

Robotic Transfer Analyzer



Applications

8.855.2043

 **Metrohm**
Ion analysis

Metrohm AG
CH-9101 Herisau
Switzerland

Robotic Transfer Analyzer



Symbols used in this document:

$c(X)$	Molar concentration of substance X in mol/L, often written as $[X]$
$M(X)$	Molar mass of substance or atomic mass (relative mass) of substance X in g/mol
$w(X)$	Mass fraction of substance X, e.g. $w(\text{NaOH}) = 40\%$
$\beta(X)$	Mass concentration of substance X, e.g. $\beta(\text{NaCl}) = 20 \text{ g/L}$
$a(X)$	Activity of substance X (only corresponds to the molar concentration in very diluted solutions; as the concentration increases, dissolved particles mutually influence each other so that their activity is lower than would be expected from the concentration).
$\text{p}K_{\text{p}}$	Autoprotolysis constant of a solvent
{ }	In this document braces such as used in the formula $\{\text{AgCl}\}$ indicate solid substances (precipitates) that do not consist of single molecules (in this case AgCl), but whose ionic components form an extended ionic lattice.

***tiamo* methods**

The methods described in this document are part of the *tiamo* installation on your PC system. To use these methods you can import them into your method groups with the built-in method manager in the *tiamo* software. Please consult the *tiamo* user manual or online-help for further instructions.

The methods can be found in the following path:

C:\Program Files\Metrohm\tiamo\examples\methods\english\855 Robotic Analyzer\Transfer

(The *tiamo* installation path of your system may be different.)

The *tiamo* user manual (in pdf file format) can be found in the following path:

C:\Program Files\Metrohm\tiamo\doc\English

Accessories of the Robotic Transfer Analyzer

1.855.0020	Robotic Titrosampler 1T/2P-V
1.800.0010	Dosino, 2x
1.802.0010	Rod Stirrer
1.804.0010	Ti Stand
6.0253.100	LL-Aquatrode plus OK
6.1414.060	Micro titration vessel lid ECO
6.1415.220	Titration vessel
6.1432.320	Sample beaker 250 mL
6.1462.040	Robotic arm (transfer)
6.1543.120	Aspiration tip M8
6.1546.030	Piston rod
6.1562.120	Pipetting tube 10 mL, with holder
6.1608.023	Bottle 1 L, 2x
6.1621.000	PE container 10 L, 2x
6.1805.060	FEP tubing / M6 / 60 cm, 3x
6.1805.120	FEP tubing / M6 / 100 cm
6.1805.510	PTFE tubing M8, 60 cm
6.1812.000	PTFE tubing 4/6 mm, 4m
6.1815.010	Spiral band 0.5 m
6.1823.010	Guiding shaft to Pipetting tubing
6.1828.000	PVDF connection nipple, 2x
6.1909.020	Stirring propeller 104 mm
6.2001.070	Support stand
6.2001.110	Bottle holder base, left side
6.2013.010	Clamping ring, 2x
6.2041.410	Sample rack 141 x 11 mL + 1 x 500 mL
6.2053.000	Cable clip, 10x
6.2061.010	Bottle holder
6.2104.020	Electrode cable 1 m
6.2151.000	Cable USB A - mini-DIN 8P
6.2308.050	KCl solution 3 mol/L (50 mL)
6.2323.000	Storage solution
6.2621.030	Hexagon key 4 mm
6.2621.070	Hexagon key 5 mm
6.2621.130	Hexagon key 2 mm
6.2621.140	Hexagon key 2.5 mm
6.2709.090	Stopper 12 mm, 2x

6.2711.070	Drip pan
6.2730.030	Stopper with nipple M10, 5x
6.2730.060	Screw nipple M16/12 mm, 2x
6.2730.080	Screw nipple M16/SGJ14
6.2740.020	Spray nozzle, 3x
6.2743.057	Sample tubes 11 mL/200 pieces, 2x
6.3032.210	Dosing Unit 10 mL
6.3032.220	Dosing Unit 20 mL
6.6056.112	<i>tiamo</i> 1.1 full version CD
E.3010.022	O-rings 5,28/1,78, 5x
T.2400.102	Ferrite cores, 4x

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1 Application Information

1.1 Introduction: titration means counting!

- Titrations are easy to carry out: The equipment and the procedures to be performed are simple. They are easy to understand – the fundamentals of titrimetric methods are widely known or can be learned in a short time.
- Titrations are carried out rapidly: If the total time for setting up the workplace to obtaining the analytical result is taken into consideration, then titrimetric determinations require much less time than other methods.
- Titration is a versatile method: Numerous titration methods have been drawn up, these range from the determination of inorganic ions up to the determination of complex organic compounds. The analyte concentrations can range from 100% or virtually 100% (analysis of ultrapure substances, purity determinations) down to the ppm range. Sample amounts of a few micrograms are adequate, amounts in the gram range are also possible.
- Titration supplies highly reproducible and correct results. A typical reproducibility is <1%. In high-precision titrations values of 0.1% are demanded and also achieved. For such demands the accuracy should lie within the limits of the standard deviation.
- Titrations can be automated: Titrimetric determinations can be automated to a high degree. This means that, for example, they are suitable for analyzing the content in routine operation (e.g. active ingredient content in tablets).

1.2 Acid / Base reactions

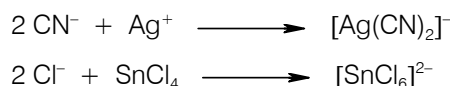
The term acid-base has been revised several times since the 18th century in accordance with the state of knowledge:

- Acids contain oxygen (Lavoisier)
- Acids contain hydrogen that can be replaced by a metal (Liebig)
- Acids dissociate in aqueous solution to release protons and bases release hydroxide ions (Arrhenius, Ostwald)

These theories can only partly explain chemical reactions. They refer to aqueous solutions; ampholytes are not included. Examples are aqueous solutions of salts: KHSO_4 reacts acidic, Na_2HPO_4 alkaline.

Brønsted defined the acid-base reaction as the exchange of protons. The *acid* acts as *proton donor* and the *base* as *proton acceptor*. A deprotonated acid is called the conjugate base and a protonated base is called the conjugate acid. In this way the theory can also be applied without any problem to ampholytes, salts and prototropes of non-aqueous solvents. The equilibrium constants of the reactions allow a quantitative reactivity series of acids and bases to be drawn up (see below).

Lewis extended the Brønsted theory (Brønsted theory). A Lewis base has a free electron pair, a Lewis acid an electron **pair gap**. Lewis acid and Lewis base react to form a covalent or coordinative compound. Examples:



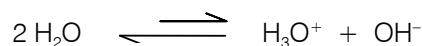
Typical Lewis acids are molecules with an incomplete valency shell, cations as the central atoms of complexes and molecules with multiple polar bonds (e.g. acid anhydrides). Typical Lewis bases are molecules and ions with free electron pairs, anions as complex ligands and molecules with multiple bonds. The Lewis model can also be used to describe precipitation and complex-forming reactions. As no universal reference acid or base exists, there is no

practical quantitative reactivity series for Lewis acids and bases; however, the Lewis conception allows numerous chemical reactions to be systemized qualitatively.

Protolysis

Polar (prototropic, protic) solvents undergo self-dissociation, i.e. they form an acid-base pair to a slight extent.

Example water:



Neither free protons nor free electrons are found in a solution. For protons this means that H^+ should not be used as an expression, but rather H_3O^+ to represent the clusters of protons and water effectively present in an aqueous solution.

According to the law of mass action, the ionic product of water K_w is calculated as follows:

$$a(\text{X}^+) \times a(\text{Y}^-) / a^2(\text{XY})$$

for H_2O this means:

$$a(\text{H}_3\text{O}^+) \times a(\text{OH}^-) / a^2(\text{H}_2\text{O}) = K_w = 10^{-14}$$

K is temperature-dependent. In tables it is usually given for 25 °C (the values for H_2O are 14.9 at 0 °C, 14.2 at 20 °C, 14.0 at 25 °C, 13.5 at 40 °C, etc.).

The relative «strength» of an acid or base in an aqueous solution is described by the equilibrium constants. These provide information about the extent to which an acid or base dissolved in water at equilibrium can accept or donate protons. Water is the reference base for each acid and the reference acid for each base. These acidity and basicity constants are known as K_a and K_b respectively. We have simplified their formulation here by using the molar concentration instead of the activities of the participating species. The molar concentration of water in a mixture is regarded as being constant for diluted solutions (55.5 mol/L) and not included.

$$K_a = [\text{H}_3\text{O}^+] \times [\text{A}^-] / [\text{HA}]$$

$$K_b = [\text{BH}^+] \times [\text{OH}^-] / [\text{B}]$$

The negative common logarithms of these values are, analogous to the pH definition, known as $\text{p}K_a$ and $\text{p}K_b$ respectively. The higher the $\text{p}K$ value, the weaker the acid or base.

The neutral point of a non-aqueous solvent is calculated in a similar manner to that for water. Clearly, the pH term only applies to purely aqueous solutions!

$$\text{H}_2\text{O, neutral pH} = -\log \sqrt{K_w} = -\log \sqrt{10^{-14.0}} = +7.0$$

The following table shows a small selection of autoprotolysis constants of prototropic solvents (at 25 °C).

Solvent	Autoprotolysis	$\text{p}K_p$
Ethanol	$2 \text{C}_2\text{H}_5\text{OH} \rightleftharpoons \text{C}_2\text{H}_5\text{OH}_2^+ + \text{C}_2\text{H}_5\text{O}^-$	19.1
Methanol	$2 \text{CH}_3\text{OH} \rightleftharpoons \text{CH}_3\text{OH}_2^+ + \text{CH}_3\text{O}^-$	16.7
Acetic acid	$2 \text{CH}_3\text{COOH} \rightleftharpoons \text{CH}_3\text{COOH}_2^+ + \text{CH}_3\text{COO}^-$	14.5
Water	$2 \text{H}_2\text{O} \rightleftharpoons \text{H}_3\text{O}^+ + \text{OH}^-$	14.0
Formic acid	$2 \text{HCOOH} \rightleftharpoons \text{HCOOH}_2^+ + \text{HCOO}^-$	6.2
Sulfuric acid	$2 \text{H}_2\text{SO}_4 \rightleftharpoons \text{H}_3\text{SO}_4^+ + \text{HSO}_4^-$	3.6

Acid-base reactions with water

In aqueous solutions the protolysis reaction with water normally predominates (as an ampholyte H_2O can act as a Brønsted acid or a Brønsted base):

acidic reaction of solvent



basic reaction of solvent



Just as with the ionic product of water, $a(\text{solvent})$ is set = 1 for dilute solutions. The resulting equilibrium constants are known as the *acidity constant* K_a and the *basicity constant* K_b .

$$K_a \times K_b = K_w$$

$$\text{p}K_a + \text{p}K_b = \text{p}K_w = 14 \text{ (25 }^\circ\text{C)}$$

The stronger the acid or base, the weaker the conjugate base or acid.

At the same time the $\text{p}K$ values of the aqueous system limit the acidic or basic strength of protolytes in aqueous solution (differentiation).

Stronger acids and bases ($\text{p}K < 0$ or $\text{p}K > 14$ for the conjugate protolyte) are converted to oxonium ions (H_3O^+) and hydroxide ions (OH^-) in aqueous solution. *Leveling* occurs – the separate determination of mixed very strong acids or mixed very strong bases in an aqueous solution is not possible by titration. (Only one endpoint is found in the titration of mixtures of e.g. KOH/NaOH or $\text{HCl/H}_2\text{SO}_4$).

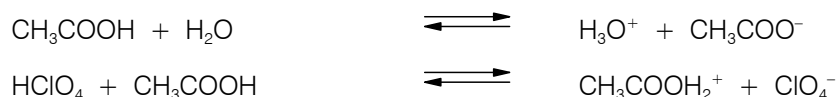
The known corresponding acid-base pairs are arranged according to increasing $\text{p}K_a$ (or decreasing $\text{p}K_b$ value) and normally divided into the following categories:

- very strong $\text{p}K_a < 0$
- strong $\text{p}K_a 0 \dots 4$
- weak $\text{p}K_a 4 \dots 10$
- very weak $\text{p}K_a 10 \dots 14$
- extremely weak $\text{p}K_a > 14$

Protolytes

As a result of the leveling effect of water, differences in the protolysis constants of strong protolytes can only be determined in non-aqueous solvents. In such solvents the relationship $\text{p}K_a + \text{p}K_b = \text{p}K_{\text{solvent}}$ also applies for the conjugate acid-base pair. This means that the solvent cation (*lyonium ion*) and the solvent anion (*lyate ion*) are always the strongest acids and bases in the affected system.

Whereas acetic acid behaves in water like a weak acid, in HClO_4 it behaves like a base:



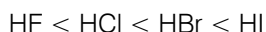
In order to be able to achieve the separate determination of the components in an aqueous solution by titration, the differences in their $\text{p}K$ values should be approx. 5 units. In contrast, in suitable non-aqueous solvents a difference of 2 to 3 $\text{p}K$ units is normally sufficient.

pK _a values of some selected acids		pK _b values of some selected bases	
Acids	pK _a	Bases	pK _b
Acetic acid	4.73	2-Picoline	7.52
Acrylic acid	4.26	Acridine	9.89
Benzoic acid	4.20	Ammonia	4.75
Boric acid	9.24	Aniline	9.42
Chloroacetic acid	2.81	Benzylamine	4.62
Hydrobromic acid	≈ -6	Calcium hydroxide	1.30
Hydrochloric acid	≈ -3	Coffeine	13.39
Hydrocyanic acid	9.40	Cyclohexylamine	3.36
Hydrofluoric acid	3.14	Diethanolamine	5.12
Hydrogen sulfide acid 1 st stage	6.90	Ethanolamine	4.56
Hydrogen sulfide acid 2 nd stage	12.9	Ethylamine	3.33
Lactic acid	3.86	Imidazole	7.00
Nitric acid	1.32	Lithium hydroxide	-0.10
Oxalic acid 1st stage	4.31	Magnesium hydroxide	3.36
Oxalic acid 2nd stage	1.42	Naphthylamine	10.08
Perchloric acid	≈ -9	o-aminobenzoic acid	10.92
Phenol	9.95	o-toluidine	9.61
Phosphoric acid 1st stage	1.96	Piperidine	2.80
Phosphoric acid 2nd stage	7.12	Pyridine	8.81
Phosphoric acid 3rd stage	12.36	Triethanolamine	6.23
Salicylic acid	2.98	Triethylamine	3.28
Sulfuric acid 1st stage	≈ -3	Trimethylamine	4.20
Sulfuric acid 2nd stage	1.92	Urea	13.8

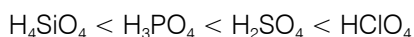
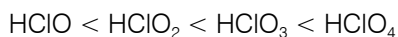
The strongest acid in the list is perchloric acid, the strongest base is lithium hydroxide.

The following empirical rules apply for the *relative acid strength* of inorganic acids:

- *Hydracids*: the acidity increases as the atomic number increases, both within the group and within the period. (This means that HI is the strongest acid.):



- *Oxyacids* are stronger the fewer H atoms and more O atoms they contain. (The strongest mono-oxyacid is HClO₄).



1.3 Acid-base titrations

Acid-base reactions occur very rapidly (proton transfer takes far less than one millionth of a second).

In mixtures of acids the titration is always carried out in the sequence of the relative acid strengths. This means that the strongest acid is always titrated first, the weakest acid last.

Example:

The titration curve of a mixture of HCl and CH₃COOH shows two equivalence points/endpoints. The consumption up to EP1 corresponds to the molar concentration of HCl and the consumption for the difference EP2 – EP1 to the molar concentration of acetic acid.

Titration of strong protolytes (strong acid with strong base and vice versa)

The salts of strong acids with strong bases do not undergo protolysis with water, which means that an equivalent amount of these acids and bases react neutrally. The equivalence

point in the titration of a strong acid with a strong base and vice versa is therefore identical with the neutral point ($\text{pH} = 7.0$ in H_2O).

