pH-potentiometry

General principles

Potentiometry is probably the most frequently used electroanalytical method. This is the method in which the potential between two electrodes is measured while the electric current (usually nearly zero) between the electrodes is controlled. The correlation between the concentration of the analyte solution and potential is given by Nernst equation:

$$E = E^0 + \frac{RT}{zF} ln \frac{c_{Ox}}{c_{Red}}$$

where E is the potential at the indicator electrode, $E°$ is the standard potential of the electrochemical reduction (a value that changes as the chemical identity of the couple changes), R is the gas law constant, T is the absolute temperature of the solution, n is the number of electrons transferred in the reduction (the value in the half reaction), F is the Faraday constant, and the c_{Ox} and c_{Red} terms are the concentrations of the oxidized and reduced chemical species, respectively, in the solution.

pH-meters (for example the Mettler-Toledo T5 apparatus) operate by measuring the difference in voltage generated between and indicator electrode and a reference electrode. A reference electrode is a half-cell having a known potential that remains constant at constant temperature and independent of the composition of the analyte solution. Calomel electrodes and silver/silver chloride electrodes are types of reference electrodes (see Figure 1).

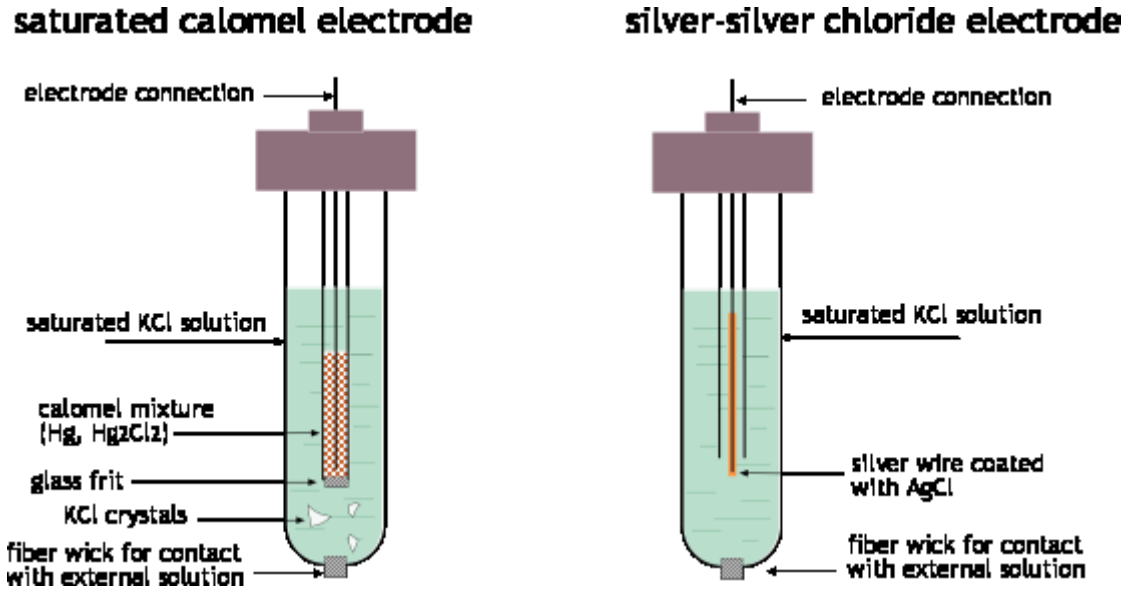


Figure 1.

An indicator electrode has a potential that varies with variations in the concentration of an analyte. Most indicator electrodes used in potentiometry are selective in their responses.

Metallic indicator electrode, ion-selective electrode and membrane electrodes are types of indicator electrodes. Ion-selective electrodes preferentially respond to a single chemical species. The potential between the indicator electrode and the reference electrode varies as the concentration or activity of that particular species varies. The selectivity of the ion-selective electrodes results from the selective interaction between the membrane and the analyte. The electrodes are classsified according to the nature of the membrane. The most common types of ion-selective electrodes are the glass electrodes.

The glass electrode for pH measurements

Typical cells for measuring pH consist of a glass indicator electrode and a saturated calomel reference electrode immersed in the solution whose pH is unknown. The indicator electrode consists of a thin, pH sensitive glass membrane sealed onto one end of a heavy-walled glass or plastic tube. A small volume of hydrochloric acid saturated with silver chloride is contained in the tube. A silver wire in this solution forms a silver/silver chloride inner-reference electrode, which is connected to one of the terminals of the potential-measuring device, pH-meter (see Figure 2.). The calomel electrode is connected to the other terminal.

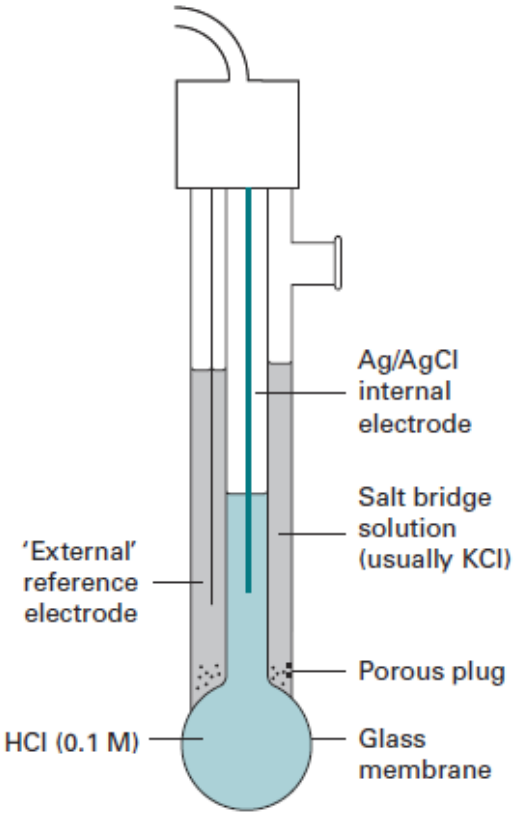
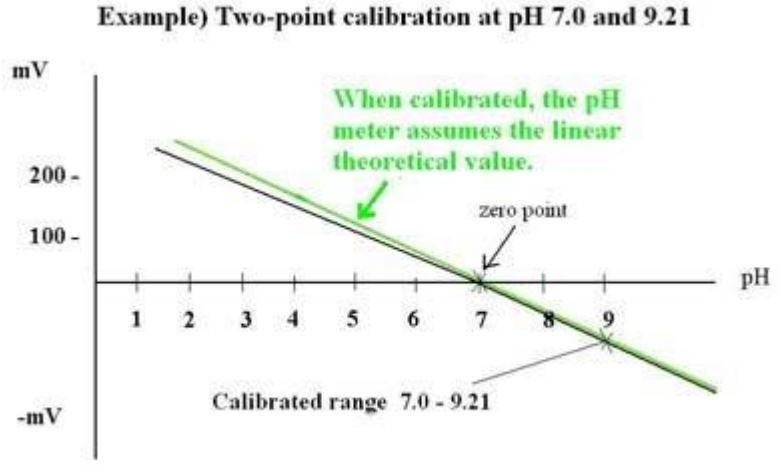
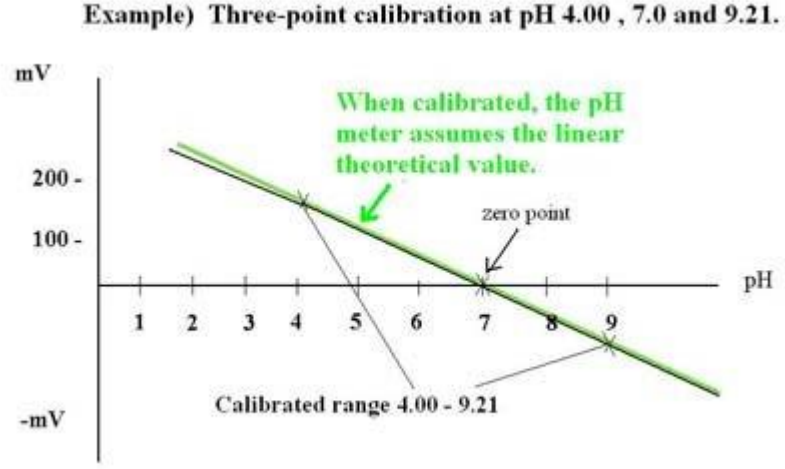


Figure 2.

