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# 736 GP Titrino

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## Applications

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8.736.0003

95.12 ks/Ti

The methods from the 6.6023.000 card are stored in different directories according to their use in different industries. The methods are ready for use. You can load them and, if necessary, modify and restore them. The methods can be restored either in the internal method memory or on the 6.2245.010 memory card. (The 6.6023.000 card is "read only".)

For use of the methods note the following:

- Adjust the parameters (the **stop criteria** may be specially important) according to your samples.
- If no printer is connected, the **report output** of the methods has to be deleted: Key <def>, >report.
- If you need other result units than the ones given in the method, it may be necessary to adjust the **calculation** values C01 and/or the formulas.
- Instead of a combined **Pt electrode** a 6.0431.100 Pt Titrode can normally be used. The direction of the titration curve changes, e.g. if the curve goes from 100 to 400 mV with a combined Pt electrode, it will run from 400 to 100 mV with the Pt Titrode and vice versa.
- Instead of a combined **Ag electrode** a 6.0430.100 Ag Titrode can normally be used. The direction of the titration curve changes, e.g. if the curve goes from 100 to 400 mV with a combined Ag electrode, it will run from 400 to 100 mV with the Ag Titrode and vice versa.

## Contents

### Index

### Introduction

	<b>Method name</b>	<b>Appl. No.</b>
<b>1. General</b>		
Strong Acid	Acid	1-1
Titer of NaOH	Tit.NaOH	1-2
Titer of HCl	Tit.HCl	1-3
Oxalic Acid	Oxalic	1-4
Oxalic Acid non-aqueous	OxalicNA	1-5
Ca <sup>2+</sup> with Amalgamated Ag Electrode	Ca++	1-6
Calcium/Magnesium in Tap Water	Ca-Mg	1-7
Metals	Metals	1-8
Iodine	Iodine	1-9
Iron (II)	Iron (II)	1-10
Chloride	Chloride	1-11
Phosphate	Phosphat	1-12
<b>2. WaterEnvir</b>		
Chloride in Tap Water	Chloride	2-1
Boric Acid	Bor.Acid	2-1
p and m Value	p+m val.	2-3
Water Hardness	Hardness	2-4
Oxidizability of Waste Water	Oxidiza.	2-5
<b>3. Petro Chem</b>		
Total Acid Number (TAN)	TAN	3-1
Total Base Number (TBN)	TBN	3-2
Bromine Index	Br-Index	3-3
Bromine Number	BrNumber	3-4
<b>4. Food</b>		
NaCl in Broth	NaCl	4-1
Formaldehyde Number in Fruit Juices	FormolNo	4-2
Calcium in Milk Products	Milk-Ca	4-3
Peroxide Number	Perox.No	4-4
Saponification Number	Sapon.No	4-5
Acid Number	Acid.No	4-6
Iodine Number	Iod.No	4-7
Vitamine C	Vit.C	4-8

	<b>Method name</b>	<b>Appl. No.</b>
<b>5. Pharma</b>		
Na <sup>+</sup> /Cl <sup>-</sup> in NaCl Isotonic Solution	Na+Cl-	5-1
Antacid	Antacid	5-2
Trypsin	Trypsin	5-3
Lipase	Lipase	5-4
2-Aminophenol	Diazo	5-5
Vitamine C	Vit.C	5-6
<b>6. Detergents</b>		
EDTA/NTA in Detergents	EDTA-NTA	6-1
Nonionic Tensides	Tenside	6-2
Cationic Tensides in Antiseptic Disinfectant	Tens.Cat	6-3
Anionic Tensides in Liquid Soap	Tens.An	6-4
Perborates in Detergents	Perborat	6-5
<b>7. Galvanics</b>		
Pure Silver Content	Silver	7-1
Metals	Metals	7-2
Aluminium	Aluminum	7-3
<b>8. Basic.Mat</b>		
Phosphorus in Fertilizer	P2O5Fert	8-1
Epoxy Number	Epoxy.No	8-2
Nitrating Acid	NitrAcid	8-3
Hydrogen Peroxide	H2O2	8-4
Phosphate	Phosphat	8-5
2-Aminophenol	Diazo	8-6
<b>9. PaperPhoto</b>		
White Liquor	W-Liquor	9-1
Silver in Fixing Baths	FixBaths	9-2
Silver in Film Emulsions	Film Ag	9-3
Kappa Number of Pulp	KappaNo	9-4
Analysis of Spinning Bath	Spinning	9-5
2-Aminophenol	Diazo	9-6
<b>10. KF Titr.</b>		
KF Titer Determination with Sodium Tartrate	TarTiter	10-1
KF Titer Determination with Water	H2OTiter	10-2
Blank Determination of Methanol	Blank_KF	10-3
Water Determination in Paper	KF-Blank	10-4
Water Determination in Honey	KF	10-5

