

# Robotic Chloride Analyzer



Applications  
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# **Robotic Chloride Analyzer**

## **Applications**



**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).
$pK_p$	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\Chloride

(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 Chloride Analyzer**

1.855.0010	855 Robotic Titrosampler
1.772.0110	Pump unit
1.800.0010	Dosino 2x
1.802.0010	Rod stirrer
6.0430.100	Ag Titrode OK
6.1236.020	Sleeve with SGJ 14/12 mm 2x
6.1458.040	Titration head insert 'Macro'
6.1459.300	Sample beakers 100x
6.1462.170	Robotic arm with sensor
6.1543.170	Aspiration tip M8
6.1546.030	Piston rod
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 2x
6.1805.510	PTFE tubing M8, 60 cm
6.1812.000	PTFE tubing 4/6 mm, 4m 2x
6.1820.050	Screw connector 2x
6.1826.100	Pump tubing PP 6.4 mm
6.1828.000	PVDF connection nipple 2x
6.1909.020	Stirring propeller 104 mm
6.2001.120	Bottle holder base, right
6.2041.840	Sample rack 59 x 120 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
6.2621.030	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
6.2711.070	Drip pan
6.2740.020	Spray nozzle 3x
6.2751.100	Safety shield
6.3032.150	Dosing Unit 5 mL
6.3032.220	Dosing Unit 20 mL
6.6056.112	tiamo 1.1 full version CD
T.2400.102	Ferrite cores 4x

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# 1 Application Information

## 1.1 Introduction: titration means counting!

Together with gravimetry, titration is one of the oldest analytical methods. Both belong to a group of analytical methods that is based on chemical reaction.

In a titration one determines the volume of a standard solution (titrant) that is necessary for complete chemical reaction with the analyte. The titrant contains a known amount of a particular substance.

Since Loschmidt and Avogadro we know that one gram molecule of a substance contains a defined number of particles. A standard solution is produced by dissolving a particular weight of a substance in a solvent. Each volume fraction of this standard solution contains a defined number of particles of the dissolved substance. This means that measuring the volume of a standard solution is a method of counting particles: Titration means counting!

Despite many new, mainly physical instrumental analytical methods, titrimetry as a «wet-chemistry method» still remains a standard procedure for quantitative analysis today. This is because it has a number of specific advantages:

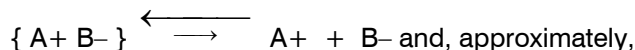
- Titration is one of the absolute content determination methods, i.e. the result of the analysis provides direct information about the amount of substance to be determined, without instrument or method-specific factors having to be calibrated (such as is normal in relative methods, for example HPLC, atomic spectroscopy or UV/VIS photometry).
- 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 Precipitation reactions

### Solubility and solubility product

The solubility of a dissociated sparingly soluble compound is determined by the maximum ionic product or *solubility product*  $K_L$ . This means that the product of the molar concentrations of the dissolved ions of a salt cannot be larger than the value  $K_L$ . In equilibrium this value becomes established when so much of the sparingly soluble compound is added to water that a precipitate is formed. This value is not exceeded even when the amount of precipitate is increased!

The solubility  $L$  is understood to be the total concentration of the substance in its saturated solution. For a binary hardly soluble salt the following applies:



$$c(\text{A}^+) \cdot c(\text{B}^-) = K_L \text{ (for the stoichiometric solubility product)}$$

$$L = c(\text{AB})_{\text{dissolved}} = c(\text{A}^+) = c(\text{B}^-)$$

$$L = \sqrt{K_L}$$

With AgCl as an example:  $K_L = 10^{-10} \text{ mol}^2/\text{L}^2$ ,  $L = 10^{-5} \text{ mol/L}$

Precipitation only begins when the product of the molar concentrations of the dissolved ions A and B, the ionic product  $c(\text{A}^+) \cdot c(\text{B}^-) > K_L$ . Tables of solubility products show that the known  $K_L$  values of salts differ by many factors of ten and in aqueous solution for 1:1 electrolytes values between approx.  $10^2$  (e.g. NaOH) and  $10^{-52}$  (e.g. HgS) are achieved. (For HgS this would mean that only  $10^{-26} \text{ mol/L}$  Hg should dissolve. However, the actual solubility is considerably higher – precipitate aging, complex formation and other effects also play a role. Strictly speaking, the molar concentrations should also be replaced by the activities.)

The equations mentioned above only apply when the dissolved ions  $\text{A}^+$  and  $\text{B}^-$  are in equilibrium with the precipitate. If one of the partners occurs in excess (*isoionic addition*), then the solubility depends on the ion with the lower concentration. With isoionic addition the solubility decreases – the solubility product is never exceeded. (This is why in gravimetry an excess of the precipitation reagent is always used.)

If foreign ions are added (*foreign ion addition*) the opposite takes place – the solubility increases. (In this case the *coefficients of activity* of all the ions present in solution – the *total ionic strength* – play a role.)

The determination limit for precipitation titrations depends very strongly on the solubility product. Concentrations below the solubility (the precipitation) can no longer be titrated. In some cases the solubility product can be reduced by the addition of an organic solvent (e.g. acetone or ethanol); this also results in a lower determination limit. (For chloride titration with  $\text{AgNO}_3$  the determination limit in  $\text{H}_2\text{O}$  is approx.  $3.5 \text{ mg/L}$ ; in glacial acetic acid, for example AOX determination, it is a few  $\mu\text{g/L}$ .)

Many precipitates formed during the titration are voluminous and inclusions could occur (sample ion and/or titrant). A more finely distributed precipitate, almost free from inclusions, is obtained by the addition of protective colloids (e.g. polyvinyl alcohol) or organic solvents (e.g. acetone or ethanol).

For  $\text{AgNO}_3$  titrations Ag electrodes (Ag Titrode) are usually used for indication of the titrations, with ISEs usually being used for the determination of other ions.

In this Application Information we are only concerned with substances that form sparingly soluble precipitates and can therefore be determined titrimetrically.

**Inorganic compounds**

Ion (analyte)	Titrant	Precipitation product
Ag <sup>+</sup>	KBr	AgBr
Ba <sub>2</sub> <sup>+</sup>	Na <sub>2</sub> SO <sub>4</sub>	BaSO <sub>4</sub>
Bromide	AgNO <sub>3</sub>	AgBr
Chloride	AgNO <sub>3</sub>	AgCl
Cyanide	AgNO <sub>3</sub>	AgCN
Fluoride	La(NO <sub>3</sub> ) <sub>3</sub>	LaF <sub>3</sub>
Iodide	AgNO <sub>3</sub>	AgI
Sulfate	BaCl <sub>2</sub>	BaSO <sub>4</sub>
Sulfide	AgNO <sub>3</sub>	Ag <sub>2</sub> S
Thiocyanate	AgNO <sub>3</sub>	AgSCN

In order to be able to titrate quantitatively the various ions present in a mixture, the differences between their solubility products must be at least three factors of ten. This means that the determination of bromide/chloride mixtures is only possible at a ratio of 1:1. If this ratio changes then problems occur or the ions can no longer be determined quantitatively in the mixture. (In contrast, chloride and iodide mixtures can be separated titrimetrically without any problems.)

The formation of precipitates from mixtures depends strictly on the solubility products of the individual components. The most sparingly soluble product is always titrated first, e.g. in the titration of halides with AgNO<sub>3</sub> the sequence is always iodide – bromide – chloride.

**Solubility products of some selected substances at 25 °C \***

Substance	KL	Substance	KL
BaCO <sub>3</sub>	8.1 x 10 <sup>-9</sup> mol <sup>2</sup> /L <sup>2</sup>	AgCl	1.56 x 10 <sup>-10</sup> mol <sup>2</sup> /L <sup>2</sup>
BaSO <sub>4</sub>	1.08 x 10 <sup>-19</sup> mol <sup>2</sup> /L <sup>2</sup>	Ag <sub>2</sub> CrO <sub>4</sub>	9 x 10 <sup>-12</sup> mol <sup>3</sup> /L <sup>3</sup>
CaF <sub>2</sub>	3.95 x 10 <sup>-11</sup> mol <sup>3</sup> /L <sup>3</sup>	AgI	1.5 x 10 <sup>-16</sup> mol <sup>2</sup> /L <sup>2</sup>
CaC <sub>2</sub> O <sub>4</sub> x H <sub>2</sub> O	2.57 x 10 <sup>-9</sup> mol <sup>2</sup> /L <sup>2</sup>	AgSCN	1.16 x 10 <sup>-12</sup> mol <sup>2</sup> /L <sup>2</sup>
PbCrO <sub>4</sub>	1.77 x 10 <sup>-14</sup> mol <sup>2</sup> /L <sup>2</sup>	AgCN	2.2 x 10 <sup>-12</sup> mol <sup>2</sup> /L <sup>2</sup>
PbSO <sub>4</sub>	1.06 x 10 <sup>-8</sup> mol <sup>2</sup> /L <sup>2</sup>	Ag <sub>2</sub> S	1.6 x 10 <sup>-49</sup> mol <sup>3</sup> /L <sup>3</sup>
PbS	3.4 x 10 <sup>-28</sup> mol <sup>2</sup> /L <sup>2</sup>	HgS	1 x 10 <sup>-52</sup> mol <sup>2</sup> /L <sup>2</sup>
AgBr	7.7 x 10 <sup>-13</sup> mol <sup>2</sup> /L <sup>2</sup>	Hg <sub>2</sub> S	1 x 10 <sup>-45</sup> mol <sup>3</sup> /L <sup>3</sup>
Ag <sub>2</sub> CO <sub>3</sub>	6.15 x 10 <sup>-12</sup> mol <sup>3</sup> /L <sup>3</sup>		

\* With most substances the solubility product increases as the temperature increases (more of the substance dissolves).

If a sensor (electrode) is used for the titration that responds to both the cation and the anion (ISE) then the titration curve is similar to that obtained for the titration of a strong acid with a strong base (pH electrode). This means that when the concentration changes by a factor of ten the electrode potential changes by approx. 59 mV (29 mV for z = 2). z corresponds to the valency, e.g. +2 for Ca. This also means that for the first 59 mV difference 90% of the

ions to be determined have been titrated, for the second 59 mV difference 99% of the ions have already been titrated, etc.

The resulting titration curves are symmetrical, and the point of inflection of the curve corresponds to the equivalence point/titration endpoint.

Self-dissociation/solubility product and dilution must be taken into account in order to obtain the true endpoint. In most cases, however, the resulting systematic error is so small that it hardly affects the result. (The modern evaluation software of the Metrohm titrators at least takes the «dilution error» into account.)

## 2 Titrant

### 2.1 Titer determination of $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.0430.100 Ag Titrode coated with  $\text{Ag}_2\text{S}$ , 6.2104.020 electrode cable

#### Reagents

- $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  (e.g. Merck no. 109081)  
or: Weigh 16.988 g  $\text{AgNO}_3$  into a 1000 mL volumetric flask and dissolve in approx. 500 mL dist.  $\text{H}_2\text{O}$ . Add 0.1 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$ , make up to the mark with dist.  $\text{H}_2\text{O}$  and mix. The solution must be stored in a dark bottle.
- $c(\text{HNO}_3) = 2 \text{ mol/L}$  (dilute conc.  $\text{HNO}_3$  with dist.  $\text{H}_2\text{O}$  1:5)
- Sodium chloride ( $\text{NaCl}$ , e.g. Merck no. 102406)

#### Titer determination

Dry the sodium chloride overnight in a drying oven at  $140 \text{ }^\circ\text{C}$  and leave to cool in a desiccator for at least 2 hours. The temperature must be constant during the titrations.

The titer is usually determined in triplicate and the mean value is stored as titer.

