Application Bulletin 102/2 e

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Conductometry

Of interest for:
Analytical laboratories in general
E L 1, 2, 4, 5, 7, 8, 16

Summary

This Bulletin is made up of two parts. The first part treats the theoretical basis of conductometry in a simple manner; the second, practice-oriented part deals with the following subjects:

- Determination of the cell constant
- Determination of the temperature coefficient
- Conductivity measurements in general
- Conductivity measurements in water samples
- Salinity (TDS – Total Dissolved Solids, expressed as NaCl content)
- Conductometric titrations

1. Theoretical part

1.1 General

The following considerations apply exclusively to solutions containing ions.

Conductometry deals with the conductivity of electrolytes. The resistance of the solution is measured by applying an alternating voltage to the measuring cell (if direct current were applied, electrolytic processes occurring within the solution could alter the resistance). Experience has shown that, due to polarization effects, more precise results are obtained if the **measuring frequency** is adapted to the measuring range, e.g. 300 Hz at low and 2.4 Hz at higher conductivities (712 Conductometer). The conductivity of a solutions depends on:

- the **number of ions.** The more ions a solution contains, the higher its conductivity.

- the **ionic mobility** in a general way. The mobility in turn depends on:

    – the **type of ion:** the smaller an ion, the more mobile it is and the better it conducts electrical current. Ions of very high conductivity are H_3O^+, OH^-, K^+ und Cl^-. If an ion is surrounded by water molecules (hydratization) and therefore becomes larger, its conductivity decreases.

    – the **solvent:** the more polar a solvent, the more completely ionized are the compounds dissolved in it. Water is an ideal solvent for ionic compounds. In alcohols the ionization decreases with increasing chain length (methanol >ethanol >propanol). In non-polar organic solvents, e.g. chlorinated and non-chlorinated hydrocarbons, there is practically no ionization.

    – the **temperature:** in contrast to what is found with solids, the conductivity of solutions increases with increasing temperature at a rate that ranges from 1 to 9% per Kelvin, depending on the ion.

    – the **viscosity:** the ionic mobility decreases with increasing viscosity, which means that the conductivity also decreases.

1.2 Measuring setup

The minimal measuring setup consists of a **conductometer** and the **conductivity cell** connected to it. Generally the following items are also used: temperature sensor, closed measuring/titrating vessel that can be thermostatted, and a magnetic stirrer.

A **conductometer** is an instrument for measuring complex resistances using alternating voltages (in contrast to the measurement of the purely ohmic resistances of metallic conductors, liquids, together with the measuring cell, constitute a network of resistances and capacities). The alternating voltages applied are available at minimum two frequencies. Quasi-ohmic conditions can be obtained, however, by the appropriate selection of the measuring frequency, cell constant and electrode material. Under these conditions the electrical conductivity can be determined from the measured resistance.

A **conductivity measuring cell** consists of two electrodes that face each other and are as inert as possible. Platinum is generally used as the electrode material. Smooth (shiny) electrodes should only be used for conductivities <20 µS/cm. For higher conductivities, platinized electrodes are used (coated with platinum black). To avoid measuring errors due to changes in the electrical field, the measurement is carried out in a strictly defined volume. This is why immersion measuring cells are usually surrounded by glass or a plastic material.

As stated above, the same cell cannot be used for the whole conductivity range. Accordingly, different cells are used that are optimized for the conductivity range to be covered. The measuring cells are characterized by their **cell constant c.** For low conductivities, cells with a small cell constant are used while cells with a high cell constant serve to measure high conductivities.

1.3 Measuring the electrical conductivity

The electrical conductivity γ is the reciprocal of the electrical resistance R (R is the reciprocal of the conductance G) multiplied by the cell constant c:

$$\gamma = \frac{1}{R} \times \frac{\ell}{A} = G \times c$$

γ = conductivity; unit: S cm^{-1} (S m^{-1})

R = resistance; unit: Ω (Ohm)

G = 1 / R   conductance; unit: S (Siemens) = Ω^{-1}

ℓ = separation; unit: cm (m)

A = cross section; unit: cm^2 (m^2)

c = ℓ / A   cell constant; unit: cm^{-1} (m^{-1})

Electrical conductivities are normally given in µS/cm or mS/cm (12.88 mS/cm = 1288 mS/m; 5 µS/cm = 500 µS/m). In American English the designations mhos and µhos are used.

