

# Robotic TAN/TBN Analyzer

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## Applications

8.855.2023

 **Metrohm**  
Ion analysis

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**Robotic TAN/TBN Analyzer**



**Symbols used in this document:**

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

***tiamo* methods**

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

The methods can be found in the following path:

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

(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 TAN/TBN Analyzer

1.855.0010	855 Robotic Titrosampler
1.772.0110	Pump Unit
1.800.0010	Dosino 2x
1.802.0010	Rod stirrer
6.0229.100	LL Solvotrode OK
6.1236.020	Sleeve with SGJ 14/12 mm 2x
6.1458.040	Titration head insert 'Macro'
6.1459.300	Sample beakers 120 mL PP
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
6.1826.160	Pump tubing Viton, 6.4 mm, 1 m
6.1828.000	PVDF connection nipple 2x
6.1909.040	Stirring propeller ETFE, 102 mm
6.2001.120	Bottle holder base
6.2041.850	Sample rack 59 x 120 mL PP
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
6.2312.010	Electrolyte LiCl 2 mol/L in EtOH
6.2320.000	Electrolyte TEABr 0.4 mol/L
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 tray
6.2740.020	Spray nozzle 3x
6.2751.100	Safety shield
6.3032.220	Dosing Unit 20 mL

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6.3032.250	Dosing Unit 50 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 ultra pure 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 General and additional remarks about non-aqueous titrations

Non-aqueous titrations are primarily carried out when:

- the substance is not soluble in water
- the samples are fats or oils, or
- components of mixtures of acids or bases have to be determined separately by titration.

In most cases only a few solvents are required. These are: acetone, ethanol, isopropanol, methanol, toluene and glacial acetic acid (concentrated acetic acid) or, occasionally, binary mixtures of them.

The titrants are also not prepared with water. Examples are: NaOH in ethanol, KOH in isopropanol, HCl in ethanol or HClO<sub>4</sub> in glacial acetic acid.

If acids or bases are dissolved in protic solvents (e.g. H<sub>2</sub>O), protolysis occurs. But salts produced during the titration can also be broken down protolytically. In such solvents the differentiation of strong acids with a similar strength is not possible by titration.

The situation is different if the titration is carried out in non-aqueous solvents. Examples are phenol and boric acid in water or ethanol or aromatic amines in water or DMF, which can easily be titrated in a non-aqueous medium.

A further reason why the titrimetric determination of acidic or basic components of a mixture is not possible in protic solvents is leveling. Instead of the strong acid or base itself lyonium or lyate ions of the solvent are produced. In the particular system these represent the strongest acids or bases that can possibly occur in the corresponding solvents. The differences in strength that were present in the original mixture of acids or bases are compensated or, as one says, leveled. A classical example of this is the aqueous solution of strong mineral acids.

With these acids water practically quantitatively forms the oxonium ion H<sub>3</sub>O<sup>+</sup>. This is why only their total can be determined in aqueous solution

Other frequently used terms:

Amphiprotic solvent

Also known as protic. Noticeable self-dissociation, e.g.:



Aprotic solvent

No self-dissociation.

*Dielectric constant (DC)*

Proportional factor between the electric flux density D and the electric field strength E in vacuum. It is strongly temperature-dependent. Solutions with a higher DC promote the dissociation of electrolytes. In nonpolar solvents with a small DC even strong electrolytes (e.g. KCl, HClO<sub>4</sub>) are only weakly dissociated. Examples of DCs:

Water	80.4 (20 °C)
Methanol	33.6 (20 °C)
Ethanol	25.1 (20 °C)
Acetone	21.2 (20 °C)
MIBK	18.5 (25 °C)
Pyridine	13.5 (20 °C)
Acetic acid	6.2 (20 °C)

Toluene 2.4 (20 °C)

*Dissociation*

Formation of ions, e.g.  $\text{CH}_3\text{OOK} \rightleftharpoons \text{CH}_3\text{COO}^- + \text{K}^+$

Solvents for non-aqueous titrations can be split up into different classes:

- a) Amphiprotic – acidic  
e.g. glacial acetic acid, trifluoroacetic acid, phenol, cresol. Solvents for bases.
- b) Amphiprotic – basic  
e.g. ethylenediamine, benzylamine, butylamine. Solvents for weak acids.
- c) Amphiprotic – neutral  
e.g. ethanol, isopropanol, ethylene glycol. Solvents for strong and weak acids and salts.
- d) Aprotic – acidic  
e.g. nitromethane, nitroethane. Solvents for strong and weak acids and salts (seldom used, explosion hazard)
- e) Aprotic – basic  
e.g. pyridine, dimethylformamide (DMF), dimethylsulfoxide (DMSO). Solvents for weak acids.
- f) Aprotic – neutral  
e.g. acetone, acetonitrile, nitrobenzene, diethyl ether, dioxan, hydrocarbons (hexane, heptane, toluene), chlorinated hydrocarbons (chloroform, methylene chloride). Carbohydrates, ether, dioxan and chlorinated hydrocarbons are only used as solubility promoters (for nonpolar substances such as fats and oils). Potentiometric indication cannot be carried out in these solvents (high electrical resistance). Used as solvents for amines or (if appropriate) for weak acids.

## 2 Titer determination

### 2.1 Titer determination for the base number

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.3032.250 Dosing Unit, 50 mL
- 6.0229.100 Solvotrode; 6.2312.010 electrolyte ( 2 mol/L LiCl in ethanol)
- 6.2104.020 electrode cable

#### General

Most standard solutions/titrants are commercially available as ready-to-use solutions and have a relatively stable titer. However, their titer is valid for 20 °C. Please note that non-aqueous solvents have a coefficient of expansion which is four times larger than that of water! This means that, for a temperature difference of 5 °C and a theoretical consumption of 10.00 mL, the actual consumption will be 50  $\mu$ L higher, i.e. a titer of 1.000 at 20 °C will be 0.995 at 25 °C. Titer determinations and sample analysis should therefore be carried out at the same temperature whenever possible. The following titrants are available from Merck:

- No. 109065 c(HClO<sub>4</sub>) = 0.1 mol/L in anhydrous acetic acid (glacial acetic acid)
- No. 100326 c(HCl) = 0.1 mol/L in isopropanol (IPA)

If the titer is to be determined then this is done by comparing it with a so-called primary titrimetric standard. The content of these hardly changes, they are available with a defined degree of purity, can be dried and are directly traceable to standard reference materials (e.g. from NIST = National Institute of Standards and Technology, USA). Such recommended primary titrimetric standards or secondary standards are:

- Fluka No. 60153 Potassium hydrogen phthalate, M = 204.23 g/mol
- Fluka No. 12349 Benzoic acid, M = 122.19 g/mol

#### Titer determination of c(HClO<sub>4</sub>) = 0.1 mol/L with KH phthalate

Weigh out approx. 200 mg KH phthalate into a beaker to 0.1 mg. Add 40 mL glacial acetic acid and stir the mixture at room temperature until everything has dissolved (5...10 min). After the addition of 40 mL toluene the electrode is immersed and the solution is titrated with HClO<sub>4</sub> until after the first endpoint (instrument parameters and an example of a curve are given in the appendix).

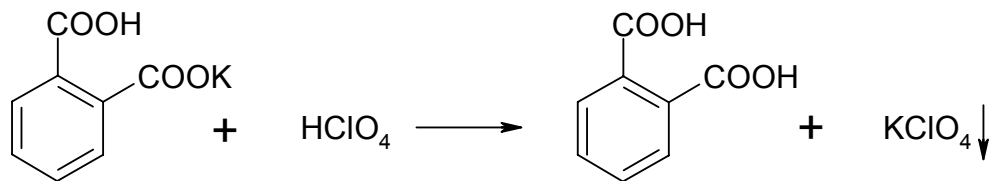
#### Calculation

$$\text{Titer} = \text{'MV.Sample size'} * 1000 / (\text{'DET U.EP\{1\}.VOL'} * \text{'DET U.CONC'} * 204.23)$$

where:

'MV.Sample size'	weight of KH phthalate [g]
1000	conversion factor for milliliter
'DET U.EP\{1\}.VOL'	titrant consumption up to the endpoint [mL]
'DET U.CONC'	concentration of the HClO <sub>4</sub> [mol/L]
204.23	molecular weight of KH phthalate [g/mol]

Reaction



Remarks

- The titer determination is normally carried out three times and the mean value is used and stored as titer.
- If you want to prepare the titrant yourself then proceed as follows:

For  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$  in glacial acetic acid:

Approx. 800 mL glacial acetic acid is placed in a 1 liter volumetric flask. It is treated with 8.6 mL (14.45 g)  $w(\text{HClO}_4) = 70\%$ , reagent grade ( $1.68 \text{ g/cm}^3$ ), made up to the mark with glacial acetic acid and mixed.

## 2.2 *tiamo* method: Titer determination of HClO<sub>4</sub> in acetic acid

### Application note

With this method the titer of HClO<sub>4</sub> in acetic acid 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 eight 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 of the primary standard followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, 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 'TAN/TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storing beaker where it is stored in the electrolyte solution used 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

The potassium hydrogen phthalate is dried over night in a drying oven at 105°C and allowed to cool down in a desiccator for at least 1 hour. For a determination approximately 200 mg potassium hydrogen phthalate are weighed out in a beaker with an accuracy of 0.1 mg.

### Procedure

The beakers are placed in the rack and the determination series started. A mixture of 30 mL acetic acid and 30 mL toluene are added automatically for dissolving. The solution is then titrated with  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 204.23 in the calculation formula is the molecular weight of potassium hydrogen phthalate 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 work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work positions. Special beaker 1 is filled with approximately 100 mL of dist. water and a special height has to be defined for this beaker as only the electrode membrane should be dipped into the water. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as inner electrolyte for the Solvotrode.