The electric potential related between the glass electrode, and the inner-reference electrode is a function of the pH value (activity of hydronium ion, a_{H3O+}) of the measured solution. So once the potential difference between glass electrode and outer-reference calomel electrode has been measured the pH value can be calculated. Modern pH electrodes are usually of the "combination" type, meaning that a single cylinder contains both a glass membrane electrode and the outer-reference calomel electrode. A change in hydronium ion concentration causes a change in composition of the glass membrane due to an ion exchange process involving the solution and the membrane. A corresponding change in membrane potential, proportional to pH, is what is measured. All other potentials are constant. In effect the membrane potential (variable) is measured against two fixed potentials, the external reference and the internal reference, both Ag/AgCl reference electrodes. Potential difference is measured using a high impedance (internal resistance) potentiometer.

pH calibration procedure





Very precise measurements necessitate that the pH meter is calibrated before each measurement. Calibration is needed because the glass electrode does not give reproducible electrostatic potential over longer periods of time. Consistent with principles of good laboratory practice, calibration is performed with at least two standard buffer solutions that span the range of pH values to be measured. For general purposes, buffers at pH 4.00 and pH 10.00 are suitable. The pH meter has one calibration control to set the meter reading equal to the value of the first standard buffer and a second control which is used to adjust the meter reading to the value of the second buffer. During a two-point calibration, the microprocessor based pH meter determines the real slope and offset error for the actual pH electrode. This information is then used to adjust the mV/pH-equation of the pH meter to match the characteristic of the electrode in use. The two-point calibration is also called bracketing calibration, since the two calibration points should bracket the range of values that will be measured.

The Gran method

A Gran plot (also known as Gran titration or the Gran method) is a common means of standardizing a titrate or titrant by estimating the equivalence volume or end point in a strong acid-strong base titration or in a potentiometric titration. The Gran method is a frequently used method to determine the equivalence volume because of the linearization of the titration curves results in more exact determination of equivalence volume and the differences come from contamination are easier detectable. Such plots have been also used to calibrate glass electrodes, to estimate the carbonate content of aqueous solutions, and to estimate the K_a values (acid dissociation constants) of weak acids and bases from titration data. Gran plots use linear approximations of the a priori non-linear relationships between the measured quantity, pH or electromotive potential (emf), and the titrant volume. In the case of strong acid and weak acid, different Gran functions will be used because the dissociation of these chemical species are different (partial for weak acids and complete for strong acids).

The following abbreviations will be used:

C_S	concentration of acid in the sample
V_0	initial volume of the sample
C_B	concentration of the alkali in the burette
V_B	volume of the alkali in the burette
V_E	equivalence volume

Titrating strong acid with strong base

Acidic region:

The excess of acid can be given by the following equation:

$$\left[H^+\right] = \frac{\left(V_E - V_B\right)C_B}{V_0 + V_B}$$

For taking into account that $[H^+] = 10^{-pH}$:

$$(V_0 + V_B)\cdot 10^{-pH} = (V_E - V_B)\cdot C_B$$

Thus, a plot of $(V_0+V_B)\cdot 10^{-pH}$ *vs.* V_B will have a linear region before equivalence, with slope $-C_B$

Alkaline region:

The excess of alkali can be given by the following equation:

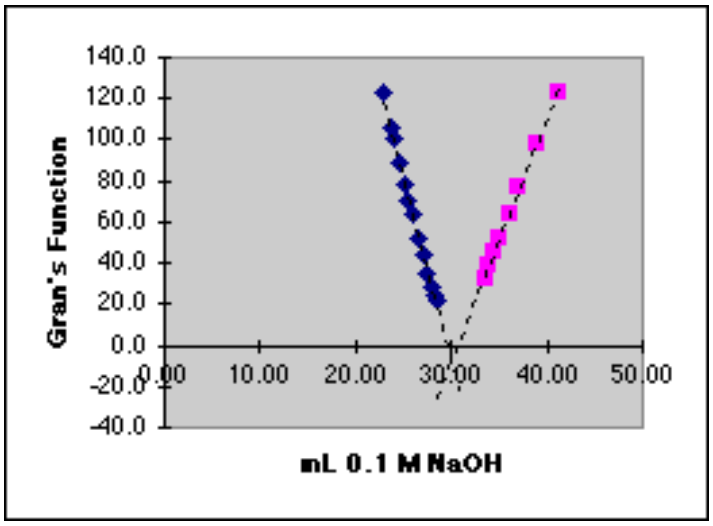
$$[OH^-] = \frac{(V_B - V_E)C_B}{V_0 + V_B}$$

For taking into account that $[OH^-] = K_v/[H^+]$, thus $[OH^-] = 10^{pH-pK_v}$:

$$(V_0 + V_B)\cdot 10^{pH-pK_v} = (V_B - V_E)\cdot C_B$$

where Kv is the water ionization constant.

Thus, a plot of $(V_0+V_B)\cdot 10^{pH}$ *vs.* V_B will have a linear region after equivalence, with slope C_B/Kv. Both plots will have $V_E=V_0\cdot C_S/C_B$ as intercept.



Titrating weak acid with strong base

The method can be used to estimate the dissociation constants of weak acids, as well as their concentrations. With an acid represented by HA, where

$$HA \leftrightarrow H^+ + A^-$$

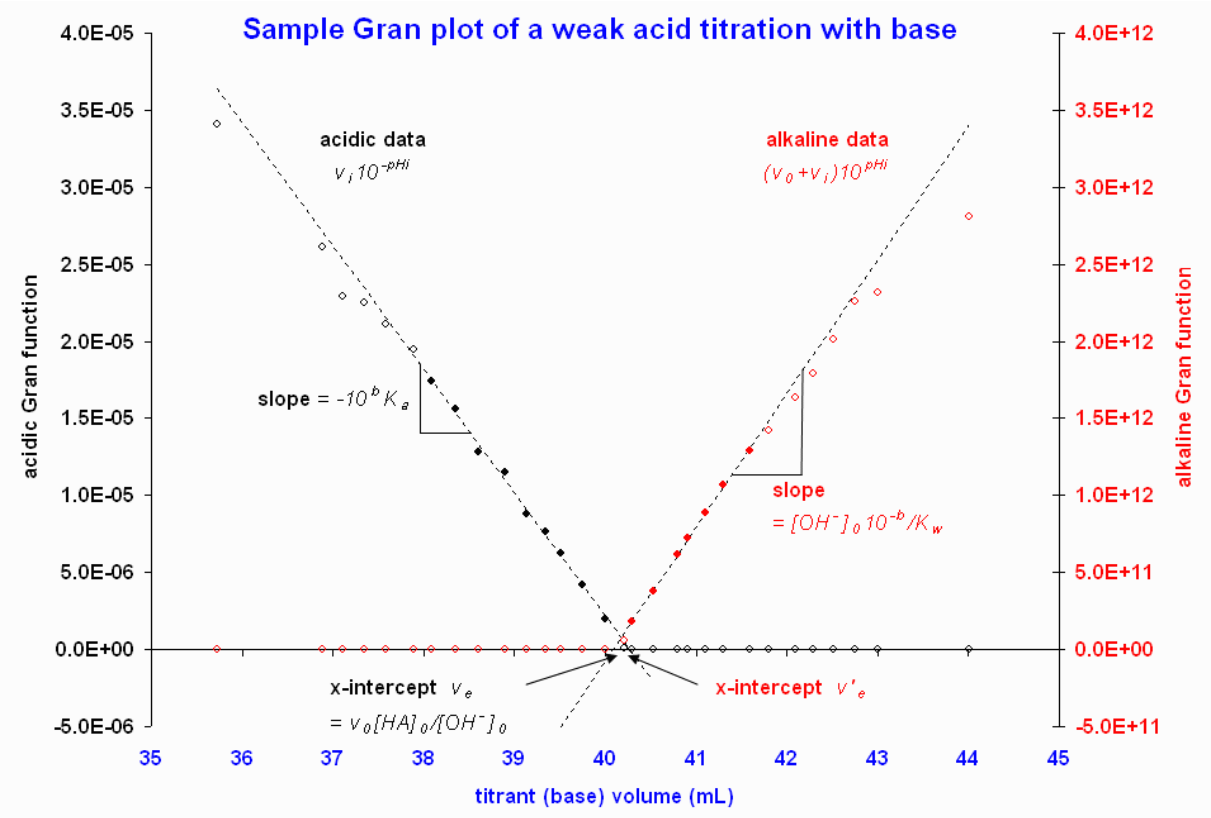
The concentration of conjugated acid and base can be given:

$$[HA] = \frac{C_B(V_E - V_B)}{V_0 + V_B} \qquad [A^-] = \frac{C_B V_B}{V_0 + V_B}$$