#### Procedure

Weigh approx. 60 mg  $\text{NaCl}$  with an accuracy of 0.1 mg into the titration beaker and dissolve in approx. 50 mL dist.  $\text{H}_2\text{O}$ . Add 1 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

#### Calculation of the titer

$$\text{Titer} = \text{'MV.Sample size'} * 1000 / (\text{'DET U.EP\{1\}.VOL'} * \text{'DET.U CONC'} * 58.442)$$

where:

'MV.Sample size'	weight of the $\text{NaCl}$ [g]
1000	conversion factor for millilitres
'DET U.EP\{1\}.VOL'	mL $\text{AgNO}_3$ consumed up to the endpoint
'DET.U CONC'	concentration of the $\text{AgNO}_3$
58.442	molecular weight of $\text{NaCl}$ [g/mol]

## 2.2 *tiamo* method: Titer determination of $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$

### Application note

With this method the titer of a solution of  $\text{AgNO}_3$  can be determined. Once the titrant bottle is open the titer value changes slightly. With the titration of a primary standard the actual titer is determined. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the dissolving of the primary standard and the titration followed by the rinsing of the electrode with water, 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 Chloride Analyzer' (can be modified). In the series end track the electrode is moved to a storage beaker where it is stored in dist. water for a proper treatment of the electrode between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is moved to the storing beaker.

### Sample preparation

The sodium chloride ( $\text{NaCl}$ ) is dried over night in a drying oven at  $140^\circ \text{C}$  and allowed to cool down in a desiccator for at least 2 hours. For a determination approx. 60 mg  $\text{NaCl}$  are weighed out in a beaker with an accuracy of 0.1 mg.

### Procedure

The beakers are placed on the rack and the determination series started. Approximately 60 mL dist. water and 1 mL of  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added automatically for dissolving. Then the solution is titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

### Remarks:

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to work position. If this requirement can not be fulfilled no beaker will be recognized. One special beaker has to be defined including its work position. Special beaker 1 is used for a proper electrode treatment between determination series and in case of an error.

**Result report (example)**



Robotic Chloride Analyzer  
Titer determination

Programm version tiamo 1.1

2005-11-02 09:04:22 UTC+1

**Results report**

**Determination**

Method . . . . . Titer determination of c(AgNO<sub>3</sub>) = 0.1 M  
 Method saving date . . . . . 2005-09-21 11:00:53 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 75565b4a:10677812b2d:-7da9  
 Determination start . . . . . 2005-09-21 11:58:15 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 17  
 User (full name) . . . . . Metrohm

**Sample data**

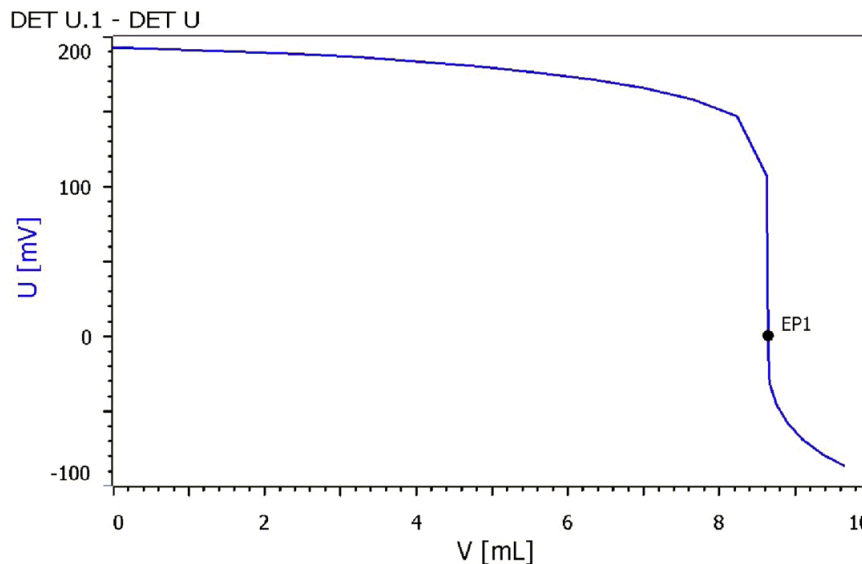
Sample Identification 1 . . . . . Titer AgNO<sub>3</sub> (0.1 M)  
 Sample size . . . . . 0.0497 g

**End points**

DET U     DET U.1  
 EP1 . . . . . 0.3 mV . . . . . 8.6497 mL

**Results**

Titer AgNO<sub>3</sub> . . . . . **0.9832**  
 Mean value titer . . . . . **1.0004**



**Statistical data (short)**



Method . . . . . Titer determination of c(AgNO<sub>3</sub>) = 0.1 mol\_L  
 Number of single determinations . . . . . 10

Result name	n	Mean value	s +/-	s rel
Titer AgNO <sub>3</sub>	10	1.0004	0.00750	0.75 %

**Statistical results (example)**

*Titer determination AgNO<sub>3</sub> (0.1M)*

Sample size [g]	Titer
0.0571	1.0001
0.0501	1.0040
0.0512	1.0029
0.0566	0.9944
0.0600	0.9960
0.0564	1.0075
0.0499	1.0077
0.0566	1.0059
0.0547	1.0019
0.0497	0.9832
Mean value	1.0004
abs. std. dev.	0.0075
rel. std. dev. %	0.75

## 3 Typical applications for the Robotic Chloride Analyzer

### 3.1 Determination of chloride in tap water

#### Recommended accessories

- 6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating, 6.2104.020 electrode cable
- 6.3032.220 Dosing Unit, 20 mL

#### Reagents

- Titrant:  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$ . Purchased ready-to-use or self-prepared: dilute  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  1:10 with dist. H<sub>2</sub>O.
- Nitric acid:  $c(\text{HNO}_3) = \text{approx. } 2 \text{ mol/L}$ ; dilute  $w(\text{HNO}_3) = 65\%$  1:5 with dist. H<sub>2</sub>O.

#### Analysis

60 mL water sample is pipetted into the titration beaker. After adding 5 mL nitric acid titrate with  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$  to after the first endpoint.

#### Calculation

$$\text{mg/L chloride} = \text{'DET.U.EP\{1\}.VOL'} * \text{'DET.U TITER'} * \text{'DET.U CONC'} * 35.453 * 1000 / \text{'MV.Sample size'}$$

where:

'DET.U.EP{1}.VOL	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U TITER'	titer of the AgNO <sub>3</sub>
'DET.U CONC'	concentration of the AgNO <sub>3</sub> [mol/L]
35.453	molecular weight of NaCl [g/mol]
1000	conversion factor for gram
'MV.Sample size'	volume of the sample [mL]

#### Remarks

- The Ag<sub>2</sub>S coating on the Ag Titrode provides short response times and increases the long-term stability of this electrode.
- If a sample contains sulfides or hydrogen sulfide then these are comprised in the first, very distinct endpoint. Chloride is then calculated from the difference EP2 – EP1.
- As the chloride content in tap water is very low, it is advisable to add a standard of known chloride concentration to the sample and calculate the chloride content of the sample after the determination.

## 3.2 *tiamo* method: Chloride in tap water

### Application note

With this method the content of chloride in tap water can be determined. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the titration followed by the rinsing of the electrode 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 Chloride Analyzer (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is rinsed and moved to the storage beaker.

### Sample preparation

60 mL water sample are pipetted into a titration beaker.

### Procedure

The beakers are placed on the rack and the determination series started. 5 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added automatically to the sample. The solution is then titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint (with sulfides after the second endpoint).

The value 35.453 in the calculation formula is the molecular weight of chloride in [g/mol].

If a sample contains sulfides or hydrogen sulfide then these are comprised in the first, very distinct endpoint. Chloride is then calculated from the difference  $\text{EP2} - \text{EP1}$ .

### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized.

One special beaker has to be defined including its own work position. Special beaker 1 is used for a proper electrode treatment between determination series and in case of an error.

**Result report (example)**



Robotic Chloride Analyzer  
Chloride in tap water

Programm version tiamo 1.1

2005-11-02 10:24:26 UTC+1

**Results report**

**Determination**

Method . . . . . Chloride in tapwater  
 Method saving date . . . . . 2005-09-21 15:35:36 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 75565b4a:10677812b2d:-7cb6  
 Determination start . . . . . 2005-09-21 15:39:07 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 22  
 User (full name) . . . . . Metrohm

**Sample data**

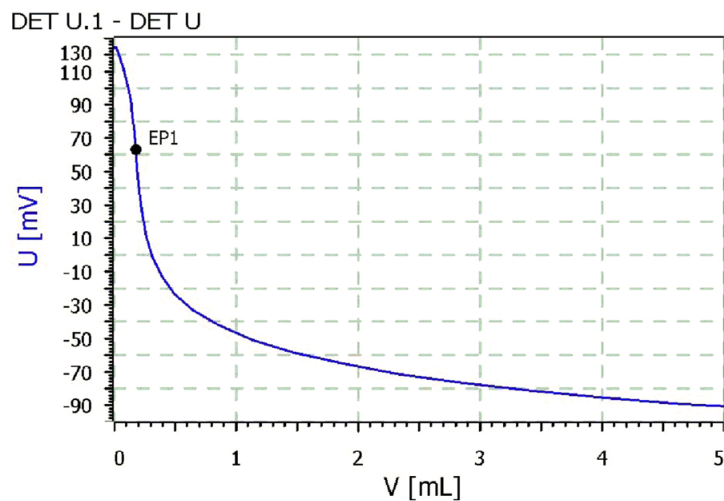
Sample Identification 1 . . . . . Tap water  
 Sample size . . . . . 60 mL

**End points**

**DET U**     **DET U.1**  
 EP1 . . . . . 63.0 mV . . . . . 0.1806 mL

**Results**

**content of chloride . . . . . 10.6773 mg/L**



**Statistical results (example)**

*Chloride in tap water*

Sample size [mL]	Content [mg/L]
60.0000	10.6773
60.0000	10.5519
60.0000	10.7684
60.0000	10.7010
60.0000	10.7223
60.0000	10.8660
60.0000	10.6539
60.0000	10.5465
60.0000	10.6228
60.0000	10.4422
Mean value	10.6552
abs. std. dev.	0.1217
rel. std. dev. %	1.14

### 3.3 Determination of chloride in wine

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating and 6.2104.020 electrode cable

#### Reagents

- Titrant:  $c(\text{AgNO}_3) = 0.05 \text{ mol/L}$ , e.g. Merck no. 111718  
OR: Weigh out 8.50 g AgNO<sub>3</sub> (e.g. Merck no. 101510) into a 1000 mL volumetric flask, add 1 mL  $w(\text{HNO}_3) = 65\%$ , dissolve in dist. H<sub>2</sub>O, make up to the mark and mix.
- Nitric acid:  $w(\text{HNO}_3) = 65\%$ , e.g. Merck no. 100462

#### General

The chloride content of wine is normally 50...200 mg/L (0.05...0.2 g/L). Contents of up to 600 mg/L have been found in wines from vineyards near the sea. In CH and the EU wines with a content of >500 mg/L chloride must be objected to. The USA and Au do not have any corresponding regulations.

The potentiometric titration method described below is a classic example of how a determination method can be simplified. The USA and SA describe methods that are preceded by complicated sample preparation steps for decoloring the wine. An additional complication is brought about by the determination itself, which is a back-titration.

#### Analysis

Pipet 50 mL sample into a beaker, treat with 1 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and approx. 25 mL dist. H<sub>2</sub>O and then titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  until after the first endpoint.