# Strong Acid

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

2 mL c(HCl) ≅ 0.1 mol/L  
50 mL dist. water

## Electrodes

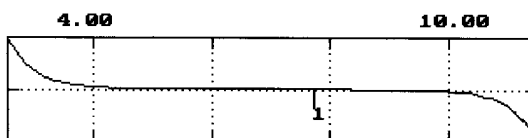
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 18:18  7
DET pH              Acid
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  titr.rate          max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  dos.element:       internal D0
  meas.input:        1
  temperature        25.0 °C
>stop conditions
  stop V:            abs.
  stop V             4 ml
  stop pH            OFF
  stop EP            9
  filling rate       max. ml/min
>statistics
  status:            OFF
>evaluation
  EPC                5
  EP recognition:    all
  fix EP1 at pH     OFF
  pK/HNP:            OFF
>preselections
  req.ident:         OFF
  req.smpl size:     OFF
  activate pulse:    OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 18:14  9
pH(init)           2.55  DET pH      Acid
smpl size          2.0 ml
EP1                1.996 ml          7.72
c(acid)             0.100 mol/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 18:14  9
start V            0.000 ml DET pH      Acid
2.0 ml/div         dpH=2.0/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 18:18  7
DET pH              Acid
>calculations
c(acid)=EP1*C01*C37/C00;3;mol/l
C00=                2.0
C01=                0.1
C37=                1.0013
-----
```

**Remarks**

- **Calculations:**  
c(acid) = concentration of acid in mol/L  
C01 = concentration of titrating agent (0.1 mol/L)  
C37 = titer of titrating agent (1.0013 common variable of "Tit.NaOH")
- Carbonate may be detected separately if present!

---

**Literature**

# Titer of NaOH

## Reagents

c(NaOH) = 0.1 mol/L; D0  
free of carbonate

## Sample

app. 300 mg potassium hydrogen phthalate dried 2 h at 105°C  
50 mL dist. water

## Electrodes

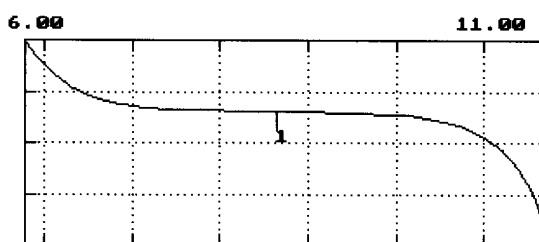
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 15:49  0
DET pH              Tit.NaOH
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  titr.rate          max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  dos.element:       internal D0
  meas.input:        1
  temperature        25.0 °C
>stop conditions
  stop V:            abs.
  stop V             20 ml
  stop pH            OFF
  stop EP            9
  filling rate       max. ml/min
>statistics
  status:            ON
  mean               n= 5
  res.tab:           original
>evaluation
  EPC                5
  EP recognition:    all
  fix EP1 at pH     OFF
  pK/HNP:           OFF
>preselections
  req.ident:         OFF
  req.smpl size:     value
  activate pulse:    OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 15:46  4
pH(init)           4.07      DET pH Tit.NaOH
smpl size           0.3021 g
EP1                 14.785 ml      8.63
Titer               1.0005
                    mean( 5)    +/-s      s/%
Titer               1.0013    0.00068      0.07
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 15:46  4
start V            12.000 ml DET pH Tit.NaOH
2.0 ml/div         dpH=1.0/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-05      time 15:49  0
DET pH              Tit.NaOH
>calculations
Titer=C00*C01/C02/EP1;4;
C00=                0.3021
C01=                10000
C02=                204.23
-----
```

**Remarks**

- **Calculations:**  
Titer = Titer of NaOH  
C01 = Theoretical consumption for 1 mol potassium hydrogen phthalate (for a solution with  $c=0.1$  mol/L = 10000 mL/mol)  
C02 = molecular mass of potassium hydrogen phthalate (204.23 g/mol)
- **Common variables**  
C37 = MN1
- Mean from 5 determinations.
- For shorter titration times, a start volume may be used, see example curve.