**Result report**



Robotic TAN / TBN Analyzer  
Titer determination

Programm version tiamo 1.1

2005-09-22 10:45:19 UTC+2

**Results report**

**Determination**

Method . . . . . Titer determination of HClO4 in acetic acid  
 Method saving date . . . . . 2005-09-21 08:41:36 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -b8acf1:106773bef9a:-7d23  
 Determination start . . . . . 2005-09-12 11:50:13 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 11  
 User (full name) . . . . . Metrohm

**Sample data**

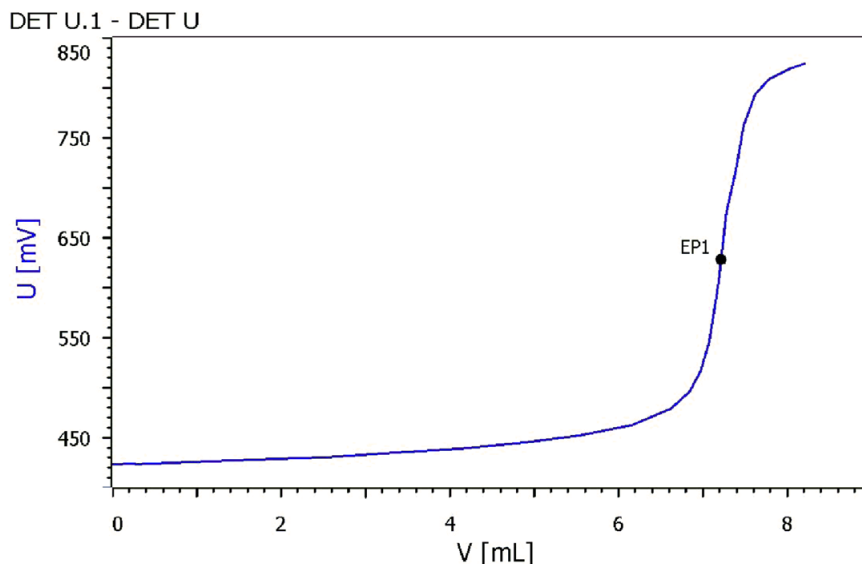
Sample Identification 1 . . . . . Titer HClO4  
 Sample size . . . . . 0.1448 g

**End points**

DET U     DET U.1  
 EP1 . . . . . 628.3 mV . . . . . 7.2179 mL

**Results**

**Titer HClO4** . . . . . **0.9823**  
**Mean value titer** . . . . . **0.9865**



**Statistical data (short)**



Method . . . . . Titer determination of HClO4 in acetic acid

Number of single determinations . . . . . 10

Result name	n	Mean value	s +/-	s rel
Titer HClO4	9	0.9865	0.00300	0.30 %

**Statistical results (example)**

*Titer determination HClO4 in acetic acid (0.1M)*

Sample size [g]	Titer
0.1919	0.9519
0.1802	0.9644
0.1730	0.9709
0.1856	0.9747
0.1513	0.9764
0.1385	0.9778
0.1820	0.9790
0.1278	0.9794
0.1572	0.9833
0.1448	0.9797
Mean value	0.9738
abs. std. dev.	0.0093
rel. std. dev. %	0.96

## 2.3 Titer determination for the acid number

### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.3032.250 Dosing Unit, 50 mL
- 6.0229.100 Solvotrode; 6.2320.000 electrolyte c(tetraethylammonium bromide) = 0.4 mol/L in ethylene glycol)
- 6.2104.020 electrode cable

### General

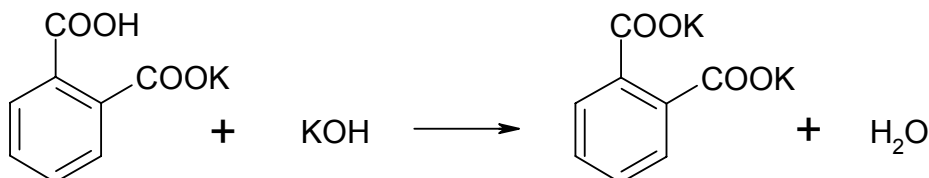
Most standard solutions/titrants are commercially available as ready-to-use solutions. Their titer is normally not stable (CO<sub>2</sub> uptake; for TBAOH additionally deamination – breakdown to form e.g. tertiary amines). The manufacturer also sets the titer for 20 °C. Please note that non-aqueous solvents have a coefficient of expansion that is four times larger than that of water! This means that for a temperature difference of 5 °C and a theoretical consumption of 10.00 mL, the actual consumption will be 50 µL higher, i.e. a titer of 1.000 at 20 °C will be 0.995 at 25 °C. Titer determinations and sample analysis should therefore always be carried out at the same temperature whenever possible. The following titrants are available from Merck:

- No. 105544 c(KOH) = 0.1 mol/L in isopropanol
- No. 109162 c(TBAOH) = 0.1 mol/L in isopropanol/methanol

The titer is determined by comparing it with a so-called primary titrimetric standard. The content of these hardly changes, they are available with a defined degree of purity, can be dried and are directly traceable to standard reference materials (e.g. National Institute of Standards and Technology, USA). Such primary titrimetric standards / secondary standards are:

- Merck No. 104874 Potassium hydrogen phthalate, M = 204.23 g/mol
- Merck No. 102401 Benzoic acid, M = 122.12 g/mol

## 2.4 Titer determination of c(KOH) = 0.1 mol/L with KH phthalate



Approx. 200 mg KH phthalate is weighed out into a beaker to 0.1 mg and dissolved in approx. 40 mL dist. H<sub>2</sub>O. After addition of 20 mL isopropanol the electrode and buret tip are immersed and the solution titrated with KOH until after the first endpoint.

### Calculation

$$\text{Titer} = \text{'MV.Sample size'} * 1000 / (\text{'DET U.EP\{1\}.VOL'} * \text{'DET U.CONC'} * 204.23)$$

where:

'MV.Sample size'	weight of KH phthalate [g]
1000	conversion factor for milliliter
'DET U.EP{1}.VOL'	titrant consumption up to the endpoint [mL]
'DET U.CONC'	concentration of the KOH [mol/L]
204.23	molecular weight of KH phthalate [g/mol]

## 2.5 *tiamo* method: Titer determination of KOH in 2-propanol

### Application note

With this method the titer of KOH in 2-propanol 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 eight 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 of the primary standard followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, which is done after every titration. In the exit track the results are calculated, a report is printed and the data is saved in the predefined database 'Robotic TAN TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storing beaker where it is stored in the electrolyte solution used 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

The potassium hydrogen phthalate is dried over night in a drying oven at 105°C and allowed to cool down in a desiccator for at least 1 hour. For a determination approximately 200 mg potassium hydrogen phthalate are weighed out in a beaker with an accuracy of 0.1 mg and dissolved at room temperature in approximately 40 mL dist. water.

### Procedure

The beakers are placed in the rack and the determination series started. 20 mL of 2-propanol are added automatically for dissolving. The solution is then titrated with  $c(\text{KOH}) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value 204.23 in the calculation formula is the molecular weight of potassium hydrogen phthalate 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 going to work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beaker have to be defined including their own work positions. Special beaker 1 is filled with distilled water and for this beaker a special height has to be defined as only the membrane of the electrode should be dipped for 1 minute. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as inner electrolyte for the Solvotrode.

**Result report**



Robotic TAN / TBN Analyzer  
Titer determination

Programm version tiamo 1.1

2005-09-13 14:43:56 UTC+2

**Results report**

**Determination**

Method . . . . . Titer determination of KOH in 2-propanol  
 Method saving date . . . . . 2005-09-06 08:53:18 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -68be1447:10629f7f76c:-7da0  
 Determination start . . . . . 2005-09-06 10:43:27 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 24  
 User (full name) . . . . . Metrohm

**Sample data**

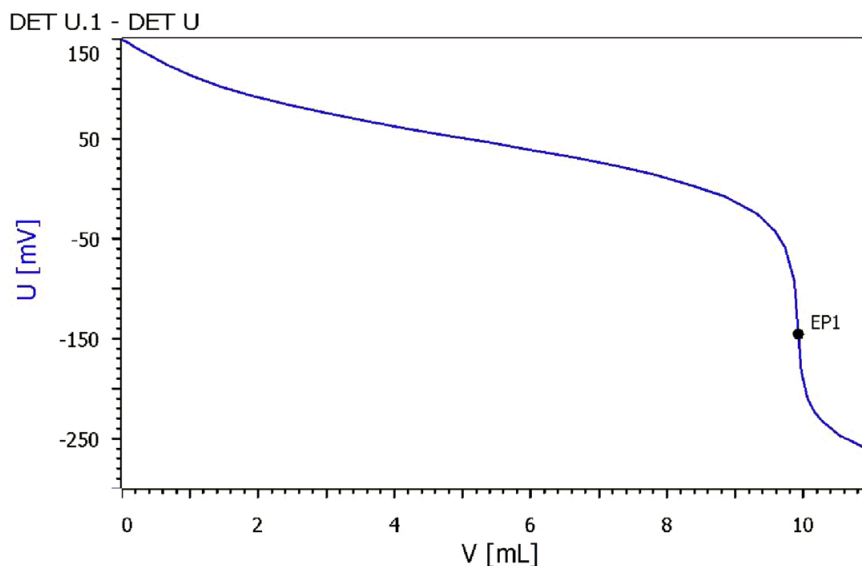
Sample Identification 1 . . . . . Titer determination KOH  
 Sample size . . . . . 0.1904 g

**End points**

DET U     DET U.1  
 EP1 . . . . . -145.6 mV . . . . . 9.9242 mL

**Results**

Titer KOH . . . . . **0.9394**  
 Mean value titer . . . . . **0.9397**



**Statistical data (short)**



Method . . . . . Titer determination of KOH in 2-propanol

Number of single determinations . . . . . 10

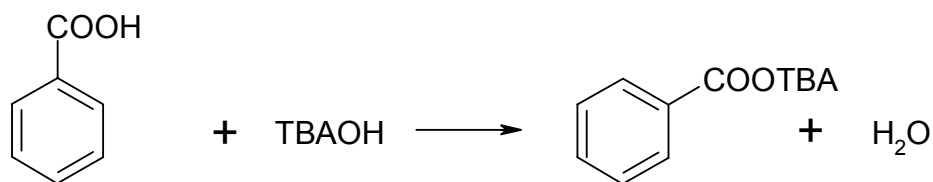
Result name	n	Mean value	s +/-	s rel
Titer KOH	10	0.9397	0.00673	0.72 %

**Statistical results (example)**

*Titer determination KOH in 2-propanol (0.1M)*

Sample size [g]	Titer
0.1656	0.9316
0.1652	0.9361
0.1928	0.9468
0.1851	0.9442
0.2085	0.9440
0.2030	0.9438
0.1852	0.9434
0.1843	0.9419
0.1592	0.9252
0.1904	0.9394
Mean value	0.9396
abs. std. dev.	0.0068
rel. std. dev. %	0.72

## 2.6 Titer determination of $c(\text{TBAOH}) = 0.1 \text{ mol/L}$ with benzoic acid



Approx. 100 mg benzoic acid is weighed out into a beaker to 0.1 mg and dissolved in 50 mL isopropanol. The electrode and buret tip are immersed and the solution titrated with TBAOH until after the first endpoint (instrument parameters as for KOH; an example of a curve is given in the appendix).

### Calculation

$$\text{Titer} = \text{'MV.Sample size'} * 1000 / (\text{'DET U.EP\{1\}.VOL'} * \text{'DET U.CONC'} * 122.12)$$

where:

'MV.Sample size'	weight of benzoic acid [g]
1000	conversion factor for milliliter
'DET U.EP\{1\}.VOL'	titrant consumption up to the endpoint [mL]
'DET U.CONC'	concentration of the $\text{HClO}_4$ [mol/L]
122.12	molecular weight of benzoic acid [g/mol]

### Remarks

- The titer determination is normally carried out three times and the mean value is used.
- The mean value of the titer is stored as titer.

