Sep 23, 2021 Version 2

Calibration of glass electrode half-cells V.2

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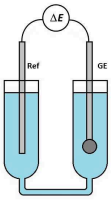

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Protocol status: In development
We are still developing and optimizing this protocol

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Abstract

The purpose of this document is give glass-electrode calibration procedure with aqueous buffers.

Troubleshooting

Before start

Turn on the thermostat and electrometer at least half an hour before starting the work.

Software

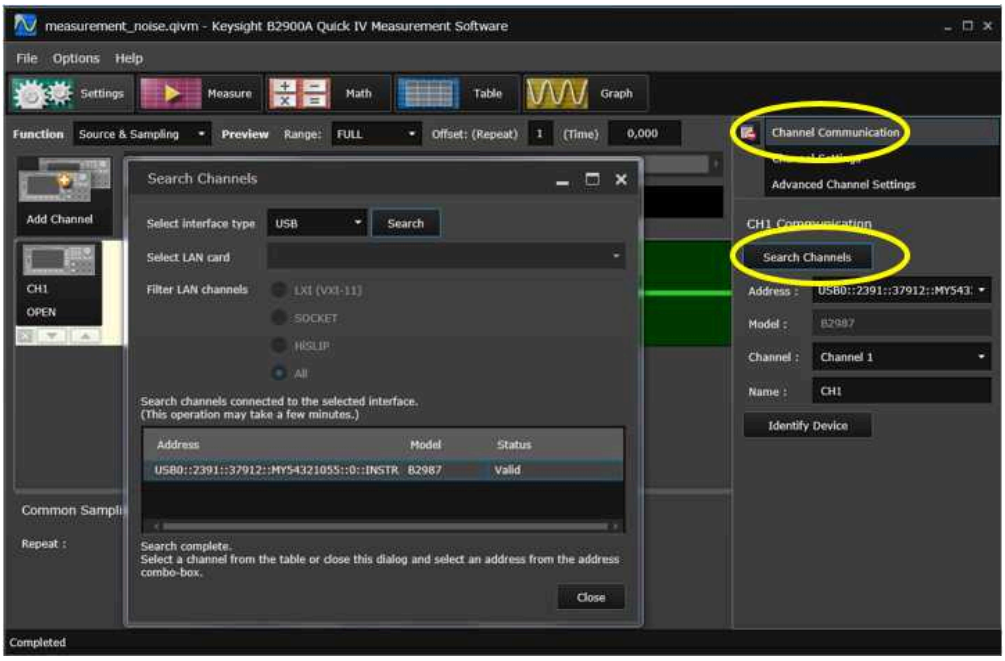
1 Start Quick IV Measurement Software.

> **Note**
>
> Computer cannot go to sleep during measurements or the communication between computer and instruments is lost and data collection stops.

2 On the left hand pick Function “Source & Sampling”.

Position of the Function choice “Source & Sampling”.

3 Right hand side click on tab “Channel Communication”. Click on “Search Channels”, which opens a new window. Select USB interface and search for the channel. After the search click on the channel and close the window.

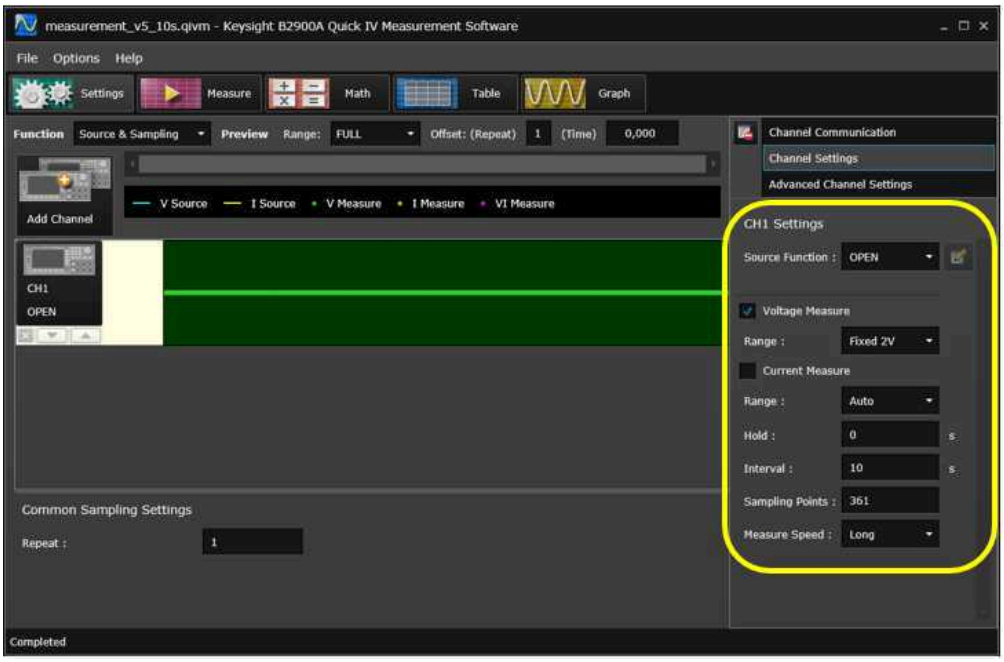


Position of the tabs “Channel Communication” and “Search Channels”.