The shape of the titration curve results from the fact that in order to increase the pH from 1 to 2 it is necessary to reduce the H_3O^+ concentration to 1/10 of the original value (or that 90% of the equivalent base has already been added). From $\text{pH} = 2$ to $\text{pH} = 3$ it is then 9%, from $\text{pH} = 3$ to $\text{pH} = 4$ then 0.9%, etc. With the excess base it is exactly the opposite; from $\text{pH} = 7$ to $\text{pH} = 8$ 0.0009% is necessary, from $\text{pH} = 8$ to $\text{pH} = 9$ requires 0.009%, from $\text{pH} = 9$ to $\text{pH} = 10$ it is 0.09% excess base, etc.

This is why typical symmetric titration curves with a steep «jump region» are obtained, whose equivalence point (titration endpoint, corresponds to the point of inflection of the curve) lies at $\text{pH} = 7.0$.

Titration of a weak acid with a strong base

Before the start of the titration it can be seen that the pH is acidic, which corresponds to the weak acid not being completely dissociated in water. After the start of the titration a buffering range (10...90% of neutralization) can be observed in which the weak acid and its salt or acid anion are both present. In this buffering range the addition of the base only alters the pH slightly. The point of inflection of the buffering range corresponds to the semi-equivalence point of the titration (half reaction) and therefore, according to the Henderson-Hasselbalch equation, the pK_a value of the weak acid is given by: $\text{pH} = \text{pK}_a + \log \frac{[\text{A}^-]}{[\text{HA}]}$, where at half reaction the molar concentrations of acid anion and acid are equal – resulting in $\log 1$, which is equal to 0.

In the above case the equivalence point no longer lies at $\text{pH} = 7$, because the added base competes with the conjugate base of the acid. At the equivalence point the *salt solution* present has an *alkaline* reaction, as the acid anions of a weak acid undergo protolysis with water (hydrolysis). The equivalence point of such a titration lies – depending on the relative strength of the acid – in the pH range 7.5...10.

The higher the concentration of acid and base in the solution, the wider the buffering range on the volume axis. The pH value achieved by adding additional base beyond the equivalence point finally approaches that of the base used as the titrant.

Titration of a weak base with a strong acid

Before the start of the titration an alkaline pH is measured that corresponds to that of the not fully dissociated weak base in water. After the start of the titration a buffering range can also be observed (10...90% of the neutralization), in which the weak base and its conjugated protonated form are both present. Addition of acid hardly alters the pH in this buffering range at all. The point of inflection of the buffering range corresponds to the semi-equivalence point of the titration (half reaction) and lies at that pH value which corresponds to the pK_b value of the base. The equivalence point is again no longer at $\text{pH} = 7$, because the added acid competes with the conjugate acid of the base. The corresponding salt solution is acidic as the protonated base undergoes protolysis with water (hydrolysis). The equivalence point lies – depending on the relative strength of the base – in the pH range 6.5...4.

Titration of a weak acid with a weak base

This titration is unsuitable for quantitative determinations! There is no marked jump in the titration curve, and the slope of the curve in the equivalence region does not achieve a maximum value. No suitable titration endpoint for a quantitative determination is reached. This means that, when selecting a titrant for the titration of weak acids and bases, weak bases and acids should not be chosen.

Titration curve

The following applies in general: The «jump» of a titration curve is more marked the higher the K_a or K_b values of the two reaction partners and the higher the concentrations of both species.

1.4 Introduction to pipetting sequences

Pipetting equipment

Together with an 800 Dosino as dosing drive (or "pipetting pump"), the Sample Processor is exceedingly suitable for pipetting liquids in a volume range from 0.5 to 9.5 mL.

Pipetting procedure

Basic principles

Measuring out the liquid to be pipetted takes place in a pipetting tubing filled with a carrier liquid (preferably water). In order to prevent any carryover or mixing an air bubble (air gap) must be inserted between the sample and the carrier liquid.

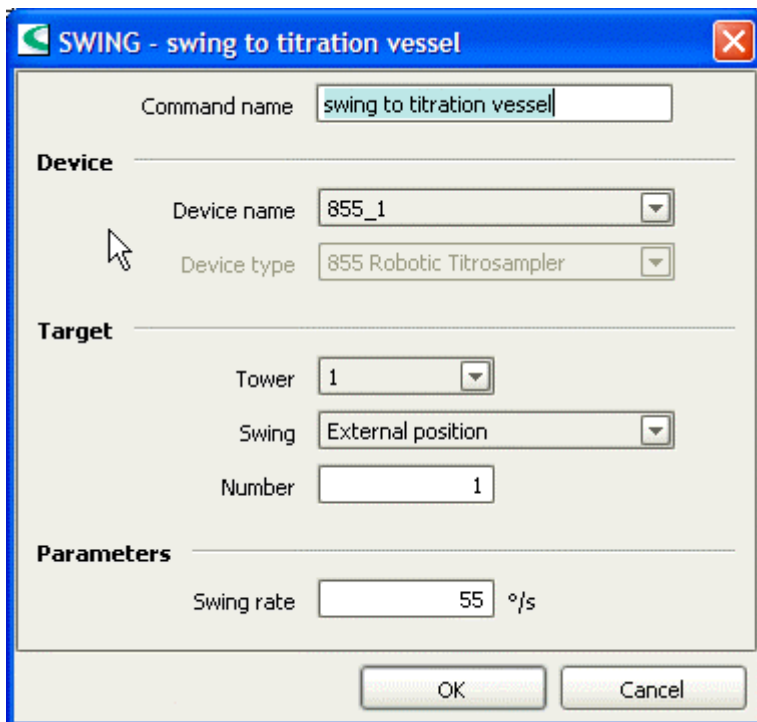
A Dosino drive is used to transfer the liquids into the pipetting tubing. The pipetting tubing is connected to Port 1 of the Dosing unit. The Dosing unit is mounted on a reagent bottle containing the carrier liquid. This can be refilled via filling port 2 of the Dosing unit. The carrier liquid can also be used for rinsing and diluting the sample.

Various pipetting procedures are possible. The following conditions must always be observed:

- The sample must always be measured out in the pipetting tubing; it must never enter the Dosing unit cylinder.
- The sample should always be aspirated after the dosing cylinder has been ejected.
- The difference in level between the surface of the sample liquid and that of the carrier liquid in the cylinder should be as small as possible.
- Aqueous sample solutions can be pipetted without having to rinse the tubing. Non-aqueous samples require the pipetting tubing to be rinsed with carrier liquid (same solvent as the sample), to avoid carryover.

Preparing the Dosing unit and the titration vessel

The Dosing unit must first be prepared, i.e. the filling tubing and pipetting tubing must be rinsed and completely filled. The cylinder contents must then be ejected.



SWING - swing to titration vessel

Command name: swing to titration vessel

Device

Device name: 855_1

Device type: 855 Robotic Titrosampler

Target

Tower: 1

Swing: External position

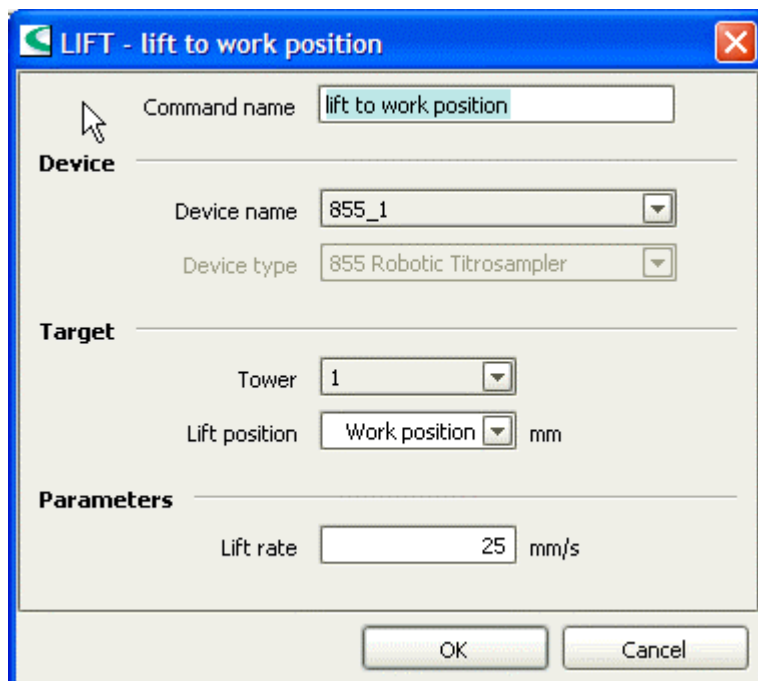
Number: 1

Parameters

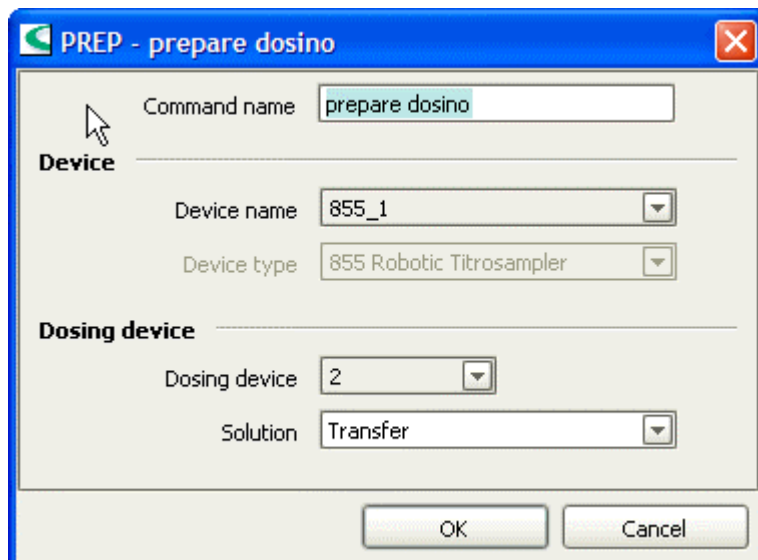
Swing rate: 55 %/s

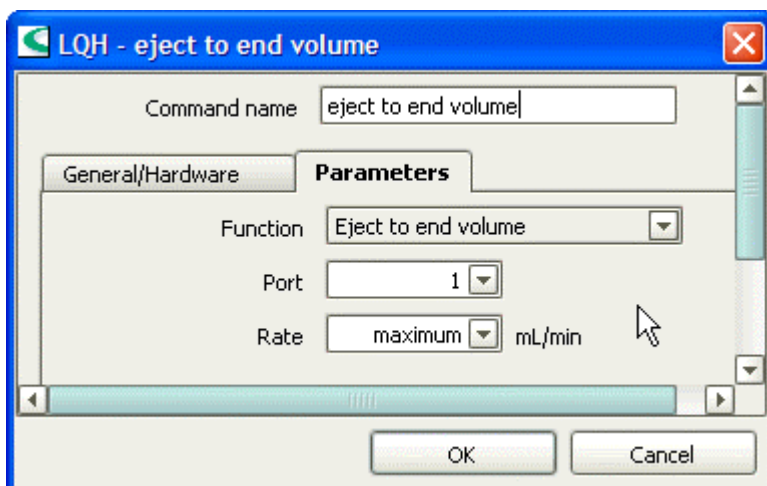
OK Cancel

The robotic arm is moved to the external titration vessel.

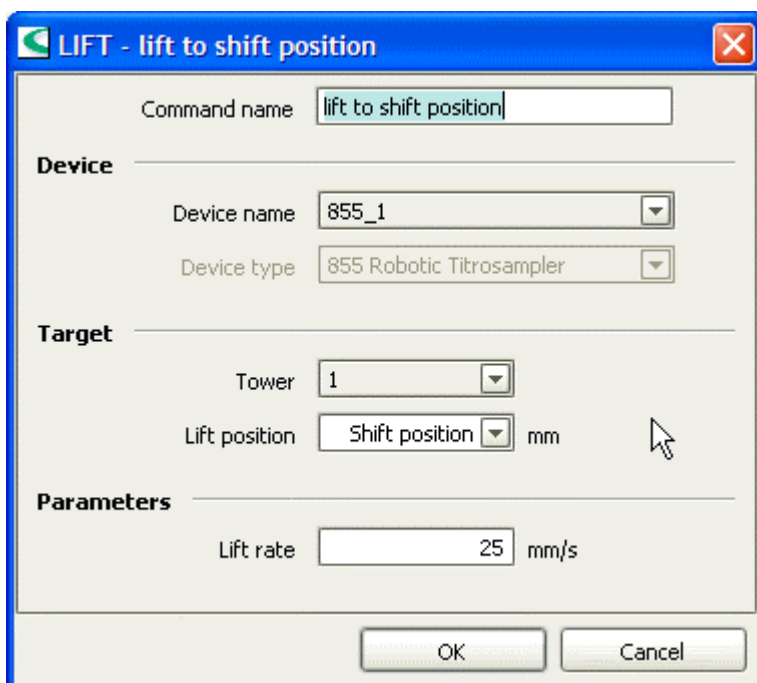


The robotic arm is lifted down to the work position where a preparation command for the Dosino is executed.





After the preparation step the complete cylinder content is ejected to end volume using Port 1. Then the tower is lifted to shift position.



These steps should be carried out before every sample series involving pipetting. Before each pipetting process the Dosing unit cylinder contents must be completely ejected. This is the only way to achieve reproducible, accurate pipetting.

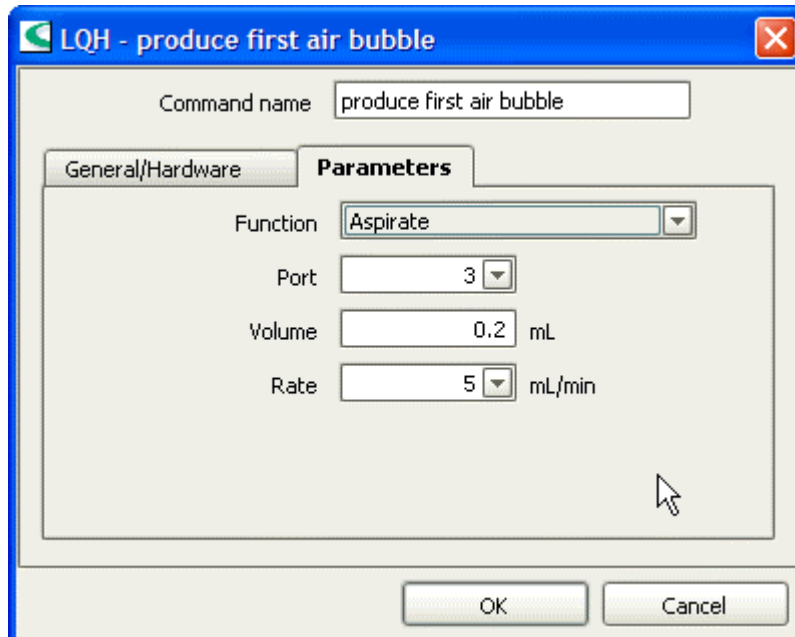
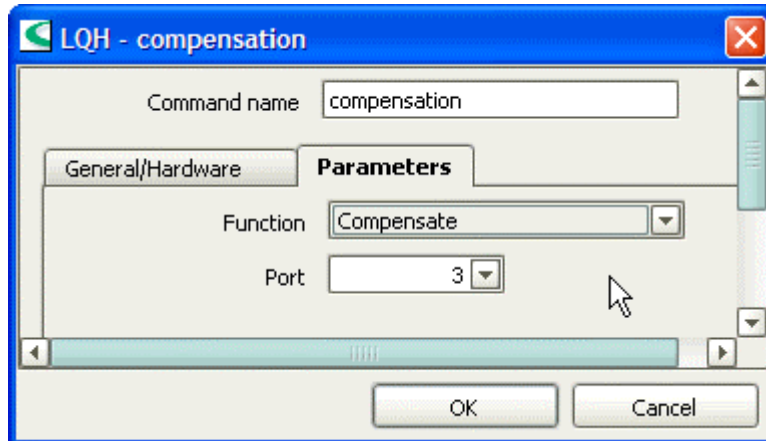
The titration cell can then be aspirated, rinsed and prepared with fresh solvent.

Pipetting

The pipetting procedure takes place in five steps:

- Ejection of cylinder contents (see above) and formation of separating bubble
- Move to sample
- Aspirate / Measure out sample
- Move to target
- Eject sample

Formation of separating bubble (air gap)



In order to prevent the carrier liquid and the sample solution from mixing, a separating air bubble must be formed that occupies a length of at least 5 mm in the pipetting tubing. However, the separating air bubble must also be measured out with sufficient accuracy. This is why a Dosing unit with max. 10 mL cylinder capacity should be used for pipetting.