## 2.7 **tiamo method: Titer determination of Tetra-n-butylammonium hydroxide (TBAOH) in 2-propanol/methanol**

With this method the titer of TBAOH in 2-propanol/methanol 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 eight different tracks. In the series start track the rack is initialized and the system prepared for the determination series. The main track handles the dissolving and the titration of the primary standard followed by the rinsing of the electrode with solvent, dist, water and ethyl alcohol 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 TAN TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in the electrolyte solution used 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:**

The benzoic acid is dried over night in a drying oven at 105°C and allowed to cool down in a desiccator for at least 1 hour. For a determination approximately 100 mg benzoic acid are weighed out in a beaker with an accuracy of 0.1 mg and dissolved at room temperature in approximately 40 mL of dist. water.

### **Procedure:**

The beakers are placed in the rack and the determination series started. 50 mL 2-propanol are added automatically for dissolving. The solution is then titrated with  $c(\text{TBAOH}) = 0.1 \text{ mol/L}$  and the titration stopped after the first endpoint.

The value '122.12' in the calculation formula is the molecular weight of benzoic acid 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 work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work positions. Special beaker 1 is filled with distilled water and a special height has to be defined for this beaker as only the membrane of the electrode should be dipped into the solution. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as inner electrolyte for the Solvotrode.

**Result report**



Robotic TAN / TBN Analyzer  
Titer determination

Programm version tiamo 1.1

2005-09-13 14:37:37 UTC+2

**Results report**

**Determination**

Method . . . . . Titer determination of TBAOH in 2-propanol\_MeOH  
 Method saving date . . . . . 2005-08-31 12:08:51 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 2e72edff:1060b146029:-7d92  
 Determination start . . . . . 2005-08-31 14:40:27 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 13  
 User (full name) . . . . . Metrohm

**Sample data**

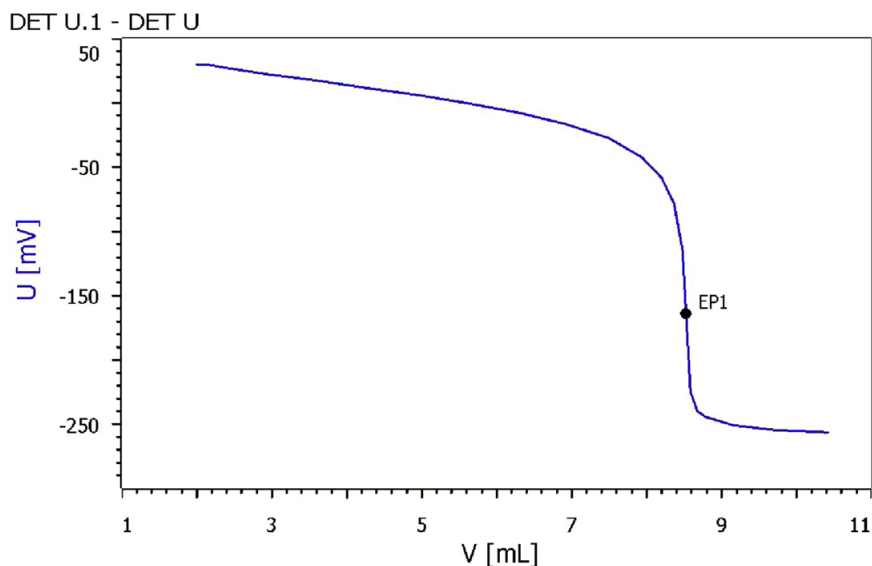
Sample Identification 1 . . . . . Titer TBAOH  
 Sample size . . . . . 0.0979 g

**End points**

DET U     DET U.1  
 EP1 . . . . . -164.0 mV . . . . . 8.5256 mL

**Results**

Titer TBAOH . . . . . **0.9403**  
 Mean value titer . . . . . **0.9431**



**Statistical data (short)**



Method . . . . . Titer determination of TBAOH in 2-propanol\_MeOH

Number of single determinations . . . . . 10

Result name	n	Mean value	s +/-	s rel
Titer TBAOH	10	0.9431	0.00670	0.71 %

**Statistical results (example)**

*Titer determination TBAOH (0.1M)*

Sample size [g]	Titer
0.1074	0.9362
0.1169	0.9434
0.1172	0.9461
0.1043	0.9384
0.111	0.9505
0.1383	0.9542
0.1133	0.9438
0.0852	0.9317
0.1076	0.9461
0.0979	0.9403
Mean value	0.9431
abs. std. dev.	0.0067
rel. std. dev. %	0.71

## 3 Typical applications for the Robotic TAN / TBN Analyzer

### 3.1 Determination of the base number

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.3032.250 Dosing Unit, 50 mL
- 6.0229.100 Solvotrode; 6.2312.010 electrolyte ( 2 mol/L LiCl in ethanol)
- 6.2104.020 electrode cable

#### General

Components in oil products that exhibit a basic reaction are determined as a cumulative total under the base number. These are chiefly primary organic and inorganic amino compounds, but the salts of weak acids and basic salts of polycarboxylic acids, as well as some heavy metal salts and detergents, are also determined. The determination is used to assess relative alterations during the working life of the product.

#### Definition

The base number is the amount of basic components, expressed as mg KOH, that is contained in 1 g sample.

#### Reagents

- Titrant:  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$  in glacial acetic acid
- Solvent mixture I: glacial acetic acid/toluene 1:1
- Solvent mixture II: glacial acetic acid/chlorobenzene 1:2\*\*
- Buffer solution pH = 4.0 (6.2307.100) and pH = 7.0 (6.2307.110)
- Ethanol, dist. H<sub>2</sub>O, hexane

\*\* For environmental reasons the use of this solvent mixture should be avoided.

#### Electrode storage, maintenance and checks

- When not in use the Solvotrode is stored in electrolyte solution (approx. 2 mol/L LiCl in ethanol).
- After each titration the electrode is rinsed with solvent mixture, then with ethanol and finally with dist. H<sub>2</sub>O. It is then immersed for 1 min in dist. H<sub>2</sub>O, rinsed with ethanol and the next sample is titrated. The ground joint of the diaphragm is loosened from time to time so that electrolyte can flow out. The joint is then lightly pressed together again. If precipitates are formed on the electrode during the titration (occurs with used oils and with some additives) then these are removed by placing the electrode in a stirred solution of hexane.
- In order to check the electrode functions (slope, response behavior, etc) please proceed as follows:

Rinse the electrode with dist. H<sub>2</sub>O and dab it dry with a soft tissue (e.g. Kleenex). Run the tiamo method 'Solvotrode function test', see 3.7.

With good electrodes a potential difference of  $>150 \text{ mV}$  is obtained at 20...25 °C. If the difference is  $<150 \text{ mV}$  then the electrode is cleaned with hexane and ethanol, immersed for 1 min in dist. H<sub>2</sub>O, the ground joint of the diaphragm is loosened slightly and then pressed together again. The measurements are then repeated. The test provides information about whether the electrode is correctly filled with electrolyte and connected correctly. Loose cable connections can also be recognized immediately.

### Analysis

Depending on the expected base number, 0.1...5 g well-mixed sample is weighed into the titration vessel. After the addition of 50...125 mL solvent mixture the solution is titrated with  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$  (instrument parameters and an example of a curve are given in the appendix).

### Calculation

Base number in mg KOH / g sample = ('DET U.EP{1}.VOL' - 'CV.Blank value TBN determination') \* 'DET U.CONC' \* 'DET U.TITER' \* 56.106 / 'MV.Sample size'

where:

'DET U.EP{1}.VOL'	titrant consumption up to the last EP**, in mL
'CV.Blank value TBN determination'	possible blank value of solvent (in mL $\text{HClO}_4$ ) <sup>++</sup>
'DET U.CONC'	0.1 (concentration $\text{HClO}_4$ in mol/L)
'DET U.TITER'	titer of titrant
56.106	molecular weight of KOH [g/mol]
'MV.Sample size'	sample size [g]

\*\* In the presence of basic components of differing strengths, several endpoints could occur. EP1 always represents the strongest base. The last endpoint is always used for calculating the total base content.

++ This value is obtained by titrating, under the same conditions, the same volume of solvent as will be used subsequently for the analysis of the sample. The result (in mL  $\text{HClO}_4$ ) is stored as common variable 'Blank value TBN determination'.

### Literature

- ASTM D 2896-88  
Standard Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
- ASTM D 4739-87  
Standard Test Method for Base Number Determination by Potentiometric Titration
- ISO 3771:1994  
Petroleum Products – Determination of Base Number – Perchloric Acid Potentiometric Titration Method
- Metrohm Application Bulletin No. 80

## 3.2 *tiamo* method: Blank value for the TBN determination

### Application note

With this method the possible blank value of the solvent used for the determination of the TBN can be determined. The method consists of eight different tracks. In the start series track the user is asked to enter the molecular weight of the used titrant, the rack is initialized and the system prepared for the measurements. The main track handles the adding of the solvent and the determination followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, 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 TAN TBN Analyzer' (can be modified). The obtained blank value is stored as common variable. In the series end track, the electrode is moved to a storage beaker where it is stored in the electrolyte solution used 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.

### Procedure

The beakers are placed on the rack and approximately 60 mL of solvent (glacial acetic acid/toluene 1:1) are added automatically. The solution is then titrated with  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$ .

### 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 going to work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work position. Special beaker 1 is filled with dist. water and a special height has to be defined for this beaker as only the membrane of the electrode should be dipped into the water. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as inner electrolyte for the Solvotrode. A common variable called "Blank value TBN determination has to be defined as well.

Before measuring, the membrane glass of the electrode is immersed in dist. water over night.