#### Calculations

The chloride content is given in g/L chloride and g/L NaCl.

$$\text{g/L Cl}^- = \text{'DET.U.EP\{1\}.VOL'} * \text{'DET.U.TITER'} * \text{'DET.U.CONC'} * 35.453 / \text{'MV.Sample size'}$$

$$\text{g/L NaCl} = \text{'DET.U.EP\{1\}.VOL'} * \text{'DET.U.TITER'} * \text{'DET.U.CONC'} * 58.442 / \text{'MV.Sample size'}$$

where:

'DET.U.EP\{1\}.VOL'	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U.TITER'	titer of the AgNO <sub>3</sub>
'DET.U.CONC'	concentration of the AgNO <sub>3</sub> [mol/L]
'MV.Sample size'	volume of the sample
35.453	molecular weight of chloride
58.442	molecular weight of sodium chloride

#### Remarks

The normal chloride content of wine is between 50...220 mg/L. Extreme values of 5 mg/L and 600 mg/L have also been found. There is normally no difference between red and white wines.

The concentration of the AgNO<sub>3</sub> should be adjusted to the sample in order to make sure that the specifications of the burette are complied.

### 3.4 *tiamo* method: Determination of chloride in wine

#### Application note

With this method the content of chloride in wine can be determined. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the titration followed by the rinsing of the electrode 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 Chloride Analyzer' (can be modified). In the series end track the electrode is moved to a storage beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is moved to the storing beaker.

#### Sample preparation

50 mL of the sample are pipetted into a titration beaker.

#### Procedure

The beakers are placed on the rack and the determination series started. Approximately 25 mL dist. water and 1 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added automatically. The solution is then titrated with  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$  and the titration stopped after the first endpoint.

Two results are calculated. One is the content of chloride and the other the content of sodium chloride in the sample. The values 35.453 and 58.442 respectively in the calculation formula are the molecular weights of chloride and sodium chloride in [g/mol].

#### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized.

One special beaker has to be defined including its work position. Special beaker 1 is used for a proper electrode treatment between determination series as well as in case of an error for both rinsing and storing.

**Result report (example)**



Robotic Chloride Analyzer  
Chloride in wine

Programm version tiamo 1.1

2005-09-28 14:42:18 UTC+2

**Results report**

**Determination**

Method . . . . . Determination of choride in wine  
 Method saving date . . . . . 2005-09-28 14:04:01 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 282da969:1069b4b5824:-7cf1  
 Determination start . . . . . 2005-09-28 14:34:50 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 10  
 User (full name) . . . . . Metrohm

**Sample data**

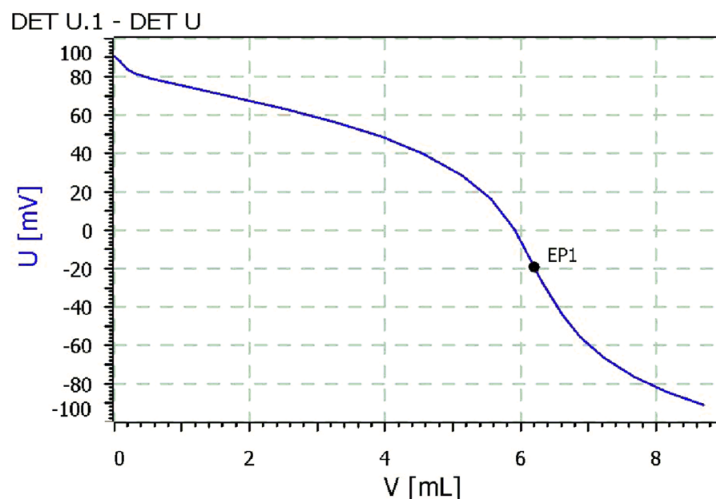
Sample Identification 1 . . . . . Red wine  
 Sample size . . . . . 50 mL

**End points**

DET U     DET U.1  
 EP1 . . . . . -19.3 mV . . . . . 6.2043 mL

**Results**

content of chloride . . . . . 0.0441 g/L  
 content of sodiumchlorid . . . . . 0.0727 g/L



**Statistical results (example)**

*Chloride in red wine*

Sample size [mL]	Content [g/L]
50.0000	0.0442
50.0000	0.0442
50.0000	0.0441
50.0000	0.0441
50.0000	0.0441
50.0000	0.0439
50.0000	0.0440
50.0000	0.0441
50.0000	0.0442
50.0000	0.0442
Mean value	0.0441
abs. std. dev.	0.0001
rel. std. dev. %	0.23

### 3.5 Determination of Ammonium chloride

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating
- 6.2104.020 electrode cable

#### Reagents

- Titrant I: c(AgNO<sub>3</sub>) = 0.1 mol/L

Any additional reagents required are mentioned under the individual substances.

#### General remarks

Direct titration with AgNO<sub>3</sub> is used for this method, which serves to determine halide contents (e.g. in hydrochlorides, salts, injection solutions) or organic halides after saponification (and reduction with Zn). The total substance content is then calculated from the halide content.

For the potentiometric indication of this titration we recommend the Ag Titrode with Ag<sub>2</sub>S coating as it has decisive advantages over the uncoated electrodes.

The Ag<sub>2</sub>S coating increases the electrode's stability compared to the uncoated Ag electrode. The potentials are established quickly and the electrode surface does not have to be re-polished – just wipe with a soft, moist paper tissue.

The Ag Titrode with Ag<sub>2</sub>S coating is a robust electrode that requires little maintenance.

#### General procedure

##### *Direct titration*

Weigh in and dissolve the sample, saponify if required, acidify and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

#### Calculations

Mass fraction  $w$  in % = 'DET U.EP{1}.VOL' \* 'DET.U TITER' \* 'DET.U CONC' \* 53.49 \* 100 / ('MV.Sample size' \* 1000)

where:

'DET U.EP{1}.VOL'	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U TITER'	titer of the AgNO <sub>3</sub>
'DET.U CONC'	concentration of the AgNO <sub>3</sub> [mol/L]
'MV.Sample size'	weight of the sample [g]
53.49	molecular weight of ammonium chloride [g/mol]
100	conversion factor for %
1000	conversion factor for liter

The following substances can also be titrated according to this method, though the calculation formula has to be adjusted:

#### **Aluminum chlorohydrate, chloride content (USP)**

Weigh approx. 0.700 g sample into a 100 mL volumetric flask, dissolve in dist. H<sub>2</sub>O, add 10 mL c(HNO<sub>3</sub>) = 2 mol/L, fill up to the mark with dist. H<sub>2</sub>O and mix. Pipet 10.0 mL of this solution (1/10 of the sample weight) into the titration beaker, add 50 mL dist. H<sub>2</sub>O and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

#### **Aluminum zirconium octachlorohydrate, chloride content (USP)**

Weigh approx. 0.250 g sample into the titration beaker and dissolve in 100 mL dist. H<sub>2</sub>O plus 10 mL c(HNO<sub>3</sub>) = 2 mol/L. Titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

#### **Ammonium bromide (Pharm.Europe)**

Weigh approx. 1.5 g sample into a 100 mL volumetric flask, dissolve in dist. H<sub>2</sub>O, fill up to the mark with dist. H<sub>2</sub>O and mix. Pipet 10.0 mL of this solution (1/10 of the sample weight) into the titration beaker, add 50 mL dist. H<sub>2</sub>O and 5 mL c(HNO<sub>3</sub>) = 2 mol/L and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (NH<sub>4</sub>Br) = 97.94 g/mol

#### **Ammonium chloride (USP)**

Weigh approx. 0.100 g sample into the titration beaker and dissolve in 50 mL dist. H<sub>2</sub>O. Add 2 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (NH<sub>4</sub>Cl) = 53.49 g/mol

#### **Diatrizoate meglumine (USP)**

Weigh approx. 0.400 g sample into a round-bottomed flask and add 30 mL c(NaOH) = 1.5 mol/L, 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist. H<sub>2</sub>O and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist. H<sub>2</sub>O. Add 5 mL glacial acetic acid to the filtrates and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>11</sub>H<sub>9</sub>I<sub>3</sub>N<sub>2</sub>O<sub>4</sub> x C<sub>7</sub>H<sub>17</sub>NO<sub>5</sub>) = 809.12 g/mol

#### **Diatrizoate sodium (USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 30 mL c(NaOH) = 1.5 mol/L, 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist. H<sub>2</sub>O and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist. H<sub>2</sub>O. Add 5 mL glacial acetic acid to the filtrates and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>11</sub>H<sub>8</sub>I<sub>3</sub>N<sub>2</sub>NaO<sub>4</sub>) = 635.88 g/mol

**Diatrizoic acid (USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 30 mL  $c(\text{NaOH}) = 1.5$  mol/L, 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 5 mL glacial acetic acid to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight ( $\text{C}_{11}\text{H}_9\text{I}_3\text{N}_2\text{O}_4$ ) = 613.90 g/mol

**Haemodialysis solution, chloride content (Pharm.Europe)**

Exactly measure into the titration beaker a sample volume corresponding to approx. 60 mg chloride. Add 50 mL dist.  $\text{H}_2\text{O}$  and 5 mL  $c(\text{HNO}_3) = 2$  mol/L and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Haemofiltration solution, chloride content (Pharm.Europe)**

Weigh into the titration beaker a sample volume corresponding to approx. 60 mg chloride. Add 50 mL dist.  $\text{H}_2\text{O}$  and 5 mL  $c(\text{HNO}_3) = 2$  mol/L and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Hydrocortisone sodium phosphate, chloride content (USP)**

Weigh approx. 3.0 g sample into the titration beaker and dissolve in 75 mL dist.  $\text{H}_2\text{O}$ . Add 1 mL  $w(\text{HNO}_3) = 65\%$  and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Iodipamide (USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 30 mL  $c(\text{NaOH}) = 1.5$  mol/L, 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 5 mL glacial acetic acid to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight ( $\text{C}_{20}\text{H}_{14}\text{I}_6\text{N}_2\text{O}_6$ ) = 1139.74 g/mol

**Iodipamide meglumine injection (USP)**

Pipet a sample volume corresponding to approx. 5 g iodipamide meglumine into a 100 mL round-bottomed flask and determine the sample weight. Fill up to the mark with  $c(\text{NaOH}) = 1.25$  mol/L and mix. Pipet 10.0 mL of this solution (1/10 of the sample weight) into a round-bottomed flask, add 20 mL  $c(\text{NaOH}) = 1.5$  mol/L, 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool down the mixture, rinse out the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the flask's contents through a sintered glass filter into a titration beaker. Rinse the flask and the filter several times with dist.  $\text{H}_2\text{O}$ . Add 5 mL glacial acetic acid to the combined filtrates and titrate with  $c(\text{AgNO}_3) = 0.1$  mol/L past the first endpoint.