Move to sample

MOVE - to sample

Command name

Device

Device name

Device type

Target

Tower

Move

OK Cancel

The robotic arm is then moved to the sample position.

LIFT - lift to work position

Command name

Device

Device name

Device type

Target

Tower

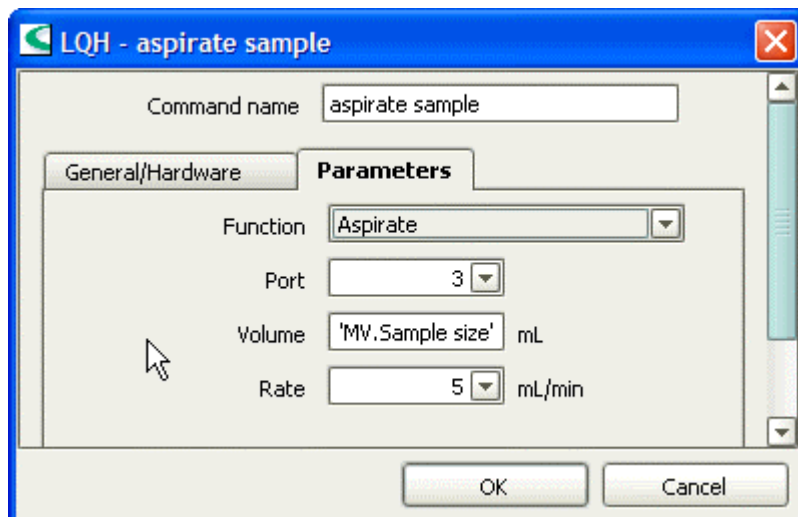
Lift position mm

Parameters

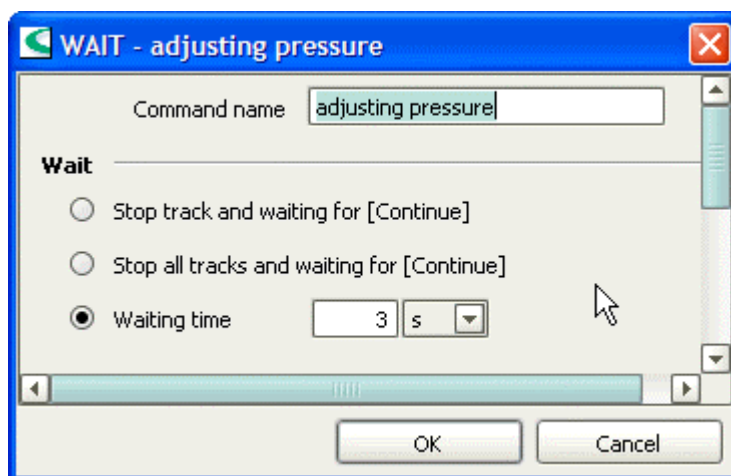
Lift rate mm/s

OK Cancel

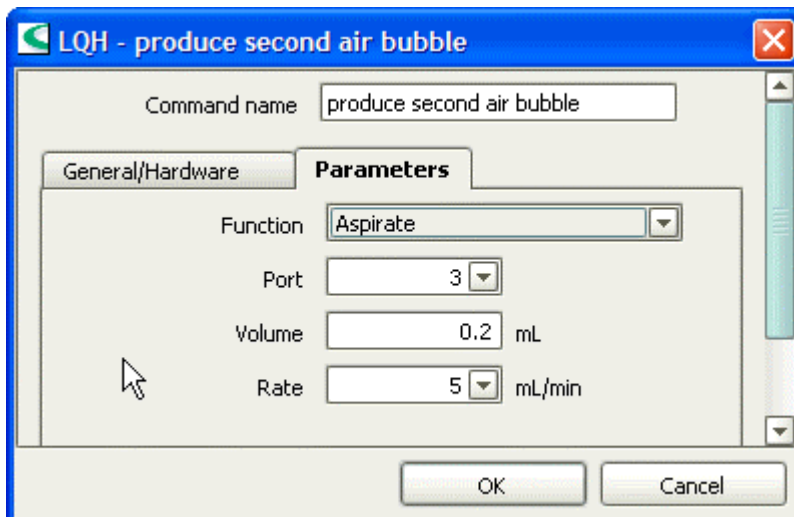
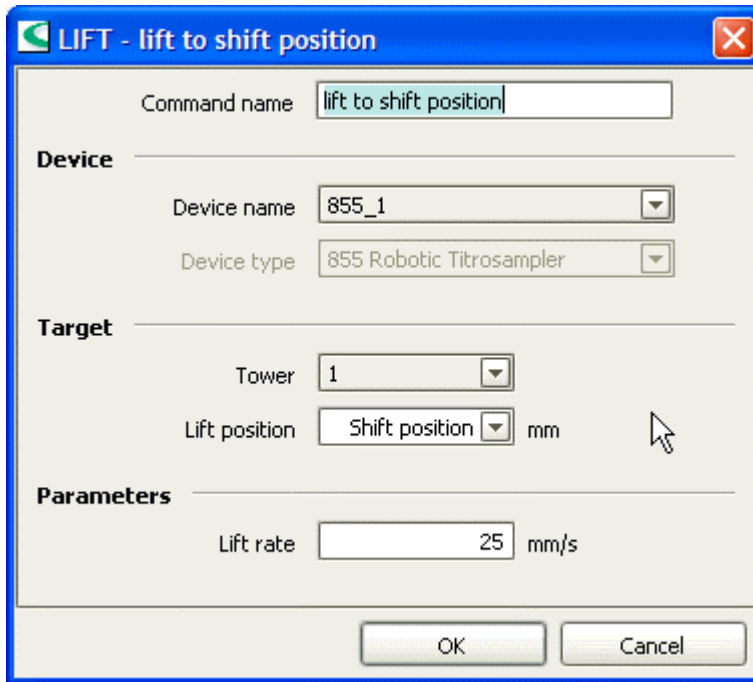
Aspirate sample



Aspiration of the sample should be carried out at a reduced filling rate (<10 mL/min).

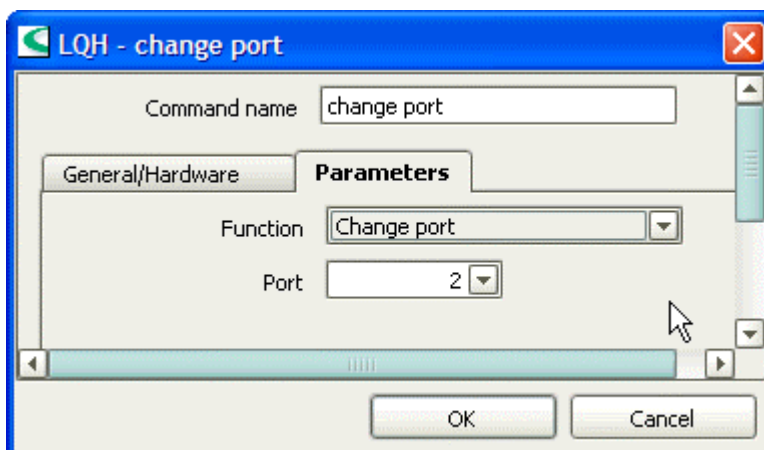


The system needs approximately 3 seconds for adjusting the pressure after the aspiration step. The waiting time can vary depending on the sample properties.

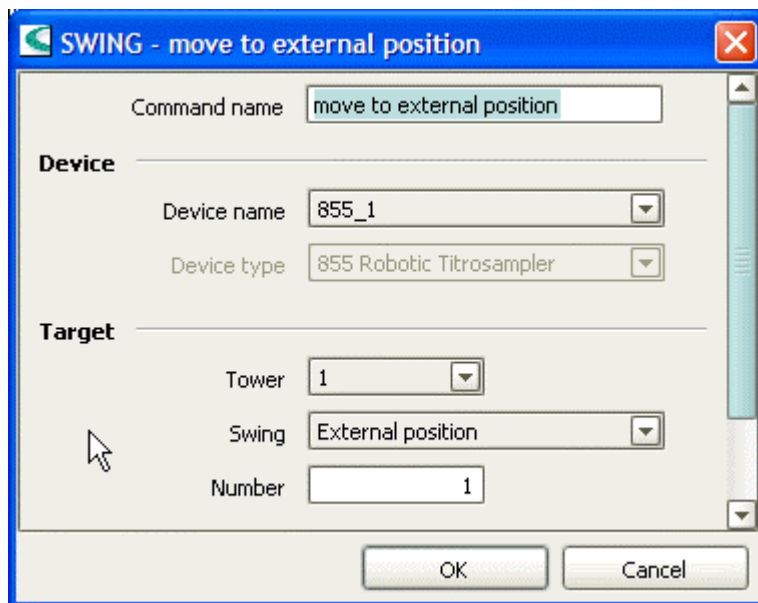


Especially with non-aqueous samples an additional small air bubble may be drawn into the pipetting tip in order to prevent the sample solution from dripping when moving from sample beaker to the titration vessel.

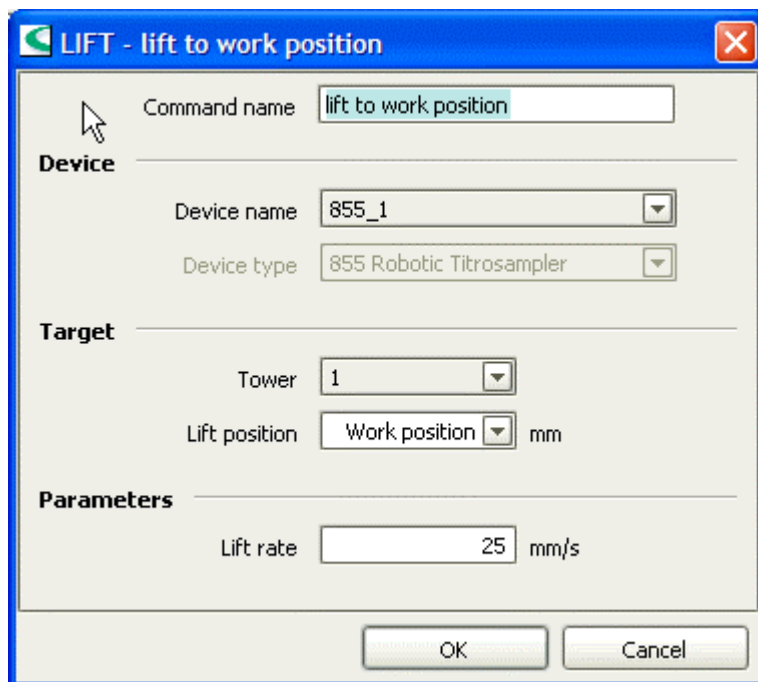
Move to target



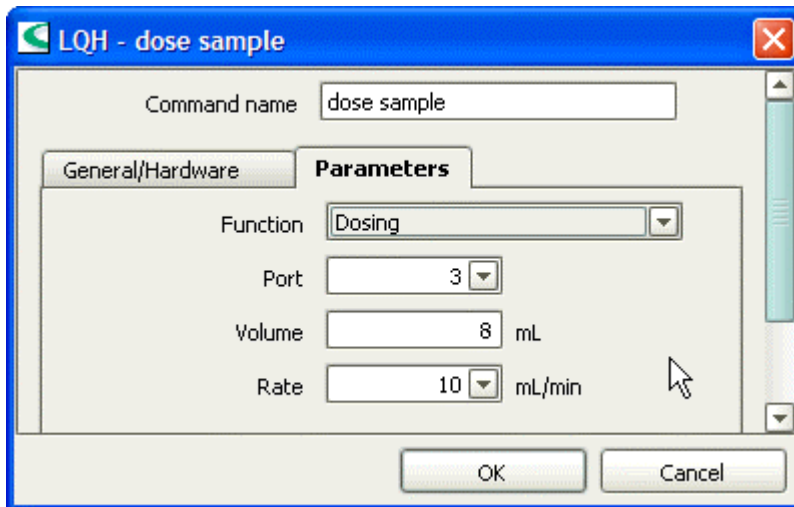
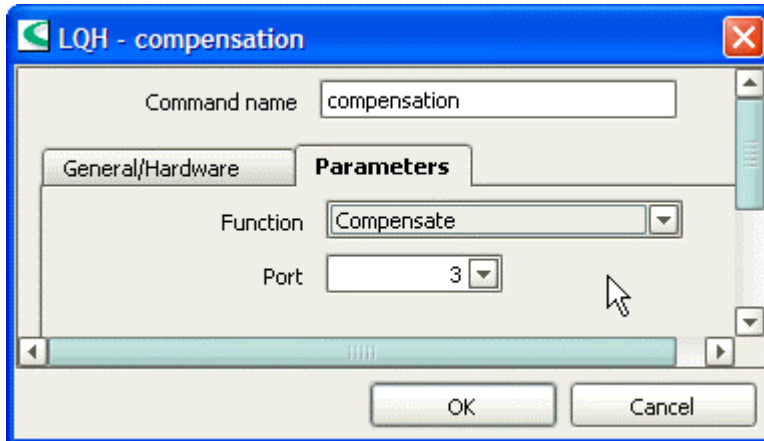
Before the pipetting tubing containing the measured out sample is moved, the corresponding Dosino port must be closed.



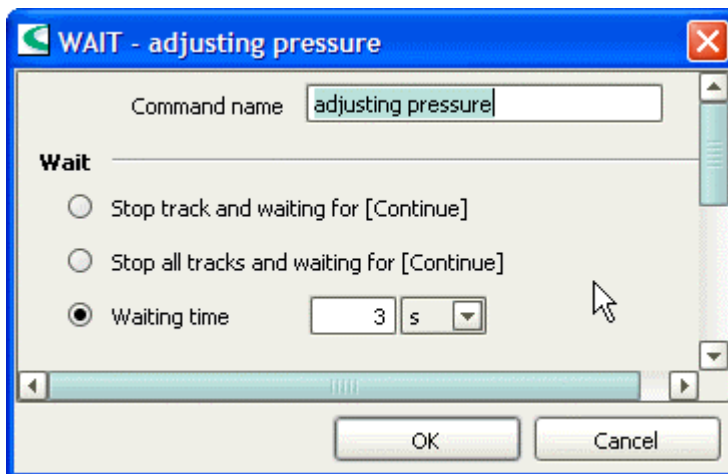
The titration cell should be filled with solvent before the sample is added so that the sample can be pipetted directly into the liquid (tip immersed).



Eject sample

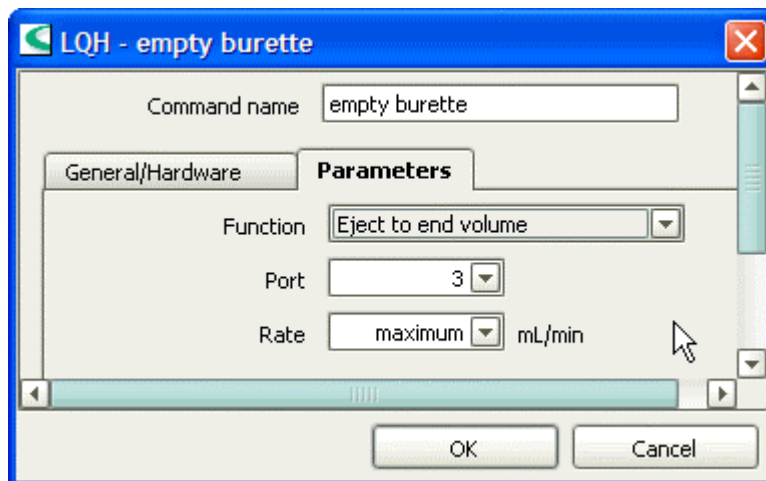


The sample should be dosed at a reduced dosing rate (<10 mL/min).



Again the system has to adjust the pressure.

Rinse



Before the next sample can be pipetted the Dosing Unit has to be prepared. For that the cylinder is emptied and a compensation command is carried out.

In order to keep sample solution carryover as low as possible, rinsing with solvent can be carried out after the sample has been ejected. This is particularly recommended for non-aqueous samples.

In preparation for the next pipetting process the cylinder should be completely ejected.