**Result report**



Robotic TAN / TBN Analyzer  
Blank value

Programm version 1.1

2005-09-19 11:16:09 UTC+2

**Results report**

**Determination**

Method . . . . . Blank value TBN determination  
 Method saving date . . . . . 2005-09-19 10:48:15 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 587941d7:1066d871327:-7ed1  
 Determination start . . . . . 2005-09-12 12:39:35 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 3  
 Run number . . . . . 15  
 User (full name) . . . . . Metrohm

**Sample data**

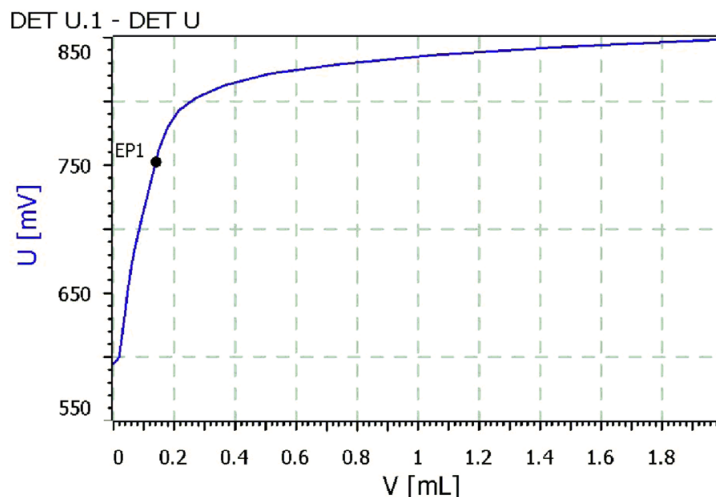
Sample Identification 1 . . . . . Blank TBN

**End points**

DET U     DET U.1  
 EP1 . . . . . 752.0 mV . . . . . 0.1410 mL

**Results**

Blank value TBN determination . . . . . 0.1410 mL  
 mean value blank . . . . . 0.1453 mL



**Statistical data (short)**

Method . . . . . Blank value TBN determination  
 Number of single determinations . . . . . 3



Robotic TAN / TBN Analyzer

Programm version 1.1

Blank value

2005-09-19 11:16:09 UTC+2

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Result name	n	Mean value	s +/-	s rel
Blank value TBN determination	3	0.1453 mL	0.00420 mL	2.89 %

---

### 3.3 *tiamo* method: Determination of the base number

#### Application note

With this method the base number of petroleum products can be determined. In this way changes of the oil during its use can be observed. The base number is the amount of basic components, expressed as mg KOH, that is contained in 1 g sample. The method consists of eight different tracks. In the start series track the user is asked to enter the molecular weight of the used titrant, the rack is initialized and the system prepared for the measurements. The main track handles the adding of the solvent and the determination followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, 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 TAN TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storage beaker where it is stored in the electrolyte solution used 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:

Depending on the expected base number about 0.1 to 5 g of the well mixed sample are weighed in a titration beaker.

#### Procedure:

The beakers are placed on the rack and approximately 60 mL of solvent (glacial acetic acid/toluene 1:1) are added automatically for dissolving. The solution is then titrated with  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$ .

#### 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 going to work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work position. Special beaker 1 is filled with dist. water and a special height has to be defined for this beaker as only the membrane of the electrode should be dipped into the water. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as inner electrolyte for the Solvotrode.

Before measuring, the membrane glass of the electrode is immersed in dist. water over night.

**Result report**



Robotic TAN / TBN Analyzer  
TBN determination

Programm version tiamo 1.1

2005-09-13 15:04:39 UTC+2

**Results report**

**Determination**

Method . . . . . TBN determination  
 Method saving date . . . . . 2005-09-12 17:58:31 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -79e2af18:1064ac1371d:-7e8b  
 Determination start . . . . . 2005-09-12 18:52:07 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 13  
 User (full name) . . . . . Metrohm

**Sample data**

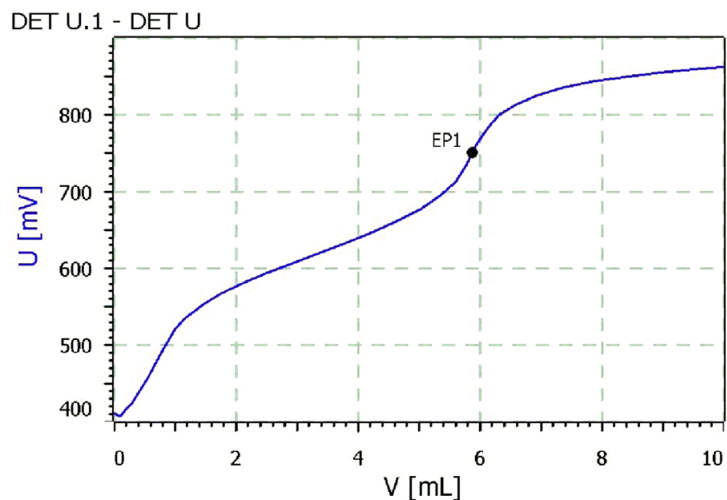
Sample Identification 1 . . . . . TBN  
 Sample size . . . . . 5.1741 g

**End points**

DET U     DET U.1  
 EP1 . . . . . 750.8 mV . . . . . 5.8795 mL

**Results**

TBN . . . . . **6.2190 mg/g**



**Statistical results (example)***TBN determination*

Sample size [g]	TBN [mg/g]
5.6062	6.3715
5.2573	6.3939
4.5197	6.4449
6.2377	6.2770
4.9078	6.3879
5.9523	6.3242
4.5381	6.3594
5.1741	6.2190
5.8565	6.2847
5.1467	6.2857
Mean value	6.3307
abs. std. dev.	0.0713
rel. std. dev. %	1.13

### 3.4 Determination of total acid number

#### Recommended accessories

- 6.3032.220 Dosing Unit, 20 mL
- 6.3032.250 Dosing Unit, 50 mL
- 6.0229.100 Solvotrode; 6.2320.000 electrolyte c(tetraethylammonium bromide) = 0.4 mol/L in ethylene glycol
- 6.2104.020 electrode cable

#### General

Components in petrochemical products that exhibit an acidic reaction are determined as a cumulative total under the acid number. These are compounds (acids, salts) with  $pK_a$  values  $< 9$ . The determination is used to assess relative alterations during the working life of the product. A global relationship between the acid number and signs of corrosion has not yet been demonstrated.

#### Definition

The acid number is the number of mg KOH that are required to neutralize 1 g sample.

#### Reagents

- Titrant:  $c(\text{KOH}) = 0.1 \text{ mol/L}$  in isopropanol/methanol (e.g. Merck No. 105544)
- Solvent: 500 mL toluene + 495 mL isopropanol + 5 mL  $\text{H}_2\text{O}$
- Buffer solutions  $\text{pH} = 4.0$  (6.2307.100) and  $\text{pH} = 7.0$  (6.2307.110)
- Ethanol, dist.  $\text{H}_2\text{O}$ , hexane

#### Storage, maintenance and checks of the electrode

- When not in use the Solvotrode is stored in electrolyte solution (TEA-Br in ethylene glycol).
- After each titration the electrode is rinsed with solvent mixture, then with ethanol and finally with dist.  $\text{H}_2\text{O}$ . It is then immersed for 1 min in dist.  $\text{H}_2\text{O}$ , rinsed with ethanol and the next sample is titrated. The ground joint of the diaphragm is loosened from time to time so that some electrolyte can flow out. The joint is then lightly pressed together again. If precipitates are formed on the electrode during the titration (occurs with used oils and with some additives) then these are removed by placing the electrode in a stirred solution of hexane.
- In order to check the electrode functions (slope, response behavior, etc) proceed as follows:

Rinse the electrode with dist.  $\text{H}_2\text{O}$  and dab it dry with a soft tissue (e.g. Kleenex). Run the tiemo method 'Solvotrode function test', see 3.7.

With good electrodes a potential difference of  $> 150 \text{ mV}$  is obtained at  $20...25 \text{ }^\circ\text{C}$ . If the difference is  $< 150 \text{ mV}$  then the electrode is cleaned with hexane and ethanol, immersed for 1 min in dist.  $\text{H}_2\text{O}$ , the ground joint of the diaphragm is loosened slightly and then pressed together again. The measurements are then repeated. The test provides information about whether the electrode is correctly filled with electrolyte and connected correctly. Loose cable connections can also be recognized immediately.

#### Analysis

Depending on the expected acid number (AN), 0.1...10 g (AN 100...250  $\Rightarrow$  0.1 g, AN 5...20  $\Rightarrow$  1 g, AN 0.05...1  $\Rightarrow$  10 g) well-mixed sample is weighed out into the titration vessel. After

the addition of 50...125 mL solvent mixture the solution is titrated with  $c(\text{KOH}) = 0.1 \text{ mol/L}$  (instrument parameters and an example of a curve are given in the appendix).

### Calculation

Acid number in mg KOH / g sample = ('MET U.EP{1}.VOL' – 'CV.Blank value TAN determination') \* 'MET U.CONC' \* 'MET U.TITER' \* 56.106 / 'MV.Sample size'

where:

'MET U.EP{1}.VOL'	titrant consumption up to the last EP**, in mL
'CV.Blank value TAN determination'	possible blank value for solvent (in mL KOH)**
'MET U.CONC'	0.1 (concentration KOH in mol/L)
'MET U.TITER'	titer of titrant
56.106	molecular weight of KOH [g/mol]
'MV.Sample size'	sample size [g]

\*\* If several strong acids are present then several endpoints may be obtained. EP1 corresponds to the strongest acid and could be used to calculate the «Strong Acid Number». The last endpoint is always used for the total acid content. If no endpoint is obtained and the titration curve increases diagonally then a so-called Fix-EP (EPA or EPB) can be defined experimentally and used for the calculation.

++ The blank value is obtained by titrating, under the same conditions, the same volume of solvent as will be used subsequently for the analysis of the sample. The result (in mL KOH) is stored as common variable 'Blank value TAN determination'.

### Literature

- ASTM D 664-95  
Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- Metrohm Application Bulletin No. 80

### 3.5 ***tiamo* method: Determination of the blank value for TAN determinations**

#### **Application note**

With this method the possible blank value of the solvent used for the determination of the TAN products can be determined. The method consists of eight different tracks. In the start series track the user is asked to enter the molecular weight of the used titrant, the rack is initialized and the system prepared for the determination. The main track handles the adding of the solvent and the determination followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, 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 TAN TBN Analyzer' (can be modified). The obtained mean blank value is stored as common variable. In the series end track, the electrode is moved to a storage beaker, where it is stored in the electrolyte solution used for a proper treatment between 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.

#### **Procedure:**

The beakers are placed on the rack and approximately 60 mL solvent (500 mL toluene + 495 mL 2-propanol + 5 mL dist. water) are added automatically. The solution is then titrated with  $c(\text{KOH}) = 0.1 \text{ mol/L}$ .

#### **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 going to work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work position. Special beaker 1 is filled with dist. water and a special position has to be defined for this beaker as only the membrane of the electrode should be dipped into the water. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as an inner electrolyte for the Solvotrode. A common variable called "Blank value TAN determination" has to be defined as well.

Before measuring, the membrane glass of the electrode is immersed in dist. water over night.