Molecular weight ( $\text{C}_{20}\text{H}_{14}\text{I}_6\text{N}_2\text{O}_6 \times 2 \text{ C}_7\text{H}_{17}\text{NO}_5$ ) = 1530.17 g/mol

**Iohexol (Pharm.Europe & USP)**

Weigh approx. 0.500 g sample into a round-bottomed flask and add 25 mL  $w(\text{NaOH}) = 42\%$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 10 mL glacial acetic acid to the filtrates and titrate immediately with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{19}\text{H}_{26}\text{I}_3\text{N}_3\text{O}_9$ ) = 821.13 g/mol

**Iopamidol (Pharm.Europe & USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 5 mL  $w(\text{NaOH}) = 42\%$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 5 mL glacial acetic acid to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{17}\text{H}_{22}\text{I}_3\text{N}_3\text{O}_8$ ) = 777.07 g/mol

**Iopanoic acid (Pharm.Europe & USP)**

Weigh approx. 0.150 g sample into a round-bottomed flask and add 5 mL  $w(\text{NaOH}) = 42\%$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 40 mL  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{11}\text{H}_{12}\text{I}_3\text{NO}_2$ ) = 570.92 g/mol

**Iothalamic acid (Pharm.Europe & USP)**

Weigh approx. 0.150 g sample into a round-bottomed flask and add 5 mL  $w(\text{NaOH}) = 42\%$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 40 mL  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{11}\text{H}_9\text{I}_3\text{N}_2\text{O}_4$ ) = 613.90 g/mol

**Ioversol (USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 12 mL  $c(\text{NaOH}) = 5 \text{ mol/L}$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 40 mL  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{18}\text{H}_{24}\text{I}_3\text{N}_3\text{O}_9$ ) = 807.10 g/mol

**Ioxaglic acid (USP)**

Weigh approx. 0.500 g (0.300 g) sample into a round-bottomed flask and add 12 mL  $c(\text{NaOH}) = 5 \text{ mol/L}$ , 20 mL dist.  $\text{H}_2\text{O}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 40 mL  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{24}\text{H}_{21}\text{I}_6\text{N}_5\text{O}_8$ ) = 1268.86 g/mol

**Ioxilan (USP)**

Weigh approx. 0.500 g (0.300 g) sample into a round-bottomed flask and add 40 mL  $c(\text{NaOH}) = 1.25 \text{ mol/L}$ , 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$ , add 5 mL glacial acetic acid and leave to cool. Filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter with  $c(\text{CH}_3\text{COOH}) = 1 \text{ mol/L}$ . Titrate the filtrates with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{18}\text{H}_{24}\text{I}_3\text{N}_3\text{O}_8$ ) = 791.10 g/mol

**Ipodate sodium (USP)**

Weigh approx. 0.300 g sample into a round-bottomed flask and add 30 mL  $c(\text{NaOH}) = 1.25 \text{ mol/L}$ , 0.5 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 1 hour. Cool the solution, rinse the condenser with 20 mL dist.  $\text{H}_2\text{O}$  and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist.  $\text{H}_2\text{O}$ . Add 5 mL glacial acetic acid to the filtrates and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{12}\text{H}_{12}\text{I}_3\text{N}_2\text{NaO}_2$ ) = 619.93 g/mol

**Ipratropium bromide (Pharm.Europe)**

Weigh approx. 0.350 g sample into the titration beaker and dissolve in 50 mL dist.  $\text{H}_2\text{O}$ . Add 3 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_{20}\text{H}_{30}\text{BrNO}_3$ ) = 412.37 g/mol

**Lindane (USP)**

Weigh approx. 0.400 g (0.150 g) sample into an Erlenmeyer flask, add 20 mL ethanol and heat up on a steam bath until the sample has dissolved completely. After the solution has cooled down, add 20 mL ethanolic  $w(\text{KOH}) = 5\%$ , mix thoroughly and leave standing for 10 min. Neutralize with  $c(\text{HNO}_3) = 2 \text{ mol/L}$ , then add another 5 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$ . Titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_6\text{H}_6\text{Cl}_6$ ) = 290.83 g/mol

**Lomustine (Pharm.Europe)**

Weigh approx. 0.200 g sample into a round-bottomed flask and dissolve in 3 mL ethanol. Add 20 mL  $w(\text{NaOH}) = 20\%$ . Mount a reflux condenser and boil for 2 hours. Add approx. 50 mL dist.  $\text{H}_2\text{O}$ , leave to cool and, using dist.  $\text{H}_2\text{O}$ , rinse quantitatively into the titration beaker. Add 4 mL  $w(\text{HNO}_3) = 65\%$  and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight ( $\text{C}_9\text{H}_{16}\text{ClN}_3\text{O}_2$ ) = 233.70 g/mol

**Mecamylamine hydrochloride, chloride content (USP)**

Weigh approx. 0.500 g (0.250 g) sample into the titration beaker and dissolve in 5 mL dist. H<sub>2</sub>O. Add 5 mL glacial acetic acid plus 50 mL methanol and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Multiple electrolyte injection, chloride content (USP)**

Weigh into the titration beaker a sample volume corresponding to approx. 50 mg chloride. Dilute with dist. H<sub>2</sub>O to approx. 50 mL, add 1 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Melphalan (USP)**

Weigh approx. 0.200 g sample into the titration beaker and dissolve in 20 mL c(NaOH) = 0.5 mol/L. Cover the beaker with a watch glass and boil the solution for 30 min. Periodically replace the water lost by evaporation by dist. H<sub>2</sub>O. After the solution has cooled down, add 50 mL dist. H<sub>2</sub>O and 5 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>13</sub>H<sub>18</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>) = 306.07 g/mol

**Meperidine hydrochloride, chloride content (USP)**

Weigh approx. 0.500 g (0.300 g) sample into the titration beaker and dissolve in 15 mL dist. H<sub>2</sub>O. Add 5 mL glacial acetic acid plus 50 mL methanol and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Methyclothiazide (USP)**

Weigh approx. 0.350 g sample into a round-bottomed flask and add 40 mL methanolic w(KOH) = 5%. Mount a reflux condenser and boil for 1 hour. After the solution has cooled down, rinse the condenser with 20 mL dist. H<sub>2</sub>O and rinse the contents of the flask into the titration beaker with two portions of 20 mL methanol each. Add 10 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>9</sub>H<sub>11</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>) = 360.24 g/mol

**Metrifonate (Pharm.Europe)**

Weigh approx. 0.300 g sample into an Erlenmeyer flask and dissolve in 30 mL ethanol. Add 10 mL ethanolamine and leave to react for 1 hour at 20...22 °C. Add 100 mL dist. H<sub>2</sub>O and 15 mL w(HNO<sub>3</sub>) = 65%, cool to room temperature and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>4</sub>H<sub>8</sub>Cl<sub>3</sub>P) = 193.44 g/mol

**Phenmetrazine hydrochloride, chloride content (USP)**

Weigh approx. 0.350 g (0.250 g) sample into the titration beaker, add 125 mL dist. H<sub>2</sub>O and 1.5 mL c(H<sub>2</sub>SO<sub>4</sub>) = 1 mol/L and leave to react for 15 min under stirring. Titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Potassium chloride (USP)**

Weigh approx. 0.200 g (0.100 g) sample into the titration beaker and dissolve in 10 mL dist. H<sub>2</sub>O. Add 10 mL glacial acetic acid and 75 mL methanol and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (KCl) = 74.55 g/mol

**Potassium iodide, oral solution and tablets (USP)**

Weigh into the titration beaker a sample quantity corresponding to approx. 200 mg KI and dissolve in 40 mL dist. H<sub>2</sub>O. Add 25 mL ethanol and 1 mL c(HNO<sub>3</sub>) = 1 mol/L and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (KI) = 166.00 g/mol

**Pralidoxime chloride, chloride content (USP)**

Weigh approx. 0.300 g (0.200 g) sample into the titration beaker and dissolve in 100 mL dist. H<sub>2</sub>O. Add 20 mL glacial acetic acid and 10 drops of (p-tert.-octylphenoxy) nonaethoxyethanol and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Pyridoxine hydrochloride, chloride content (USP)**

Weigh approx. 0.500 g (0.250 g) sample into the titration beaker and dissolve in 50 mL methanol. Add 5 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Ringer's solution, Ringer's dextrose injection, chloride content (USP)**

Pipet 10.0 mL sample into the titration beaker. Add 75 mL methanol and 10 mL glacial acetic acid and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Sodium amidotrizoate (Pharm.Europe)**

Weigh approx. 0.150 g sample into a round-bottomed flask and add 5 mL w(NaOH) = 42%, 20 mL dist. H<sub>2</sub>O, 1 g zinc powder and a few boiling stones. Mount a reflux condenser and boil for 30 min. Cool the solution, rinse the condenser with 20 mL dist. H<sub>2</sub>O and filter the contents of the flask through a sintered glass filter into the titration beaker. Rinse flask and filter several times with dist. H<sub>2</sub>O. Add 40 mL c(H<sub>2</sub>SO<sub>4</sub>) = 1 mol/L to the filtrates and titrate with c(AgNO<sub>3</sub>) = 0.1 mol/L past the first endpoint.

Molecular weight (C<sub>11</sub>H<sub>8</sub>I<sub>3</sub>N<sub>2</sub>NaO<sub>4</sub>) = 635.88 g/mol

**Sodium chloride (Pharm.Europe & USP)**

Weigh approx. 1.0 g sample into a 100 mL volumetric flask, dissolve in dist. H<sub>2</sub>O, fill up to the mark with dist. H<sub>2</sub>O and mix. Pipet 10.0 mL of this solution (1/10 of the sample weight) into the titration beaker, add 50 mL dist. H<sub>2</sub>O and 5 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight (NaCl) = 58.44 g/mol

**Sodium nitroprusside (Pharm.Europe & USP)**

Weigh approx. 0.200 g sample into the titration beaker and dissolve in 100 mL dist. H<sub>2</sub>O. Add 0.1 mL  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

1 mL  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  corresponds to 13.10 mg  $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}]$  (C01)

**Succinylcholine chloride, chloride content (USP)**

Weigh approx. 0.400 g (0.200 g) sample into the titration beaker and dissolve in 5 mL dist. H<sub>2</sub>O. Add 5 mL glacial acetic acid and 50 mL methanol and titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

**Zolazepam hydrochloride, chloride content (USP)**

Weigh approx. 0.400 g sample into the titration beaker, add 5 mL dist. H<sub>2</sub>O, 5 mL glacial acetic acid and 50 mL methanol and dissolve the sample. Titrate with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  past the first endpoint.

Molecular weight of chloride: 35.453 g/mol

### 3.6 *tiamo* method: Determination of ammonium chloride

#### Application note

With this method the mass fraction of ammonium chloride can be determined. The method consists of six different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the dissolving and the titration followed by the rinsing of the electrode with dist. water 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 Chloride Analyzer (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is moved to the storing beaker.

#### Sample preparation

Approximately 50 mg of the sample are weighed into a titration beaker with an accuracy of 0.1 mg.

#### Procedure

The beakers are placed on the rack and the determination series started. Approximately 60 mL of dist. water and 2 mL of  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added automatically for dissolving. The solution is then titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 53.49 in the calculation formula is the molecular weight of ammonium chloride in [g/mol]. The factor 100 is needed to receive percent as result.

#### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized .

Two special beaker have to be defined including their own work positions. Special beaker 1 is used for the rinsing of the electrode in case of an error. Special beaker 2 is used for the storing of the electrode between the determination series.