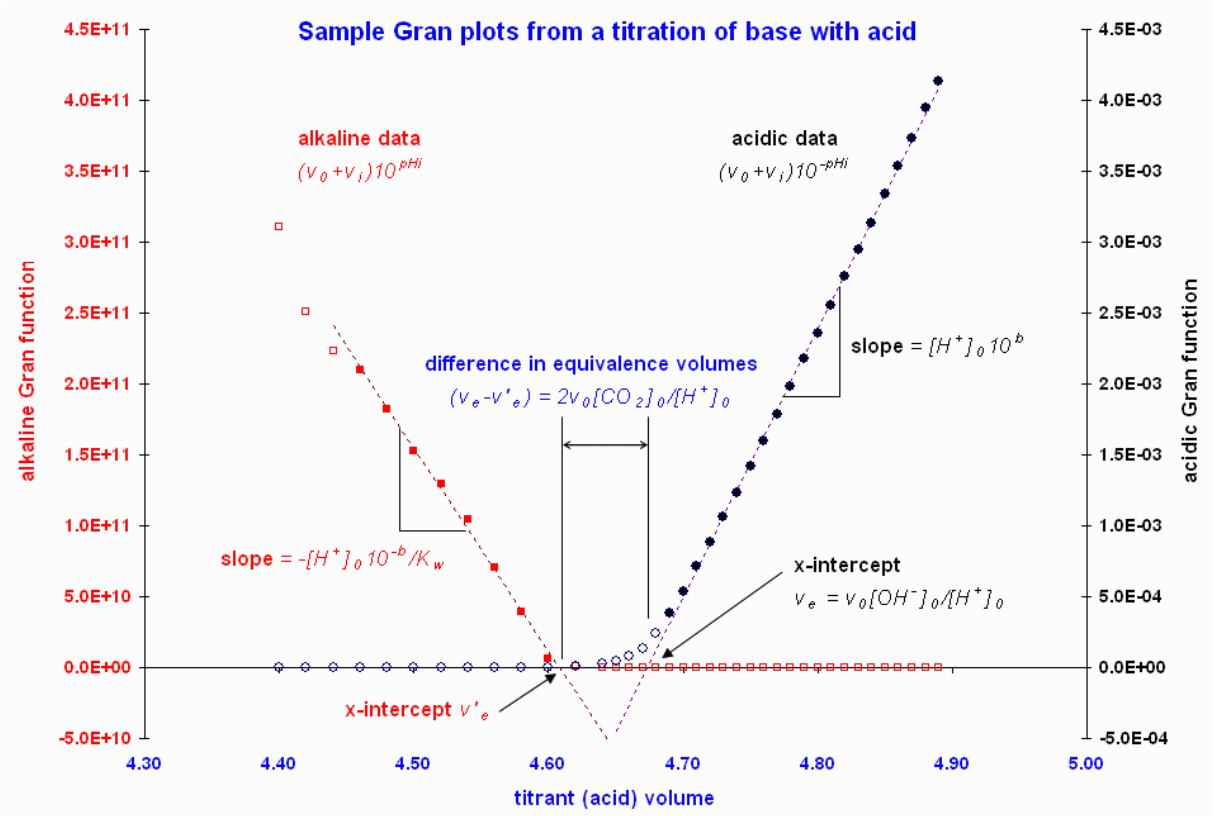
Thus and for taking into account that $[H^+] = 10^{-pH}$:

$$V_B 10^{-pH} = (V_E - V_B) \cdot 10^{-pK_S}$$

A plot of $V_B \cdot 10^{-pH}$ *vs.* V_B will have a linear region with an extrapolated x-intercept V_E.



Finally, it is important to note that the Gran function is able to estimate the adventitious carbon-dioxide in the base solution. This contamination is illustrated by the following figure.



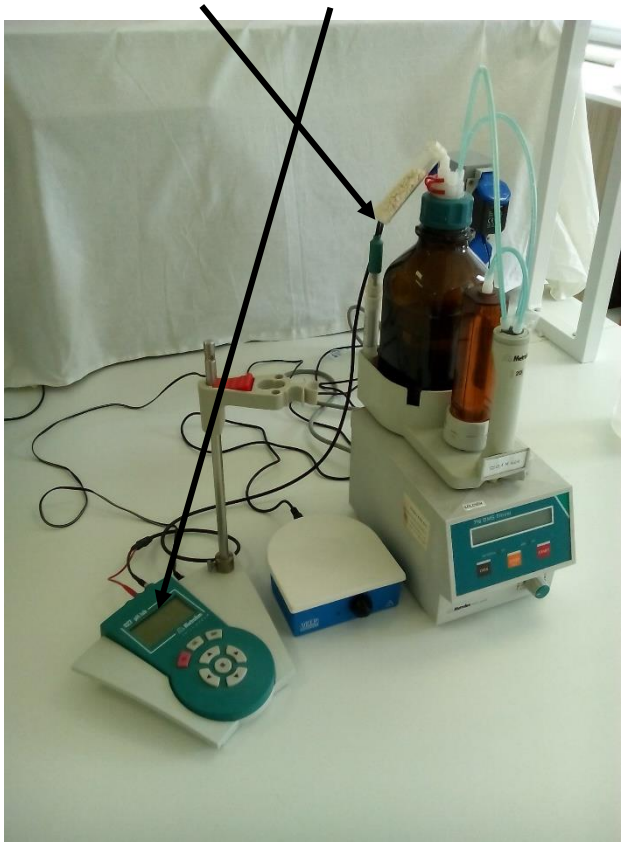
In that situation, the extra acid used to neutralize the carbonate. This effect results in difference in the calculated equivalence volumes.

Review questions:

1. Concept of pH and principles of its instrumental measurement.
2. Typical titration curves during the determination of weak and strong acids with strong base.
3. Acid-base concepts
4. Properties of electrodes used in pH-metry.
5. Calibration of electrodes used in pH-metry.
6. Reference electrodes and the principles of their use.
7. Advantages of pH-metric titrations.
8. Principles of recording and evaluating pH-metric titration curves.
9. Gran functions.
10. Titration curves of multiprotic acids.
11. Titration curves of mixtures of acids.

Practical part

How to use Titrino titrator + Metrohm pH-meter



Picture 1: Work station for pH-metric

- **Guide for the use of the electrode:**

○The electrode is equilibrated in 1 M KCl solution to avoid the ion transport between the internal and external part of the membrane.

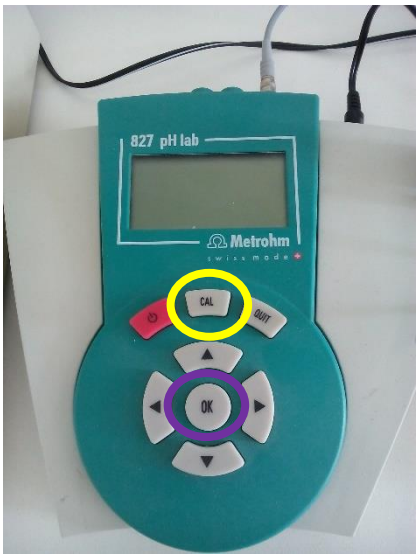
○ On the top of the electrode a ***filling gap (Picture 2)*** can be found that must be opened before beginning the measurement and must be closed at the end of the practice.

Picture 2: filling gap

○In order to clean the electrode, rinse it with large volume of distilled water. Dry the electrode with paper but do not rub the membrane!

Picture 3: glass frit

○ Int he case of the Metrohm pH-meter the electrode is covered by plastic and has three plastic „legs" to avoid it from any harm. Therefore the electrode has to be cleaned more carefully, the area around the membrane should be rinsed with distilled water and dried with paper.



Picture 4: Metrohm pH-meter ((on/off button. CAL.

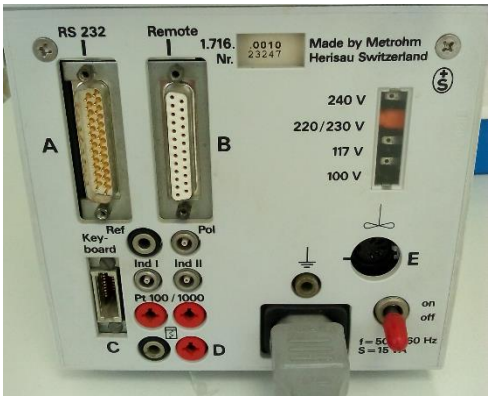
○In the case of the Metrohm pH-meter the black ring above the plastic legs should be in the sample. The electrode must be cleaned each time before immersed in an other solution.

○Avoid leaving the electrode drying that causes the deposition of KCl on the glass frit. Also avoid leaving the electrode in highly basic solution at the end of a titration.

- **Calibration**

- Immerse the cleaned and dried electrode in the **third** buffer solution
- In case of the Metrohm pH-meter ***(Picture 4)*** After switching on (***red button***) calibration process can be started by pressing ***CAL*** button. Confirm, that the electrode is immersed in the buffer by pressing ***OK***. After that the temperature can be set, accept the default value of 25.0 °C by pressing ***OK***. The equipment shows the pH value to be calibrated to, accept is by pressing ***OK***. After the pH is stabilized (disappearance of the *drifting* sign and the graph) accept the value on the screen by pressing ***OK***. The pH-meter is ready to calibrate on the next buffer solutions in decreasing order of pH, press ***OK***, the method is similar as described above. In the case of this pH-meter the potential values in mV are also shown for each buffer.
- At the end of the calibration procedure the slope (SLP) and the pH(0) value appears on the screen of the pH-meter. The value of sensitivity (SLP) should be around 100% and must be registered in the lab manuals immediately because it disappears in a few seconds.