**Result report**



Robotic TAN / TBN Analyzer  
Blank value

Programm version 1.1

2005-10-28 11:13:52 UTC+2

**Results report**

**Determination**

Method . . . . . Blank value TAN determination  
 Method saving date . . . . . 2005-09-06 11:32:00 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . -7448934e:107363b6f68:-7f03  
 Determination start . . . . . 2005-09-07 13:10:12 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 4  
 User (full name) . . . . . Metrohm

**Sample data**

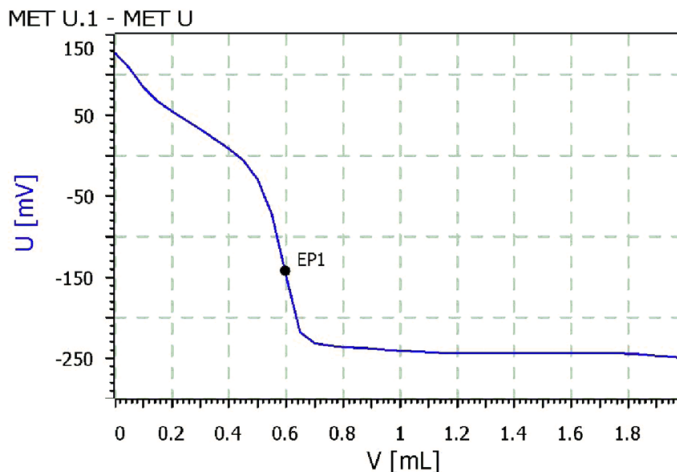
Sample Identification 1 . . . . . Blank TAN

**End points**

MET U MET U.1  
 EP1 . . . . . -141.9 mV . . . . . 0.5961 mL

**Results**

Blank value TAN . . . . . 0.596 mL  
 mean value blank . . . . . 0.595 mL



**Statistical data (short)**

Method . . . . . Blank value TAN determination  
 Number of single determinations . . . . . 3



Robotic TAN / TBN Analyzer

Programm version 1.1

Blank value

2005-10-28 11:13:52 UTC+2

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Result name	n	Mean value	s +/-	s rel
Blank value TAN	2	0.595 mL	0.0014 mL	0.24 %

---

### 3.6 *tiamo* method: Determination of the acid number

#### Application note

With this method the acid number of petroleum products can be determined. In this way changes of the oil during its use can be observed. The acid number is given in milligrams KOH that were used to neutralize 1 g of the sample. The method consists of eight different tracks. In the start series track the user is asked to enter the molecular weight of the used titrant, the rack is initialized and the system prepared for the determination. The main track handles the adding of the solvent and the determination followed by the rinsing of the electrode with solvent, dist. water and ethyl alcohol, 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 TAN TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storage beaker, where it is stored in the electrolyte solution used for a proper treatment between 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:

Depending on the expected acid number about 0.1 to 10 g of the well mixed sample are weighed in a titration beaker.

#### Procedure:

The beakers are placed on the rack and approximately 60 mL solvent (500 mL toluene + 495 mL 2-propanol + 5 mL dist. water) are added automatically for dissolving. The solution is then titrated with  $c(\text{KOH}) = 0.1 \text{ mol/L}$ .

#### 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 going to work position. If this requirement can not be fulfilled no beaker will be recognized. Three special beakers have to be defined including their work position. Special beaker 1 is filled with dist. water and a special position has to be defined for this beaker as only the membrane of the electrode should be dipped into the water. Special beaker 2 contains ethyl alcohol in which the complete electrode is immersed for 10 seconds. Special beaker 3 is filled with the electrolyte solution used as an inner electrolyte for the Solvotrode.

Before measuring, the membrane glass of the electrode is immersed in dist. water over night.

**Result report**



Robotic TAN / TBN Analyzer  
TAN determination

Programm version tiamo 1.1

2005-09-13 14:49:22 UTC+2

**Results report**

**Determination**

Method . . . . . TAN determination  
 Method saving date . . . . . 2005-09-08 13:22:50 UTC+2  
 Method version . . . . . 1  
 Method state . . . . . original  
 Determination ID . . . . . 46ab124e:10634555f36:-7f2d  
 Determination start . . . . . 2005-09-08 13:25:28 UTC+2  
 Determination state . . . . . original  
 Determination version . . . . . 1  
 Run number . . . . . 3  
 User (full name) . . . . . Metrohm

**Sample data**

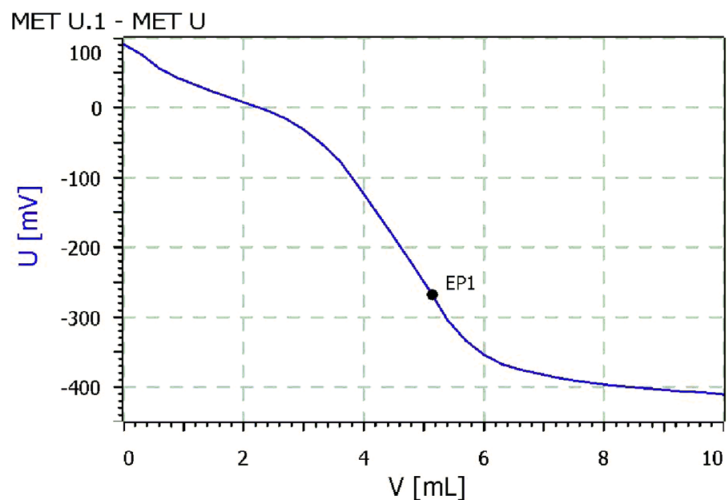
Sample Identification 1 . . . . . TAN  
 Sample size . . . . . 10.1403 g

**End points**

**MET U**     **MET U.1**  
 EP1 . . . . . -268.4 mV . . . . . 5.1507 mL

**Results**

**TAN** . . . . . **2.4394 mg/g**



**Statistical results (example)***TAN determination (rinsing with hexane)*

Sample size [g]	TAN [mg/g]
10.2767	2.4421
10.1403	2.4394
10.2437	2.4216
10.3890	2.4239
10.7227	2.3521
10.1912	2.4017
10.2230	2.4091
10.0095	2.3501
10.9244	2.3928
10.6484	2.3380
Mean value	2.3971
abs. std. dev.	0.0381
rel. std. dev. %	1.59

### **3.7 *tiamo* method: Solvotrode function test**

This electrode test provides information about the function of the Solvotrode. The method consists of five different tracks. In the start series track the rack is initialized and the system prepared for the measurements. The main track handles the measuring of the buffer solutions followed by the rinsing of the electrode with dist. water.

In the exit track the difference of the potentials is calculated, a report is printed and the data is saved in the predefined database 'Robotic TAN TBN Analyzer' (can be modified). In the series end track, the electrode is moved to a storage beaker, where it is stored in the electrolyte solution used for a proper treatment between determination series.

#### **Procedure**

Two beakers are placed on the rack. The first beaker is filled with buffer 7, the second one with buffer 4. Then the measurement is started.

#### **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 going to work position. If this requirement can not be fulfilled no beaker will be recognised. Two special beakers have to be defined including their work positions. Special beaker 1 is filled with dist. water. Special beaker 2 is filled with the electrolyte solution used as an inner electrolyte for the Solvotrode.

Before measuring, the membrane glass of the electrode is immersed in dist. water over night.

**Result report**



Robotic TAN / TBN Analyzer  
Solvotrode function test

Programm version tiamo 1.1

2005-10-03 10:18:57 UTC+2

**Results report**

**Determination**

Method . . . . . Solvotrode function test  
Method saving date . . . . . 2005-10-03 08:50:23 UTC+2  
Method version . . . . . 1  
Method state . . . . . original  
Determination ID . . . . . -43483cfc:106b5080fb1:-7e2a  
Determination start . . . . . 2005-10-03 08:51:22 UTC+2  
Determination state . . . . . original  
Determination version . . . . . 1  
Run number . . . . . 3  
User (full name) . . . . . Metrohm

**Sample data**

Sample Identification 1 . . . . . Solvotrode function test

**Results**

**potential buffer 7** . . . . . **24.3997 mV**  
**potential buffer 4** . . . . . **193.8709 mV**  
**difference** . . . . . **169.4712 mV**

## 4 Troubleshooting Solvotrode

### 4.1 Troubleshooting glass membrane

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

## 4.2 Cleaning and care of pH-electrodes with fixed-sleeve diaphragm

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

## 4.3 Storage

Solvotrode:      Store in electrolyte.  
                          Lift sleeve ring before first measurement.  
                          Condition glass membrane in dist. water before next measurement.

## 5 Method reports

### 5.1 Titer determination of HClO4 in acetic acid



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-13 14:16:38 UTC+2

#### Method parameters

Method . . . . . Titer determination of HClO4 in acetic acid  
 Method saving date . . . . . 2005-09-13 13:29:21 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 - 31  
 2005-09-13 14:16:38 UTC+2

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

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

Name . . . . . **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 - 31

2005-09-13 14:16:38 UTC+2

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 position**  
 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**

**dose solvent mixture**  
 General/Hardware  
 Device  
     Device name ..... 855\_1  
 Dosing device  
     Dosing device ..... 2  
     Solution ..... Toluene/acetic acid  
     Tandem dosing ..... off  
 Stirrer  
     Stirrer ..... off  
     Stirring rate ..... 8  
     Switch off automatically ..... on  
 Dosing parameters



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-13 14:16:38 UTC+2

Volume . . . . . 60 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 . . . . . HClO4 in acetic acid  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 600 s

**Titration parameters**

Titration rate  
 Titration rate . . . . . user  
 Measured value acceptance  
 Signal drift . . . . . 50.0 mV/min  
 Min. waiting time . . . . . 0 s  
 Max. waiting time . . . . . 26 s  
 Dosing of increments  
 Measuring point density . . . . . 4  
 Min. increment . . . . . 100.0 µL  
 Max. increment . . . . . off µL



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31

2005-09-13 14:16:38 UTC+2

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 . . . . . 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

**CALL call**

Call text	Track name	Condition	Condition
start rinsing	solvent rinsing	off	

**SERIES Series start track**

**START**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate solvent**

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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31  
 2005-09-13 14:16:38 UTC+2

Pump(s) . . . . . 2  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 7 s

**PUMP rinse and aspirate**

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

**PUMP aspirate rinsing solution**

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

**CALL call water rinsing**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK water rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to water beaker**

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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31

2005-09-13 14:16:38 UTC+2

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

**WAIT electrode in water**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 90  
     Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call alcohol rinsing**

Call text	Track name	Condition	Condition
start ethyl alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to alcohol 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

**WAIT electrode in alcohol**

Wait  
 Stop track and waiting for [Continue] . . . . . off



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31  
 2005-09-13 14:16:38 UTC+2

Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s  
 Message  
     Record message . . . . . on  
     Message by e-mail . . . . . off  
     Acoustic signal . . . . . off

**EXIT Exit track**

**CALC calculations**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer HClO4	= 'MV.Sample size' * 1000 / ( 'DET U.EP{1}.VOL' * 'DET U.CONC' * 204.23 )		4	RS01	on
Mean value titer	= 'RS.Titer HClO4.MNV'		4	RS02	off

Result name . . . . . **Titer HClO4**  
 Formula . . . . . = 'MV.Sample size' \* 1000 / ( 'DET U.EP {1}.VOL' \* 'DET U.CONC' \* 204.23 )  
 Unit . . . . .  
 Decimal places . . . . . 4  
 Assignment . . . . . RS01  
 Statistics . . . . . on  
 Description . . . . . RS.'Result name'[.VAL]Result value.  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . on  
     Solution name . . . . . HClO4 in acetic acid

Result name . . . . . **Mean value titer**  
 Formula . . . . . = 'RS.Titer HClO4.MNV'  
 Unit . . . . .  
 Decimal places . . . . . 4  
 Assignment . . . . . RS02  
 Statistics . . . . . off  
 Description . . . . .  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . off  
     Solution name . . . . .