**Result report (example)**



Robotic Chloride Analyzer  
 Determination of ammonium chloride

Programm version tiamo 1.1

2005-09-26 10:28:10 UTC+2

**Results report**

**Determination**

Method . . . . . Determination of ammonium chloride  
 Method saving date . . . . . 2005-09-26 09:23:18 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -1667c48e:10691179cc0:-7ed9  
 Determination start . . . . . 2005-09-26 09:52:35 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 5  
 User (full name) . . . . . Metrohm

**Sample data**

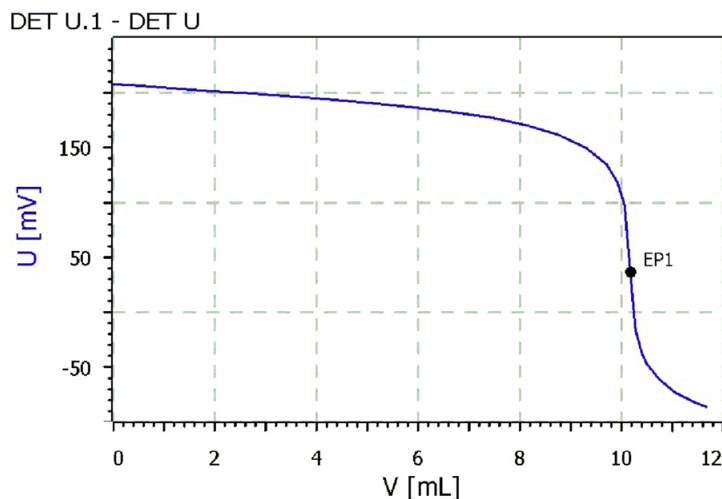
Sample Identification 1 . . . . . Ammoniumchlorid  
 Sample size . . . . . 0.0543 g

**End points**

DET U     DET U.1  
 EP1 . . . . . 36.6 mV . . . . . 10.1843 mL

**Results**

Mass fraction of chloride . . . . . **96.6114 %**



**Statistical results (example)***Chloride in ammonium chloride*

---

Sample size [mL]	Mass fraction [%]
50.0000	95.8703
50.0000	95.8037
50.0000	95.9569
50.0000	96.0416
50.0000	96.0601
50.0000	96.1211
50.0000	96.2679
50.0000	95.8551
50.0000	96.2017
50.0000	96.4129

---

Mean value	96.0591
abs. std. dev.	0.1961
rel. std. dev. %	0.20

### 3.7 Determination of Chloride content in acidic copper baths

#### Recommended accessories

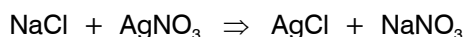
- 6.3032.220 Dosing Unit, 20 mL
- 6.0430.100 Ag Titrode coated with Ag<sub>2</sub>S, 6.2104.020 electrode cable

#### Reagents

- c(AgNO<sub>3</sub>) = 0.01 mol/L, c(AgNO<sub>3</sub>) = 0.1 mol/L diluted with dist. H<sub>2</sub>O 1:10.

#### General

Acidic copper baths contain only small amounts of chloride. The titration is therefore carried out with diluted AgNO<sub>3</sub> solution. Silver nitrate solution precipitates chloride ions as sparingly soluble silver chloride.



#### Analysis

Pipet 25.0 mL sample into the glass beaker and add approx. 35 mL dist. H<sub>2</sub>O. Titrate with c(AgNO<sub>3</sub>) = 0.01 mol/L past the first endpoint.

#### Calculations

mg/L chloride = 'DET.U.EP{1}.VOL' \* 'DET.U TITER' \* 'DET.U CONC' \* 35.453 \* 1000 / 'MV.Sample size'

where:

mg/L NaCl	'RS. content of chloride' * 1.64898
'DET.U.EP{1}.VOL'	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U TITER'	titer of AgNO <sub>3</sub>
'DET.U CONC'	concentration of AgNO <sub>3</sub>
35.453	molecular weight of chloride [g/mol]
1000	conversion factor for gram
'MV.Sample size'	volume of the sample [ml]
'RS. content of chloride'	determined content of chloride of the sample [mg/L]
1.64898	calculation factor

### 3.8 *tiamo* method: Chloride content in acidic copper baths

#### Application note

With this method the content of chloride in acidic copper baths can be determined. Acidic copper baths contain only small amounts of chloride. Therefore the titration is carried out with diluted  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$  solution. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the titration and the rinsing of the electrode 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 Chloride Analyzer (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is moved to the storing beaker.

#### Sample preparation

25 mL of the copper bath are pipetted in a sample beaker.

#### Procedure

The beakers are placed on the rack and the determination series is started. Approximately 30 mL of dist. water are added automatically. The solution is then titrated with  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 35.453 in the calculation formula is the molecular weight of chloride in  $[\text{g/mol}]$ . 1000 is a correction factor for the millilitres. The factor 1.64898 is used to calculate the content of sodium chloride.

#### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized.

Two special beaker have to be defined including their own work positions. Special beaker 2 is used for a proper electrode treatment between the determination series. In case of an error the electrode is moved to special beaker 1 where it is rinsed and then it is moved to special beaker 2.

**Result report (example)**



Robotic Chloride Analyzer  
Chloride in acidic copper baths

Programm version tiamo 1.1

2005-10-31 14:59:14 UTC+1

**Results report**

**Determination**

Method . . . . . Chloride in acidic copper baths  
 Method saving date . . . . . 2005-09-27 10:36:36 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -4194d45f:1069643070f:-7d77  
 Determination start . . . . . 2005-09-27 10:58:57 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 22  
 User (full name) . . . . . Metrohm

**Sample data**

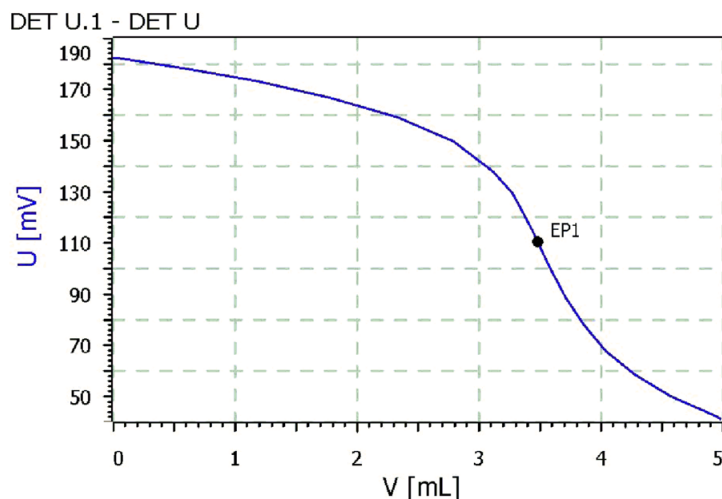
Sample Identification 1 . . . . . Acidic copper bath  
 Sample size . . . . . 25 mL

**End points**

DET U     DET U.1  
 EP1 . . . . . 110.3 mV . . . . . 3.4831 mL

**Results**

content of chloride . . . . . **49.4982 mg/L**  
 content of sodiumchlorid . . . . . **81.6215 mg/L**



**Statistical results (example)**
*Chloride in acidic copper baths*

Sample size [mL]	Content [mg/L]
25.0000	81.8248
25.0000	82.1294
25.0000	81.9918
25.0000	81.8631
25.0000	81.7045
25.0000	81.6085
25.0000	81.6215
25.0000	81.3078
25.0000	81.2583
25.0000	81.4487
Mean value	81.6758
abs. std. dev.	0.2853
rel. std. dev. %	0.35

### 3.9 Chloride in broth

#### Recommended accessories

- 6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating, 6.2104.020 electrode cable
- 6.3032.220 Dosing Unit, 20 mL

#### Reagents

- Titrant:  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$
- Nitric acid:  $c(\text{HNO}_3) = \text{approx. } 2 \text{ mol/L}$   
 $w(\text{HNO}_3) = 65\%$  is diluted 1:5 with dist. H<sub>2</sub>O
- Protective colloid: polyvinyl alcohol solution  
 40 mg polyvinyl alcohol are dissolved in 100 mL hot dist. water and allowed to cool down.

#### Sample preparation

Powdered, granulated and pasty samples: 10.0 g are dissolved in approximately 500 mL hot dist. water, allowed to cool down, made up to 1 liter and mixed.  
 Liquid seasonings: 25.0 g are diluted to 1 liter with dist. water.

#### Analysis

15 mL of the prepared sample solution are pipetted into the titration beaker and diluted with dist. water to approx. 60 mL. After the addition of 2 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and 5 mL protective colloid the mixture is titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  to after the first endpoint.

#### Calculations

The chloride content is given in % NaCl (g/100 g).

$$\% \text{ NaCl} = \frac{\text{'DET U.EP\{1\}.VOL' * 'DET.U TITER' * 'DET.U CONC' * 58.443 * 0.1}}{\text{'MV.Sample size'}}$$

'DET U.EP{1}.VOL'	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U TITER'	titer of AgNO <sub>3</sub>
'DET.U CONC'	concentration of AgNO <sub>3</sub> [mol/L]
58.443	molecular weight of NaCl [g/mol]
0.1	conversion factor for %
'MV.Sample size'	0.15 [g] (corresponds to 15 mL of the sample solution)

### 3.10 *tiamo* method: Chloride in broth

#### Application note

This is a method to determine the content of chloride in broth. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the titration followed by the rinsing of the electrode 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 Chloride Analyzer (can be modified). In the series end track, the electrode is moved to a storing beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is rinsed and moved to the storing beaker.

#### Sample preparation

Powdered, granulated and pasty samples: 10.0 g are dissolved in approximately 500 mL hot dist. water, allowed to cool down, made up to 1 liter and mixed. For a determination 15 mL of the prepared sample (corresponds to 0.15 g of the solid sample) are pipetted into a titration beaker and 5 mL of a polyvinyl alcohol solution (80 mg polyvinyl alcohol dissolved in 200 mL dist. water) are added.

#### Analysis

The beakers are placed on the rack and the determination series started. Approximately 35 mL of dist. water and 2 mL of  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added. The solution is then titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 58.443 in the calculation formula is the molecular weight of sodium chloride in [g/mol].

#### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized.

Two special beaker have to be defined including their own work positions. Two special beaker have to be defined including their own work positions. Special beaker 1 is used for the rinsing of the electrode in case of an error. Special beaker 2 is used for the storing of the electrode between the determination series.

**Result report (example)**



Robotic Chloride Analyzer  
Chloride in broth

Programm version tiamo 1.1

2005-10-31 14:56:35 UTC+1

**Results report**

**Determination**

Method . . . . . Chloride in broth  
 Method saving date . . . . . 2005-10-31 14:50:37 UTC+1  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 4c621812:10745c61df2:-7bf5  
 Determination start . . . . . 2005-09-23 09:10:25 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 16  
 User (full name) . . . . . Metrohm

**Sample data**

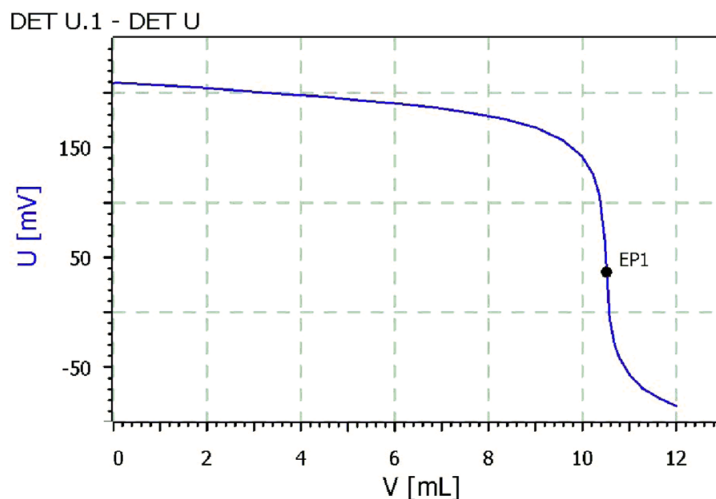
Sample Identification 1 . . . . . Broth  
 Sample size . . . . . 0.15 g

**End points**

DET U     DET U.1  
 EP1 . . . . . 36.6 mV . . . . . 10.5224 mL

**Results**

content of sodium chloride . . . . . **41.0136 %**



**Statistical results (example)***Chloride in broth*

Sample size [mL]	Content [%]
15.0000	40.9843
15.0000	40.9898
15.0000	41.0063
15.0000	41.0145
15.0000	41.0076
15.0000	41.0059
15.0000	41.0020
15.0000	41.0287
15.0000	41.0136
15.0000	41.0249
Mean value	41.0078
abs. std. dev.	0.0138
rel. std. dev. %	0.03

### 3.11 Chloride (NaCl) in washing agents and soaps

#### Recommended accessories

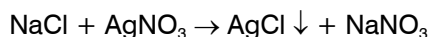
- 6.3032.220 Dosing Unit, 20 mL
- 6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating
- 6.2104.020 electrode cable

#### Reagents

- Titrant:  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$   
1.699 g AgNO<sub>3</sub> (e.g. Merck no. 101512) is weighed out into a 1000 mL volumetric flask and dissolved in approx. 500 mL dist. H<sub>2</sub>O. The solution is treated with 0.5 mL conc. HNO<sub>3</sub> and made up to the mark with dist. H<sub>2</sub>O.
- Nitric acid:  $c(\text{HNO}_3) = 2 \text{ mol/L}$

#### General

Chlorides (e.g. NaCl) can be titrated with AgNO<sub>3</sub> in washing agents without any problem:



To prevent other sample ingredients from interfering, the titration is carried out under acidic conditions. The Ag Titrode is provided with an Ag<sub>2</sub>S coating to obtain a more stable signal.