- **Guide for the use of the titrator:**



Picture 5a: Titrino titrator

- Turn on the titrator ***(Picture 5a)*** by clicking the ***on/off tumbler switch*** on the back of the equipment.
- The equipment fills the burette automatically while the valve moves in front of the burette, because the valve moves to “fill” position, the container is connected with the burette, the piston lowers. After filling the burette the valve moves back to “titration” position and the burette is connected with the plastic tube to let out the base solution, the piston will lift.
- Base solution can be added through this plastic tube (behind the burette, on the right) by holding the **DOS** button ***(Picture 5b).*** This procedure is applied to remove the gas bubbles from the line connecting the burette and the glass tube. After letting out some ml of the solution and carefully hitting the line some small bubbles can still remain. In this case the titration can be started but simultaneously pay attention to the bubbles not to let them out in the sample.

Picture 5b: Titrino titrator

- Fill the burette by pressing the **STOP/FILL** button, and wash and dry the plastic tube of the titrator then immerse it IN the sample placed on the magnetic stirrer at least 1 cm deep.
- Drop a clean and dry stirrer bar in the baker and immerse the dry electrode in the sample, too.
- Pay attention while setting of the equipment that everything should be distant from the side of the baker except the stirrer bar and keep the electrode, the plastic tube and the stirrer bar away from each other.
- Start the mixing (intensely but causing no whirl in the sample).
- After registering the stabilized pH of the solution add the first, given increment of the base solution. Add a volume as close as possible to the given increment to the sample solution. Avoid the short, several press of the button, add a volume increment of base solution by ***holding the button only once***. It is more important to ***register the exact volume added*** instead of the closely approached given increment.

- In a certain range of the titration the stabilization of pH would take a longer time but after 2 minutes the value must be registered and the titration must be continued by adding the next increment.
- After finishing the titration fill the burette and clean the plastic tube, the electrode and the magnetic stirrer bar, then start the titration of the next sample.
- At the end of the practice the clean and dry electrode back in the 1 M KCl solution, close the filling gap, and switch off each equipment.

How to use ABU titrator + Thermo Orion 2 Star pH-meter

Picture 1: Work station for pH-metric titrations

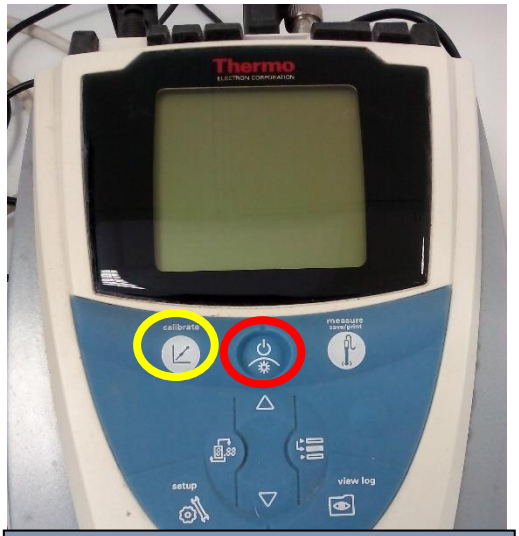
- **Guide for the use of the electrode:**
  - The electrode is equilibrated in 1 M KCl solution to avoid the ion transport between the internal and external part of the membrane.
  - On the top of the electrode a ***filling gap (Picture 2)*** can be found that must be opened before beginning the measurement and must be closed at the end of the practice.

Picture 2: filling gap

  - In order to clean the electrode, rinse it with large volume of distilled water. Dry the electrode with paper but do not rub the membrane!

Picture 3: glass frit

  - The ***glass frit (Picture 3.)*** (small white spot) on the bottom of the electrode is responsible for the transport between the internal and external solution, therefore it should be in the sample. The electrode must be cleaned each time before immersed in an other solution.
  - Avoid leaving the electrode drying that causes the deposition of KCl on the glass frit. Also avoid leaving the electrode in highly basic solution at the end of a titration.

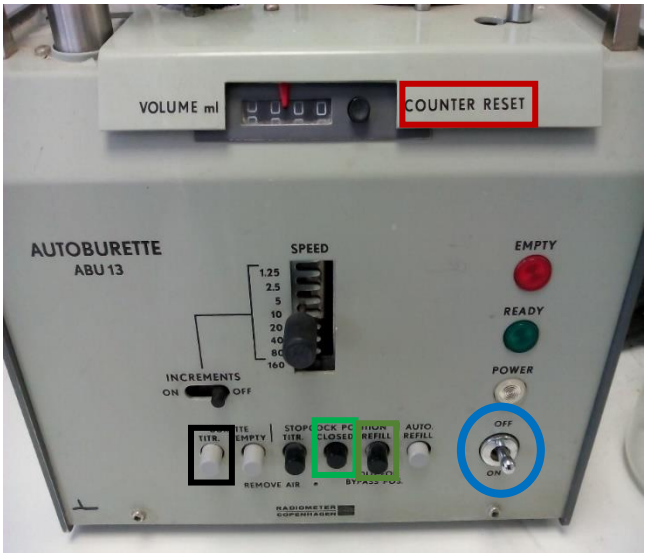


Picture 4: Thermo Orion 2 Star pH-meter (on/off button, Calibrate button)

- **Calibration**
  - Immerse the cleaned and dried electrode in the first buffer solution.

- In case of the Thermo Orion 2 Star pH-meter ***(Picture 4)*** after switching on (***middle button on the top***) calibration process can be started by pressing ***Calibrate*** button, then accept the pH value by pressing it repeatedly after the equilibrium is stabilized (*pH* sign stops blinking on the screen). After that the electrode must be cleaned and dried and immersed in the next buffer solutions in increasing order of pH followed by the acceptance of the stabilized pH value similarly.
- At the end of the calibration procedure the slope (SLP) and the pH(0) value appears on the screen of the pH-meter. The value of sensitivity (SLP) should be around 100% and must be registered in the lab manuals immediately because it disappears in a few seconds.

- **Guide for the use of the titrator:**
  - Turn on the titrator ***(Picture 5)*** by clicking the ***on/off tumbler switch***.
  - Press the ***Closed*** button.
  - Fill the burette by shortly pressing the black ***Refill*** button (waiting time can be ca. 20-30 seconds). The cog wheel valve moves to "fill" position, the container is connected with the burette and the piston lowers. After filling the burette the valve moves back to "titration" position, the burette is connected with the glass tube and the piston can lift. The green light of the ***Ready*** lamp shows that the titration can be started.



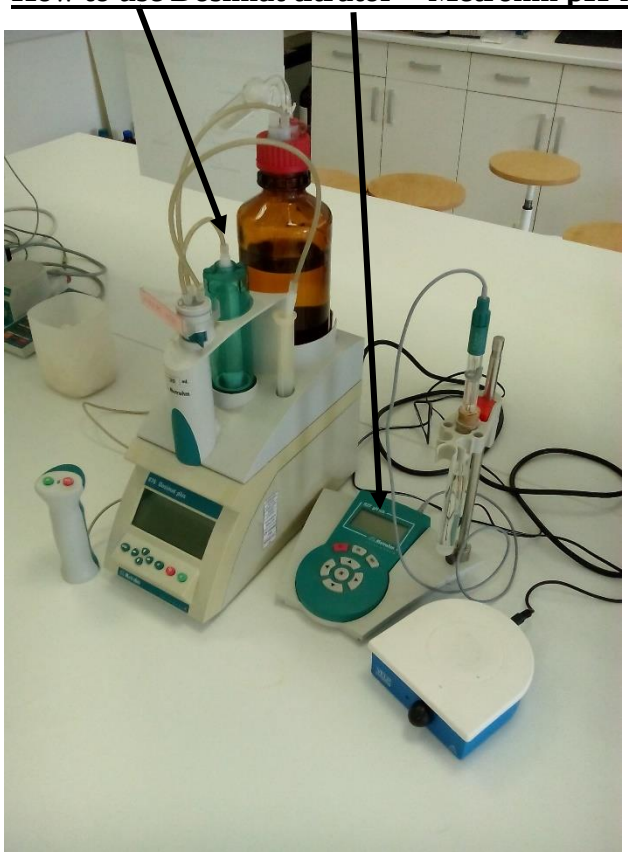
Picture 5: ABU titrator (on/off tumbler switch, CLOSED, REFILL, TITR., and COUNTER RESET buttons)

  - Base solution can be added through the glass tube by holding the grey ***TITR.*** button. This procedure is applied to remove the gas bubbles from the line connecting the burette and the glass tube (the white plastic tube is hardly can be seen through, at least 4 ml base solution has to be let out in a baker).
  - Fill the burette and press the ***Counter Reset*** button, wash and dry the glass tube of the titrator then immerse it IN the sample placed on the magnetic stirrer at least 1 cm deep.
  - Drop a clean and dry stirrer bar in the baker and immerse the dry electrode in the sample, too.
  - Pay attention while setting of the equipment that everything should be distant from the side of the baker except the stirrer bar and keep the electrode, the glass tube and the stirrer bar away from each other.
  - Plug in the magnetic stirrer and start the mixing (intensely but causing no whirl in the sample).