The electrical conductivity strongly depends on the temperature. The value measured at a given temperature is therefore often converted to a **reference temperature.** The usual reference temperatures are 20 °C and 25 °C. The conversion is done applying the **temperature coefficient TC.**

$$TC(\Theta - 25) = \frac{1}{\gamma_{25}} \left(\frac{\gamma_\Theta - \gamma_{25}}{\Theta - 25} \right) \times 100$$

25 stands for the reference temperature (25 °C) and Θ °C is the temperature at which the electrical conductivity was measured.

The temperature coefficient can be given in % per Kelvin or **% per °C.**

The temperature coefficient depends mainly on the ions that are in the solution but its behavior is seldom linear. That's why we recommend to have it determined automatically by the 712 Conductometer.

Low **conductances** are determined with a low **measuring frequency,** high conductances with a high measuring frequency (note the difference between conductance and conductivity).

1.4 Concepts

Ionic product of water

Water dissociates in a process that is called auto-protolysis:

$$2\ H_2O\ \rightarrow\ H_3O^+\ +\ OH^-$$
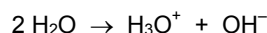
Because of this dissociation, pure water has an electrical conductivity of 0.055 µS/cm at 25 °C or 0.039 µS/cm at 20 °C. Please note the high temperature coefficient of 5.8 %/°C.

Molar conductivity

The molar conductivity Λ is defined as the ratio between the specific conductivity γ and the concentration c [mol/L] of the dissolved substance:

$$\Lambda = \frac{1000 \times \gamma}{c} = S\,/\,cm\,/\,mol$$
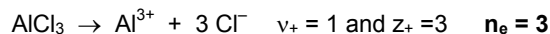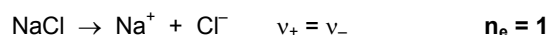
Equivalent conductivity

The equivalent conductivity Λ^* is mainly used in theoretical investigations. It is calculated from the molar conductivity Λ and the electrochemical valency n_e:

$$\Lambda^*\ =\ \Lambda\,/\,n_e$$

For a molecule that dissociates into ν_- anions and ν_+ cations with the corresponding charges z_- and z_+, the electrochemical valency n_e is calculated as follows:

$$n_e\ =\ \nu_-\ ^*\ z_-\ =\ \nu_+\ ^*\ z_+$$

Examples:

$NaCl \rightarrow Na^+ + Cl^-$ $\nu_+ = \nu_-$ **n_e = 1**

$AlCl_3 \rightarrow Al^{3+} + 3\ Cl^-$ $\nu_+ = 1$ and $z_+ = 3$ **n_e = 3**

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Dielectric constant D

In electrochemistry the dielectric constant D is of significance wherever there is interaction between opposed charges. Examples for this are ionic **dissociation** and ionic interaction. The higher the (relative) dielectric constant, the more complete the dissociation of ionic compounds in the corresponding solvent. Here are some examples:

Solvent	D 20 °C
Formamide	110
Water	80
Methanol	34
Ethanol	25
Acetone	21
Propanol	19
Chloroform	4.8
Hexane	1.9

Ionic mobility l_i

The ionic mobility is the product of the migration rate u_i and the Faraday constant F, where u_i refers to a standard field having a field strength of 1 V/cm. The distance covered by the ion i is given in cm. The ionic mobility depends on temperature and concentration. It is usually given for 25 °C and infinite dilution (measuring series extrapolated to $c_i = 0$). A few examples:

Cation	l_+ [cm^2/Ω]	Anion	l_- [cm^2/Ω]
H^+	350	OH^-	199
Li^+	39	Cl^-	76
Na^+	50	NO_3^-	71
K^+	74	CH_3COO^-	41
½ Mg^{2+}	53	½ SO_4^{2-}	80
½ Ca^{2+}	60	½ CO_3^{2-}	69

Non-electrolytes

As their name implies, non-electrolytes do not conduct the electrical current and thus do not contribute to the electrical conductivity. Such substances dissolved in water are, for example, non-ionic surfactants, sugars and suspended oils. The content of ionic impurities can, however, be used for analytical purposes. An example for this is the **determination of the ash content of sugars.**

In an emulsion the charge of the suspended phase is opposed to that of the dispersant. Water droplets suspended in an oil can migrate in an electric field. This causes phenomena that, looked at superficially, are similar to electrical conductivity. Thus, the **stability of emulsions** can be checked by conductivity measurements.

1.5 Conductometric titration

Apart from color indicators, conductivity measurement is one of the oldest methods for the indication of titration endpoints.