#### Analysis

For reasons of accuracy (sample weight) a sample solution is first prepared. Approx. 8 g are weighed out into a 1 L volumetric flask with an accuracy of 0.1 mg and dissolved (as far as possible) in approx. 500 mL dist. H<sub>2</sub>O under gentle heating (40 °C). After cooling down it is carefully made up to the mark with dist. H<sub>2</sub>O and mixed.

50.0 mL of this sample solution are pipetted into the titration beaker and treated with 2 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$  and approx. 10 mL dist. H<sub>2</sub>O. The solution is titrated with  $c(\text{AgNO}_3) = 0.01 \text{ mol/L}$ .

#### Calculation

$$\% \text{ NaCl} = \frac{\text{'DET.U.EP\{1\}.VOL'} * \text{'DET.U.TITER'} * \text{'DET.U.CONC'} * 58.443 * 100}{\text{'MV.Sample size'} * 1000}$$

where:

'DET.U.EP{1}.VOL	mL AgNO <sub>3</sub> consumed up to the endpoint
'DET.U.TITER'	titer of AgNO <sub>3</sub>
'DET.U.CONC'	concentration of AgNO <sub>3</sub> [mol/L]
58.443	molecular weight of NaCl [g/mol]
100	conversion factor for %
'MV.Sample size'	0.4 [g] corresponds to 50 mL of the prepared solution
1000	correction factor for milliliters

#### Reference

- Metrohm Application Bulletin no. 130  
Chloride titrations with potentiometric indication

### 3.12 *tiamo* method: Chloride in washing agents and soaps

#### Application note

This is a method to determine the percentage of sodium chloride in washing agents and soaps. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the determination series. The main track handles the determination followed by the rinsing of the electrode 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 Chloride Analyzer (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in dist. water for a proper electrode treatment between the determination series. In case of an error, the error track is carried out, which guarantees that the electrode is moved to the storing beaker.

#### Sample preparation

For accuracy reasons a sample solution is made in a first step. Approximately 8 g are weighed out in a 1 L volumetric flask and dissolved in 500 mL dist. water at about 40°C. After cooling down the flask is filled to the mark with dist. water. For a determination 50 mL of the prepared sample (corresponds to approximately 0.4 g of the solid sample) are pipetted into a titration beaker.

#### Procedure

The beakers are placed on the rack and the determination series started. Approximately 10 mL dist. water and 2 mL of  $c(\text{HNO}_3) = 2 \text{ mol/L}$  are added automatically. The solution is then titrated with  $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 58.443 in the calculation formula is the molecular weight of sodium chloride in [g/mol]. The factor 100 is needed to receive percent as result.

#### Remarks

To run this method the settings of the 855 Robotic Titrosampler have to be adjusted. The lift positions have to be defined according to the rack and beakers used. As the robotic swing arm is equipped with a beaker sensor the head has to touch the beaker brim when moving to the work position. If this requirement can not be fulfilled no beaker will be recognized. Two special beakers have to be defined including their work positions. Special beaker 1 is used for rinsing the electrode in case of an error. Special beaker 2 is used for a proper electrode treatment between the determination series and in case of an error.

**Result report (example)**



Robotic Chloride Analyzer  
Chloride (NaCl) in washing agents and soaps

Programm version tiamo 1.1

2005-09-27 11:09:16 UTC+2

**Results report**

**Determination**

Method . . . . . Chloride (NaCl) in washing agents and soaps  
 Method saving date . . . . . 2005-09-27 08:37:44 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -4194d45f:1069643070f:-7f10  
 Determination start . . . . . 2005-09-27 09:16:09 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 11  
 User (full name) . . . . . Metrohm

**Sample data**

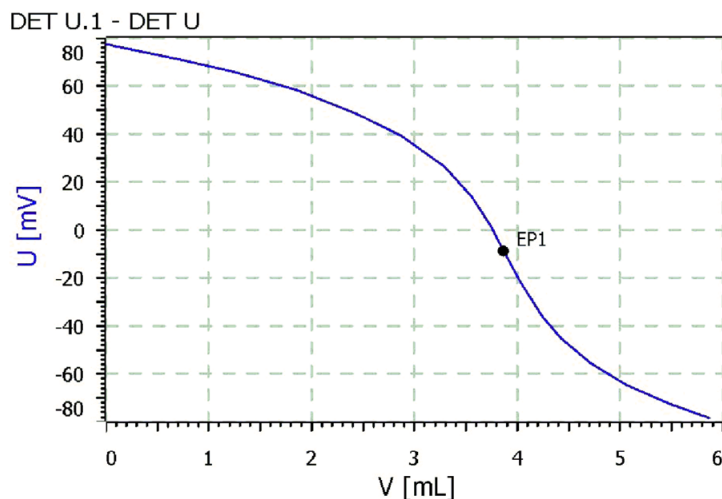
Sample Identification 1 . . . . . Washing agent  
 Sample size . . . . . 0.4061 g

**End points**

DET U     DET U.1  
 EP1 . . . . . -9.0 mV . . . . . 3.8687 mL

**Results**

content of sodiumchloride . . . . . 0.5579 %



**Statistical results (example)***Chloride (NaCl) in washing agents and soaps*

Sample size [mL]	Content [%]
50.0000	0.5624
50.0000	0.5627
50.0000	0.5598
50.0000	0.5613
50.0000	0.5600
50.0000	0.5607
50.0000	0.5644
50.0000	0.5637
50.0000	0.5626
50.0000	0.5579
Mean value	0.5616
abs. std. dev.	0.0020
rel. std. dev. %	0.35

## 4 Storage

Ag-Titrode: Store in dist. water only

## 5 Method reports

### 5.1 Titer determination of $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:51 UTC+1

#### Method parameters

Method . . . . . Titer determination of  $c(\text{AgNO}_3) = 0.1 \text{ M}$   
 Method saving date . . . . . 2005-10-31 14:41:44 UTC+1  
 Method version . . . . . 1  
 Method group . . . . . Robotic Chloride 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 - 36

2005-10-31 14:41:51 UTC+1

Message by e-mail . . . . . off  
 Mail to . . . . .  
 Subject . . . . . Message from tiamo - Method 'New method 7' - 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 7' - 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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:51 UTC+1

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

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

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

**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

**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

**ADD**

**add HNO3**



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:41:51 UTC+1

General/Hardware

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . HNO3  
 Tandem dosing . . . . . off  
 Stirrer  
 Stirrer . . . . . off  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on  
 Dosing parameters  
 Volume . . . . . 1 mL  
 Dosing rate . . . . . maximum mL/min  
 Filling rate . . . . . maximum mL/min  
 Fill automatically at end . . . . . on

DET U

DET U

General/Hardware

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . AgNO3  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Ag Titrode  
 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 mL  
 Dosing rate . . . . . maximum mL/min  
 Start measured value  
 Start measured value . . . . . off mV  
 Dosing rate . . . . . 5 mL/min  
 Start slope  
 Start slope . . . . . off mV/mL  
 Dosing rate . . . . . 5 mL/min  
 Pause



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:51 UTC+1

Pause ..... 120 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 ..... 6  
 Min. increment ..... 10.0 µL  
 Max. increment ..... off µL  
 Dosing rate ..... maximum mL/min  
 Temperature  
 Temperature ..... 25.0 °C

**Stop conditions**

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

**Potentiometric evaluation**

Evaluation without window ..... on  
 EP criterion ..... 5  
 EP recognition ..... greatest  
 Evaluation with measured value window (U) ..... 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

**PUMP**

**aspirate sample solution**

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



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:41:51 UTC+1

Duration . . . . . on  
 Time . . . . . 10.0 s

**PUMP**

**rinse**

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**

**empty beaker**

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

**SERIES START**

**Series start track**

**RACK**

**initialize rack**

Device  
 Device name . . . . . 855\_1  
 Rack test . . . . . off

**EXIT**

**Exit track**

**CALC**

**calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer AgNO3	= 'MV.Sample size' * 1000 / ( 'DET U.EP{1}.VOL' * 'DET U.CONC' * 58.442 )		4	RS01	on
Mean value titer	= 'RS.Titer AgNO3.MNV'		4	RS02	off

Result name . . . . . **Titer AgNO3**  
 Formula . . . . . = 'MV.Sample size' \* 1000 / ( 'DET U.EP {1}.VOL' \* 'DET U.CONC' \* 58.442 )  
 Unit . . . . .



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

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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 AgNO3.MNV'  
 Unit . . . . .  
 Decimal places . . . . . 4  
 Assignment . . . . . RS02  
 Statistics . . . . . off  
 Description . . . . .  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . on  
     Solution name . . . . . AgNO3

**REPORT**

**report**  
 Report template  
     Report template . . . . . Titer determination AgNO3  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Titer determination AgNO3.pdf  
     Send e-mail . . . . . off

**DATABASE**

**database**  
 Database  
 Robotic Chloride Analyzer

**SERIES  
END**

**Series end track**  
 Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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2005-10-31 14:41:51 UTC+1

Number ..... 1  
 Beaker test  
     Display message ..... on  
     Stop determination ..... off  
     Stop determination and series ..... off  
 Parameters  
     Shift rate ..... 20 %/s  
     Shift direction ..... auto  
     Swing rate ..... 55 %/s

**PUMP add dist water**

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**

**MOVE to storage beaker**

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

**PUMP fill beaker with water**

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



License ID 124049905  
Client name TITRATION14  
User Metrohm

Program version tiamo 1.1 - 36  
2005-10-31 14:41:51 UTC+1

Switch off ..... off  
Duration ..... on  
Time ..... 20 s

## 5.2 Chloride in tap water



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:13 UTC+1

### Method parameters

Method . . . . . Chloride in tap water  
 Method saving date . . . . . 2005-10-31 14:39:55 UTC+1  
 Method version . . . . . 1  
 Method group . . . . . Robotic Chloride 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 - 36

2005-10-31 14:42:13 UTC+1

Subject . . . . . Message from tiamo - Method 'New method 3' - 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 3' - 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 - 36

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Check at start . . . . . on  
 Comment . . . . . Sample identification 1

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

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

**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

**ADD**

**add HNO3**

General/Hardware  
 Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . HNO3  
 Tandem dosing . . . . . off  
 Stirrer  
 Stirrer . . . . . off  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on  
 Dosing parameters  
 Volume . . . . . 5 mL  
 Dosing rate . . . . . maximum mL/min



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:42:13 UTC+1

Filling rate . . . . . maximum mL/min  
 Fill automatically at end . . . . . on

DET U

DET U

**General/Hardware**

Device

Device name . . . . . 855\_1

Dosing device

Dosing device . . . . . 1

Solution . . . . . AgNO3

Sensor

Measuring input . . . . . 1

Sensor . . . . . Ag Titrode

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 mL

Dosing rate . . . . . maximum mL/min

Start measured value

Start measured value . . . . . off mV

Dosing rate . . . . . 5 mL/min

Start slope

Start slope . . . . . off mV/mL

Dosing rate . . . . . 5 mL/min

Pause

Pause . . . . . 15 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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:42:13 UTC+1