- After registering the stabilized pH of the solution add the first, given increment of the base solution. Add a volume as close as possible to the given increment to the sample solution. The red triangle on the counter marks the decimal point. Avoid the short, several press of the button, add a volume increment of base solution by ***holding the button only once***. It is more important to ***register the exact volume added*** instead of the closely approached given increment. Exact volume to three decimals should be registered by determining the last number according to the position of the second decimal on the counter.

- In a certain range of the titration the stabilization of pH would take a longer time but after 2 minutes the value must be registered and the titration must be continued by adding the next increment.
- After finishing the titration fill the burette and clean the glass tube, the electrode and the magnetic stirrer bar, then start the titration of the next sample.
- At the end of the practice press the ***Closed*** button and place the clean and dry electrode back in the 1 M KCl solution, close the filling gap, lift up the glass tube in the same height of the level of the base solution and put a baker under it. Switch off each equipment and plug out the magnetic stirrer.

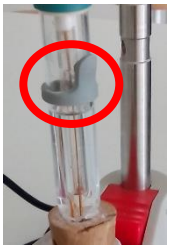
How to use Dosimat titrator + Metrohm pH-meter



Picture 1: Work station for pH-metric

- **Guide for the use of the electrode:**

○ The electrode is equilibrated in 1 M KCl solution to avoid the ion transport between the internal and external part of the membrane.



Picture 2: filling gap

○ On the top of the electrode a ***filling gap (Picture 2)*** can be found that must be opened before beginning the measurement and must be closed at the end of the practice.

Picture 3: glass frit

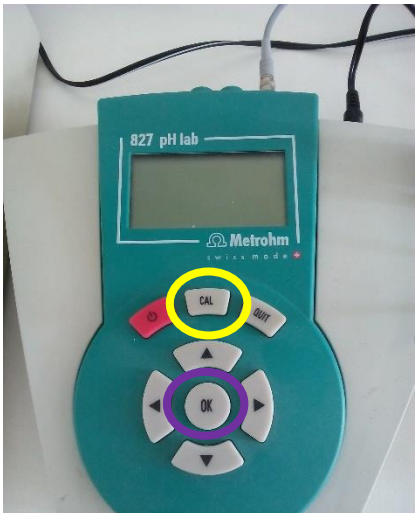
○ In order to clean the electrode, rinse it with large volume of distilled water. Dry the electrode with paper but do not rub the membrane!

○ The ***glass frit (Picture 3.)*** (small white spot) on the bottom of the electrode is responsible for the transport between the internal and external solution, therefore it

should be in the sample. The electrode must be cleaned each time before immersed in an other solution.

- Avoid leaving the electrode drying that causes the deposition of KCl on the glass frit. Also avoid leaving the electrode in highly basic solution at the end of a titration.

- **Calibration**
  - Immerse the cleaned and dried electrode in the first buffer solution.
  - In case of the Metrohm pH-meter ***(Picture 4)*** After switching on (***red button***) calibration process can be started by pressing ***CAL*** button. Confirm, that the electrode is immersed in the buffer by pressing ***OK***. After that the temperature can be set, accept the default value of 25.0 °C by pressing ***OK***. The equipment shows the pH value to be calibrated to, accept is by pressing ***OK***. After the pH is stabilized (disappearance of the *drifting* sign and the graph) accept the value on the screen by pressing ***OK***. The pH-meter is ready to calibrate on the next buffer solutions in increasing order of pH, press ***OK***, the method is similar as described above. In the case of this pH-meter the potential values in mV are also shown for each buffer.



Picture 4: Metrohm pH-meter (on/off button, CAL, OK)

  - At the end of the calibration procedure the slope (SLP) and the pH(0) value appears on the screen of the pH-meter. The value of sensitivity (SLP) should be around 100% and must be registered in the lab manuals immediately because it disappears in a few seconds.

- **Guide for the use of the titrator:**
  - Turn on the titrator ***(Picture 5)*** by pressing the **FILL** button then press **OK**.
  - The equipment fills the burette automatically while the valve moves in front of the burette, because the valve moves to “fill” position, the container is connected with the burette, the piston lowers. After filling the burette the valve moves back to “titration” position and the burette is connected with the plastic tube to let out the base solution, the piston will lift.

Picture 5: Dosimat titrator

- Base solution can be added through this plastic tube (behind the burette, on the right) by holding **GO** button. This procedure is applied to remove the gas bubbles from the line connecting the burette and the glass tube. After letting out some ml of the solution and carefully hitting the line some small bubbles can still remain. In this case the titration can be started but simultaneously pay attention to the bubbles not to let them out in the sample.
- Fill the burette by pressing the **FILL** button, reset the counter by pressing it again and wash and dry the plastic tube of the titrator then immerse it IN the sample placed on the magnetic stirrer at least 1 cm deep.
- Drop a clean and dry stirrer bar in the baker and immerse the dry electrode in the sample, too.
- Pay attention while setting of the equipment that everything should be distant from the side of the baker except the stirrer bar and keep the electrode, the plastic tube and the stirrer bar away from each other.
- Start the mixing (intensely but causing no whirl in the sample).

- After registering the stabilized pH of the solution add the first, given increment of the base solution. Add a volume as close as possible to the given increment to the sample solution. Avoid the short, several press of the button, add a volume increment of base solution by ***holding the button only once***. It is more important to ***register the exact volume added*** instead of the closely approached given increment.
- In a certain range of the titration the stabilization of pH would take a longer time but after 2 minutes the value must be registered and the titration must be continued by adding the next increment.
- After finishing the titration fill the burette and clean the plastic tube, the electrode and the magnetic stirrer bar, then start the titration of the next sample.
- At the end of the practice the clean and dry electrode back in the 1 M KCl solution, close the filling gap, and switch off each equipment.

How to use the Mettler T5 apparatus (Figure 3.)

To turn on:

1. Open LabX 2017 on the PC and wait for starting the program.
2. Push the ⏻ buttom on the Mettler T5 titrator to turn it on.
3. Push the OK buttom on the control panel (Figure 4.*) ábra*) in the push-up window for recognizing the burette.

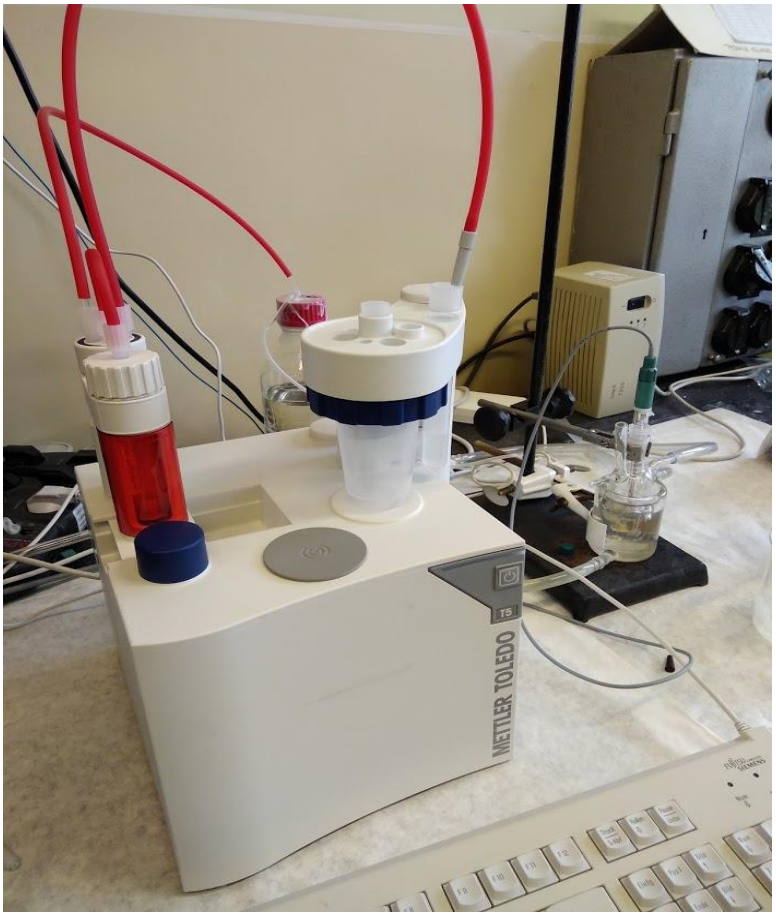


Figure 3. Mettler T5 titrator

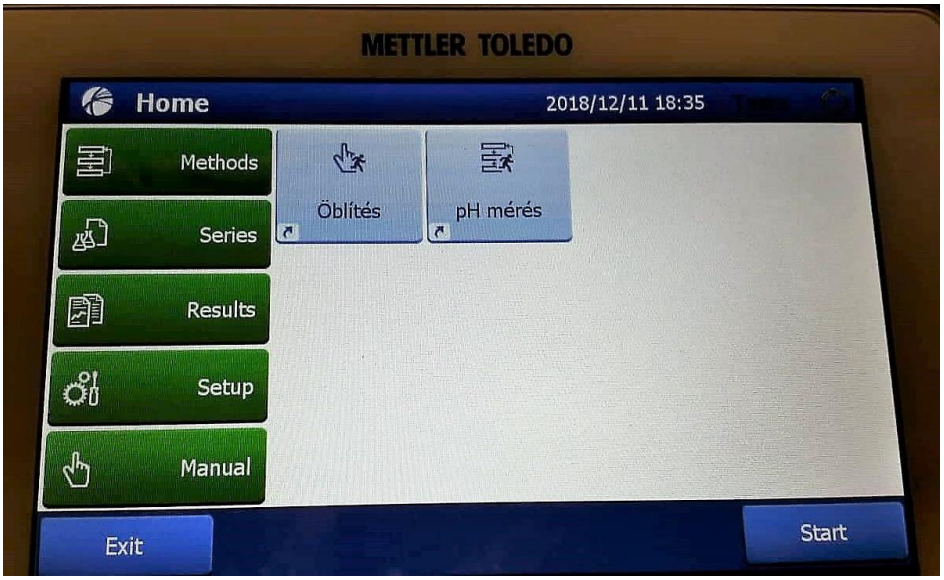


Figure 4. Control panel of Mettler T5

After opening the software the **Method Tools** (Figure 5.) window appears.