**REPORT report**



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31

2005-09-13 14:16:38 UTC+2

Report template  
     Report template . . . . . Titer determination non aqueous  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\Titer HClO4.pdf  
     Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic TAN TBN Analyzer

**SERIES END Series end track**

**MOVE to storing beaker**

Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 3  
 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

**ERROR Error track**

**MOVE to storage beaker**

Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 3  
 Beaker test  
     Display message . . . . . off  
     Stop determination . . . . . on  
     Stop determination and series . . . . . off  
 Parameters  
     Shift rate . . . . . 20 °/s  
     Shift direction . . . . . auto



License ID 124049905  
Client name TITRATION14  
User Metrohm

Program version tiamo 1.1 - 31  
2005-09-13 14:16:38 UTC+2

---

Swing rate . . . . . 55 °/s

## 5.2 Titer determination of KOH in 2-propanol



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-13 13:55:46 UTC+2

### Method parameters

Method . . . . . Titer determination of KOH in 2-propanol  
 Method saving date . . . . . 2005-09-13 13:55:07 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 - 31  
 2005-09-13 13:55:46 UTC+2

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 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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31

2005-09-13 13:55:46 UTC+2

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 position**

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**

**dose 2-propanol**

General/Hardware  
 Device  
 Device name ..... 855\_1  
 Dosing device  
 Dosing device ..... 2  
 Solution ..... 2-propanol  
 Tandem dosing ..... off  
 Stirrer  
 Stirrer ..... off  
 Stirring rate ..... 8  
 Switch off automatically ..... on  
 Dosing parameters



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 User Metrohm 2005-09-13 13:55:46 UTC+2

Volume . . . . . 20 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 . . . . . KOH in 2-propanol  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 60 s

**Titration parameters**

Titration rate  
 Titration rate . . . . . user  
 Measured value acceptance  
 Signal drift . . . . . 50.0 mV/min  
 Min. waiting time . . . . . 0 s  
 Max. waiting time . . . . . 26 s  
 Dosing of increments  
 Measuring point density . . . . . 4  
 Min. increment . . . . . 100 µL  
 Max. increment . . . . . off µL



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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 . . . . . 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

**CALL**

**call**

Call text	Track name	Condition	Condition
start rinsing	solvent rinsing	off	

**SERIES START**

**Series start track**

**RACK**

**initialize rack**

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

**TRACK**

**solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP**

**asiprate sample solution**

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



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Pump(s) . . . . . 2  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 7 s

**PUMP rinse and aspirate**

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

**PUMP aspirate**

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

**CALL call 2**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK water rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to water beaker**

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



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

**WAIT electrode in water**

**Wait**  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 90  
     Unit . . . . . s  
**Message**  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call 3**

Call text	Track name	Condition	Condition
start ethyl alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to alcohol 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

**WAIT electrode in alcohol**

**Wait**  
 Stop track and waiting for [Continue] . . . . . off



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Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s  
 Message  
     Record message . . . . . on  
     Message by e-mail . . . . . off  
     Acoustic signal . . . . . off

**EXIT Exit track**

**CALC calculations**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer KOH	= 'MV.Sample size' * 1000 / ( 'DET U.EP {1}.VOL' * 'DET U.CONC' * 204.23 )		4	RS01	on
Mean value titer	= 'RS.Titer KOH.MNV'		4	RS02	off

Result name . . . . . **Titer KOH**  
 Formula . . . . . = 'MV.Sample size' \* 1000 / ( 'DET U.EP {1}.VOL' \* 'DET U.CONC' \* 204.23 )  
 Unit . . . . .  
 Decimal places . . . . . 4  
 Assignment . . . . . RS01  
 Statistics . . . . . on  
 Description . . . . . RS.'Result name'[.VAL]Result value.  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . off  
     Solution name . . . . .

Result name . . . . . **Mean value titer**  
 Formula . . . . . = 'RS.Titer KOH.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 . . . . . KOH in 2-propanol

**REPORT report**



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Report template  
 Report template . . . . . Titer determination non aqueous  
 Report output  
 Printer . . . . . off  
 PDF file . . . . . on  
 PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\Titer determination  
 KOH.pdf  
 Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic TAN TBN Analyzer

**SERIES Series end track  
 END**

**MOVE to storing beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 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

**ERROR Error track**

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 Beaker test  
 Display message . . . . . off  
 Stop determination . . . . . on  
 Stop determination and series . . . . . off  
 Parameters  
 Shift rate . . . . . 20 %/s  
 Shift direction . . . . . auto



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Swing rate . . . . . 55 °/s

### 5.3 Titer determination of TBAOH



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 Client name TITRATION14  
 User Metrohm 2005-09-13 13:58:13 UTC+2

#### Method parameters

Method . . . . . Titer determination of TBAOH in 2-propanol\_MeOH  
 Method saving date . . . . . 2005-09-13 13:47:54 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 - 31  
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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



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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 position**  
 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**

**dose 2-propanol**  
 General/Hardware  
 Device  
     Device name ..... 855\_1  
 Dosing device  
     Dosing device ..... 2  
     Solution ..... 2-propanol  
     Tandem dosing ..... off  
 Stirrer  
     Stirrer ..... off  
     Stirring rate ..... 8  
     Switch off automatically ..... on  
 Dosing parameters



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 Client name TITRATION14  
 User Metrohm 2005-09-13 13:58:13 UTC+2

Volume . . . . . 70 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 . . . . . TBAOH  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 2 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 . . . . . 90 s

**Titration parameters**

Titration rate  
 Titration rate . . . . . user  
 Measured value acceptance  
 Signal drift . . . . . 50.0 mV/min  
 Min. waiting time . . . . . 0 s  
 Max. waiting time . . . . . 26 s  
 Dosing of increments  
 Measuring point density . . . . . 4  
 Min. increment . . . . . 100 µL  
 Max. increment . . . . . off µL



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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 . . . . . 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

**CALL call**

Call text	Track name	Condition	Condition
start rinsing	solvent rinsing	off	

**SERIES START Series start track**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate sample solution**

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



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 User Metrohm

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Pump(s) . . . . . 2  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 7 s

**PUMP rinse and aspirate**

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

**PUMP aspirate**

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

**CALL call 2**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK water rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to water beaker**

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



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

**WAIT electrode in water**

**Wait**  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 90  
     Unit . . . . . s  
**Message**  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call 3**

Call text	Track name	Condition	Condition
start ethyl alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to alcohol 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

**WAIT electrode in alcohol**

**Wait**  
 Stop track and waiting for [Continue] . . . . . off



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Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s  
 Message  
     Record message . . . . . on  
     Message by e-mail . . . . . off  
     Acoustic signal . . . . . off

**EXIT Exit track**

**CALC calculations**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Titer TBAOH	= 'MV.Sample size' * 1000 / ( 'DET U.EP {1}.VOL' * 'DET U.CONC' * 122.12 )		4	RS01	on
Mean value titer	= 'RS.Titer TBAOH.MNV'		4	RS02	off

Result name . . . . . **Titer TBAOH**  
 Formula . . . . . = 'MV.Sample size' \* 1000 / ( 'DET U.EP {1}.VOL' \* 'DET U.CONC' \* 122.12 )  
 Unit . . . . .  
 Decimal places . . . . . 4  
 Assignment . . . . . RS01  
 Statistics . . . . . on  
 Description . . . . . RS.'Result name'[.VAL]Result value.  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . off  
     Solution name . . . . .

Result name . . . . . **Mean value titer**  
 Formula . . . . . = 'RS.Titer TBAOH.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 . . . . . TBAOH

**REPORT report**



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 Client name TITRATION14  
 User Metrohm

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Report template  
 Report template . . . . . Titer determination non aqueous  
 Report output  
 Printer . . . . . off  
 PDF file . . . . . on  
 PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\Titer determination of TBAOH.pdf  
 Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic TAN TBN Analyzer

**SERIES END Series end track**

**MOVE to storing beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 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

**ERROR Error track**

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 Beaker test  
 Display message . . . . . off  
 Stop determination . . . . . on  
 Stop determination and series . . . . . off  
 Parameters  
 Shift rate . . . . . 20 %/s  
 Shift direction . . . . . auto



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Swing rate . . . . . 55 °/s

5.4 Blank value TBN determination



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-14 16:31:12 UTC+2

Method parameters

Method . . . . . Blank value TBN determination  
 Method saving date . . . . . 2005-09-14 16:27:48 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 . . . . . 3  
 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 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



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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 position**

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 solvent**

General/Hardware  
 Device  
     Device name ..... 855\_1  
 Dosing device  
     Dosing device ..... 2  
     Solution ..... Toluene/acetic acid  
     Tandem dosing ..... off  
 Stirrer  
     Stirrer ..... off  
     Stirring rate ..... 8  
     Switch off automatically ..... on  
 Dosing parameters  
     Volume ..... 60 mL



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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 . . . . . HClO4 in acetic acid  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 10 mV/min  
 Min. waiting time . . . . . 0 s  
 Max. waiting time . . . . . 15 s  
 Dosing of increments  
 Measuring point density . . . . . 4  
 Min. increment . . . . . 10.0 µL  
 Max. increment . . . . . off µL  
 Dosing rate . . . . . maximum mL/min



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Temperature  
 Temperature . . . . . 25.0 °C

**Stop conditions**

Stop volume . . . . . 2 mL  
 Stop measured value . . . . . off mV  
 Stop EP . . . . . 9  
 Volume after EP . . . . . off 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

**CALL call solvent rinsing**

Call text	Track name	Condition	Condition
start solvent rinsing	solvent rinsing	off	

**SERIES START Series start track**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate sample solution**

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



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Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 10 s

**PUMP rinse and aspirate**

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

**PUMP aspirate rinsing solution**

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

**CALL call hexane rinsing**

Call text	Track name	Condition	Condition
start hexane rinsing	hexane rinsing	off	

**TRACK hexane rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to hexane beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 1  
 Beaker test  
 Display message . . . . . on



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

**STIR**

**stirrer on**

Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . on  
 Switch off . . . . . off  
 Duration . . . . . off

**WAIT**

**electrode in hexane**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 10  
 Unit . . . . . s  
 Message  
 Record message . . . . . off  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**STIR**

**stirrer off**

Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . on  
 Duration . . . . . off

**CALL**

**call water rinsing**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	



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**TRACK water rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to water beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 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

**WAIT electrode immersed**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 90  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call alcohol rinsing**

Call text	Track name	Condition	Condition
start alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to alcohol beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3



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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

WAIT

**electrode in alcohol**

Wait

Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s

Message

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

EXIT

**Exit track**

CALC

**calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Blank value TBN determination	= 'DET U.EP{1}.VOL'	mL	4	RS01	on
mean value blank	= 'RS.Blank value TBN determination.MNV'	mL	4	RS02	off

Result name . . . . . **Blank value TBN determination**  
 Formula . . . . . = 'DET U.EP{1}.VOL'  
 Unit . . . . . mL  
 Decimal places . . . . . 4  
 Assignment . . . . . RS01  
 Statistics . . . . . on  
 Description . . . . .  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . off  
     Name . . . . .  
 Save result as titer . . . . . off  
     Solution name . . . . .