2 Calibration

2.1 Calibrating the pH glass electrode

Recommended accessory

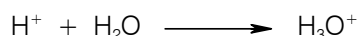
- Comb. pH glass electrode with built-in Pt 1000 temperature sensor, e.g. 6.0257.000 Aquatrode Plus

Reagents

- Buffer solutions pH = 4.00, 7.00 and 9.00, e.g. 6.2307.100, 6.2307.110 and 6.2307.120

General

Free protons (H^+ ions) occur in solutions just as little as free electrons. They combine with water to form oxonium ions:



The pH value is defined as the negative logarithm of the oxonium ion activity, i.e. of the concentration of free, dissociated oxonium ions in mol/L: $\text{pH} = -\log [\text{H}_3\text{O}^+]$

Strictly speaking, the term pH only applies to purely aqueous solutions.

The pH scale ranges from 0 to 14 with the neutral point at $\text{pH} = 7.0$, where the H_3O^+ and OH^- ions are present in equilibrium. pH values below 7 result from an H_3O^+ excess, pH values above 7 from an OH^- ion excess. The more acidic a solution the higher its H_3O^+ ion concentration and the lower its pH value.

Weak acids, e.g. tartaric acid, do not dissociate completely. This means that only a small fraction (approx. 2...3%) of their acid ions are released. This also means that only on very rare occasions can the pH value be used as a measure of the concentration of acids or bases.

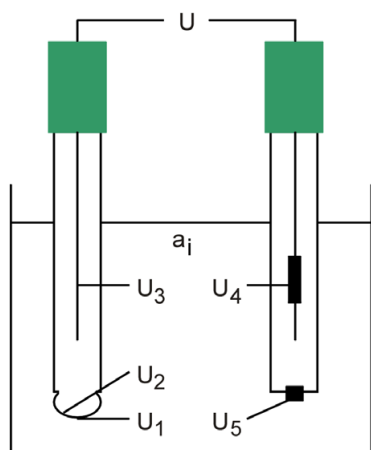
As the pH scale is logarithmic this means that small differences in pH correspond to large differences in the concentration of H_3O^+ ions. For example, at $\text{pH} = 3.0$ there are ten times more H_3O^+ ions present than at $\text{pH} = 4.0$, and at $\text{pH} = 3.1$ there are twice as many H_3O^+ ions present than at $\text{pH} = 3.4$.

The measuring setup for potentiometric measurements always consists of two electrodes – a *measuring or indicator electrode* and a *reference electrode*. For practical reasons these two electrodes are usually contained in a single *combined* electrode.

The indicator electrode (in this case the pH glass electrode) produces a potential that is dependent on the composition of the sample solution.

The reference electrode (usually Ag/AgCl) has the task of providing a potential that is as independent as possible of the sample solution (reference potential).

The potential measurement itself takes place virtually current-free by using a «voltmeter» (in this case a Titrimo) with a high-impedance measuring input (this is necessary to avoid unwanted potential drops). The measured potential U is made up from the individual potentials produced by the indicator and reference electrodes. The following illustration shows a schematic diagram with a separate pH glass electrode (left) and a reference electrode (right).



- U_1 : Galvani potential between measuring electrode and measuring solution
- U_2 : Galvani potential between internal buffer and glass membrane
- U_3 : Galvani potential between internal reference electrode and internal buffer
- U_4 : Galvani potential of reference electrode
- U_5 : Diffusion potential at the diaphragm

The individual potentials U_2 , U_3 and U_4 are determined by the construction of the electrodes and are therefore constant for a given electrode pair. The diffusion potential U_5 should be kept relatively constant and low by taking suitable measures. These measures include an optimal and clean diaphragm, constant stirrer speed during the measurements as well as a suitable reference electrolyte solution whose anions and cations have similar ionic mobilities – e.g. KCl. In this way the potential U_1 measured between the electrodes depends only on the sample solution. In the pH measurement this potential is again dependent on the activity a_i of the measuring ion (H_3O^+ ion / OH^- ion). This relationship is described by the *Nernst equation*:

$$U = U_0 + \frac{2.303 \times R \times T}{z_i \times F} \times \log a_i = U_0 + U_N \times \log a_i$$

where:

- U measured difference in potential between indicator and reference electrode
- U_0 standard potential of the combined electrode (depends on its construction)
- R gas constant (8.31441 J / (K mol))
- T absolute temperature in K (273.15 + t / °C)
- z_i charge on the measuring ion i including its sign (+1 for H_3O^+ and –1 for OH^-)
- a_i activity of measuring ion
- U_N Nernst slope (59.16 mV at 25 °C and $z = 1$)
- 2.303 conversion factor from natural to common logarithm

The Nernst slope U_N describes the theoretical electrode slope and corresponds to the change in potential produced by altering a_i by a factor of ten. It depends on the temperature and charge z of the measuring ion. **Please note: The instrument compensates the effect of temperature on U_N but not on the pH value of the solution!**

The following table shows values of U_N as a function of $t / ^\circ\text{C}$ for $z = 1$:

Temperature $t / ^\circ\text{C}$	U / mV	Temperature $t / ^\circ\text{C}$	U / mV
0	54.20	40	62.14
10	56.18	50	64.12
20	58.17	60	66.10
25	59.16	70	68.09
30	60.15	80	70.07
38	61.74	90	72.06

An ideal pH glass electrode has a slope of 1 (100% of the Nernst slope) and an electrode zero point pH_{as} of 7.0, the latter corresponding to $U_{\text{as}} = 0$ mV.

Things are different in practice. The electrode zero point should have a value for U_{as} of ± 15 mV (corresponds to $\text{pH}_{\text{as}} = 6.75 \dots 7.25$) and the slope should be > 0.95 (> 56.2 mV / pH at 25°C).

In order to «inform» the instrument of the true electrode data it is necessary to calibrate the electrode. Buffer solutions have a defined pH value which, however, is temperature-dependent. The relevant information about the buffer solution is entered during the calibration procedure. The following table shows the pH values of Metrohm buffer solutions as a function of the temperature:

Temperature $t / ^\circ\text{C}$	pH = 4.00 ± 0.02	pH = 7.00 ± 0.02	pH = 9.00 ± 0.02
10	3.99	7.06	9.13
20	3.99	7.02	9.04
25	4.00	7.00	9.00
30	4.00	6.99	8.96
38	4.02	6.98	8.91
40	4.02	6.98	8.90
50	4.04	6.97	8.84
60	4.07	6.97	8.79
70	4.11	6.98	8.74
80	4.15	7.00	8.71
90	4.20	7.01	8.68

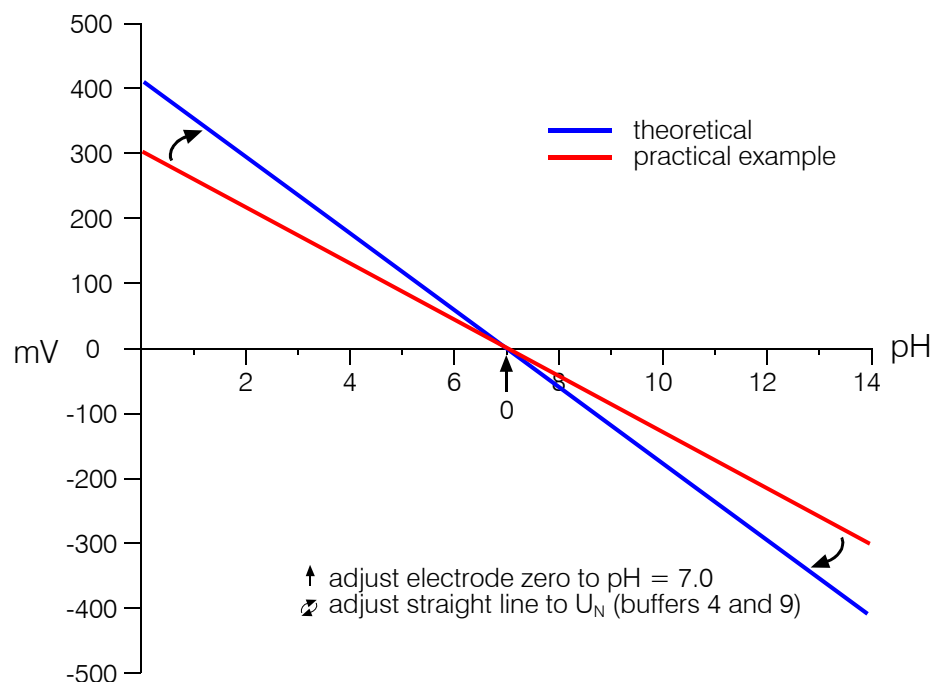
Buffer solutions are not stable!! They can be decomposed by bacteria and/or molds or – this applies to alkaline buffer solutions – alter their pH value by absorbing CO_2 from the atmosphere. This is why you should always use only fresh buffer solutions and reject them after use, i.e. not pour them back into the storage bottle.

Electrode calibration and electrode handling

We recommend the following procedure for **calibrating** a pH glass electrode:

- Remove the electrode from its storage vessel, attach a cable if necessary and connect it to the instrument.
- Open the electrolyte filling opening and, if necessary, top up the electrolyte solution.
- Rinse the electrode thoroughly with dist. H₂O and dab dry with a soft paper tissue (do not rub).
- Fill pH = 7.0 buffer solution into a beaker and add a stirrer bar.
- Immerse the electrode into the buffer solution and stir for approx. 1 min.
- On the **tiamo** enter the pH value of the buffer solution (at the corresponding temperature) and start the calibration with buffer 1 under stirring.
- When the measured value has been accepted, remove the electrode from the solution, rinse it thoroughly with dist. H₂O and dab dry with a soft paper tissue.
- Add pH = 4.0 or 9.0 buffer solution to a second beaker, add a stirrer bar, immerse the electrode and stir for approx. 1 min (the second buffer solution must have the same temperature as the first one).
- On the **tiamo** enter the pH value of the second buffer solution (at the corresponding temperature) and continue the calibration under stirring.
- After the measured value has been accepted, end the calibration. Remove the electrode from the solution, rinse the electrode thoroughly with dist. H₂O and dab dry with a soft paper tissue.

What happens in the instrument during calibration can be seen in the following plot:



Handling

Laboratory electrodes should have a long lifetime. Their characteristics (slope, response behavior, pH_{as} / U_{as}) must lie within the given criteria. In order to ensure this a few basic rules must be observed:

- After use rinse the electrode thoroughly with dist. H_2O and dab dry with a soft paper tissue. Close off the electrolyte filling opening and store the electrode by immersing it in electrolyte solution – usually $c(KCl) = 3 \text{ mol/L}$ – to an adequate depth. Dry storage leads to delayed and poor response behavior. The electrolyte solution may become concentrated and pH_{as} / U_{as} could alter. Storage in dist. H_2O could result in the diaphragm being blocked by $AgCl$.
- If the electrode responds sluggishly and/or the slope is unsatisfactory then the electrode membrane must be etched. This is done by immersing the membrane in a 10% solution of ammonium difluoride (NH_4HF_2 , use plastic beaker) for 1 min, then swirling it for approx. 10 s in $c(HCl) = 5 \text{ mol/L}$, rinsing it thoroughly with dist. H_2O and then wiping off the silicate residue with a moist tissue. In order to build up a new gel layer the electrode is placed in $c(KCl) = 3 \text{ mol/L}$ for 24 h (or for 5 h in the same solution at $50 \text{ }^\circ\text{C}$).
- If the diaphragm becomes blocked please refer to the electrode data sheet that accompanies each electrode. Removing such a blockage is complicated and time-consuming – it is better to send the electrode to your local Metrohm distributor for this.
- Contamination by fats, oils, lacquers, paints, etc.: remove the contamination with an organic solvent (acetone, petroleum benzine, toluene), rinse thoroughly with ethanol and dist. H_2O , dab dry and place in electrolyte solution.
- Contamination by proteins: immerse the electrode in a solution of 5% pepsin in $c(HCl) = 0.1 \text{ mol/L}$ for a few hours. Then rinse thoroughly with dist. H_2O , dab dry and place in electrolyte solution.

3 Titrants

3.1 Preparing the most important alkaline and acidic titrants

General

Titrants are standard solutions, i.e. solutions that contain a defined content of a reactant. This content is given as the molar concentration c in mol/L. The «normality», which was frequently used previously, is no longer valid today and should therefore not be used.

Example:

- $0.1 \text{ N HCl} \Rightarrow c(\text{HCl}) = 0.1 \text{ mol/L}$

Not all titrants have a stable titer, i.e. their concentration can vary with time.

Example:

- Hydroxides absorb CO_2 from the atmosphere to form carbonates

This all means that the titer of the titrant may alter as time passes. In order to know the true titer concentration the titer must be determined at regular intervals.

The so-called standard titrimetric substances are used for determining the titer. Their content hardly changes, they are available with a defined degree of purity, can be dried and can be traced back directly to standard reference materials (e.g. National Institute of Standards and Technology – NIST, USA).

Such standard titrimetric substances /secondary standards are:

- For bases potassium hydrogen phthalate, $M = 204.23 \text{ g/mol}$
- For acids tris(hydroxymethyl)-aminomethane, $M = 121.14 \text{ g/mol}$

Most standard solutions/titrants are commercially available as ready-to-use solutions with a titer adjusted by the manufacturer at $20 \text{ }^\circ\text{C}$ to 1.000. We recommend that you purchase such ready-to-use solutions and do not prepare them yourselves.

In principle titer determinations should always be carried out at the same temperature at which the analyses are later to be carried out. Please note that solutions expand as their temperature increases.

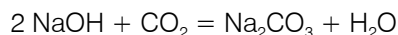
For aqueous solutions a temperature difference of $5 \text{ }^\circ\text{C}$ for a theoretical consumption of 10.00 mL results in a difference in volume of $12.5 \mu\text{L}$. This means that a titer of 1.0000 at $20 \text{ }^\circ\text{C}$ becomes a titer of 0.9988 at $25 \text{ }^\circ\text{C}$ – and for non-aqueous solutions this difference is even larger. In such a case a titer of 1.0000 at $20 \text{ }^\circ\text{C}$ becomes a titer of 0.9950 at $25 \text{ }^\circ\text{C}$ (possible error 0.5%)!

Normally the titer determination is carried out three times and the mean value is used. The mean value of the titer** is saved as «TITER» in the titrant list of tiamo.

** In Europe a dimensionless factor, with at least 4 decimal places (e.g. 1.0015).
In the USA already multiplied by c , with at least 4 decimal places (e.g. 1.0015 mol/L).

3.2 Titer determination of an alkaline titrant

Alkaline titrants do not have a stable titer. They may absorb CO₂ from the atmosphere to form carbonates, e.g.:



This not only reduces the titer. The different strengths of the bases NaOH and Na₂CO₃ may negatively influence the titration curves and therefore the results – high-bias results are simulated, particularly for weak acids. In order to reduce CO₂ absorption as much as possible, soda lime (e.g. Merck no. 106839) is placed in the drying/absorber tube of the Exchange Unit.

– c(NaOH) = 0.1 mol/L

If possible this titrant should be bought ready for use. Otherwise dissolve 4.0 g NaOH in CO₂-free dist. H₂O, make up to 1 liter and mix.

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Titer determination

Potassium hydrogen phthalate is dried over night in a drying oven at 105 °C and allowed to cool down in a desiccator for at least 1 h.

Approx. 2, 3 and 4 g of the dried potassium hydrogen phthalate are weighed out into three 100 mL volumetric flasks with an accuracy of 1 mg, dissolved with dist. water and filled up to the mark. The sample tubes are filled with the prepared solutions and placed on the rack. Then the determination series is started.

Calculation

$$\text{TITER NaOH} = \text{'MV.Sample size'} * \text{'MV.Weight of PPh'} * 1000 / (\text{'DET pH.EP \{1\}.VOL'} * \text{'DET pH.CONC'} * 204.2 * 100)$$

where:

'MV.Sample size'	Volume of the sample [mL]
'MV.Weight of PPh'	Weight of the potassium hydrogen phthalate in the sample solution [g]
1000	Conversion factor for mL
'DET pH.EP {1}.VOL'	Volume NaOH used up to the endpoint [mL]
'DET pH.CONC'	Concentration of the used NaOH [mol/L]
204.2	Molecular weight of potassium hydrogen phthalate [g/mol]
100	Volume of the sample solution [mL]

3.3 *tiamo* method: Titer determination an alkaline titrant

Application note

With this method the titer of $c(\text{NaOH}) = 0.1 \text{ mol/L}$ can be determined. Alkaline titrants tend to absorb CO_2 from the atmosphere which results in a reduced titer value. With the titration of a primary standard the actual titer is determined. The method consists of five different tracks. In the series start track the user is asked to enter the weighed out potassium hydrogen phthalate, the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

Potassium hydrogen phthalate is dried overnight in a drying oven at $105 \text{ }^\circ\text{C}$ and allowed to cool down in a desiccator for at least 1 h.