---

**Literature**

- Metrohm Application Bulletin No. 206: Titer determination in potentiometry

# Titer of HCl

## Reagents

c(HCl) = 0.1 mol/L; D0

## Sample

app. 100 mg tris(hydroxymethyl)aminomethane dried 2 h at 105°C  
50 mL dist. water

## Electrodes

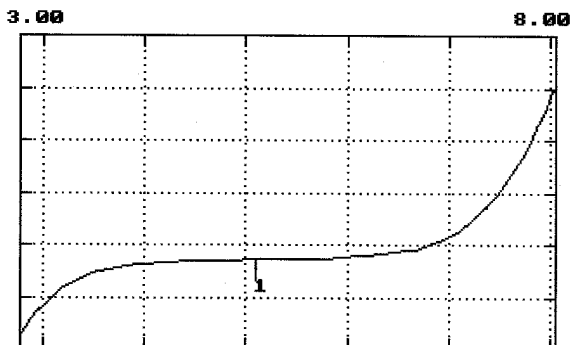
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-07      time 11:10   20
DET pH              Tit.HCl
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  titr.rate          max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           rel.
  factor             50
  dos.rate           max. ml/min
  pause              0 s
  dos.element:       internal D0
  meas.input:        1
  temperature        25.0 °C
>stop conditions
  stop V:            abs.
  stop V             99.99 ml
  stop pH            2.8
  stop EP            9
  filling rate       max. ml/min
>statistics
  status:            ON
  mean               n= 10
  res.tab:           original
>evaluation
  EPC                5
  EP recognition:    all
  fix EP1 at pH     OFF
  pK/HNP:           OFF
>preselections
  req.ident:         OFF
  req.smpl size:     value
  activate pulse:    OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-07      time 10:54   19
pH(init)           10.13   DET pH   Tit.HCl
smpl size          0.09959 g
EP1                8.286 ml           5.09
Titer              0.9922
                    mean( 5)  +/-s    s/%
Titer              0.9922  0.00044    0.04
stop meas.val.reached
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-07      time 10:54   19
start V            4.980 ml   DET pH   Tit.HCl
1.0 ml/div         dpH=1.0/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-07      time 11:10   20
DET pH              Tit.HCl
>calculations
Titer=C00*C01/C02/EP1;4;
C00=                1.0
C01=                10000
C02=                121.14
-----
```

**Remarks**

- **Calculations:**  
Titer = Titer of HCl  
C01 = Theoretical consumption for 1 mol tris(hydroxymethyl)aminomethane (for a solution with  $c=0.1$  mol/L = 10000 mL/mol)  
C02 = molecular mass of tris(hydroxymethyl)aminomethane (121.14 g/mol)
- **Common variables**  
C36 = MN1
- Mean from 5 determinations.
- This method may be used for the GLP validation, see Metrohm Bulletin 252.  
To shorten the titration times, a relative start volume is used for the GLP validation.

---

**Literature**

- Metrohm Application Bulletin No. 206: Titer determination in potentiometry
- Metrohm Application Bulletin No. 252: Validation of Metrohm titrators (potentiometric) according to GLP/ISO9001

# Oxalic Acid

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

2 mL c(C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>) ≅ 0.1 mol/L; HOOC-COOH pK<sub>1</sub> = 1.42, pK<sub>2</sub> = 4.31  
50 mL dist. water

## Electrodes

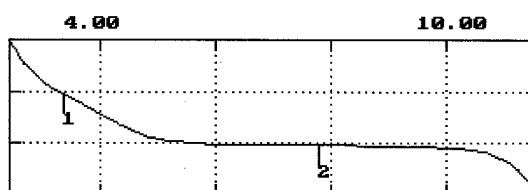
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 10:11    6
DET pH              Oxalic
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift     50 mV/min
  equilibr.time    26 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         6 ml
  stop pH        OFF
  stop EP        9
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at pH OFF
  pK/HNP:      ON
>preselections
  req.ident:    OFF
  req.smpl size: OFF
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 09:57    6
pHc(init)         2.41   DET pH   Oxalic
smpl size         2.00 ml
EP1               2.110 ml           3.36
EP2               4.098 ml           7.77
pK1               2.71
pK2               4.17
Oxalic            0.103 mol/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 09:57    6
start V           0.000 ml DET pH   Oxalic
2.0 ml/div       dpH=2.0/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 10:11    6
DET pH              Oxalic
>calculations
Oxalic=EP2*C01*C37/C02/C00;3;mol/l
C00=                2.00
C01=                0.1
C02=                2
C37=                1.0013
-----
```