Result name . . . . . **mean value blank**  
 Formula . . . . . = 'RS.Blank value TBN determination.MNV'



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 Client name TITRATION14  
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Unit . . . . . mL  
 Decimal places . . . . . 4  
 Assignment . . . . . RS02  
 Statistics . . . . . off  
 Description . . . . . RS.'Result name'[,VAL]Result value.  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . on  
     Name . . . . . CV.Blank value TBN determination  
 Save result as titer . . . . . off  
     Solution name . . . . .

**REPORT**

**report**  
 Report template  
     Report template . . . . . Blank value  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\Blank value TBN.pdf  
     Send e-mail . . . . . off

**DATABASE**

**database**  
 Database  
 Robotic TAN TBN Analyzer

**SERIES  
END**

**Series end track**

**MOVE**

**to storing beaker**  
 Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 4  
 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

**ERROR**

**Error track**

**CALL**

**call rinsing procedure**



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 Client name TITRATION14  
 User Metrohm

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Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1

Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 4

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

## 5.5 TBN determination



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-13 15:10:26 UTC+2

### Method parameters

Method . . . . . TBN determination  
 Method saving date . . . . . 2005-09-13 15:08:08 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 - 31

2005-09-13 15:10:26 UTC+2

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

Value . . . . . off. . . . .



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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 position**

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 solvent**

General/Hardware  
 Device  
 Device name . . . . . 855\_1

Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . Toluene/acetic acid  
 Tandem dosing . . . . . off

Stirrer  
 Stirrer . . . . . off  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on

Dosing parameters  
 Volume . . . . . 55 mL  
 Dosing rate . . . . . maximum mL/min



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 Client name TITRATION14  
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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 . . . . . HClO4 in acetic acid  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 10 mV/min  
 Min. waiting time . . . . . 0 s  
 Max. waiting time . . . . . 10 s  
 Dosing of increments  
 Measuring point density . . . . . 4  
 Min. increment . . . . . 100 µL  
 Max. increment . . . . . off µL  
 Dosing rate . . . . . maximum mL/min  
 Temperature



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 Client name TITRATION14  
 User Metrohm

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Temperature . . . . . 25.0 °C

**Stop conditions**

Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 Stop EP . . . . . 9  
 Volume after EP . . . . . off mL  
 Stop time . . . . . off s  
 Filling rate . . . . . maximum mL/min

**Potentiometric evaluation**

Evaluation without window . . . . . on  
 EP criterion . . . . . 5  
 EP recognition . . . . . last  
 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

**CALL call**

Call text	Track name	Condition	Condition
start solvent rinsing	solvent rinsing	off	

**SERIES START Series start track**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate sample solution**

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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 7 s

**PUMP rinse and aspirate**

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

**PUMP aspirate rinsing solution**

Device  
 Device name . . . . . 855\_1  
 Pumps  
 Tower . . . . . 1  
 Pump(s) . . . . . 2  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 4 s

**CALL call hexane rinsing**

Call text	Track name	Condition	Condition
start hexane rinsing	hexane rinsing	off	

**TRACK hexane rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to hexane beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 1  
 Beaker test  
 Display message . . . . . on  
 Stop determination . . . . . off



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

**STIR**

**stirrer on**  
 Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . on  
 Switch off . . . . . off  
 Duration . . . . . off

**WAIT**

**electrode in hexane**  
 Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 10  
 Unit . . . . . s  
 Message  
 Record message . . . . . off  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**STIR**

**stirrer off**  
 Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . on  
 Duration . . . . . off

**CALL**

**call water rinsing**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK**

**water rinsing**



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Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to water beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 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

**WAIT electrode immersed**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 120  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call alcohol rinsing**

Call text	Track name	Condition	Condition
start alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to alcohol beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 Beaker test



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 User Metrohm

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

**STIR**

**start stirrer**

Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . on  
 Switch off . . . . . off  
 Duration . . . . . off

**WAIT**

**electrode in alcohol**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 10  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**STIR**

**stop stirrer**

Device  
 Device name . . . . . 855\_1  
 Stirrer  
 Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8  
 Action  
 Switch on . . . . . off  
 Switch off . . . . . on  
 Duration . . . . . off

**EXIT**

**Exit track**

**CALC**

**calculation**



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 Client name TITRATION14  
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Result name	Formula	Unit	Decimal places	Assignment	Statistics
TBN	= ('DET U.EP{1}.VOL' - 'CV.Blank value TBN determination') * 'DET U.CONC' * 'DET U.TITER' * 56.106 / 'MV.Sample size'	mg/g	4	RS01	off

Result name ..... **TBN**  
 Formula ..... = ('DET U.EP{1}.VOL' - 'CV.Blank value TBN determination') \* 'DET U.CONC' \* 'DET U.TITER' \* 56.106 / 'MV.Sample size'  
 Unit ..... mg/g  
 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 ..... TBN determination  
 Report output  
     Printer ..... off  
     PDF file ..... on  
     PDF file ..... C:\Programme\Metrohm\tiamo\Data\TBN determination.pdf  
     Send e-mail ..... off

**DATABASE**

**database**  
 Database  
 Robotic TAN TBN Analyzer

**SERIES END**

**Series end track**  
**MOVE to storing beaker**  
 Device  
     Device name ..... 855\_1  
 Target  
     Tower ..... 1  
     Move ..... Special beaker  
     Number ..... 4  
 Beaker test  
     Display message ..... off



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

**ERROR Error track**

**CALL call rinsing procedure**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 4  
 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

### 5.6 Blank value TAN determination



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-14 16:31:01 UTC+2

#### Method parameters

Method . . . . . Blank value TAN determination  
 Method saving date . . . . . 2005-09-14 16:29:05 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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  
 Message by e-mail . . . . . off



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31

2005-09-14 16:31:01 UTC+2

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 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



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 position**

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 solvent**

General/Hardware  
 Device  
     Device name ..... 855\_1  
 Dosing device  
     Dosing device ..... 2  
     Solution ..... Toluene/2-propanol/water  
     Tandem dosing ..... off  
 Stirrer  
     Stirrer ..... off  
     Stirring rate ..... 8  
     Switch off automatically ..... on  
 Dosing parameters  
     Volume ..... 60 mL



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

**MET U**

**MET U**

**General/Hardware**

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . KOH in 2-propanol  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 30 s

**Titration parameters**

Titration rate  
 Titration rate . . . . . user  
 Measured value acceptance  
 Signal drift . . . . . 40.0 mV/min  
 Min. waiting time . . . . . 3 s  
 Max. waiting time . . . . . 6 s  
 Dosing of increments  
 Volume increment . . . . . 0.05 mL  
 Dosing rate . . . . . maximum mL/min  
 Temperature  
 Temperature . . . . . 25.0 °C



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 User Metrohm

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**Stop conditions**

Stop volume . . . . . 2 mL  
 Stop measured value . . . . . off mV  
 Stop EP . . . . . 9  
 Volume after EP . . . . . off mL  
 Stop time . . . . . off s  
 Filling rate . . . . . maximum mL/min

**Potentiometric evaluation**

Evaluation without window . . . . . on  
 EP criterion . . . . . 30 mV  
 EP recognition . . . . . last  
 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

**CALL call solvent rinsing**

Call text	Track name	Condition	Condition
start solvent rinsing	solvent rinsing	off	

**SERIES START Series start track**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate sample solution**

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



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 Client name TITRATION14  
 User Metrohm

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Switch off . . . . . off  
 Duration . . . . . on  
 Time . . . . . 10 s

**PUMP rinse and aspirate**

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

**PUMP aspirate rinsing solution**

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

**CALL call hexane rinsing**

Call text	Track name	Condition	Condition
start hexane rinsing	hexane rinsing	off	

**TRACK hexane rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to hexane 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



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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Parameters

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

**STIR**

**stirrer on**

Device

Device name . . . . . 855\_1

Stirrer

Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8

Action

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

**WAIT**

**electrode in hexane**

Wait

Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s

Message

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

**STIR**

**stirrer off**

Device

Device name . . . . . 855\_1

Stirrer

Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8

Action

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

**CALL**

**call water rinsing**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK**

**water rinsing**

Return immediately . . . . . off



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Delete all data . . . . . off

**MOVE to water beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 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

**WAIT electrode immersed**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 30  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call alcohol rinsing**

Call text	Track name	Condition	Condition
start alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to ethyl alcohol beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 Beaker test  
 Display message . . . . . off



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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

**WAIT electrode in alcohol**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**EXIT Exit track**

**CALC calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Blank value TAN	= 'MET U.EP{1}.VOL'	mL	3	RS01	on
mean value blank	= 'RS.Blank value TAN.MNV'	mL	3	RS02	off

Result name . . . . . **Blank value TAN**  
 Formula . . . . . = 'MET U.EP{1}.VOL'  
 Unit . . . . . mL  
 Decimal places . . . . . 3  
 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 blank**  
 Formula . . . . . = 'RS.Blank value TAN.MNV'  
 Unit . . . . . mL  
 Decimal places . . . . . 3  
 Assignment . . . . . RS02



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

Program version tiamo 1.1 - 31  
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Statistics . . . . . off  
 Description . . . . . RS.'Result name'[,VAL]Result value.  
 Result monitoring . . . . . off  
 Save result as common variable . . . . . on  
     Name . . . . . CV.Blank value TAN determination  
 Save result as titer . . . . . off  
     Solution name . . . . .

**REPORT**

**report**  
 Report template  
     Report template . . . . . Blank value  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\Blank value TAN.pdf  
     Send e-mail . . . . . off

**DATABASE**

**database**  
 Database  
 Robotic TAN TBN Analyzer

**SERIES  
END**

**Series end track**

**MOVE**

**to storing beaker**  
 Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 4  
 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

**ERROR**

**Error track**

**CALL**

**call rinsing procedure**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	



License ID 124049905  
Client name TITRATION14  
User Metrohm

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**MOVE to storage beaker**

Device

Device name . . . . . 855\_1

Target

Tower . . . . . 1

Move . . . . . Special beaker

Number . . . . . 4

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

## 5.7 TAN determination



License ID 124049905 Program version tiamo 1.1 - 31  
 Client name TITRATION14  
 User Metrohm 2005-09-13 15:10:31 UTC+2

### Method parameters

Method . . . . . TAN determination  
 Method saving date . . . . . 2005-09-13 15:06:29 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 . . . . .