Approx. 2, 3 and 4 g of the dried potassium hydrogen phthalate are weighed out into three 100 mL volumetric flasks with an accuracy of 1 mg, dissolved with dist. water and filled up to the mark.

Procedure

The 11 mL sample tubes are filled with the prepared solution and placed on the rack. 3 to 6 mL of the sample solution are pipetted to the titration vessel and titrated with $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and sample vessels used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

The value 204.2 in the calculation formula is the molecular weight of potassium hydrogen phthalate in gram per mole. The number 1000 is a calculation factor for liter.

Result report



Robotic Acid / Base Analyzer
Titer determination

Programm version tiamo 1.1

2005-11-21 10:05:51 UTC+1

Results report

Determination

Method Titer determination of an alkaline titrant
 Method saving date 2005-10-19 08:48:05 UTC+2
 Method version 1
 Method state original
 Determination ID 544c53b3:1070767168f:-7eca
 Determination start 2005-10-19 09:47:55 UTC+2
 Determination state original
 Determination version 1
 Run number 14
 User (full name) Metrohm

Sample data

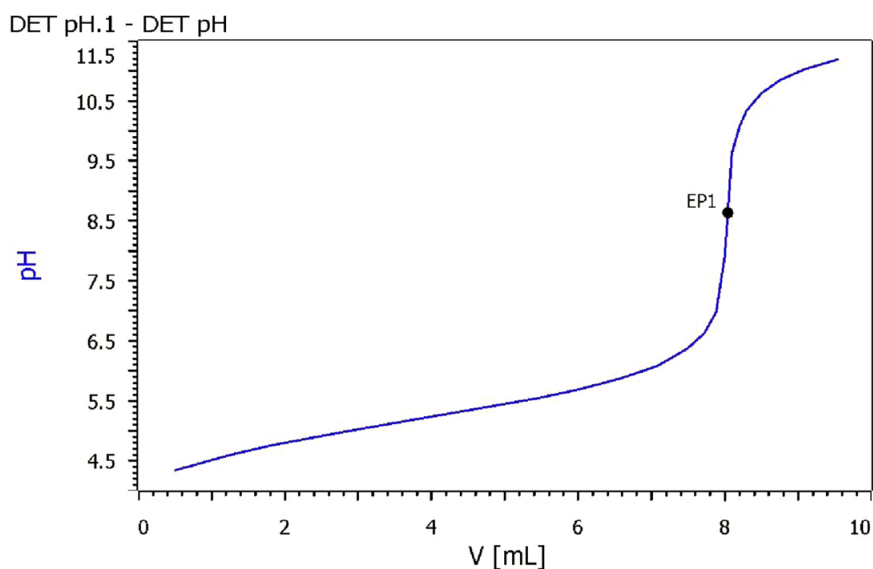
Sample Identification 1 Titer determination
 Weight of PPh in 100 mL 4.084 g
 Sample size 4 mL

End points

DET pH **DET pH.1**
 EP1 8.630 pH 8.0422 mL

Results

Titer NaOH **0.9948**
Mean value titer **0.9954**



Robotic Acid / Base Analyzer
Titer determination

Programm version tiamo 1.1

2005-11-21 10:05:51 UTC+1

Statistical data (short)Method Titer determination of an alkaline titrant
Number of single determinations 10

Result name	n	Mean value	s +/-	s rel
Titer NaOH	10	0.9954	0.00122	0.12 %

3.4 Titer determination of an acidic titrant

– $c(\text{HCl}) = 0.1 \text{ mol/L}$ in H_2O

If possible this titrant should be bought ready for use. Otherwise place approx. 800 mL dist. H_2O in a 1000 mL volumetric flask, add 9.8 mL $w(\text{HCl}) = 32\%$, make up to the mark with dist. H_2O or ethanol and mix.

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Titer determination

Tris(hydroxymethyl)-aminomethane (TRIS) is stored for at least 24 h in a desiccator before use.

Approx. 1.2, 1.8 and 2.4 g TRIS are weighed out into three 100 mL volumetric flasks with an accuracy of 1 mg, dissolved in dist. H_2O and filled up to the mark. The sample tubes are filled with the prepared solutions and placed on the rack. Then the determination series is started.

Calculation

$$\text{TITER HCl} = \frac{\text{'MV.Sample size'} * \text{'MV.Weight of TRIS'} * 1000}{(\text{'DET pH.EP \{1\}.VOL'} * \text{'DET pH.CONC'} * 121.14 * 100)}$$

where:

'MV.Sample size'	Volume of the sample [mL]
'MV.Weight of TRIS'	Weight of the TRIS in the sample solution [g]
1000	Conversion factor for mL
'DET pH.EP {1}.VOL'	Volume HCl used up to the endpoint [mL]
'DET pH.CONC'	Concentration of the used HCl [mol/L]
121.14	Molecular weight of TRIS [g/mol]
100	Volume of the sample solution [mL]

3.5 ***tiamo* method: Titer determination of an acidic titrant**

Application note

With this method the titer of $c(\text{HCl}) = 0.1 \text{ mol/L}$ can be determined. Once the bottle of titrant is open the titer value changes. With the titration of a primary standard the actual titer is determined. The method consists of five different tracks. In the series start track the user is asked to enter the weighed out TRIS (tris(hydroxymethyl)-aminoethane), the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

Tris(hydroxymethyl)-aminomethane (TRIS) is stored for at least 24 h in a desiccator before use.

Approx. 1.2, 1.8 and 2.4 g TRIS are weighed out into three 100 mL volumetric flasks with an accuracy of 1 mg, dissolved in dist. H_2O and filled up to the mark.

Procedure

The sample tubes are filled with the prepared solution and placed on the rack. 3 to 6 mL of the sample solution are transferred to the titration vessel and titrated with $c(\text{HCl}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and beakers used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

The value 121.14 in the calculation formula is the molecular weight of TRIS in gram per mole. The number 1000 is a calculation factor for liter.

Result report



Robotic Acid / Base Analyzer
Titer determination

Programm version tiamo 1.1

2005-11-21 17:10:53 UTC+1

Results report

Determination

Method Titer determination of an acidic titrant
 Method saving date 2005-10-18 10:43:57 UTC+2
 Method version 1
 Method state original
 Determination ID 4d350777:1070246a555:-7cd4
 Determination start 2005-10-18 11:50:18 UTC+2
 Determination state original
 Determination version 1
 Run number 31
 User (full name) Metrohm

Sample data

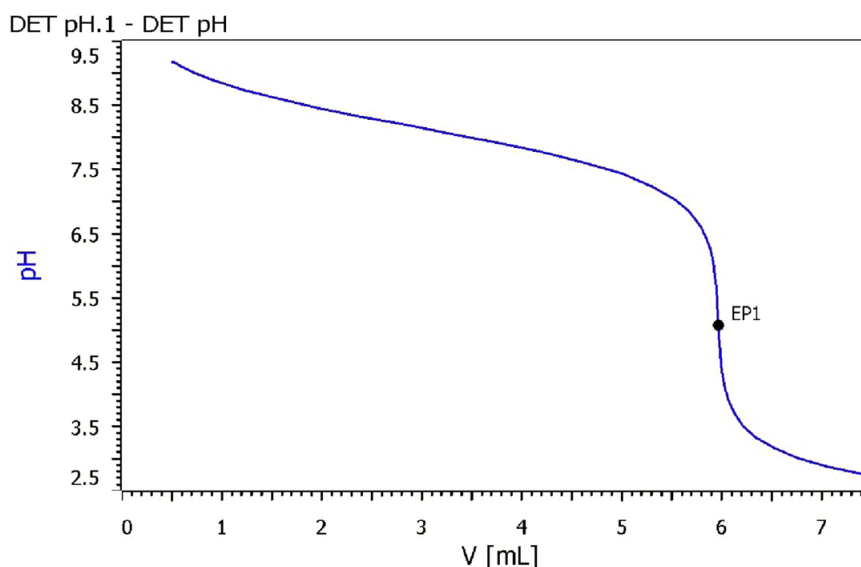
Sample Identification 1 Titer c(HCl) = 0.1M
 Weight of TRIS in 100 mL 2.4292 g
 Sample size 3 mL

End points

DET pH **DET pH.1**
 EP1 5.070 pH 5.9696 mL

Results

Titer HCl **1.0077**
 Mean value titer **1.0065**



Robotic Acid / Base Analyzer
Titer determination

Programm version tiamo 1.1

2005-11-21 17:10:53 UTC+1

Statistical data (short)Method Titer determination of an acidic titrant
Number of single determinations 10

Result name	n	Mean value	s +/-	s rel
Titer HCl	10	1.0065	0.00095	0.09 %

4 Typical applications for the Robotic Transfer Analyzer

4.1 Determination of $c(\text{HCl}) = 0.1 \text{ mol/L}$

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Reagents

- $c(\text{NaOH}) = 0.1 \text{ mol/L}$
- $c(\text{HCl}) = 0.1 \text{ mol/L}$
- dist. water

Analysis

The sample tubes are filled with the sample solution and placed on the rack. Then the determination series can be started.

Calculation

'DET pH.EP {1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / 'MV.Sample size'

where:

'DET pH.EP {1}.VOL'	Volume NaOH used up to the endpoint [mL]
'DET pH.TITER'	Titer of the used NaOH [mol/L]
'DET pH.CONC'	Concentration of the used NaOH [mol/L]
'MV.Sample size'	Volume of the sample [mL]

4.2 *tiamo* method: Determination of $c(\text{HCl}) = 0.1 \text{ mol/L}$

Application note

With this method the content of $c(\text{HCl}) = 0.1 \text{ mol/L}$ can be determined. The method consists of five different tracks. In the series start track the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

No sample preparation is necessary.

Procedure

The sample tubes are filled with the sample and placed on the rack. 3 to 6 mL of the sample solution are transferred to the titration vessel and titrated with $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and beakers used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

Result report



Robotic Transfer Analyzer
 Determination of c(HCl) = 0.1 M

Programm version tiamo 1.1

2005-10-21 14:52:50 UTC+2

Results report

Determination

Method Determination of c(HCl) = 0.1 M
 Method saving date 2005-10-19 11:53:43 UTC+2
 Method version 1
 Method state original
 Determination ID 544c53b3:1070767168f:-7c64
 Determination start 2005-10-19 13:53:24 UTC+2
 Determination state original
 Determination version 1
 Run number 39
 User (full name) Metrohm

Sample data

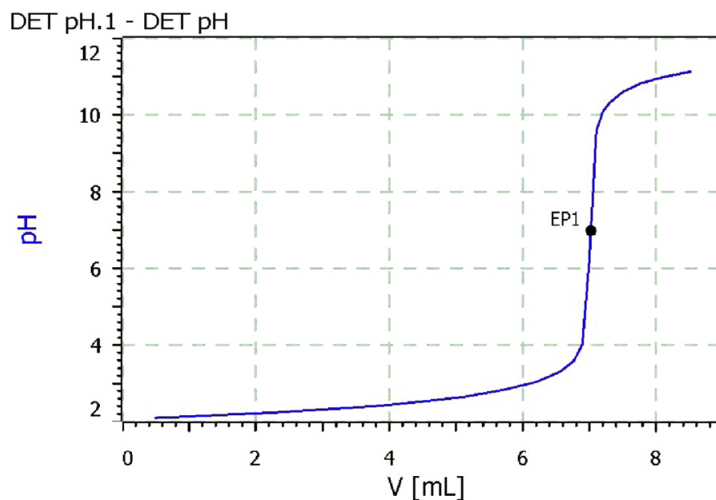
Sample Identification 1 Hydrochloric acid
 Sample size 7 mL

End points

DET pH **DET pH.1**
 EP1 6.968 pH 7.0272 mL

Results

Content **0.0999 mol/L**



Statistical results (example)
Determination of $c(\text{HCl}) = 0.1\text{M}$

Sample size [mL]	Content
5.000	0.0999
6.000	0.0996
7.000	0.0997
5.000	0.0999
6.000	0.0995
7.000	0.0997
5.000	0.0999
6.000	0.0995
7.000	0.0997
5.000	0.0999
Mean value	0.0997
abs. std. dev.	0.0002
rel. std. dev. %	0.16

4.3 Determination of $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Reagents

- $c(\text{NaOH}) = 0.1 \text{ mol/L}$
- $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$
- dist. water

Analysis

The sample tubes are filled with the sample solution and placed on the rack. Then the determination series can be started.

Calculation

$$\text{'DET pH.EP \{1\}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / ('MV.Sample size' * 2)}$$

where:

'DET pH.EP {1}.VOL'	Volume NaOH used up to the endpoint [mL]
'DET pH.TITER'	Titer of the used NaOH [mol/L]
'DET pH.CONC'	Concentration of the used NaOH [mol/L]
'MV.Sample size'	Volume of the sample [mL]
2	Stoichiometric factor

4.4 ***tiamo* method: Determination of $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$**

Application note

With this method the content of $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ can be determined. The method consists of five different tracks. In the series start track the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

No sample preparation is necessary.

Procedure

The sample tubes are filled with the sample and placed on the rack. 3 to 6 mL of the sample solution are transferred to the titration vessel and titrated with $c(\text{NaOH}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and beakers used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

The number 2 in the calculation formula is needed to factor the two protons of the sulphuric acid into the calculation.

Result report



Robotic Transfer Analyzer
 Determination of c(H₂SO₄) = 0.1 M

Programm version tiamo 1.1

2005-10-21 14:51:51 UTC+2

Results report

Determination

Method Determination of c(H₂SO₄) = 0.1 M
 Method saving date 2005-10-19 11:45:18 UTC+2
 Method version 1
 Method state original
 Determination ID 544c53b3:1070767168f:-7d1a
 Determination start 2005-10-19 12:40:10 UTC+2
 Determination state original
 Determination version 1
 Run number 27
 User (full name) Metrohm

Sample data

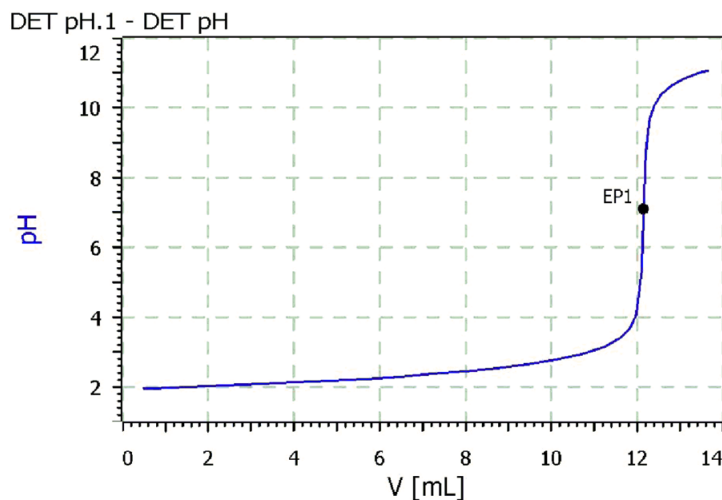
Sample Identification 1 Sulfuric acid
 Sample size 6 mL

End points

DET pH **DET pH.1**
 EP1 7.103 pH 12.1536 mL

Results

Content **0.1008 mol/L**



Statistical results (example)
Determination of $c(\text{H}_2\text{SO}_4) = 0.1\text{M}$

Sample size [mL]	Content
5.000	0.1010
6.000	0.1008
3.000	0.1010
4.000	0.1009
5.000	0.1009
6.000	0.1008
3.000	0.1010
4.000	0.1009
5.000	0.1010
6.000	0.1008
Mean value	0.1009
abs. std. dev.	0.0001
rel. std. dev. %	0.09

4.5 Determination of $c(\text{NaOH}) = 0.1 \text{ mol/L}$

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Reagents

- $c(\text{HCl}) = 0.1 \text{ mol/L}$
- $c(\text{NaOH}) = 0.1 \text{ mol/L}$
- dist. water

Analysis

The sample tubes are filled with the sample solution and placed on the rack. Then the determination series can be started.