As at any stage of the titration the sum of all ions present determines the conductivity, there result the typical V-shaped titration curves.

Example: titration of HCl with NaOH:

$$H^+ + Cl^- + Na^+ + OH^- = H_2O + Cl^- + Na^+$$
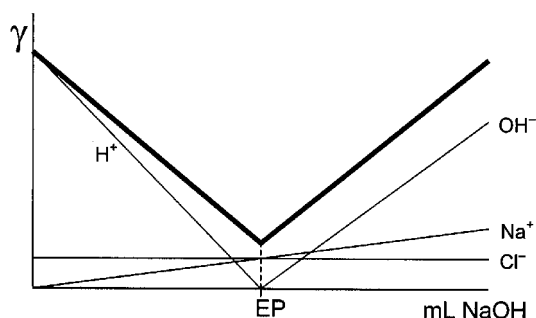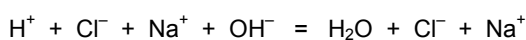
Thin lines: contributions of the individual ions to the conductivity
Bold line: total conductivity (determines the titration curve)

The **endpoint of the titration** is given by the minimum of the conductivity

1.6 Tables

a) Conductivity and temperature coefficient of KCl solutions at different temperatures

Temperature [°C]	c(KCl) = 0.1 mol/L		c(KCl) = 0.01 mol/L	
	γ [mS/cm]	TC_{20} [%/°C]	γ [mS/cm]	TC_{20} [%/°C]
18	11.19	2.06	1.225	2.07
19	11.34	2.06	1.251	2.11
20	11.67	–	1.278	–
21	11.91	2.06	1.305	2.11
22	12.15	2.06	1.332	2.11
23	12.39	2.06	1.359	2.11
24	12.64	2.07	1.386	2.11
25	12.88	2.07	1.413	2.11

b) Conductivity of KCl solutions at 20 °C and 25 °C

c(KCl) [mol/L]	γ 20 °C	γ 25 °C
0.0005	67 µS/cm	74 µS/cm
0.001	133 µS/cm	147 µS/cm
0.005	654 µS/cm	720 µS/cm
0.010	1.28 mS/cm	1.41 mS/cm
0.020	2.51 mS/cm	2.77 mS/cm
0.050	6.06 mS/cm	6.70 mS/cm
0.100	11.67 mS/cm	12.90 mS/cm
0.200	22.44 mS/cm	24.80 mS/cm

c) Conductivity of NaCl and H_3PO_4 solutions at 20 °C

% G/V NaCl	γ [mS/cm]	c(H_3PO_4) [mol/L]	γ [mS/cm]
5	65	0.05	7.5
10	113	0.10	10.2
15	148	0.50	32
20	175	1.00	57
25	191	2.00	110
		3.00	165
		4.00	190
		5.00	210

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2. Practical part

2.1 Determination of the cell constant c

Metrohm conductivity cells are delivered with a given cell constant. During the lifetime of a cell, the cell constant may change. For this reason, if precision measurements are to be performed, the cell constant must be checked periodically and redetermined. KCl solutions of known concentration are used for the cell constant determinations. To achieve the necessary precision (weighing error), a stock solution of $c(KCl)$ = 0.1000 mol/L is prepared or purchased. Lower concentrations are produced from this stock solution by dilution with **low-conductivity water** (electrical conductivity $\leq$1 µS/cm).

c(KCl) = 0.1000 mol/L (Metrohm Nr. 6.2301.060)

AR-grade KCl (e.g. Merck no. 104936) is dried for 2 h at 105 °C and left to cool in a desiccator. 7.456 g are dissolved in low-conductivity water and made up to 1000 mL with it. The conductivity of this solution is:

– 11.67 mS/cm at 20 °C

– 12.88 mS/cm at 25 °C

c(KCl) = 0.010 mol/L

10.0 mL $c(KCl)$ = 0.1 mol/L are diluted to 100 mL with low-conductivity water. The conductivity of this solutions is:

– 1.28 mS/cm at 20 °C

– 1.41 mS/cm at 25 °C

c(KCl) = 0.001 mol/L

As its shelf-life is very short, this solution must be prepared immediately before use. The carbon dioxide is driven out of the low-conductivity water before its use by sparging with nitrogen. 10.0 mL $c(KCl)$ = 0.01 mol/L are made up to 100 mL with CO_2-free low-conductivity water. The conductivity of this solution is:

– 133 µS/cm at 20 °C

– 147 µS/cm at 25 °C

Certified conductivity standards covering a wide range (1 µS/cm ... 100 mS/cm) are available, for example, from Reagecon, Ireland (www.reagecon.com).

