requires a password for any changes to be made to it.

¹This determines the number of times the assay is run on the entire cassette. Each sample is counted once during each assay cycle.

²This defines the number of times each sample is counted prior to counting the next sample

³If performing analysis of multiple samples from the same electrolyte, the user can set a desired number of vials to be averaged together following counting.

Calibrating the instrument:

SNC (Self Normalization and Calibration) function is the performance check for the instrument and should be conducted prior to each sample analysis. Note: the “SNC” assay uses values that are unique to the standard vials to be used in this calibration step. If any of the vials have been replaced the values written on the new vials should be included in appropriate places in the IPA assay.

1. Place the cartridge containing the calibration vials in the unit. Make certain the vials in the cartridge match the labels on the side of the cartridge. Note: The empty vial must be the same type of vial as the one that contains the sample.
2. Move the tab on the flag to the left.
3. Highlight “SNC” in the protocol tree. Press on the green flag.
4. At the end of the calibration process a report will print. If it is reported that the system cannot be normalized do not use the instrument for analysis. Call for service.
5. Following a successful normalization, the instrument will continue to test any other cartridge whose flag is pushed to left in the instrument.

To Execute a protocol:

Note: An assay called “Electrolyte check” is saved in assay folder. This assay has been determined to be the optimum test parameters for routine LiOD electrolyte checks. Its parameters cannot be changed.

1. Make sure calibration of the system has been conducted.
2. Associate the assay of choice to the protocol to be used by following these steps:
 - a) In main screen, in the “protocol” tab on the bottom left corner, right click on the protocol appropriate flag number in the protocols tree and select “Associate Assay” option from the sub-menu. (You may have to “dissociate’ first if that flag is already associated to an assay)
 - b) Select the assay to be used in the “assays” folder. Press OK
 - c) Enter name in the “user” window. Press OK
 - d) Click on OK button on the main window to save the selections. The assay file name will appear in the protocols tree with by the associated flag number.
3. To start the analysis, select the desired protocol by clicking on it in the protocol tree on the left side of the screen. It will be highlighted in blue.
4. Click the green flag in the top left-hand corner to start. The user can stop the run at any time by using the red stop button located next to the green flag. The instrument will complete the requested assay and stop automatically. “Instrument Idle” will appear in the dialog box at the top once the assay is complete.

Notes:

- The top indicator bar below the file menus should show the protocol number you selected, cassette number you filled with samples, and the dialog box shift from “Instrument Idle” to “Searching”. If any of these are different from what you expect, either the green flag was not selected to start the assay, or the instrument has loaded the incorrect cartridge.
 - The screen will switch from report summary display to spectra count view as the protocol proceeds to take data.
5. Report will be printed automatically if chosen so in the assay set up. Additional reports can be printed by selecting the “report” function in the protocols tree in the main window.

Reviewing Previous Assay Results:

1. On the bottom left-hand side, choose the tab for “Replay” to view completed assays.
2. Select the assay to be viewed from the file drop down menu that will open.

Waste:

Samples:

Once the analysis of a sample is completed the vial containing the sample should be placed in an empty original box or double bags in clear plastic to be disposed of.

Materials used for test set up:

Any leftover scintillation cocktail that does not contain any sample should be emptied into the glass jar labeled "LSC waste". Empty LSC bottles can also be placed in the solid waste container.

Dry items such as gloves, paper towels etc should be placed into solid waste container labeled "Quantulus waste".

The beakers which contained LSC should be wiped with paper towel and rinsed with water. Paper towel can be put in the solid waste. The beaker can be washed to be reused.

The pipette tips should be placed into the sharps container only.

Spills: Any small spilled electrolyte sample or its mixture with the scintillation cocktail should be picked up with a paper towel placed into separate solid waste container. For larger spills absorbent should be used to pick up the liquid.

Any calibration liquid that may have been broken should be picked up with an absorbent and placed into a glass jar and capped tightly. The calibration liquids contain toluene which is flammable and evaporates quickly.