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 User Metrohm

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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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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 position**

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 solvent**

General/Hardware  
 Device  
 Device name . . . . . 855\_1

Dosing device  
 Dosing device . . . . . 2  
 Solution . . . . . Toluene/2-propanol/water  
 Tandem dosing . . . . . off

Stirrer  
 Stirrer . . . . . off  
 Stirring rate . . . . . 8  
 Switch off automatically . . . . . on

Dosing parameters  
 Volume . . . . . 50 mL  
 Dosing rate . . . . . maximum mL/min



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

**MET U**

**MET U**

**General/Hardware**

Device  
 Device name . . . . . 855\_1  
 Dosing device  
 Dosing device . . . . . 1  
 Solution . . . . . KOH in 2-propanol  
 Sensor  
 Measuring input . . . . . 1  
 Sensor . . . . . Solvotrode  
 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 . . . . . 40.0 mV/min  
 Min. waiting time . . . . . 5 s  
 Max. waiting time . . . . . 26 s  
 Dosing of increments  
 Volume increment . . . . . 0.3 mL  
 Dosing rate . . . . . maximum mL/min  
 Temperature  
 Temperature . . . . . 25.0 °C

**Stop conditions**



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Stop volume . . . . . 20 mL  
 Stop measured value . . . . . off mV  
 Stop EP . . . . . 9  
 Volume after EP . . . . . off mL  
 Stop time . . . . . off s  
 Filling rate . . . . . maximum mL/min

**Potentiometric evaluation**

Evaluation without window . . . . . on  
 EP criterion . . . . . 30 mV  
 EP recognition . . . . . last  
 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

**CALL call solvent rinsing**

Call text	Track name	Condition	Condition
start solvent rinsing	solvent rinsing	off	

**SERIES START Series start track**

**RACK initialize rack**

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

**TRACK solvent rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**PUMP aspirate sample solution**

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 . . . . . 7 s

**PUMP rinse and aspirate**

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

**PUMP aspirate rinsing solution**

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

**CALL call hexane rinsing**

Call text	Track name	Condition	Condition
start hexane rinsing	hexane rinsing	off	

**TRACK hexane rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to hexane 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



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

**STIR**

**stirrer on**

Device

Device name . . . . . 855\_1

Stirrer

Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8

Action

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

**WAIT**

**electrode in hexane**

Wait

Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
     Time . . . . . 10  
     Unit . . . . . s

Message

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

**STIR**

**stirrer off**

Device

Device name . . . . . 855\_1

Stirrer

Stirrer . . . . . 1  
 Stirrer type . . . . . unknown  
 Stirring rate . . . . . 8

Action

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

**CALL**

**call water rinsing**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**TRACK**

**water rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off



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**MOVE to water beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 2  
 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

**WAIT electrode immersed**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 90  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**CALL call alcohol rinsing**

Call text	Track name	Condition	Condition
start alcohol rinsing	ethyl alcohol rinsing	off	

**TRACK ethyl alcohol rinsing**

Return immediately . . . . . off  
 Delete all data . . . . . off

**MOVE to ethyl alcohol beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 3  
 Beaker test  
 Display message . . . . . off  
 Stop determination . . . . . off



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

**WAIT electrode in alcohol**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 2  
 Unit . . . . . s  
 Message  
 Record message . . . . . on  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**EXIT Exit track**

**CALC calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
TAN	= ('MET U.EP{1}.VOL' - 'CV.Blank value TAN determination') * 'MET U.CONC' * 'MET U.TITER' * 56.106 / 'MV.Sample size'	mg/g	4	RS01	off

Result name . . . . . **TAN**  
 Formula . . . . . = ('MET U.EP{1}.VOL' - 'CV.Blank value TAN determination') \* 'MET U.CONC' \* 'MET U.TITER' \* 56.106 / 'MV.Sample size'  
 Unit . . . . . mg/g  
 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 . . . . . TAN determination  
 Report output  
 Printer . . . . . off



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PDF file . . . . . on  
 PDF file . . . . . C:\Programme\Metrohm\tiamo\Data\TAN determination.pdf  
 Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic TAN TBN Analyzer

**SERIES END Series end track**

**MOVE to storing beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 4  
 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

**ERROR Error track**

**CALL call rinsing procedure**

Call text	Track name	Condition	Condition
start water rinsing	water rinsing	off	

**MOVE to storage beaker**

Device  
 Device name . . . . . 855\_1  
 Target  
 Tower . . . . . 1  
 Move . . . . . Special beaker  
 Number . . . . . 4  
 Beaker test  
 Display message . . . . . off  
 Stop determination . . . . . on  
 Stop determination and series . . . . . off  
 Parameters  
 Shift rate . . . . . 20 %/s



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

5.8 Solvotrode function test



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 Client name TITRATION14  
 User Metrohm 2005-10-24 14:53:34 UTC+2

Method parameters

Method . . . . . Solvotrode function test  
 Method saving date . . . . . 2005-10-24 14:53:25 UTC+2  
 Method version . . . . . 1  
 Method group . . . . . Robotic TAN TBN 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 document

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 . . . . .



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 Client name TITRATION14  
 User Metrohm

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Subject . . . . . Message from tiamo - Method 'Solvotrode function test' -  
 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 'Solvotrode function test' -  
 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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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 buffer 7**

Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Sample position  
 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

**MEAS U**

**measure buffer 7**

**General/Hardware**

Device  
     Device name . . . . . 855\_1  
 Sensor  
     Measuring input . . . . . 1  
     Sensor . . . . . Solvotrode  
     Temperature measurement . . . . . automatic  
 Stirrer  
     Stirrer . . . . . 1  
     Stirring rate . . . . . 8  
     Switch off automatically . . . . . on

**Measuring parameters**

Measurement  
     Measurement with drift control . . . . . on



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Signal drift . . . . . 10.0 mV/min  
 Min. waiting time . . . . . 60 s  
 Max. waiting time . . . . . 70 s  
 Measuring interval . . . . . 2.0 s  
 Stop measured value . . . . . off mV  
 Measurement without drift control . . . . . off  
 Temperature  
 Temperature . . . . . 25.0 °C

**Evaluations**

Fix end point 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 . . . . . 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

**WAIT electrode immersed**

Wait  
 Stop track and waiting for [Continue] . . . . . off  
 Stop all tracks and waiting for [Continue] . . . . . off  
 Waiting time . . . . . on  
 Time . . . . . 10  
 Unit . . . . . s  
 Message  
 Record message . . . . . off  
 Message by e-mail . . . . . off  
 Acoustic signal . . . . . off

**MOVE to buffer 4**

Device  
 Device name . . . . . 855\_1



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Target  
 Tower ..... 1  
 Move ..... Rack position  
 Number ..... = 'MV.Sample position' + 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

**MEAS U**

**measure buffer 4**

**General/Hardware**

Device  
 Device name ..... 855\_1  
 Sensor  
 Measuring input ..... 1  
 Sensor ..... Solvotrode  
 Temperature measurement ..... automatic  
 Stirrer  
 Stirrer ..... 1  
 Stirring rate ..... 8  
 Switch off automatically ..... on

**Measuring parameters**

Measurement  
 Measurement with drift control ..... on  
 Signal drift ..... 10.0 mV/min  
 Min. waiting time ..... 60 s  
 Max. waiting time ..... 70 s  
 Measuring interval ..... 2.0 s  
 Stop measured value ..... off mV  
 Measurement without drift control ..... off  
 Temperature  
 Temperature ..... 25.0 °C

**Evaluations**

Fix end point 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 water beaker**

Device



License ID 124049905  
 Client name TITRATION14  
 User Metrohm

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```

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

WAIT      wait 10 sec
Wait
Stop track and waiting for [Continue] . . . . . off
Stop all tracks and waiting for [Continue] . . . . . off
Waiting time . . . . . on
Time . . . . . 10
Unit . . . . . s
Message
Record message . . . . . off
Message by e-mail . . . . . off
Acoustic signal . . . . . off

SERIES   Series start track
START

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

TRACK    message track
Return immediately . . . . . off
Delete all data . . . . . off

REQUEST  message
Sample data request
Sample position . . . . . off
ID1 . . . . . off
ID2 . . . . . off
ID3 . . . . . off
ID4 . . . . . off
ID5 . . . . . off
ID6 . . . . . off
ID7 . . . . . off
ID8 . . . . . off
    
```



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Sample size . . . . . off  
 Unit . . . . . off  
 Message . . . . . on

The absolute potential difference is smaller than 150 mV. The Solvotrode should be rinsed with hexane and ethyl alcohol. After rinsing the Solvotrode is immersed for 1 min. in dist. water, the ground joint of the diaphragm is loosened slightly and then closed again. The measurements are repeated after cleaning the electrode.

**EXIT**      **Exit track**

**CALC**      **calculation**

Result name	Formula	Unit	Decimal places	Assignment	Statistics
Potential buffer 7	= 'measure buffer 7.EME'	mV	4	RS01	off
Potential buffer 4	= 'measure buffer 4.EME'	mV	4	RS02	off
Absolute difference	=Abs( 'RS.potential buffer 7' - 'RS.potential buffer 4')	mV	4	RS03	off

Result name . . . . . **Potential buffer 7**  
 Formula . . . . . = 'measure buffer 7.EME'  
 Unit . . . . . mV  
 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 . . . . . **Potential buffer 4**  
 Formula . . . . . = 'measure buffer 4.EME'  
 Unit . . . . . mV  
 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 . . . . .

Result name . . . . . **Absolute difference**  
 Formula . . . . . =Abs( 'RS.potential buffer 7' - 'RS.potential buffer 4')



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Unit . . . . . mV  
 Decimal places . . . . . 4  
 Assignment . . . . . RS03  
 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 . . . . .

**CALL call message track**

Call text	Track name	Condition	Condition
show message	message track	on	= 'RS.Absolute difference' < 150

**REPORT report**

Report template  
     Report template . . . . . Solvotrode function test  
 Report output  
     Printer . . . . . off  
     PDF file . . . . . on  
     PDF file . . . . . C:\Programme\Metrohm\tiamo\solvotrode check.pdf  
     Send e-mail . . . . . off

**DATABASE database**

Database  
 Robotic TAN TBN Analyzer

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

**MOVE to storing beaker**

Device  
     Device name . . . . . 855\_1  
 Target  
     Tower . . . . . 1  
     Move . . . . . Special beaker  
     Number . . . . . 4  
 Beaker test  
     Display message . . . . . on  
     Stop determination . . . . . off  
     Stop determination and series . . . . . off  
 Parameters  
     Shift rate . . . . . 20 %/s  
     Shift direction . . . . . auto



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Swing rate . . . . . 55 °/s





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