Temperature . . . . . 25.0 °C

**Stop conditions**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 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 . . . . . 10  
 EP recognition . . . . . greatest  
 Evaluation with measured value window (U) . . . . . 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

**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**

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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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2005-10-31 14:42:13 UTC+1

**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

**SERIES START Series start track**

**RACK initialize rack**  
 Device  
   Device name . . . . . 855\_1  
 Rack test . . . . . off

**EXIT Exit track**

**CALC calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
content of chloride	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 35.453 * 1000 / 'MV. Sample size'	mg/L	4	RS01	off

Result name . . . . . **content of chloride**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 35.453 \* 1000 / 'MV. Sample size'  
 Unit . . . . . mg/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 . . . . . Chloride in tap water  
 Report output



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:13 UTC+1

Printer . . . . . off  
 PDF file . . . . . on  
 PDF file . . . . . C:\Programme\Metrohm\tiamo\Chloride in tap water.pdf  
 Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic Chloride Analyzer

**SERIES Series end track  
 END**

**MOVE to storing beaker**

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

**PUMP add dist water**

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**

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker



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Client name TITRATION14  
User Metrohm

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Number ..... 1

Beaker test

  Display message ..... off

  Stop determination ..... on

  Stop determination and series ..... off

Parameters

  Shift rate ..... 20 %/s

  Shift direction ..... auto

  Swing rate ..... 55 %/s

**PUMP**   **fill beaker with water**

Device

  Device name ..... 855\_1

Pumps

  Tower ..... 1

  Pump(s) ..... 1

Action

  Switch on ..... off

  Switch off ..... off

  Duration ..... on

  Time ..... 20 s

### 5.3 Determination of chloride in wine



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:41:59 UTC+1

#### Method parameters

Method . . . . . Determination of choride in wine  
 Method saving date . . . . . 2005-10-31 14:41:11 UTC+1  
 Method version . . . . . 1  
 Method group . . . . . Robotic Chloride 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 - 36

2005-10-31 14:41:59 UTC+1

Subject . . . . . Message from tiamo - Method 'New method 6' - 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 6' - 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

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Check at start . . . . . on  
 Comment . . . . . Sample identification 1

Name . . . . . **ID2**  
 Type . . . . . Text  
 Assignment . . . . . on . . . . . ID2  
 Value . . . . . off . . . . .

Check at start . . . . . on  
 Comment . . . . . Sample identification 2

Name . . . . . **ID3**  
 Type . . . . . Text  
 Assignment . . . . . on . . . . . ID3  
 Value . . . . . off . . . . .

Check at start . . . . . on  
 Comment . . . . . Sample identification 3

**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

**PUMP add water**

Device  
 Device name . . . . . 855\_1

Pumps  
 Tower . . . . . 1  
 Pump(s) . . . . . 1

Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 6 s

**ADD add HNO3**

General/Hardware  
 Device



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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Device name . . . . . 855\_1  
 Dosing device  
   Dosing device . . . . . 2  
   Solution . . . . . HNO3  
   Tandem dosing . . . . . off  
 Stirrer  
   Stirrer . . . . . off  
   Stirring rate . . . . . 8  
   Switch off automatically . . . . . on  
 Dosing parameters  
   Volume . . . . . 1 mL  
   Dosing rate . . . . . maximum mL/min  
   Filling rate . . . . . maximum mL/min  
   Fill automatically at end . . . . . on

**DET U**

**DET U**

**General/Hardware**

Device  
   Device name . . . . . 855\_1  
 Dosing device  
   Dosing device . . . . . 1  
   Solution . . . . . AgNO3 0.01  
 Sensor  
   Measuring input . . . . . 1  
   Sensor . . . . . Ag Titrode  
   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 mL  
   Dosing rate . . . . . maximum mL/min  
 Start measured value  
   Start measured value . . . . . off mV  
   Dosing rate . . . . . 5 mL/min  
 Start slope  
   Start slope . . . . . off mV/mL  
   Dosing rate . . . . . 5 mL/min  
 Pause  
   Pause . . . . . 10 s

**Titration parameters**



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

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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**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 Stop EP . . . . . 1  
 Volume after EP . . . . . 2.5 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 (U) . . . . . 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

**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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

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**PUMP fill beaker**  
 Device  
     Device name . . . . . 855\_1  
 Pumps  
     Tower . . . . . 1  
     Pump(s) . . . . . 1  
 Action  
     Switch on . . . . . off  
     Switch off . . . . . off  
     Duration . . . . . on  
     Time . . . . . 20 s

**STIR stir 10 sec**  
 Device  
     Device name . . . . . 855\_1  
 Stirrer  
     Stirrer . . . . . 1  
     Stirrer type . . . . . unknown  
     Stirring rate . . . . . 13  
 Action  
     Switch on . . . . . off  
     Switch off . . . . . off  
     Duration . . . . . on  
     Time . . . . . 10.0 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 . . . . . 15 s

**SERIES START Series start track**

**RACK initialize rack**  
 Device  
     Device name . . . . . 855\_1  
 Rack test . . . . . off

**EXIT Exit track**

**CALC calculation**



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:59 UTC+1

Result name	Formula	Unit	Decimal places	Assignment	Statistics
content of chloride	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 35.453 / 'MV.Sample size'	g/L	4	RS01	off
content of sodium chlorid	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 58.442 / 'MV.Sample size'	g/L	4	RS02	off

Result name . . . . . **content of chloride**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 35.453 / 'MV.Sample size'  
 Unit . . . . . g/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 . . . . .

Result name . . . . . **content of sodium chlorid**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 58.442 / 'MV.Sample size'  
 Unit . . . . . g/L  
 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 . . . . . off  
     Solution name . . . . .

**REPORT**

**report**  
 Report template  
     Report template . . . . . Chloride in wine  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Chloride in wine.pdf  
     Send e-mail . . . . . off

**DATABASE database**



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:59 UTC+1

Database

Robotic Chloride Analyzer

**SERIES  
END**

**Series end track**

**MOVE**

**to storing beaker**

Device

Device name . . . . . 855\_1

Target

Tower . . . . . 1

Move . . . . . Special beaker

Number . . . . . 2

Beaker test

Display message . . . . . on

Stop determination . . . . . off

Stop determination and series . . . . . off

Parameters

Shift rate . . . . . 20 %/s

Shift direction . . . . . auto

Swing rate . . . . . 55 %/s

**PUMP**

**add dist water**

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**

**MOVE**

**to storage beaker**

Device

Device name . . . . . 855\_1

Target

Tower . . . . . 1

Move . . . . . Special beaker

Number . . . . . 2

Beaker test

Display message . . . . . off

Stop determination . . . . . on

Stop determination and series . . . . . off



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

2005-10-31 14:41:59 UTC+1

Parameters

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

**PUMP aspirate**

Device

Device name . . . . . 855\_1

Pumps

Tower . . . . . 1  
 Pump(s) . . . . . 2

Action

Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 5 s

**PUMP rinsing**

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 empty rinsing beaker**

Device

Device name . . . . . 855\_1

Pumps

Tower . . . . . 1  
 Pump(s) . . . . . 2

Action

Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 3 s

**PUMP fill beaker with water**

Device

Device name . . . . . 855\_1

Pumps

Tower . . . . . 1  
 Pump(s) . . . . . 1

Action



License ID 124049905  
Client name TITRATION14  
User Metrohm

Program version tiamo 1.1 - 36  
2005-10-31 14:41:59 UTC+1

---

Switch on ..... off  
Switch off ..... off  
Duration ..... on  
Time ..... 20 s

## 5.4 Determination of ammonium chloride



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:12 UTC+1

### Method parameters

Method ..... Determination of ammonium chloride  
 Method saving date ..... 2005-10-31 14:40:27 UTC+1  
 Method version ..... 1  
 Method group ..... Robotic Chloride 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 - 36

2005-10-31 14:42:12 UTC+1

Mail to .....  
 Subject ..... Message from tiamo - Method 'New method 5' - 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 5' - 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 . . . . . on  
 Comment . . . . . Sample identification 1

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

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

**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

**PUMP add water**

Device  
 Device name . . . . . 855\_1  
 Pumps  
 Tower . . . . . 1  
 Pump(s) . . . . . 1  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 18 s

**ADD add HNO3**

General/Hardware



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36  
 2005-10-31 14:42:12 UTC+1

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . HNO3  
 Tandem dosing . . . . . off  
 Stirrer  
 Stirrer . . . . . off  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on  
 Dosing parameters  
 Volume . . . . . 2 mL  
 Dosing rate . . . . . maximum mL/min  
 Filling rate . . . . . maximum mL/min  
 Fill automatically at end . . . . . on

DET U

DET U

**General/Hardware**

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . AgNO3  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Ag Titrode  
 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 mL  
 Dosing rate . . . . . maximum mL/min  
 Start measured value  
 Start measured value . . . . . off mV  
 Dosing rate . . . . . 5 mL/min  
 Start slope  
 Start slope . . . . . off mV/mL  
 Dosing rate . . . . . 5 mL/min  
 Pause  
 Pause . . . . . 200 s



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**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 . . . . . 100.0 µL  
 Max. increment . . . . . off µL  
 Dosing rate . . . . . maximum mL/min  
 Temperature  
 Temperature . . . . . 25.0 °C

**Stop conditions**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 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 (U) . . . . . 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

**PUMP**

**aspirate sample solution**

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



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```

Time ..... 10.0 s

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

STIR    stir 10 sec
Device
  Device name ..... 855_1
Stirrer
  Stirrer ..... 1
  Stirrer type ..... unknown
  Stirring rate ..... 13
Action
  Switch on ..... off
  Switch off ..... off
  Duration ..... on
  Time ..... 10.0 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 ..... 15 s