Calculation

'DET pH.EP {1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / 'MV.Sample size'

where:

'DET pH.EP {1}.VOL' Volume HCl used up to the endpoint [mL]

'DET pH.TITER' Titer of the used HCl [mol/L]

'DET pH.CONC' Concentration of the used HCl [mol/L]

'MV.Sample size' Volume of the sample [mL]

4.6 **tiamo method: Determination of $c(\text{NaOH}) = 0.1 \text{ mol/L}$**

Application note

With this method the content of $c(\text{NaOH}) = 0.1 \text{ mol/L}$ can be determined. The method consists of five different tracks. In the series start track the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

No sample preparation is necessary.

Procedure

The sample tubes are filled with the sample and placed on the rack. 3 to 6 mL of the sample solution are transferred to the titration vessel and titrated with $c(\text{HCl}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and beakers used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

Result report



Robotic Transfer Analyzer
 Determination of c(NaOH) = 0.1 M

Programm version tiamo 1.1

2005-10-21 14:34:49 UTC+2

Results report

Determination

Method Determination of c(NaOH) = 0.1 M
 Method saving date 2005-10-18 13:00:03 UTC+2
 Method version 1
 Method state original
 Determination ID 4d350777:1070246a555:-7b8b
 Determination start 2005-10-18 13:45:51 UTC+2
 Determination state original
 Determination version 1
 Run number 48
 User (full name) Metrohm

Sample data

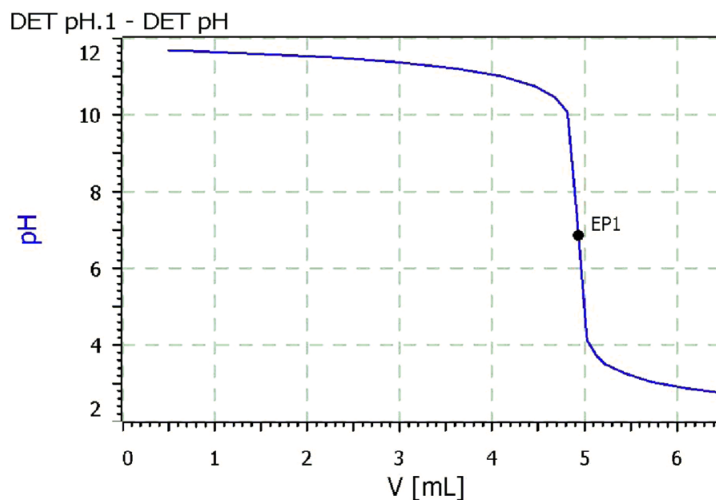
Sample Identification 1 NaOH 0.1 M
 Sample size 5 mL

End points

DET pH DET pH.1
 EP1 6.853 pH 4.9363 mL

Results

Content 0.0994 mol/L



Statistical results (example)
Determination of $c(\text{NaOH}) = 0.1\text{M}$

Sample size [mL]	Content
5.000	0.0993
6.000	0.0993
4.000	0.0993
5.000	0.0995
6.000	0.0997
4.000	0.0993
5.000	0.0995
6.000	0.0994
4.000	0.0993
5.000	0.0994
Mean value	0.0994
abs. std. dev.	0.0001
rel. std. dev. %	0.14

4.7 Determination of $c(\text{NH}_4\text{OH}) = 0.1 \text{ mol/L}$

Recommended accessories

- Comb. pH glass electrode for aqueous titrations 6.0257.000 Aquatrode Plus
- 20 mL Dosing Unit: 6.3032.220
- 10 mL Dosing Unit: 6.3032.210

Reagents

- $c(\text{HCl}) = 0.1 \text{ mol/L}$
- $c(\text{NaOH}) = 0.1 \text{ mol/L}$
- dist. water

Analysis

The sample tubes are filled with the sample solution and placed on the rack. Then the determination series can be started.

Calculation

'DET pH.EP {1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / 'MV.Sample size'

where:

'DET pH.EP {1}.VOL' Volume HCl used up to the endpoint [mL]

'DET pH.TITER' Titer of the used HCl [mol/L]

'DET pH.CONC' Concentration of the used HCl [mol/L]

'MV.Sample size' Volume of the sample [mL]

4.8 tiamo method: Determination of $c(\text{NH}_4\text{OH}) = 0.1 \text{ mol/L}$

Application note

With this method the content of $c(\text{NH}_4\text{OH}) = 0.1 \text{ mol/L}$ can be determined. The method consists of five different tracks. In the series start track the rack is initialized and the system prepared for the measurements. The main track handles the addition of water, the transferring of the sample and the determination followed by the rinsing of the electrode and the titration vessel which is done after every determination.

In the exit track the results are calculated, a report is printed and the data is saved in the predefined database Robotic Transfer Analyzer (can be modified).

Finally, in the series end track, the titration vessel is filled with water in order to avoid the drying out of the electrode.

In case of an error, the error track is carried out, which means that water is given into the titration vessel.

Sample preparation

No sample preparation is necessary.

Procedure

The sample tubes are filled with the sample and placed on the rack. 3 to 6 mL of the sample solution are transferred to the titration vessel and titrated with $c(\text{HCl}) = 0.1 \text{ mol/L}$.

Remarks

To run this method the 855 Robotic Titrosampler has to be adjusted. The lift positions have to be defined according to the rack and beakers used. For titration and the rinsing procedure an external position has to be defined, as well as a corresponding shift and work position respectively.

Result report



Robotic Transfer Analyzer
 Determination of c(NH₄OH) = 0.1 M

Programm version tiamo 1.1

2005-10-21 14:37:35 UTC+2

Results report

Determination

Method Determination of c(NH₄OH) = 0.1 M
 Method saving date 2005-10-18 15:19:06 UTC+2
 Method version 1
 Method state original
 Determination ID 4d350777:1070246a555:-79ec
 Determination start 2005-10-18 15:46:29 UTC+2
 Determination state original
 Determination version 1
 Run number 69
 User (full name) Metrohm

Sample data

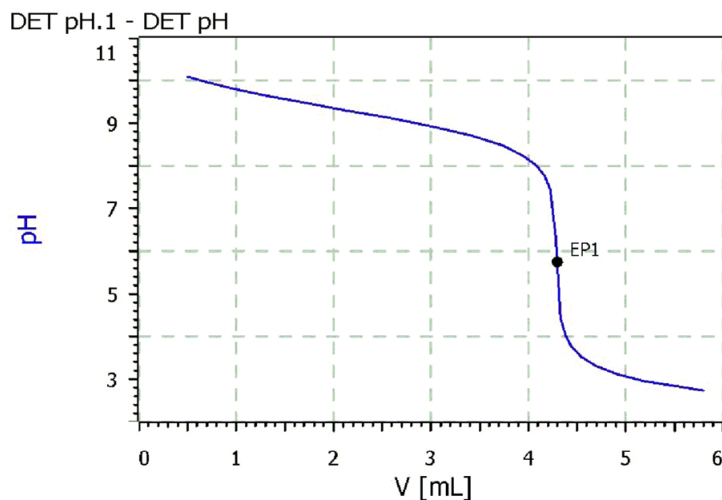
Sample Identification 1 NH₄OH 0.1 M
 Sample size 5 mL

End points

DET pH **DET pH.1**
 EP1 5.753 pH 4.2993 mL

Results

Content **0.0865 mol/L**



Statistical results (example)
Determination of $c(\text{NH}_4\text{OH}) = 0.1\text{M}$

Sample size [mL]	Content
5.000	0.0848
6.000	0.0853
4.000	0.0851
4.000	0.0853
5.000	0.0855
6.000	0.0851
4.000	0.0845
5.000	0.0847
6.000	0.0850
4.000	0.0849
Mean value	0.0850
abs. std. dev.	0.0003
rel. std. dev. %	0.37

5 Troubleshooting pH glass electrodes

5.1 Troubleshooting Aquatrode

Source of error	Effects	Cleaning	Alternatives
HF-containing solutions	Etching and dissolution of the glass membrane → corrosion potential during the measurement/short working life		Use of the Sb electrode
High pH value and high alkali content	Increased alkali error → pH too low		Use of electrodes with U glass
High temperatures	Rapid rise in membrane resistance by aging → increased polarizability and drift		Use of electrodes with U glass. Careful treatment with etching salt and HCl
Measurements at low temperatures	High membrane resistance → polarization effects		Use of electrodes with T glass and Idrolyte as reference electrolyte
Dry storage	Zero point drift	Storage in 6.2323.000 storage solution	
Non-aqueous media	Reduced sensitivity	Store in water	T glass / non-aqueous electrolyte solution
Deposition of solids on membrane surface	Slow response, zero point shift, slope reduction	Solvent or strong acids	
Deposition of proteins on membrane surface	Slow response, zero point shift, slope reduction	5% pepsin in 0.1 mol/L HCl	

5.2 Cleaning and care of pH-electrodes with fixed-sleeve diaphragm

Diaphragm type	Type of contamination	Cleaning
General	Precipitates of silver halides and silver sulfides	Immerse diaphragm for several hours in a solution of 7% thiourea in 0.1 mol/L HCl.
	Proteins, polypeptides	Immerse diaphragm for several hours in a solution of 5% pepsin in 0.1 mol/L HCl.
	Suspensions, solids, resins, glues, oils, fats	Clean electrode with suitable solvent
Fixed ground joint	All types of contamination	Aspirate off reference electrolyte and immerse electrode in the corresponding cleaning solution.
Separable ground joint	All types of contamination	Loosen the ground-joint sleeve (using hot water if necessary) and clean according to the type of contamination.

5.3 Storage

Aquatrode Plus: Store in 6.2323.000 storage solution only! The filling plug for the bridge electrolyte must not be opened.

6 Method reports

6.1 Titer determination of an alkaline titrant



License ID 124049905 Program version tiamo 1.1 - 32
 Client name TITRATION14
 User Metrohm 2005-10-21 14:24:00 UTC+2

Method parameters

Method Titer determination of an alkaline titrant
 Method saving date 2005-10-21 14:23:54 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START

Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics on
 Number of single determinations not defined
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
Weight of PHPH	Number	ID2		Sample identification 2	off
Dilution factor	Number	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on, Sample size
 Value off,
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:24:00 UTC+2

Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample position**
 Type Number
 Assignment on. Sample position
 Value off.
 Check at start on
 Comment Sample position number
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Weight of PPh**
 Type Number
 Assignment on. ID2
 Value off.
 Check at start off
 Comment Sample identification 2
 Variable monitoring off
 Lower limit
 Upper limit



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Dilution factor**
 Type Number
 Assignment on ID3
 Value off
 Check at start off
 Comment Sample identification 3
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample size unit**
 Type Text
 Assignment on Sample size unit
 Value off
 Check at start on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:24:00 UTC+2

Comment Sample size unit

Name **ID1**
 Type Text
 Assignment on. ID1
 Value off.
 Check at start off
 Comment Sample identification 1

PUMP

add water

Device
 Device name 855_1

Pumps
 Tower 1
 Pump(s) 1

Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters
 Function Compensate
 Port 3

LQH

produce first air bubble

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters
 Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

MOVE

to sample



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Device
 Device name 855_1
 Target
 Tower 1
 Move Sample position
 Beaker test
 Display message off
 Stop determination off
 Stop determination and series on
 Parameters
 Shift rate 20 %/s
 Shift direction auto
 Swing rate 55 %/s

LIFT lift to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH aspirate sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT adjusting pressure

Wait
 Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:24:00 UTC+2

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

LQH change port

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 °/s

LIFT go down to work position

Device
 Device name 855_1
 Target
 Tower 1



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH

compensation

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Compensate
 Port 3

LQH

dose sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Dosing
 Port 3
 Volume ='MV.Sample size' + 2 mL
 Rate 10 mL/min

WAIT

pressure adjustment

Wait

Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s

Message

Record message off
 Message by e-mail off
 Acoustic signal off

LIFT

to shifting position

Device

Device name 855_1

Target

Tower 1
 Lift position Shift position mm

Parameters

Lift rate 25 mm/s



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:24:00 UTC+2

DET pH

DET pH

General/Hardware

Device

Device name 855_1

Dosing device

Dosing device 1

Solution NaOH

Sensor

Measuring input 1

Sensor Aquatrode Plus

Temperature measurement automatic

Stirrer

Stirrer 1

Stirring rate 8

Switch off automatically on

Start conditions

Initial measured value

Signal drift off mV/min

Min. waiting time 0 s

Max. waiting time 1 s

Start volume

Start volume 0.5 mL

Dosing rate maximum mL/min

Start measured value

Start measured value pH off

Dosing rate 5 mL/min

Start slope

Start slope off pH/mL

Dosing rate 5 mL/min

Pause

Pause 5 s

Titration parameters

Titration rate

Titration rate user

Measured value acceptance

Signal drift 50.0 mV/min

Min. waiting time 0 s

Max. waiting time 26 s

Dosing of increments

Measuring point density 4

Min. increment 100 µL

Max. increment off µL

Dosing rate maximum mL/min

Temperature

Temperature 25.0 °C

Stop conditions



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1



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 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:24:00 UTC+2

Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

EXIT Exit track

CALC calculation

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer NaOH	= 'MV.Sample size' * 'MV.Weight of PPh' * 1000 / ('DET pH.EP{1}.VOL' * 'DET pH.		4	RS01	on



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 Client name TITRATION14
 User Metrohm

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Result name	Formula	Unit	Decimal places	Assignment	Statistics
Mean value titer	= 'RS.Titer NaOH.MNV'		4	RS02	off

Result name **Titer NaOH**
 Formula = 'MV.Sample size' * 'MV.Weight of
 PPh' * 1000 / ('DET pH.EP{1}.VOL' *
 'DET pH.CONC' * 204.2 * 'MV.Dilution
 factor')
 Unit
 Decimal places 4
 Assignment RS01
 Statistics on
 Description RS.'Result name[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

Result name **Mean value titer**
 Formula = 'RS.Titer NaOH.MNV'
 Unit
 Decimal places 4
 Assignment RS02
 Statistics off
 Description RS.'Result name[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer on
 Solution name NaOH

REPORT

report
 Report template
 Report template titer determination
 Report output
 Printer off
 PDF file on
 PDF file C:\Dokumente und Einstellungen\demo\Eigene
 Dateien\mma\855 Robotic Titrosampler tiamo 1.1\Robotic
 Transfer Analyzer\Reports\Titer NaOH 0.1 M.pdf
 Send e-mail off

DATABASE database



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Mean value titer	= 'RS.Titer NaOH.MNV'		4	RS02	off

Result name **Titer NaOH**
 Formula = 'MV.Sample size' * 'MV.Weight of
 PHPH' * 1000 / ('DET pH.EP{1}.VOL' *
 'DET pH.CONC' * 204.2 * 'MV.Dilution
 factor')
 Unit
 Decimal places 4
 Assignment RS01
 Statistics on
 Description RS:'Result name'[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

Result name **Mean value titer**
 Formula = 'RS.Titer NaOH.MNV'
 Unit
 Decimal places 4
 Assignment RS02
 Statistics off
 Description RS:'Result name'[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer on
 Solution name NaOH

REPORT

report
 Report template
 Report template titer determination
 Report output
 Printer off
 PDF file on
 PDF file C:\Dokumente und Einstellungen\demo\Eigene
 Dateien\mma\855 Robotic Titrosampler tiamo 1.1\Robotic
 Transfer Analyzer\Reports\Titer NaOH 0.1 M.pdf
 Send e-mail off

DATABASE database



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Database

Robotic Transfer Analyzer

SERIES START **Series start track**

REQUEST **sample size**

Sample data request

Sample position off
 ID1 off
 ID2 on
 ID3 on
 ID4 off
 ID5 off
 ID6 off
 ID7 off
 ID8 off
 Sample size off
 Unit off
 Message on
 Enter the value of the weighed in potassium hydrogen phthalate and the dillution factor.