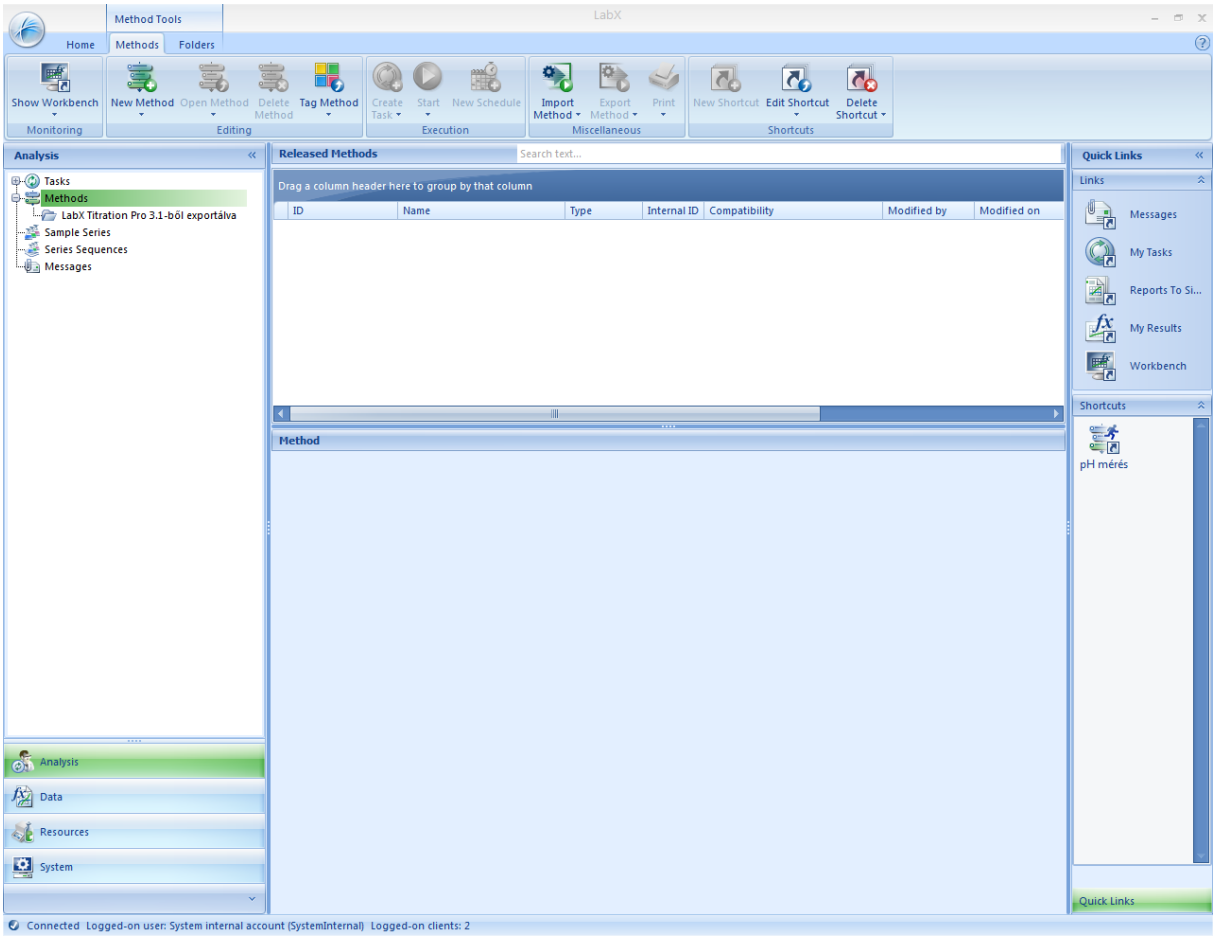


Figure 5. ***Method Tools***

Measuring the pH of a potassium-hydrogenphthalate (KHP) buffer solution:

Left-click once on the **Workbench** and select T5 in the pop-up window. Now the **Workbench** (Figure 6.) window appears. In this window select **Sensor** from the vertical menupanel on the right. Within this window press the  buttom to start measuring the pH of the solution (Figure 7.). The results of measuring the pH can also be seen on the control panel. After the stabilization of the pH-readings (ca. 20 min.) the measurement can be terminated by clicking on the buttom.