Example of a determination at 20 °C with the 712 Conductometer

Instruments and accessories:

*   2.712.0010 Conductometer, Magnetic Stirrer
*   Conductivity measuring cell, e.g. 6.0907.110
*   Printer and printer cable, thermostat, nitrogen from pressure cylinder
*   6.1414.010 titration-vessel lid and 6.1418.250 titration vessel with thermostatic jacket
*   6.1440.010 gas inlet and sparging tube

Reagents:

* $c(KCl)$ = 0.0100 mol/L
*   Acetone and low-conductivity water

Procedure:

Immerse the measuring cell in acetone for 30 min. Rinse it with low-conductivity water and store it there for at least 2 h, preferably overnight. Rinse the titration vessel first with $c(KCl)$ = 0.01 mol/L and the fill it up with this solution. Rinse the measuring cell also with $c(KCl)$ = 0.01 mol/L and insert it into the titration vessel. Stir the solution in the titration vessel and adjust its temperature to 20.0 °C by means of a thermostat, sparging nitrogen through the solution for at least 10 min. After this, make the nitrogen pass over the solution. Make sure there are no gas bubbles left inside the measuring cell.

Set the 712 Conductometer's parameters as follows (see also parameter report):

Cell constant	1.0 /cm
Measuring temperature	20.0 °C
Reference temperature	20.0 °C
TC selection	const.
TC const.	0.0 %/°C
Frequency	auto
Measurement type	standard
Conductivity limits	OFF
Standard	1.278 mS/cm

Start the automatic determination at the 712 Conductometer

If no thermostat is available, proceed as follows:

Preparation of the measuring cell and the solutions used are exactly the same as described above. The measurements are also performed in a closed measuring/titration vessel. Instead of thermostatting the solution, connect a Pt 1000 or Pt 100 temperature sensor (6.1110.100 or 6.1103.000). The settings on the 712 Conductometer are also the same except for the temperature coefficient, which is set to **2.11%/°C.**

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The calculation:

New cell constant = nominal conductance / measured conductance

Example:

Nominal conductance = 1.28 mS
Measured conductance = 1.533 mS

c = 1.28 / 1.533 → **c = 0.835 cm^{-1}**

6.0907.110 conductivity measuring cell:

c = AVG(10) = **0.8195 ± 0.007 cm^{-1}**

2.2 Determination of the temperature coefficient of $c(Na_2SO_4)$ = 0.05 mol/L

As mentioned in the theoretical part, the conductivity of ionic solutions is strongly temperature-dependent and this dependency is seldom linear. We therefore recommend to determine the TC in the temperature range of interest automatically with the 712 Conductometer.

Instruments and accessories:

- 2.712.0010 Conductometer
  Magnetic Stirrer

- E.g. 6.0912.110 conductivity cell with built-in temperature sensor (Pt 1000)

- Printer and printer cable, thermostat

- 6.1414.010 titration-vessel lid and 6.1418.250 titration vessel with thermostatic jacket

Reagents:

- $c(Na_2SO_4)$ = 0.05 mol/L:
  Dissolve 7.10 g Na_2SO_4 or 16.11 g Na_2SO_4 x 10 H_2O in low-conductivity water and make up to 1000 mL with it.

- Low-conductivity water

Procedure:

Rinse the conditioned measuring cell with low-conductivity water and Na_2SO_4 solution. Place a sufficient amount of Na_2SO_4 solution in the measuring vessel and insert the measuring cell, making sure that the cell is free of air bubbles.

Set the conductometer's parameters as follows (see also parameter report):

Start temperature	40.0 °C
Stop temperature	15.0 °C
Reference temperature	20.0 °C
Frequency	auto
Temperature coefficient	0.00 %
Cell constant	actual value

Switch on the stirrer and heat the solution to approx. 45 °C with the thermostat. Start the automatic determination and let the solution cool down slowly, i.e not faster than 1 °C/min.

Example:

Measured value:

TC = AVG(8) = **2.35 ± 0.09 %/°C**

2.3 General remarks concerning conductivity measurements

The measuring cell

- The cell constant must be adapted to the measuring solution. A low cell constant is required for low conductivities, a high one for high conductivities:

  – c ≈ 0.1 cm^{-1} for low-conductivity solutions such as partially or completely desalinated water

  – c ≈ 1 cm^{-1} for solutions having moderate conductivity such as drinking water, surface water, ground water and waste water

  – c ≈ 10 cm^{-1} for solutions having a good conductivity such as sea water and rinse water, physiological solutions, etc.