**Remarks**

- **Calculations:**  
Oxalic = concentration of oxalic acid in mol/L  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = factor for "normality" (2)  
C37 = titer of titrating agent (1.0013 common variable of "Tit.NaOH")
  - Compare also titration in non aqueous medium, application No. 1-5
  - For pK determinations, the electrode should be calibrated.
- 

**Literature**

# Oxalic Acid Non-aqueous

## Reagents

c(TBAOH) = 0.1 mol/L; D0  
 TBAOH = Tetrabutyl ammonium hydroxide

## Sample

2 mL c(C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>) ≅ 0.1 mol/L; HOOC-COOH  
 25 mL ethanol

## Electrodes

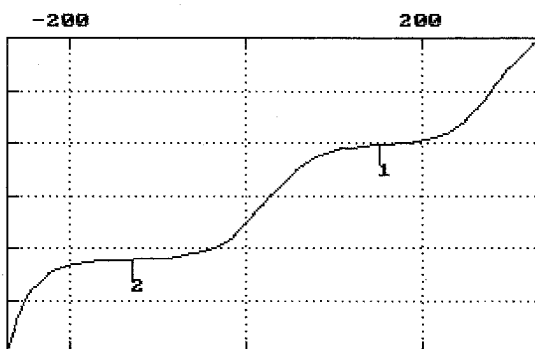
6.0103.100 pH glass electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (LiCl sat. in ethanol)

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-18      time 10:52  6
DET U              OxalicNA
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            50 ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V              6 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-18      time 10:47  6
U(init)            341 mV DET U OxalicNA
smpl size          2 ml
EP1                2.048 ml      152 mV
EP2                4.221 ml      -130 mV
Oxalic             0.106 mol/l
stop V reached
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-18      time 10:48  6
start V           0.000 ml DET U OxalicNA
1.0 ml/div        dU=200.0 mV/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-18      time 10:53  6
DET U              OxalicNA
>calculations
Oxalic =EP2*C01/C02/C00;3;mol/l
C00=                2
C01=                0.1
C02=                2
-----
```

---

**Remarks**

- **Calculations:**  
Oxalic = concentration of oxalic acid in mol/L  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = factor for "normality" (2)
- Compare also titration in aqueous medium, application No. 1-4

---

**Literature**

# Ca<sup>2+</sup> with Amalgamated Ag Electrode

## Reagents

c(Na<sub>2</sub>EDTA) = 0.1 mol/L; D0

## Sample

2 mL c(CaCl<sub>2</sub>) ≅ 0.1 mol/L  
 10 mL buffer pH = 10  
 40 mL dist. water

## Electrodes

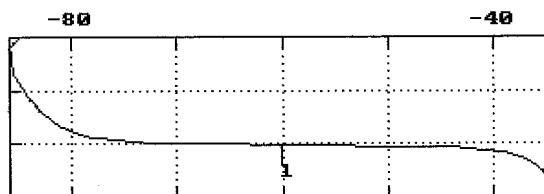
6.0430.100 Ag Titrode amalgamated; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 11:22    4
MET U              Ca++
parameters
>titration parameters
V step             0.10 ml
titr.rate          max. ml/min
signal drift      OFF mV/min
equilibr.time     10 s
start V:          OFF
pause             0 s
dos.element:     internal D0
meas.input:       1
temperature       25.0 °C
>stop conditions
stop V:           abs.
stop V            3 ml
stop U            OFF mV
stop EP           9
filling rate     max. ml/min
>statistics
status:           OFF
>evaluation
EPC               30 mV
EP recognition:   all
fix EP1 at U     OFF mV
pK/HNP:          OFF
>preselections
req.ident:        OFF
req.smpl size:   OFF
activate pulse:  OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 11:19    4
U(init)           -82 mV MET U    Ca++
smpl size         2 ml
EP1               2.043 ml          -60 mV
c(Ca++)           0.102 mol/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 11:19    4
start V           0.000 ml MET U    Ca++
1.0 ml/div        dU=10.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 11:22    4
MET U              Ca++
>calculations
c(Ca++)=EP1*C01/C00;3;mol/l
C00=                2
C01=                0.1
-----
```