SERIES  Series start track
START

RACK    initialize rack
Device
  Device name ..... 855_1
  Rack test ..... off

EXIT    Exit track

CALC    calculation
    
```





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Stop determination . . . . . off  
 Stop determination and series . . . . . off  
 Parameters  
   Shift rate . . . . . 20 %/s  
   Shift direction . . . . . auto  
   Swing rate . . . . . 55 %/s

**PUMP    add dist water**  
 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**

**MOVE    to rinse beaker**  
 Device  
   Device name . . . . . 855\_1  
 Target  
   Tower . . . . . 1  
   Move . . . . . Special beaker  
   Number . . . . . 1  
 Beaker test  
   Display message . . . . . off  
   Stop determination . . . . . on  
   Stop determination and series . . . . . off  
 Parameters  
   Shift rate . . . . . 20 %/s  
   Shift direction . . . . . auto  
   Swing rate . . . . . 55 %/s

**PUMP    aspirate**  
 Device  
   Device name . . . . . 855\_1  
 Pumps  
   Tower . . . . . 1  
   Pump(s) . . . . . 2  
 Action  
   Switch on . . . . . off  
   Switch off . . . . . off  
   Duration . . . . . on  
   Time . . . . . 5 s



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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**PUMP rinsing**

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 empty rinsing beaker**

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

**PUMP fill beaker with water**

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

### 5.5 Chloride content in acidic copper baths



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36  
 2005-10-31 14:42:45 UTC+1

#### Method parameters

Method ..... Chloride content in acidic copper baths  
 Method saving date ..... 2005-10-31 14:38:55 UTC+1  
 Method version ..... 1  
 Method group ..... Robotic Chloride 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 - 36

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Mail to .....  
Subject ..... Message from tiamo - Method 'New method 4' - 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 4' - 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



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 Client name TITRATION14  
 User Metrohm

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

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

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

**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

**PUMP**

**add water**

Device  
 Device name . . . . . 855\_1  
 Pumps  
 Tower . . . . . 1  
 Pump(s) . . . . . 1  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 8 s

**DET U**

**DET U  
 General/Hardware**



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:45 UTC+1

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . AgNO3 0.01  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Ag Titrode  
 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 mL  
 Dosing rate . . . . . maximum mL/min  
 Start measured value  
 Start measured value . . . . . off mV  
 Dosing rate . . . . . 5 mL/min  
 Start slope  
 Start slope . . . . . off mV/mL  
 Dosing rate . . . . . 5 mL/min  
 Pause  
 Pause . . . . . 10 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**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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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 . . . . . greatest  
 Evaluation with measured value window (U) . . . . . 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

**MOVE to rinsing beaker**

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

**PUMP asirate 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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 36

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**PUMP rinse**  
 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

**SERIES START Series start track**

**RACK initialize rack**  
 Device  
     Device name . . . . . 855\_1  
 Rack test . . . . . off

**EXIT Exit track**

**CALC calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
content of chloride	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 35.453 * 1000 / 'MV. Sample size'	mg/L	4	RS01	off
content of sodiumchlorid	= 'RS.content of chloride' * 1.64898	mg/L	4	RS02	off

Result name . . . . . **content of chloride**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 35.453 \* 1000 / 'MV. Sample size'  
 Unit . . . . . mg/L



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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 . . . . .

---

Result name . . . . . **content of sodiumchlorid**  
 Formula . . . . . = 'RS.content of chloride' \* 1.64898  
 Unit . . . . . mg/L  
 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 . . . . . off  
     Solution name . . . . .

**REPORT**

**report**  
 Report template  
     Report template . . . . . Chloride in acidic copper baths  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Acidic copper bath.pdf  
     Send e-mail . . . . . off

**DATABASE**

**database**  
 Database  
 Robotic Chloride Analyzer

**SERIES  
END**

**Series end track**  
 Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 2



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 Client name TITRATION14  
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Beaker test

Display message . . . . . on  
 Stop determination . . . . . off  
 Stop determination and series . . . . . off

Parameters

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

**PUMP fill beaker with water**

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**

**MOVE to rinse beaker**

Device

Device name . . . . . 855\_1

Target

Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 1

Beaker test

Display message . . . . . off  
 Stop determination . . . . . on  
 Stop determination and series . . . . . off

Parameters

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

**PUMP aspirate**

Device

Device name . . . . . 855\_1

Pumps

Tower . . . . . 1  
 Pump(s) . . . . . 2

Action

Switch on . . . . . off  
 Switch off . . . . . off



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Duration . . . . . on  
 Time . . . . . 3 s

**PUMP**

**rinsing**

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

**PUMP**

**empty rinsing beaker**

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

**MOVE**

**to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 Beaker test  
 Display message . . . . . off  
 Stop determination . . . . . on  
 Stop determination and series . . . . . off  
 Parameters  
 Shift rate . . . . . 20 %/s  
 Shift direction . . . . . auto  
 Swing rate . . . . . 55 %/s

**PUMP**

**add dist water**

Device  
 Device name . . . . . 855\_1  
 Pumps  
 Tower . . . . . 1



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Client name TITRATION14  
User Metrohm

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---

Pump(s) ..... 1  
Action  
Switch on ..... off  
Switch off ..... off  
Duration ..... on  
Time ..... 20 s

5.6 Chloride in broth



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:39 UTC+1

Method parameters

Method . . . . . Chloride in broth  
 Method saving date . . . . . 2005-10-31 14:39:31 UTC+1  
 Method version . . . . . 1  
 Method group . . . . . Robotic Chloride 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 - 36

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Subject . . . . . Message from tiamo - Method 'New method 2' - 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 2' - 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.



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 Client name TITRATION14  
 User Metrohm

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Check at start . . . . . on  
 Comment . . . . . Sample identification 1

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

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

**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

**PUMP**

**add water**  
 Device  
     Device name . . . . . 855\_1  
 Pumps  
     Tower . . . . . 1  
     Pump(s) . . . . . 1  
 Action  
     Switch on . . . . . off  
     Switch off . . . . . off  
     Duration . . . . . on  
     Time . . . . . 10 s

**ADD**

**add HNO3**  
 General/Hardware  
     Device



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 User Metrohm 2005-10-31 14:42:39 UTC+1

Device name . . . . . 855\_1  
 Dosing device  
   Dosing device . . . . . 2  
   Solution . . . . . HNO3  
   Tandem dosing . . . . . off  
 Stirrer  
   Stirrer . . . . . off  
   Stirring rate . . . . . 8  
   Switch off automatically . . . . . on  
 Dosing parameters  
   Volume . . . . . 2 mL  
   Dosing rate . . . . . maximum mL/min  
   Filling rate . . . . . maximum mL/min  
   Fill automatically at end . . . . . on

**DET U**

**DET U**

**General/Hardware**

Device  
   Device name . . . . . 855\_1  
 Dosing device  
   Dosing device . . . . . 1  
   Solution . . . . . AgNO3  
 Sensor  
   Measuring input . . . . . 1  
   Sensor . . . . . Ag Titrode  
   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 mL  
   Dosing rate . . . . . maximum mL/min  
 Start measured value  
   Start measured value . . . . . off mV  
   Dosing rate . . . . . 5 mL/min  
 Start slope  
   Start slope . . . . . off mV/mL  
   Dosing rate . . . . . 5 mL/min  
 Pause  
   Pause . . . . . 15 s

**Titration parameters**



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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**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 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 (U) . . . . . 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

**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 s



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**PUMP rinse**  
 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 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

**SERIES START Series start track**

**RACK initialize rack**  
 Device  
     Device name . . . . . 855\_1  
 Rack test . . . . . off

**EXIT Exit track**

**CALC calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
content of sodiumchloride	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 58.443 * 0.1 / 'MV.Sample size'	%	4	RS01	off

Result name . . . . . **content of sodiumchloride**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 58.443 \* 0.1 / 'MV.Sample size'  
 Unit . . . . . %  
 Decimal places . . . . . 4  
 Assignment . . . . . RS01



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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 . . . . . Chloride in broth  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Chloride in broth.pdf  
     Send e-mail . . . . . off

**DATABASE**

**database**  
 Database  
 Robotic Chloride Analyzer

**SERIES  
END**

**Series end track**

**MOVE**

**to storing beaker**  
 Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 2  
 Beaker test  
     Display message . . . . . on  
     Stop determination . . . . . off  
     Stop determination and series . . . . . off  
 Parameters  
     Shift rate . . . . . 20 °/s  
     Shift direction . . . . . auto  
     Swing rate . . . . . 55 °/s

**PUMP**

**add dist water**  
 Device  
     Device name . . . . . 855\_1  
 Pumps  
     Tower . . . . . 1  
     Pump(s) . . . . . 1



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

**ERROR Error track**

**MOVE to rinse beaker**

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

**PUMP aspirate**

Device  
 Device name ..... 855\_1  
 Pumps  
 Tower ..... 1  
 Pump(s) ..... 2  
 Action  
 Switch on ..... off  
 Switch off ..... off  
 Duration ..... on  
 Time ..... 5 s

**PUMP rinsing**

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 empty rinsing beaker**



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

PUMP fill beaker with water

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

## 5.7 Chloride in washing agents and soaps



License ID 124049905 Program version tiamo 1.1 - 36  
 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:41 UTC+1

### Method parameters

Method ..... Chloride (NaCl) in washing agents and soaps  
 Method saving date ..... 2005-10-31 14:37:46 UTC+1  
 Method version ..... 1  
 Method group ..... Robotic Chloride 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



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 Client name TITRATION14  
 User Metrohm

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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



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

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

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

**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

**PUMP add water**

Device  
 Device name . . . . . 855\_1  
 Pumps  
 Tower . . . . . 1  
 Pump(s) . . . . . 1  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 5 s

**ADD add HNO3**

General/Hardware



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 Client name TITRATION14  
 User Metrohm

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Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . HNO3  
 Tandem dosing . . . . . off  
 Stirrer  
 Stirrer . . . . . 1  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on  
 Dosing parameters  
 Volume . . . . . 2 mL  
 Dosing rate . . . . . maximum mL/min  
 Filling rate . . . . . maximum mL/min  
 Fill automatically at end . . . . . on

DET U

DET U

**General/Hardware**

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . AgNO3 0.01  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Ag Titrode  
 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 mL  
 Dosing rate . . . . . maximum mL/min  
 Start measured value  
 Start measured value . . . . . off mV  
 Dosing rate . . . . . 5 mL/min  
 Start slope  
 Start slope . . . . . off mV/mL  
 Dosing rate . . . . . 5 mL/min  
 Pause  
 Pause . . . . . 10 s



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 Client name TITRATION14  
 User Metrohm

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**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 . . . . . 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 . . . . . off mV  
 Stop EP . . . . . 1  
 Volume after EP . . . . . 2 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 (U) . . . . . 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

**PUMP**

**aspirate sample solution**

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



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```

Time ..... 15 s

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

STIR    stir 10 sec
Device
  Device name ..... 855_1
Stirrer
  Stirrer ..... 1
  Stirrer type ..... unknown
  Stirring rate ..... 13
Action
  Switch on ..... off
  Switch off ..... off
  Duration ..... on
  Time ..... 10.0 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 ..... 15 s

SERIES  Series start track
START

RACK    initialize rack
Device
  Device name ..... 855_1
Rack test ..... off

EXIT    Exit track

CALC    calculation
    
```



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 Client name TITRATION14  
 User Metrohm 2005-10-31 14:42:41 UTC+1

Result name	Formula	Unit	Decimal places	Assignment	Statistics
content of sodiumchloride	= 'DET U.EP{1}.VOL' * 'DET U.TITER' * 'DET U.CONC' * 58.443 * 100 / ( 'MV.Sample size' * 1000 )	%	4	RS01	off

Result name . . . . . **content of sodiumchloride**  
 Formula . . . . . = 'DET U.EP{1}.VOL' \* 'DET U.TITER' \* 'DET U.CONC' \* 58.443 \* 100 / ( 'MV.Sample size' \* 1000 )  
 Unit . . . . . %  
 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 . . . . . Chloride in washing agents and soaps  
 Report output  
 Printer . . . . . off  
 PDF file . . . . . on  
 PDF file . . . . . C:\Programme\Metrohm\tiamo\Chloride in washing agents and soaps.pdf  
 Send e-mail . . . . . off  
 Mail to . . . . .  
 Subject . . . . . Message from tiamo - Method 'Titer determination of c (AgNO3) = 0.1 mol\_L' - Command 'Report 9'  
 Configuration  
 User . . . . .  
 Password . . . . . \*\*\*\*\*  
 Mail from . . . . .  
 SMTP Server . . . . .  
 POP3 Server . . . . .

**DATABASE database**

Database  
 Robotic Chloride Analyzer

**SERIES Series end track**  
**END**



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**MOVE to storing beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 Beaker test  
 Display message . . . . . on  
 Stop determination . . . . . off  
 Stop determination and series . . . . . off  
 Parameters  
 Shift rate . . . . . 20 °/s  
 Shift direction . . . . . auto  
 Swing rate . . . . . 55 °/s

**PUMP add dist water**

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**

**MOVE to rinse beaker**

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

**PUMP aspirate**

Device



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 User Metrohm

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Device name ..... 855\_1  
 Pumps  
   Tower ..... 1  
   Pump(s) ..... 2  
 Action  
   Switch on ..... off  
   Switch off ..... off  
   Duration ..... on  
   Time ..... 5 s

**PUMP rinsing**

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 empty rinsing beaker**

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

**PUMP fill beaker with water**

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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