RACK **initialize rack**

Device

Device name 855_1
 Rack test off

SEQUENCE **prepare vessel and dosino**

SWING **swing to titration vessel**

Device

Device name 855_1

Target

Tower 1
 Swing External position
 Number 1

Parameters

Swing rate 55 %/s

PUMP **empty titration vessel**

Device

Device name 855_1

Pumps

Tower 1
 Pump(s) 2

Action

Switch on off
 Switch off off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
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Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP clean titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:24:00 UTC+2

Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

SERIES END Series end track

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

ERROR Error track

PUMP empty vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP add water to vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1



License ID 124049905
Client name TITRATION14
User Metrohm

Program version tiamo 1.1 - 32
2005-10-21 14:24:00 UTC+2

Action

Switch on off
Switch off off
Duration on
Time 20 s

6.2 Titer determination of an acidic titrant



License ID 124049905 Program version tiamo 1.1 - 32
 Client name TITRATION14
 User Metrohm 2005-10-21 14:42:57 UTC+2

Method parameters

Method Titer determination of an acidic titrant
 Method saving date 2005-10-21 14:42:16 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics on
 Number of single determinations not defined
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
Weight of TRIS	Number	ID2		Sample identification 2	off
Dillution factor	Number	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on. Sample size
 Value off.
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:42:57 UTC+2

Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample position**
 Type Number
 Assignment on. Sample position
 Value off.
 Check at start on
 Comment Sample position number
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Weight of TRIS**
 Type Number
 Assignment on. ID2
 Value off.
 Check at start off
 Comment Sample identification 2
 Variable monitoring off
 Lower limit
 Upper limit



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:42:57 UTC+2

Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Dilution factor**
 Type Number
 Assignment on ID3
 Value off
 Check at start off
 Comment Sample identification 3
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample size unit**
 Type Text
 Assignment on Sample size unit
 Value off
 Check at start on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:42:57 UTC+2

Comment Sample size unit
 Name ID1
 Type Text
 Assignment on. ID1
 Value off.
 Check at start off
 Comment Sample identification 1

PUMP

add water
 Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Compensate
 Port 3

LQH

produce first air bubble
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

MOVE to sample



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
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Device
 Device name 855_1
 Target
 Tower 1
 Move Sample position
 Beaker test
 Display message off
 Stop determination off
 Stop determination and series on
 Parameters
 Shift rate 20 %/s
 Shift direction auto
 Swing rate 55 %/s

LIFT lift to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH aspirate sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT adjusting pressure

Wait
 Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:42:57 UTC+2

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

LQH change port

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 °/s

LIFT go down to work position

Device
 Device name 855_1
 Target
 Tower 1



License ID 124049905
 Client name TITRATION14
 User Metrohm

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Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH

compensation

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Compensate
 Port 3

LQH

dose sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Dosing
 Port 3
 Volume 8 mL
 Rate 10 mL/min

WAIT

pressure adjustment

Wait

Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s

Message

Record message off
 Message by e-mail off
 Acoustic signal off

LIFT

to shifting position

Device

Device name 855_1

Target

Tower 1
 Lift position Shift position mm

Parameters

Lift rate 25 mm/s



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:42:57 UTC+2

DET pH

DET pH

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 1
 Solution HCl
 Sensor
 Measuring input 1
 Sensor Aquatrode Plus
 Temperature measurement automatic
 Stirrer
 Stirrer 1
 Stirring rate 8
 Switch off automatically on

Start conditions

Initial measured value
 Signal drift off mV/min
 Min. waiting time 0 s
 Max. waiting time 1 s
 Start volume
 Start volume 0.5 mL
 Dosing rate maximum mL/min
 Start measured value
 Start measured value pH off
 Dosing rate 5 mL/min
 Start slope
 Start slope off pH/mL
 Dosing rate 5 mL/min
 Pause
 Pause 5 s

Titration parameters

Titration rate
 Titration rate user
 Measured value acceptance
 Signal drift 50.0 mV/min
 Min. waiting time 0 s
 Max. waiting time 26 s
 Dosing of increments
 Measuring point density 4
 Min. increment 10.0 µL
 Max. increment off µL
 Dosing rate maximum mL/min
 Temperature
 Temperature 25.0 °C

Stop conditions



License ID 124049905
 Client name TITRATION14
 User Metrohm

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 2005-10-21 14:42:57 UTC+2

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:42:57 UTC+2

Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

EXIT Exit track

CALC calculation

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer HCl	= 'MV.Sample size' * 'MV.Weight of TRIS' * 1000 / ('DET pH.EP{1}.VOL' * 'DET pH.		4	RS01	on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:42:57 UTC+2

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Mean value titer	= 'RS.Titer HCl.MNV'		4	RS02	off

Result name **Titer HCl**
 Formula = 'MV.Sample size' * 'MV.Weight of TRIS' * 1000 / ('DET pH.EP{1}.VOL' * 'DET pH.CONC' * 121.14 * 'MV.Dilution factor')
 Unit
 Decimal places 4
 Assignment RS01
 Statistics on
 Description RS.'Result name[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

Result name **Mean value titer**
 Formula = 'RS.Titer HCl.MNV'
 Unit
 Decimal places 4
 Assignment RS02
 Statistics off
 Description RS.'Result name[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer on
 Solution name HCl

REPORT

report
 Report template
 Report template titer determination
 Report output
 Printer off
 PDF file on
 PDF file C:\Dokumente und Einstellungen\demo\Eigene Dateien\mma\855 Robotic Titrosampler tiamo 1.1\Robotic Transfer Analyzer\Reports\Titer HCl 0.1 M.pdf
 Send e-mail off

DATABASE database



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:42:57 UTC+2

Database

Robotic Transfer Analyzer

SERIES START **Series start track**

REQUEST **sample size**

Sample data request
 Sample position off
 ID1 off
 ID2 on
 ID3 on
 ID4 off
 ID5 off
 ID6 off
 ID7 off
 ID8 off
 Sample size off
 Unit off
 Message on
 Enter the value of the weighed in TRIS and the dillution factor.

RACK **initialize rack**

Device
 Device name 855_1
 Rack test off

SEQUENCE **prepare vessel and dosino**

SWING **swing to titration vessel**

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 %/s

PUMP **empty titration vessel**

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:42:57 UTC+2

Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP clean titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:42:57 UTC+2

Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

SERIES END Series end track

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

ERROR Error track

PUMP empty vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP add water to vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1



License ID 124049905
Client name TITRATION14
User Metrohm

Program version tiamo 1.1 - 32
2005-10-21 14:42:57 UTC+2

Action

Switch on off
Switch off off
Duration on
Time 20 s

6.3 Determination of c(HCl) = 0.1 mol/L



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:01:45 UTC+2

Method parameters

Method Determination of c(HCl) = 0.1 M
 Method saving date 2005-10-21 14:01:40 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START

Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics off
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
ID2	Text	ID2		Sample identification 2	off
ID3	Text	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on Sample size
 Value off
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to



License ID 124049905
Client name TITRATION14
User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:01:45 UTC+2

Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
User
Mail from
SMTP Server
POP3 Server
Acoustic signal off
Action off
Stop determination on
Stop determination and series off

Name **Sample position**
Type Number
Assignment on. Sample position
Value off.
Check at start on
Comment Sample position number
Variable monitoring off
Lower limit
Upper limit
Message
Display message on
Record message on
Message by e-mail off

Mail to
Subject Message from tiamo - Method 'New method 1' - Command 'Main track'

User
Mail from
SMTP Server
POP3 Server
Acoustic signal off
Action off
Stop determination on
Stop determination and series off

Name **Sample size unit**
Type Text
Assignment on. Sample size unit
Value off.
Check at start on
Comment Sample size unit

Name **ID1**
Type Text
Assignment on. ID1
Value off.



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

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Check at start off
 Comment Sample identification 1

Name ID2
 Type Text
 Assignment on. ID2
 Value off.
 Check at start off
 Comment Sample identification 2

Name ID3
 Type Text
 Assignment on. ID3
 Value off.
 Check at start off
 Comment Sample identification 3

PUMP

add water
 Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Compensate
 Port 3

LQH

produce first air bubble
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:01:45 UTC+2

Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

MOVE

to sample

Device

Device name 855_1

Target

Tower 1

Move Sample position

Beaker test

Display message off
 Stop determination off
 Stop determination and series on

Parameters

Shift rate 20 °/s
 Shift direction auto
 Swing rate 55 °/s

LIFT

lift to working position

Device

Device name 855_1

Target

Tower 1

Lift position Work position mm

Parameters

Lift rate 25 mm/s

LQH

aspirate sample

General/Hardware

Device

Device name 855_1

Dosing device

Dosing device 2

Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT

adjusting pressure

Wait

Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:01:45 UTC+2

Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

LQH change port

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1



License ID 124049905
 Client name TITRATION14
 User Metrohm

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Parameters

Swing rate 55 %/s

LIFT go down to work position

Device

Device name 855_1

Target

Tower 1

Lift position Work position mm

Parameters

Lift rate 25 mm/s

LQH compensation

General/Hardware

Device

Device name 855_1

Dosing device

Dosing device 2

Solution Transfer

Parameters

Function Compensate

Port 3

LQH dose sample

General/Hardware

Device

Device name 855_1

Dosing device

Dosing device 2

Solution Transfer

Parameters

Function Dosing

Port 3

Volume ='MV.Sample size' + 2 mL

Rate 10 mL/min

WAIT pressure adjustment

Wait

Stop track and waiting for [Continue] off

Stop all tracks and waiting for [Continue] off

Waiting time on

Time 3

Unit s

Message

Record message off

Message by e-mail off

Acoustic signal off

LIFT to shifting position



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Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

DET pH

DET pH

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 1
 Solution NaOH
 Sensor
 Measuring input 1
 Sensor Aquatrode Plus
 Temperature measurement automatic
 Stirrer
 Stirrer 1
 Stirring rate 8
 Switch off automatically on

Start conditions

Initial measured value
 Signal drift off mV/min
 Min. waiting time 0 s
 Max. waiting time 1 s
 Start volume
 Start volume 0.5 mL
 Dosing rate maximum mL/min
 Start measured value
 Start measured value pH off
 Dosing rate 5 mL/min
 Start slope
 Start slope off pH/mL
 Dosing rate 5 mL/min
 Pause
 Pause 5 s

Titration parameters

Titration rate
 Titration rate user
 Measured value acceptance
 Signal drift 50.0 mV/min
 Min. waiting time 0 s
 Max. waiting time 26 s
 Dosing of increments



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 Client name TITRATION14
 User Metrohm

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Measuring point density 4
 Min. increment 100 µL
 Max. increment off µL
 Dosing rate maximum mL/min
 Temperature
 Temperature 25.0 °C

Stop conditions

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

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Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
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EXIT **Exit track**

CALC **calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Content	= 'DET pH.EP{1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / ('MV.Sample size')	mol/L	4	RS01	off

Result name **Content**
 Formula = 'DET pH.EP{1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / ('MV.Sample size')
 Unit mol/L
 Decimal places 4
 Assignment RS01
 Statistics off
 Description RS.'Result name[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

REPORT **report**

Report template
 Report template Determination of c(HCl) = 0.1 M
 Report output
 Printer off
 PDF file on
 PDF file C:\Programme\Metrohm\tiamo\Det HCl 0.1 M.pdf
 Send e-mail off

DATABASE **database**

Database
 Robotic Transfer Analyzer

SERIES START **Series start track**

RACK **initialize rack**

Device
 Device name 855_1
 Rack test off

SEQUENCE **prepare vessel and dosino**

SWING **swing to titration vessel**

Device



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:01:45 UTC+2

Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 %/s

PUMP empty titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP clean titration vessel



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:01:45 UTC+2

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

SERIES END Series end track

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

ERROR Error track

PUMP empty vessel



License ID 124049905
Client name TITRATION14
User Metrohm

Program version tiamo 1.1 - 32
2005-10-21 14:01:45 UTC+2

Device
Device name 855_1
Pumps
Tower 1
Pump(s) 2
Action
Switch on off
Switch off off
Duration on
Time 10.0 s

PUMP

add water to vessel
Device
Device name 855_1
Pumps
Tower 1
Pump(s) 1
Action
Switch on off
Switch off off
Duration on
Time 20 s

6.4 Determination of $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$



License ID 124049905 Program version tiamo 1.1 - 32
 Client name TITRATION14
 User Metrohm 2005-10-21 13:57:52 UTC+2

Method parameters

Method Determination of $c(\text{H}_2\text{SO}_4) = 0.1 \text{ M}$
 Method saving date 2005-10-21 13:57:42 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics off
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
ID2	Text	ID2		Sample identification 2	off
ID3	Text	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on Sample size
 Value off
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 13:57:52 UTC+2

Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample position**
 Type Number
 Assignment on Sample position
 Value off
 Check at start on
 Comment Sample position number
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample size unit**
 Type Text
 Assignment on Sample size unit
 Value off
 Check at start on
 Comment Sample size unit

Name **ID1**
 Type Text
 Assignment on ID1



License ID 124049905
 Client name TITRATION14
 User Metrohm

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Value off.
 Check at start off
 Comment Sample identification 1

Name **ID2**
 Type Text
 Assignment on. ID2
 Value off.
 Check at start off
 Comment Sample identification 2

Name **ID3**
 Type Text
 Assignment on. ID3
 Value off.
 Check at start off
 Comment Sample identification 3

PUMP

add water
 Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Compensate
 Port 3

LQH

produce first air bubble
General/Hardware
 Device
 Device name 855_1
 Dosing device



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 13:57:52 UTC+2

Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

MOVE to sample

Device
 Device name 855_1
 Target
 Tower 1
 Move Sample position
 Beaker test
 Display message off
 Stop determination off
 Stop determination and series on
 Parameters
 Shift rate 20 %/s
 Shift direction auto
 Swing rate 55 %/s

LIFT lift to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH aspirate sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT adjusting pressure

Wait
 Stop track and waiting for [Continue] off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
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Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

LQH change port

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

```

Number . . . . . 1
Parameters
Swing rate . . . . . 55 %/s

LIFT go down to work position
Device
Device name . . . . . 855_1
Target
Tower . . . . . 1
Lift position . . . . . Work position mm
Parameters
Lift rate . . . . . 25 mm/s

LQH compensation
General/Hardware
Device
Device name . . . . . 855_1
Dosing device
Dosing device . . . . . 2
Solution . . . . . Transfer
Parameters
Function . . . . . Compensate
Port . . . . . 3

LQH dose sample
General/Hardware
Device
Device name . . . . . 855_1
Dosing device
Dosing device . . . . . 2
Solution . . . . . Transfer
Parameters
Function . . . . . Dosing
Port . . . . . 3
Volume . . . . . =MV.Sample size' + 2 mL
Rate . . . . . 10 mL/min

WAIT pressure adjustment
Wait
Stop track and waiting for [Continue] . . . . . off
Stop all tracks and waiting for [Continue] . . . . . off
Waiting time . . . . . on
Time . . . . . 3
Unit . . . . . s
Message
Record message . . . . . off
Message by e-mail . . . . . off
Acoustic signal . . . . . off
    
```



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

LIFT to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

DET pH DET pH

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 1
 Solution NaOH
 Sensor
 Measuring input 1
 Sensor Aquatrode Plus
 Temperature measurement automatic
 Stirrer
 Stirrer 1
 Stirring rate 8
 Switch off automatically on

Start conditions

Initial measured value
 Signal drift off mV/min
 Min. waiting time 0 s
 Max. waiting time 1 s
 Start volume
 Start volume 0.5 mL
 Dosing rate maximum mL/min
 Start measured value
 Start measured value pH off
 Dosing rate 5 mL/min
 Start slope
 Start slope off pH/mL
 Dosing rate 5 mL/min
 Pause
 Pause 5 s

Titration parameters

Titration rate
 Titration rate user
 Measured value acceptance
 Signal drift 50.0 mV/min
 Min. waiting time 0 s



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

Max. waiting time 26 s
 Dosing of increments
 Measuring point density 4
 Min. increment 100 µL
 Max. increment off µL
 Dosing rate maximum mL/min
 Temperature
 Temperature 25.0 °C

Stop conditions

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

Parameters

Lift rate 25 mm/s

EXIT Exit track

CALC calculation

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Content	= 'DET pH.EP{1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / ('MV.Sample size' * 2)	mol/L	4	RS01	off