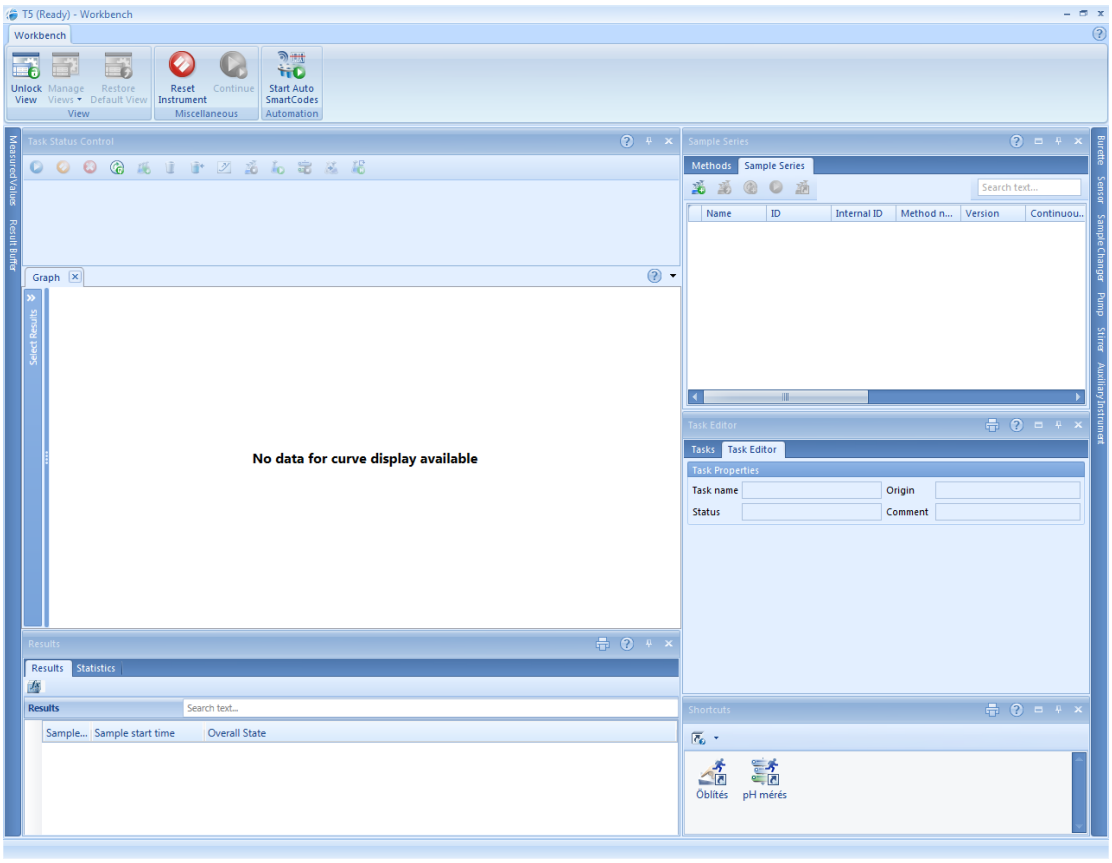


Figure 6. ***Workbench***

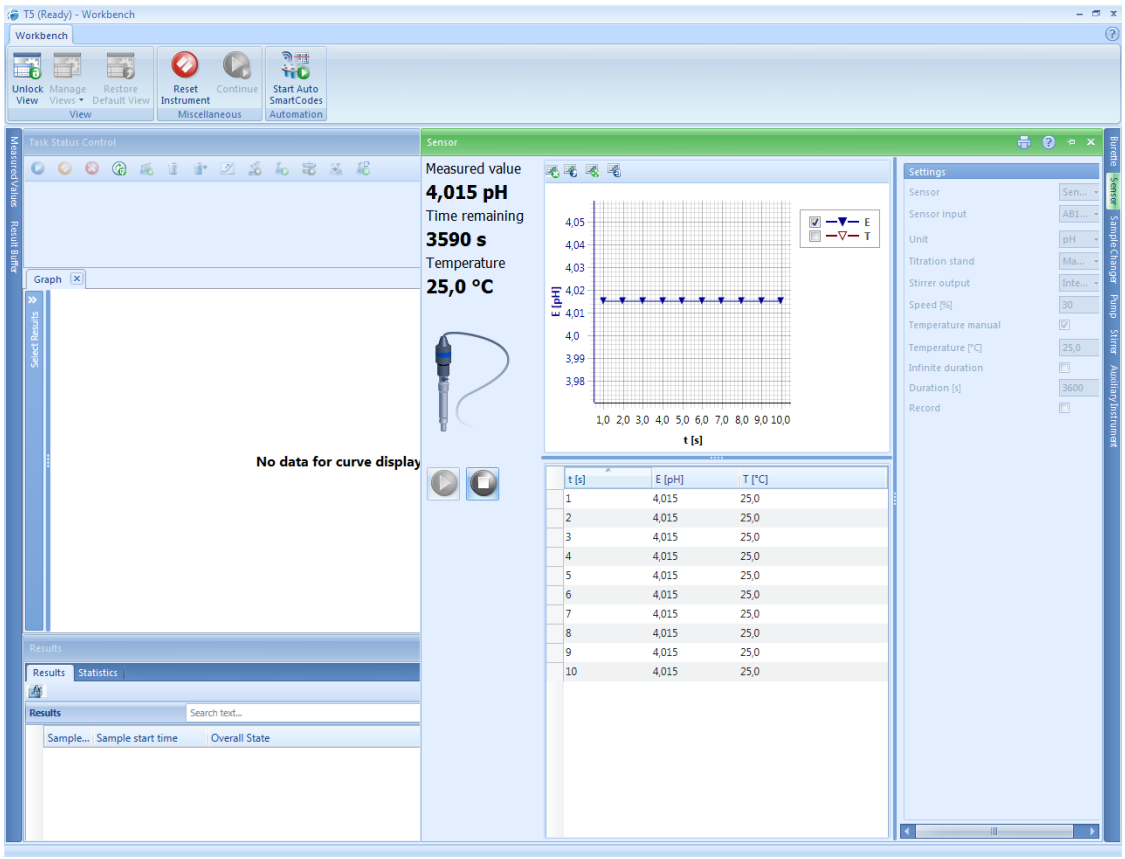


*Figure 7. Measuring pH in the **Workbench** using the Sensor window*

Calibration of the electrode system:

In the window **Method Tools**

Click on the **Methods/Exported** from LabX Titration Pro 3.1 and select **Calibration**, and click **Start**, select T5 and OK (Figure *8.*) in the pop-up **Task Editor** window. In ca.1.5 min. when the potential measurement is completed a **Sample Completed** window pops up and here press OK. If required, the current pH can be checked again by pressing **Sensor** (see measuring pH); the value should be 4.008 after the KHP calibration.

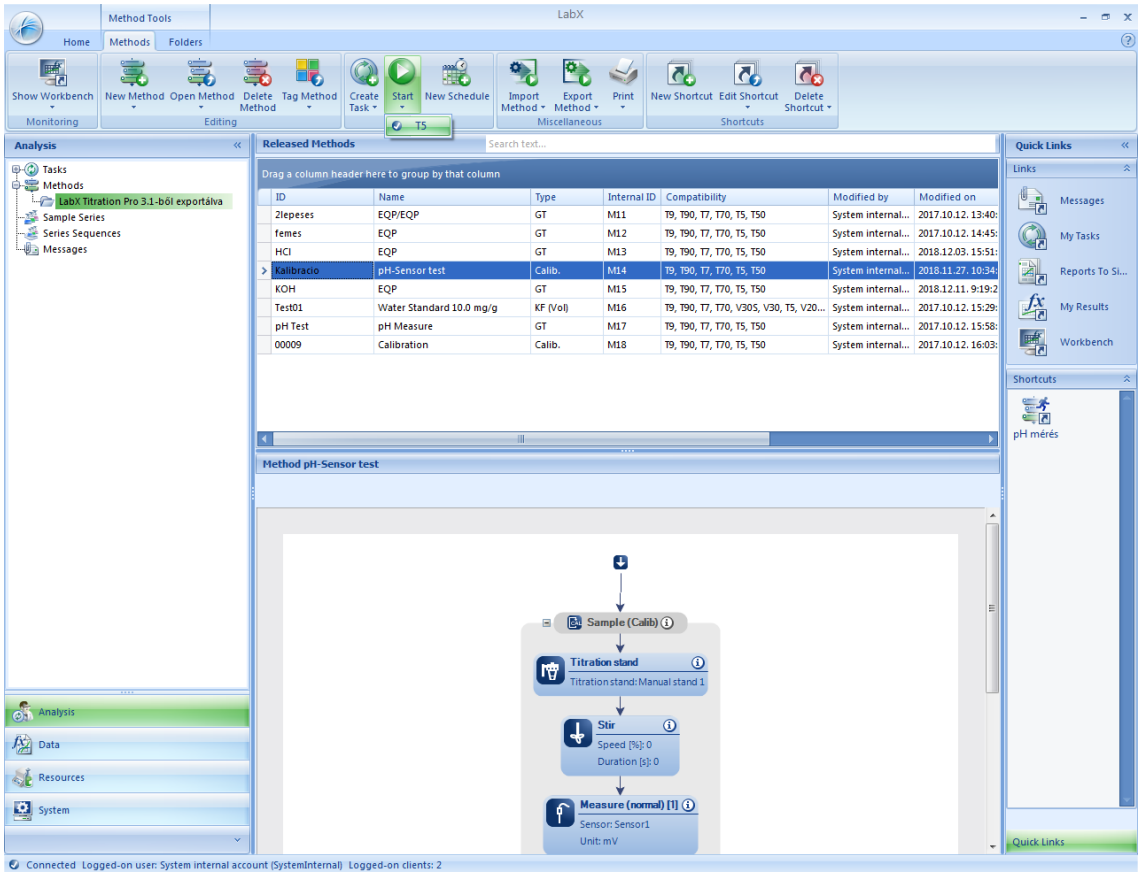


Figure 8. Calibration

Performing the titration of an unknown solution:

1. step: Remove the KHP solution from the titration vessel, rinse the electrode, put the unknown sample (e.g. acid, base etc.) and immerse the dried electrode into the sample.
2. step: Removal of any bubbles from the base capillary.

Go to **Workbench**/Burette. You can select either **Rinse** or **Disperse**. In the case of **Rinse** you can set up the number of rinses and the volume expressed in % of the total volme of the burette. In the case of **Dispense** you can set up the volume of base used to flush the capillary (or add to the sample in one portion). During rinsing the gas bubbles can be removed from the cappillary by gentle hitting of it.

After the removal of any bubbles from the capillary rinse it outside with distilled water and dried with a small filter paper. The base introducing capillary is now ready to be immersed into the sample. Put also the other capillary providing the stirring gas for the titration into the sample.

3. Go to the **Sensor** window (see pH-measurements) and start measuring the starting pH of the sample for reaching a pH equilibrium (10-15 min). Meanwhile set up the measuring parameters before the tiration. After getting a steady pH for the sample to be titrated terminate the pH-measurement with the ⏹ buttom.
4. Set up the measuring parameters in the **Method Tools** window. Double click on the method you want to use. After this the **Method Editor** flow chart can be seen (Figure 9).