  – c ≈ 100 cm^{-1} for solutions having a very good conductivity such as brines, acids, bases, electroplating baths, etc.

- The measuring cell must be well prepared and conditioned. Measuring cells that have been stored dry are immersed in acetone for 30 min, then rinsed with low-conductivity water and stored in it for at least 2 h, preferably overnight. If used frequently, the measuring cells are stored in low-conductivity water or 20% ethanol (to prevent the growth of microorganisms). Measuring cells that are only used sporadically should be stored dry.

- Contaminated measuring cells cannot be used for measurements any more and must be cleaned (rinse thoroughly with low-conductivity water after cleaning). Possible sources of contamination are:

  – Calcium carbonate or barium sulfate deposits: rinse with HCl. If there are $BaSO_4$ deposits, immerse the measuring cell overnight into a stirred solution of $w(Na_2EDTA)$ = 10% in $c(NaOH)$ = 0.1 mol/L.

  – Fat and oil residues: rinse with acetone. In severe cases treat with ethanolic $c(NaOH)$ = 1 mol/L at approx. 40 °C (saponification).

  – Contamination by algae or bacteria: immerse in hot chromic acid.

  – Proteins: immerse in $w(pepsin)$ = 5% in $c(HCl)$ = 0.1 mol/L for 1 to 2 h under stirring.

The conductometer

- Set the measuring frequency to «auto». This means that the instrument measures with the optimum frequency. The following general rule applies: measure low conductivities with low frequency and high conductivities with high frequency.

- Temperature coefficient (TC): if it is known, set it on the instrument, otherwise it has to be determined. Another possibility is to measure at the reference temperature in a thermostatted solution. In this case the TC does not have to be set.

- Temperature: preferably use a measuring cell with an integrated temperature sensor. Otherwise connect a separate temperature sensor or a thermometer; in the latter case enter the measuring temperature on the instrument.

- Cell constant: enter the cell constant indicated on the measuring cell or redetermine the cell constant.

- Reference temperature: normally 20.0 °C. Some applications prescribe 25.0 °C (water samples or tropical countries, for example). The 712 Conductometer accepts any reference temperature.

The measurement

Use only well-conditioned measuring cells. Preferably rinse the cell with the measuring solution before measurement. Make sure the measuring cell is free of bubbles and immersed deeply enough in the measuring solution. The lateral openings must be fully immersed. Immerse and pull out the cell a few times. Wait for the measuring cell / solution to reach a constant temperature.

2.4 Conductivity measurements in water samples

a) Waste water, ground water, mineral water, surface and drinking water

The usual reference temperature is 25.0 °C. To avoid errors due to faulty setting of the temperature coefficient (TC), it is recommended or prescribed to thermostat the sample solution to 25.0 °C. If this is not desirable, a TC can be entered according to the table below or «TC ident.» can be set to «DIN»*.

Sample temperature [°C]	TC [%/°C]
5 … 10	2.62
10 … 15	2.41
15 … 20	2.23
20 … 25	2.08
25 … 30	1.94
30 ... 35	1.79

* Refers to water samples that contain mainly calcium and hydrogen carbonate ions and only small amounts of magnesium, sulfate, chloride and nitrate ions.

Examples:

Tape water Herisau, 25 °C:

AVG(10) = **512.5 ± 8.3 µS/cm**

Mineral water, 25 °C:

AVG(10) = **1.813 ± 0.013 mS/cm**

b) Demineralized water

The interferences possible with these samples demand a special procedure for water samples with conductivities <5 µS/cm and especially for those with conductivities <1 µS/cm! The main interferences are:

- Entrainment of CO_2 (or other gases that increase conductivity) from ambient air.

- Leaching of Na and Ca traces from the glassware.

In both cases there results a slow adjustment of the measuring value and too high, i.e. faulty conductivities are obtained. To eliminate these interferences, we recommend to proceed as follows:

Variant 1:

Flow-though measurement. Because of the small volume of the measuring setup we recommend to screw the 6.0912.110 conductivity measuring cell (PP shaft, integrated Pt 1000 temperature sensor) into the 6.1420.100 flow-through vessel. Let the water flow through the setup and determine the conductivity in the usual manner.

Variant 2:

Use as large a volume as possible for the measurement. Pass nitrogen or argon through and over the solution and stir it. Whenever possible work with a closed or covered vessel.