Result name **Content**
 Formula = 'DET pH.EP{1}.VOL' * 'DET pH.TITER' * 'DET pH.CONC' / ('MV.Sample size' * 2)
 Unit mol/L
 Decimal places 4
 Assignment RS01
 Statistics off
 Description RS:'Result name'[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

REPORT report

Report template
 Report template Determination of c(H2SO4) = 0.1 M
 Report output
 Printer off
 PDF file on
 PDF file C:\Programme\Metrohm\tiamo\Det H2SO4 0.1 M.pdf
 Send e-mail off

DATABASE database

Database
 Robotic Transfer Analyzer

SERIES START Series start track

RACK initialize rack

Device
 Device name 855_1
 Rack test off

SEQUENCE prepare vessel and dosino



License ID 124049905
 Client name TITRATION14
 User Metrohm

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SWING swing to titration vessel

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 °/s

PUMP empty titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 13:57:52 UTC+2

Rate maximum mL/min

PUMP clean titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

**SERIES Series end track
 END**

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 13:57:52 UTC+2

ERROR Error track

PUMP empty vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP add water to vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

6.5 Determination of c(NaOH) = 0.1 mol/L



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

2005-10-21 14:04:17 UTC+2

Method parameters

Method Determination of c(NaOH) = 0.1 M
 Method saving date 2005-10-21 14:04:12 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START

Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics off
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
ID2	Text	ID2		Sample identification 2	off
ID3	Text	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on Sample size
 Value off
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:04:17 UTC+2

Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample position**
 Type Number
 Assignment on, Sample position
 Value off,
 Check at start on
 Comment Sample position number
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample size unit**
 Type Text
 Assignment on, Sample size unit
 Value off,
 Check at start on
 Comment Sample size unit

Name **ID1**
 Type Text
 Assignment on, ID1



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

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Value off
 Check at start off
 Comment Sample identification 1

Name **ID2**
 Type Text
 Assignment on ID2
 Value off
 Check at start off
 Comment Sample identification 2

Name **ID3**
 Type Text
 Assignment on ID3
 Value off
 Check at start on
 Comment Sample identification 3

PUMP

add water

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Compensate
 Port 3

LQH

produce first air bubble

General/Hardware

Device
 Device name 855_1
 Dosing device



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:04:17 UTC+2

Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.2 mL
 Rate 5 mL/min

MOVE to sample

Device
 Device name 855_1
 Target
 Tower 1
 Move Sample position
 Beaker test
 Display message off
 Stop determination off
 Stop determination and series on
 Parameters
 Shift rate 20 °/s
 Shift direction auto
 Swing rate 55 °/s

LIFT lift to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH aspirate sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT adjusting pressure

Wait
 Stop track and waiting for [Continue] off



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32
 2005-10-21 14:04:17 UTC+2

Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Aspirate
 Port 3
 Volume 0.2 mL
 Rate 5 mL/min

LQH change port

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position



License ID 124049905
 Client name TITRATION14
 User Metrohm

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```

        Number ..... 1
    Parameters
        Swing rate ..... 55 %/s

LIFT    go down to work position
    Device
        Device name ..... 855_1
    Target
        Tower ..... 1
        Lift position ..... Work position mm
    Parameters
        Lift rate ..... 25 mm/s

LQH    compensation
    General/Hardware
    Device
        Device name ..... 855_1
    Dosing device
        Dosing device ..... 2
        Solution ..... Transfer
    Parameters
        Function ..... Compensate
        Port ..... 3

LQH    dose sample
    General/Hardware
    Device
        Device name ..... 855_1
    Dosing device
        Dosing device ..... 2
        Solution ..... Transfer
    Parameters
        Function ..... Dosing
        Port ..... 3
        Volume ..... 8 mL
        Rate ..... 10 mL/min

WAIT   pressure adjustment
    Wait
        Stop track and waiting for [Continue] ..... off
        Stop all tracks and waiting for [Continue] ..... off
        Waiting time ..... on
            Time ..... 3
            Unit ..... s
    Message
        Record message ..... off
        Message by e-mail ..... off
        Acoustic signal ..... off
    
```



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LIFT to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

DET pH DET pH

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 1
 Solution HCl
 Sensor
 Measuring input 1
 Sensor Aquatrode Plus
 Temperature measurement automatic
 Stirrer
 Stirrer 1
 Stirring rate 8
 Switch off automatically on

Start conditions

Initial measured value
 Signal drift off mV/min
 Min. waiting time 0 s
 Max. waiting time 1 s
 Start volume
 Start volume 0.5 mL
 Dosing rate maximum mL/min
 Start measured value
 Start measured value pH off
 Dosing rate 5 mL/min
 Start slope
 Start slope off pH/mL
 Dosing rate 5 mL/min
 Pause
 Pause 5 s

Titration parameters

Titration rate
 Titration rate user
 Measured value acceptance
 Signal drift 50.0 mV/min
 Min. waiting time 0 s



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Max. waiting time 26 s
 Dosing of increments
 Measuring point density 4
 Min. increment 100 µL
 Max. increment off µL
 Dosing rate maximum mL/min
 Temperature
 Temperature 25.0 °C

Stop conditions

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2



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Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm



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Parameters

Lift rate 25 mm/s

EXIT Exit track

CALC calculation

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Content	= 'DET pH.EP{1}.VOL' * 'DET pH.CONC' * 'DET pH.TITER' / 'MV.Sample size'	mol/L	4	RS01	off

Result name **Content**
 Formula = 'DET pH.EP{1}.VOL' * 'DET pH.CONC' * 'DET pH.TITER' / 'MV.Sample size'
 Unit mol/L
 Decimal places 4
 Assignment RS01
 Statistics off
 Description RS.'Result name'[,VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

REPORT report

Report template
 Report template Determination of c(NaOH) = 0.1 M
 Report output
 Printer off
 PDF file on
 PDF file C:\Programme\Metrohm\tiamo\DET NaOH 0.1 M.pdf
 Send e-mail off

DATABASE database

Database
 Robotic Transfer Analyzer

SERIES START Series start track

RACK initialize rack

Device
 Device name 855_1
 Rack test off

SEQUENCE prepare vessel and dosino



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SWING swing to titration vessel

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 %/s

PUMP empty titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Eject to end volume
 Port 3



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Rate maximum mL/min

PUMP clean titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

**SERIES Series end track
 END**

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s



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ERROR Error track

PUMP empty vessel

Device
Device name 855_1
Pumps
Tower 1
Pump(s) 2
Action
Switch on off
Switch off off
Duration on
Time 10.0 s

PUMP add water to vessel

Device
Device name 855_1
Pumps
Tower 1
Pump(s) 1
Action
Switch on off
Switch off off
Duration on
Time 20 s

6.6 Determination of $c(\text{NH}_4\text{OH}) = 0.1 \text{ mol/L}$



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Method parameters

Method Determination of $c(\text{NH}_4\text{OH}) = 0.1 \text{ M}$
 Method saving date 2005-10-21 14:07:29 UTC+2
 Method version 1
 Method group Robotic Transfer Analyzer
 Method status original
 Method saved by (full name) Metrohm
 Method saved by (short name) Metrohm

START Main track

General

Workplace view
 Current view on
 Track view for live window
 Live display 1 Main track
 Live display 2 Main track
 Statistics on
 Number of single determinations not defined
 Conditioning
 Automatic conditioning off

Application note

See attached documents

Method variables

Name	Type	Assignment	Value	Comment	Monitoring
Sample size	Number	Sample size		Sample size	off
Sample size unit	Text	Sample size unit		Sample size unit	off
Sample position	Number	Sample position		Sample position number	off
ID1	Text	ID1		Sample identification 1	off
ID2	Text	ID2		Sample identification 2	off
ID3	Text	ID3		Sample identification 3	off

Name **Sample size**
 Type Number
 Assignment on Sample size
 Value off
 Check at start on
 Comment Sample size
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on



License ID 124049905
 Client name TITRATION14
 User Metrohm

Program version tiamo 1.1 - 32

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Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample position**
 Type Number
 Assignment on. Sample position
 Value off.
 Check at start on
 Comment Sample position number
 Variable monitoring off
 Lower limit
 Upper limit
 Message
 Display message on
 Record message on
 Message by e-mail off
 Mail to
 Subject Message from tiamo - Method 'New method 1' - Command 'Main track'
 User
 Mail from
 SMTP Server
 POP3 Server
 Acoustic signal off
 Action off
 Stop determination on
 Stop determination and series off

Name **Sample size unit**
 Type Text
 Assignment on. Sample size unit
 Value off.
 Check at start on
 Comment Sample size unit

Name **ID1**
 Type Text



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 User Metrohm

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Assignment on. ID1
 Value off.
 Check at start off
 Comment Sample identification 1

Name **ID2**
 Type Text
 Assignment on. ID2
 Value off.
 Check at start off
 Comment Sample identification 2

Name **ID3**
 Type Text
 Assignment on. ID3
 Value off.
 Check at start on
 Comment Sample identification 3

PUMP

add water
 Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off
 Duration on
 Time 20 s

SEQUENCE sample transfer

LQH

compensate
General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Compensate
 Port 3

LQH

produce first air bubble
General/Hardware
 Device
 Device name 855_1



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 Client name TITRATION14
 User Metrohm

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Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

MOVE to sample

Device
 Device name 855_1
 Target
 Tower 1
 Move Sample position
 Beaker test
 Display message off
 Stop determination off
 Stop determination and series on

Parameters

Shift rate 20 %/s
 Shift direction auto
 Swing rate 55 %/s

LIFT lift to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH aspirate sample

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters

Function Aspirate
 Port 3
 Volume = 'MV.Sample size' mL
 Rate 5 mL/min

WAIT adjusting pressure

Wait



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Stop track and waiting for [Continue] off
 Stop all tracks and waiting for [Continue] off
 Waiting time on
 Time 3
 Unit s
 Message
 Record message off
 Message by e-mail off
 Acoustic signal off

LIFT lift to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

LQH produce second air bubble

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Aspirate
 Port 3
 Volume 0.1 mL
 Rate 5 mL/min

LQH change port

General/Hardware
 Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer
Parameters
 Function Change port
 Port 2

SWING move to external position

Device
 Device name 855_1
 Target
 Tower 1



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 User Metrohm

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```

    Swing . . . . . External position
    Number . . . . . 1
    Parameters
    Swing rate . . . . . 55 %/s

LIFT    go down to work position
    Device
    Device name . . . . . 855_1
    Target
    Tower . . . . . 1
    Lift position . . . . . Work position mm
    Parameters
    Lift rate . . . . . 25 mm/s

LQH    compensation
    General/Hardware
    Device
    Device name . . . . . 855_1
    Dosing device
    Dosing device . . . . . 2
    Solution . . . . . Transfer
    Parameters
    Function . . . . . Compensate
    Port . . . . . 3

LQH    dose sample
    General/Hardware
    Device
    Device name . . . . . 855_1
    Dosing device
    Dosing device . . . . . 2
    Solution . . . . . Transfer
    Parameters
    Function . . . . . Dosing
    Port . . . . . 3
    Volume . . . . . 8 mL
    Rate . . . . . 10 mL/min

WAIT   pressure adjustment
    Wait
    Stop track and waiting for [Continue] . . . . . off
    Stop all tracks and waiting for [Continue] . . . . . off
    Waiting time . . . . . on
    Time . . . . . 3
    Unit . . . . . s
    Message
    Record message . . . . . off
    Message by e-mail . . . . . off
    
```



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 Client name TITRATION14
 User Metrohm 2005-10-21 14:07:35 UTC+2

Acoustic signal off

LIFT to shifting position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

DET pH DET pH

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 1
 Solution HCl
 Sensor
 Measuring input 1
 Sensor Aquatrode Plus
 Temperature measurement automatic
 Stirrer
 Stirrer 1
 Stirring rate 8
 Switch off automatically on

Start conditions

Initial measured value
 Signal drift off mV/min
 Min. waiting time 0 s
 Max. waiting time 1 s
 Start volume
 Start volume 0.5 mL
 Dosing rate maximum mL/min
 Start measured value
 Start measured value pH off
 Dosing rate 5 mL/min
 Start slope
 Start slope off pH/mL
 Dosing rate 5 mL/min
 Pause
 Pause 5 s

Titration parameters

Titration rate
 Titration rate user
 Measured value acceptance
 Signal drift 50.0 mV/min



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 User Metrohm

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Min. waiting time 0 s
 Max. waiting time 26 s
 Dosing of increments
 Measuring point density 4
 Min. increment 50 µL
 Max. increment off µL
 Dosing rate maximum mL/min
 Temperature
 Temperature 25.0 °C

Stop conditions

Stop volume 20 mL
 Stop measured value pH off
 Stop EP 1
 Volume after EP 1.5 mL
 Stop time off s
 Filling rate maximum mL/min

Potentiometric evaluation

Evaluation without window on
 EP criterion 5
 EP recognition all
 Evaluation with measured value window (pH) off
 Evaluation with volume window (mL) off

Additional evaluations

Fix end point evaluation off
 pK/HNP evaluation off
 Minimum evaluation off
 Maximum evaluation off
 Break point evaluation off

Additional measured values

Additional calculated measured values off
 Additional external measured values off

SEQUENCE clean vessel

LIFT down to working position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

LQH empty burette

General/Hardware

Device
 Device name 855_1
 Dosing device



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 User Metrohm

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Dosing device 2
 Solution Transfer

Parameters

Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP aspirate sample solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10.0 s

PUMP rinse and aspirate

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 7 s

PUMP empty beaker

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT go to shift position

Device
 Device name 855_1
 Target
 Tower 1



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Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

EXIT Exit track

CALC calculation

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Content	= 'DET pH.EP{1}.VOL' * 'DET pH.CONC' * 'DET pH.TITER' / 'MV.Sample size'	mol/L	4	RS01	off

Result name **Content**
 Formula = 'DET pH.EP{1}.VOL' * 'DET pH.CONC' * 'DET pH.TITER' / 'MV.Sample size'
 Unit mol/L
 Decimal places 4
 Assignment RS01
 Statistics off
 Description RS.'Result name'[.VAL]Result value.
 Result monitoring off
 Save result as common variable off
 Name
 Save result as titer off
 Solution name

REPORT report

Report template
 Report template Determination of c(NH4OH) = 0.1 M
 Report output
 Printer off
 PDF file on
 PDF file C:\Programme\Metrohm\tiamo\DET NH4OH 0.1 M.pdf
 Send e-mail off

DATABASE database

Database
 Robotic Transfer Analyzer

SERIES START Series start track

RACK initialize rack

Device
 Device name 855_1
 Rack test off



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SEQUENCE prepare vessel and dosino

SWING swing to titration vessel

Device
 Device name 855_1
 Target
 Tower 1
 Swing External position
 Number 1
 Parameters
 Swing rate 55 %/s

PUMP empty titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

LIFT lift to work position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Work position mm
 Parameters
 Lift rate 25 mm/s

PREP prepare dosino

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

LQH eject to end volume

General/Hardware

Device
 Device name 855_1
 Dosing device
 Dosing device 2
 Solution Transfer

Parameters



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Function Eject to end volume
 Port 3
 Rate maximum mL/min

PUMP clean titration vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1+2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 10 s

PUMP aspirate solution

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 2
 Action
 Switch on off
 Switch off off
 Duration on
 Time 3 s

LIFT lift to shift position

Device
 Device name 855_1
 Target
 Tower 1
 Lift position Shift position mm
 Parameters
 Lift rate 25 mm/s

**SERIES Series end track
 END**

PUMP fill vessel

Device
 Device name 855_1
 Pumps
 Tower 1
 Pump(s) 1
 Action
 Switch on off
 Switch off off



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Client name TITRATION14
User Metrohm

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Duration on
Time 20 s

ERROR Error track

PUMP empty vessel

Device
Device name 855_1
Pumps
Tower 1
Pump(s) 2
Action
Switch on off
Switch off off
Duration on
Time 10.0 s

PUMP add water to vessel

Device
Device name 855_1
Pumps
Tower 1
Pump(s) 1
Action
Switch on off
Switch off off
Duration on
Time 20 s



Metrohm Ltd., CH-9101 Herisau

Switzerland

Phone

+41 71 353 85 85

Fax

+41 71 353 89 01

E-Mail

info@metrohm.com

Internet

www.metrohm.com