Note

This must be done every time the program is started.

4 Settings can be saved as QIVM file, which can be used for measurements. The saved file can be opened under tab “File”. The settings will remain the same between the measurements if the program is not closed.



Program settings.

Note

Range is "Fixed 20V" in case of EST-0601 calibration.

Filling the cell

8m

5

25.0 °C

Equipment	
VistaShield	NAME
Faraday cage	TYPE
Gamry	BRAND
VistaShield	SKU

Note

The side ports must be open.

Note

A beaker can also be used if temperature is controlled.

6 Fill capillary with standard aqueous buffer with pH 7.00. 50 mL 2m

7 Insert glass electrode and a reference electrode. 1m

Equipment

K401	NAME
Calomel reference electrode	TYPE
Radiometer	BRAND
K401	SKU

7.1 Electrode is rinsed with water, gently dried, and then rinsed with solution to be measured. 1m

8 Connect electrodes to the instrument.

Equipment

B2987A Electrometer / High Resistance Meter	NAME
Electrometer	TYPE
Keysight	BRAND
B2987A	SKU
https://www.keysight.com/zz/en/home.html	LINK

8.1 Crocodile clips are as following:
red is signal and connects to the glass electrode,
black is shield and is connected to reference electrode and
green is guard. Green stays unused.

Note

Cable goes through the port in Faraday cage. Electrometer is not in the Faraday cage.

Equipment

N1415A Triax to Alligator Cable	NAME
Cable	TYPE
Keysight Technologies	BRAND
N1415A	SKU
https://www.keysight.com/zz/en/product/N1415A/triax-alligator-cable-200-v-1-5-m.html	LINK

Measurement

1h

9 Start the measurement by clicking on the "Measure" button on the upper panel.

10 Data collection. Point is taken at 10 s interval. 1h

> Note
>
> Calibration duration must be the same as later used in pH measurements.

11 Save data. Files are named as Date_GE_vs_ref_pHx.

> Note
>
> **SAVE THE FILE BEFORE NEW MEASUREMENT!** Otherwise, data is lost. Data is given in volts and seconds.

11.1 Go to table view, right click on the table, choose “Export as CSV...” and save the results. The data can be automatically imported into excel file while measuring or after (both under “Excel Navigation”), but this causes constant shift in the comma position.

Washing

12 Remove the electrodes.

13 Remove the solution.

14 Rinse three times with plenty of water 20 mL each

15 Rinse once with acetone to ease the drying. 2 mL

16 Dry cell with compressed air.

Measurement

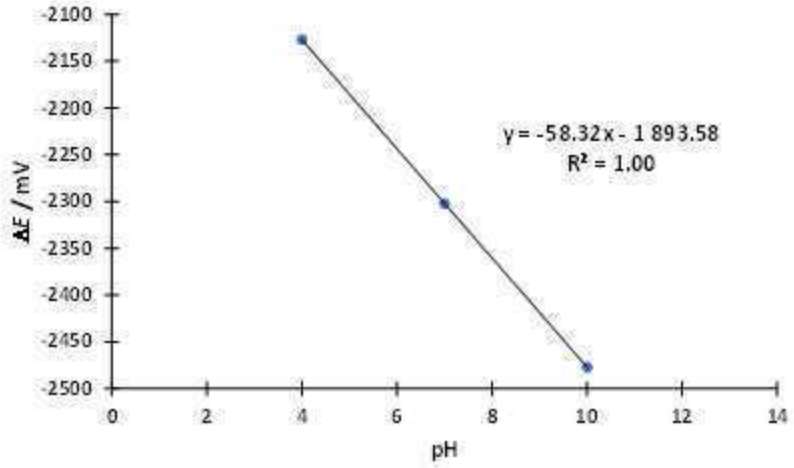
17 Repeat measurements with standard buffers pH 4 and pH 10.

Data analysis

18 Points from 30 min to 60 min are used for analysis.

19 Take the average of the chosen data points.

20 Plot Δ*E* vs pH.



Calibration graph of a glass electrode half-cell.

21 Obtain the slope and intercept of the glass electrode-half cell.