Please note the high TC (approx. 5.8 %/°C) of these water samples!

2.5 Salinity

In certain applications it is not the conductivity that is of primary interest but the total dissolved salt content. A separation into the individual ions is not possible with conductivity measurement as each ion species contributes differently to the total conductivity. For this reason, in these determinations, the conductivity of the sample solution is considered to be due to an NaCl solution. The 712 Conductometer performs this conversion automatically if the measurement type **TDS** (**T**otal **D**issolved **S**olids) is selected. The TDS value is displayed on the 712 Conductometer as mg/L or g/L NaCl in lieu of the cell constant.

Examples:

Tap water Herisau, 25 °C:

AVG(10) = **245.6 ± 4.2 mg/L NaCl**

Mineral water, 25 °C:

AVG(10) = **894.8 ± 6.7 mg/L NaCl**

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2.6 Conductometric titration

Conductometric titrations usually do not require knowledge of the cell constant. A double Pt-sheet electrode serves as the measuring electrode for the titration, which is normally carried out in a glass beaker. This electrode is easy to clean. Thermostatting is not required for simple titrations – work is done at room temperature. Changes in conductivity due to temperature fluctuations are negligible.

Instruments and accessories:

- 2.712.0010 Conductometer
- Titrino or Titrando with Dosino or Dosimat and with Magnetic Stirrer
- Exchange Unit
- 6.0309.100 double Pt-sheet electrode with 6.2104.080 electrode cable
- 6.2116.000 connecting cable 712 – Titrino
- PC with connecting cable to the Titrino plus printer and printer cable
- For example Metrodata VESUV 3.0 Light for the manual evaluation of L- and V-shaped titration curves (VESUV = Metrodata Application Software; **Ve**rification **Su**pport for **V**alidation).

Reagents:

- $c(AgNO_3)$ = 0.01 mol/L as titrant (in the Exchange Unit)
- $c(KCl)$ = 0.1000 mol/L, e.g. Metrohm no. 6.2301.060
- Low-conductivity water and acetone

Procedure:

Interconnect the instruments as described in the 712 Instructions for Use. Transfer 0.25 mL $c(KCl)$ = 0.1 mol/L and 30 mL each of acetone and low-conductivity water into the beaker. Immerse the electrode and the buret tip and start the titration with $c(AgNO_3)$ = 0.01 mol/L after having set the parameters as follows:

712 Conductometer:

>cond/parameters

cell constant	1.0 /cm
meas.temp.	20.0 °C
ref.temp.	20.0 °C
TC selection:	const.
TC const.	0.0 %/°C
frequency:	auto
meas.type:	titration
cond/limits	OFF

>cond/analog output

status:	ON
polarity	+
1 V range:	1 mS/cm
0 V at:	0 µS/cm
offset:	0 mV

>cond/limits

status:	OFF

>cond/plot margins

left:	0 µS/cm
right:	1000 µS/cm

799 GPT Titrino:

MET U   cond. titr.

>titration parameters

V step	0.10 mL
dos.rate	max. mL/min
signal drift	50 mV/min
equilibr.time	26 s
start V:	OFF
pause	0 s

>stop conditions

stop V:	abs.
stop V	4.0 mL
filling rate	max. mL/min

>evaluation

EP criterion (EPC)	30 mV
EP recognition:	all

Example of a titration curve (VESUV 3.0):
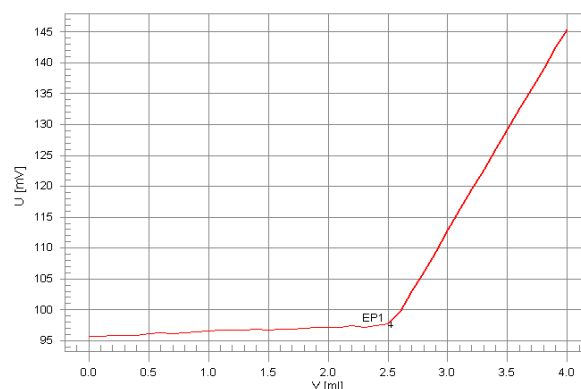
Evaluation:

L- and V-shaped titration curves are evaluated as follows:

Fit tangents to the two branches of the curve. The endpoint of the titration is defined by the intersection of the tangents.

With Metrodata VESUV 3.0, proceed as follows:

Move tangent 1 with the <CTRL> key and the left mouse key, tangent 2 with the <Shift> key and the left mouse key along the curve.

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