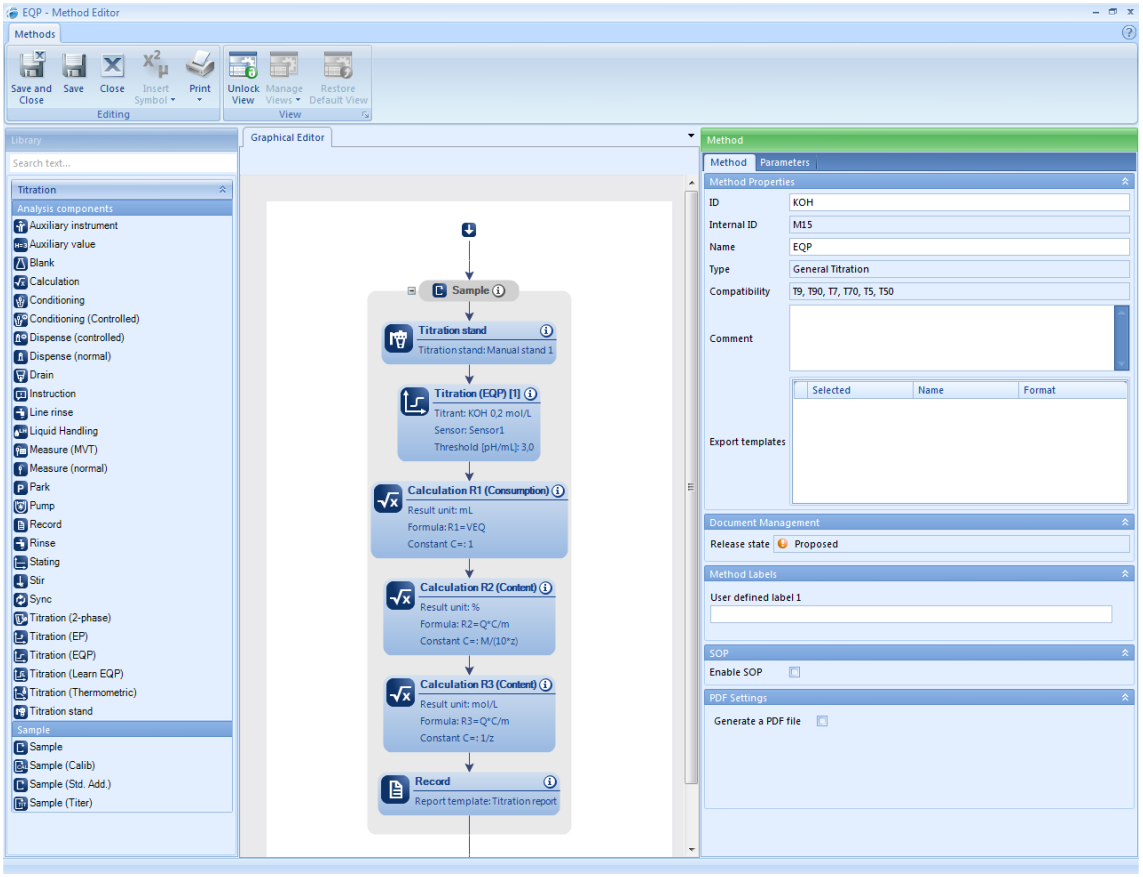
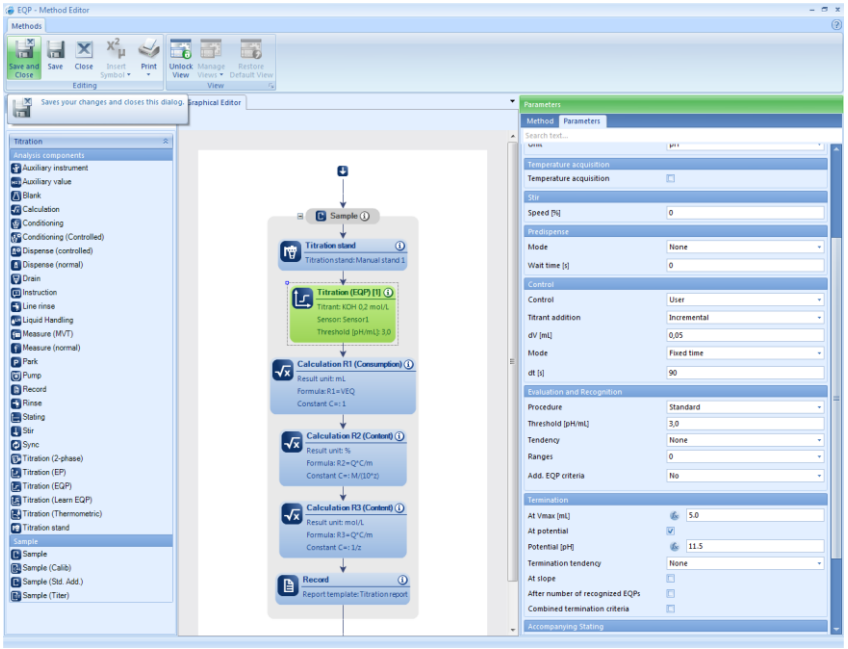


Figure 9. Flow chart of Method Editor

Click to the **Titration (EQP)** [1] bouble once to select it. It turns green in colour. Now you can



set up or modify the various parameteres (Table 1) in the **Parameters** window on the right (Figure 10).

Figure 10. The window with the various parameters

Table 1. Initial parameters of the various methods

		KOH ($V_{tot.}$=25 ml, c_{KHP}=0.01 M)	HCl ($V_{tot.}$=25 ml, c_{HCl}=$8 \cdot 10^{-3}$ M)	Ligand	M-L systems
Method		KOH	HCl	HCl	femes
Control					
Dosage	dV [ml]	0.05	0.05	depending on the system	depending on the system
	Mode	Fixed time	Fixed time	Fixed time	Equilibrium controlled
	dE [mV]	-	-	-	0,1
Waiting time	dt [s]	90	120	120	90
	t(min) [s]	-	-	-	120
	t(max) [s]	-	-	-	300-1800 (depending on the system)
Termination					
	Potential [pH]	11.5	11.6	11.5	11.0

If you change any parameters press Save and Close.

5. Initiating the measurement: Select the appropiate **Method** with the correct parameters by clicking once on it. Now press **Start/T5** and press **Start/OK** in **the Task Editor** pop-up window. The measurement can be monitoted either in the **Workbench** window or on the touch screen panel.

Saving the data:

At the end of the titration in the LabX 2017 program an **Enter Sample Size** window appears; press 1 and Enter and Enter again. (This can be done either on the panel or on the screen.) Double click in the **LabX/Data/My Latest Results** window on the data file to be saved. Now the **Results Editor** window appears. Select **Measured Values** and highlight the necessary volume-pH data pairs with Ctrl+A and copy them with Ctrl+C and Ctrl+V into an empty Excel worksheet and save the excel file on a pendrive. Once done close the **Results Editor** window.

To turn off the instrument:

Use the control panel as follows:

1. Press twice OK buttom on the panel.
2. After returning of the computer to the main menu press **Exit/Offline/Shut** down.

1. Preparation of 0.05 mol/dm^3 potassium hydrogen phthalate solution

Prepare 100 cm^3 0.05 mol/dm^3 potassium hydrogen phthalate solution. This solution will be used for the calibration (pH = 4.01), and for the determination of concentration of KOH solution. For calibrating the electrode, the guideline placed near the instrument must be followed.

2. Determination of the concentration of ca. 0.1 mol/dm^3 KOH solution

The concentration of the titrant (KOH solution) is determined using the solution of potassium hydrogen phthalate already prepared. In a 150 cm^3 beaker, prepare 100.0 cm^3 of a solution with the following concentrations:

KH-phthalate: 0.005 mol/dm^3 (10 cm^3 from 0.05 mol/dm^3)

KCl: 0.2 mol/dm^3 (10 cm^3 from 2 mol/dm^3)

Titrate this sample with the KOH solution under continuous stirring. Use the following increment sequence:

0 - 3 cm^3: add 0.5 cm^3 titrant between two points

3 - 6 cm^3: add 0.2 cm^3 titrant between two points

6 - 8 cm^3: add 0.5 cm^3 titrant between two points

Draw a graph showing the titration curve and determine the equivalence points using Gran functions.

3. Determination of the concentration of ca. 0.1 mol/dm^3 hydrochloric acid

hydrochloric acids

In a 150 cm^3 beaker, prepare 100.0 cm^3 of a solution with the following concentrations:

HCl: ca. 0.005 mol/dm^3 (5 cm^3 from ca. 0.1 mol/dm^3)

KCl: 0.2 mol/dm^3 (10 cm^3 from 2 mol/dm^3)

Titrate this sample with the KOH solution under continuous stirring. Use the following increment sequence:

0 - 4 cm^3: add 0.5 cm^3 titrant between two points

4 - 6 cm^3: add 0.2 cm^3 titrant between two points

6 - 8 cm^3: add 0.5 cm^3 titrant between two points

Draw a graph showing the titration curve and determine the equivalence points using Gran functions.

4. Determination of the concentration of a mixture containing acetic and hydrochloric acids

In a 150 cm^3 beaker, prepare 100.0 cm^3 of a solution with the following concentrations:

HCl: ca. 0.003 mol/dm^3 (3 cm^3 from ca. 0.1 mol/dm^3)

Acetic acid: ca. 0.003 mol/dm^3 (3 cm^3 from ca. 0.1 mol/dm^3)

KCl: 0.2 mol/dm^3 (10 cm^3 from 2 mol/dm^3)

Titrate this sample with the KOH solution under continuous stirring. Use the following increment sequence:

0 - 9 cm^3: add 0.2 cm^3 titrant between two points

Draw a graph showing the titration curve. Determine the concentrations of both acids after carefully considering which Gran functions are useful for this purpose.

The Mettler T5 pH-potentiometric titration apparaus, presented and used in the laboratory course, was purchased through the GINOP-2.3.2-15-2016-00008 project, funded by the European Union, co-funded by the European Regional Development Fund.