**Remarks**

- **Calculations:**  
Ca<sup>++</sup> = concentration of Ca<sup>2+</sup> in mol/L  
C01 = concentration of titrating agent (0.1 mol/L)
  - **Coating of Ag Titrode:**  
Clean Ag Titrode first by immersing it in conc. HNO<sub>3</sub>, then immerse it shortly in Hg.
  - **Buffer pH = 10:**  
Dissolve 54 g NH<sub>4</sub>Cl and 350 mL w(NH<sub>3</sub>) = 0.25 (25%) in dist water and fill up of 1 liter.
  - If several metal ions which form EDTA complexes are present, their sum is determined.
  - For a greater break, add Hg-EDTA complex.
- 

**Literature**

# Calcium/Magnesium in Tap Water

## Reagents

$c(\text{Na}_2\text{EDTA}) = 0.05 \text{ mol/L}$  in  $c(\text{KOH}) = 0.1 \text{ mol/L}$ ; D0

## Sample

100 mL tap water  
 15 mL  $c(\text{Acetylacetone}) = 0.1 \text{ mol/L}$  in  $c(\text{Trishydroxymethylamino-methane}) = 0.1 \text{ mol/L}$  (auxiliary complexing agent, pH app. 8.5)

## Electrodes

6.0504.100  $\text{Ca}^{2+}$  sensitive indicator electrode; input 1  
 6.0733.100 Ag/AgCl reference electrode ( $c(\text{KCl}) = 3 \text{ mol/L}$ )

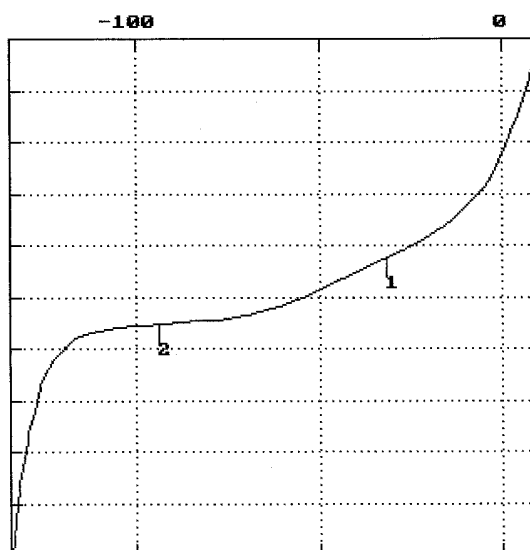
## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 15:08  1
DET U              Ca-Mg
parameters
>titration parameters
  meas.pt.density  1
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     20 mV/min
  equilibr.time    38 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         10 ml
  stop U         OFF mV
  stop EP       9
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: OFF
  activate pulse: OFF
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 15:02  1
U(init)            10 mV DET U    Ca-Mg
smpl size          100 ml id#1    Metrohm
id#2              Herisau id#3    TapWater
EP1               4.245 ml      -32 mV
EP2               5.535 ml      -94 mV
Ca++              2.12 mmol/l
Mg++              0.64 mmol/l
Total             2.77 mmol/l
stop V reached
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 15:02  1
start V           0.000 ml DET U    Ca-Mg
1.0 ml/div        dU=50.0 mV/div
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 15:08  1
DET U              Ca-Mg
>calculations
Ca++=EP1*C01*C02/C00;2;mmol/l
Mg++=(EP2-EP1)*C01*C02/C00;2;mmol/l
Total=EP2*C01*C02/C00;2;mmol/l
C00= 100
C01= 0.05
C02= 1000
```



**Remarks**

- 1st break:  $\text{Ca}^{2+}$   
2nd break:  $\text{Mg}^{2+}$
- **Calculations:**  
Ca<sup>++</sup> = calcium hardness in mmol/L  
Mg<sup>++</sup> = magnesium hardness in mmol/L  
Total = total hardness in mmol/L  
C01 = concentration of titrating agent (0.05 mol/L)  
C02 = factor for the conversion mol  $\Rightarrow$  mmol (1000)
- **Electrode preparation:**  
Ca electrodes should be conditioned for 10 min. in  $c(\text{CaCl}_2) = 0.01$  mol/L before use.
- The volume of auxiliary reagent can be optimised for the magnesium content. Rule of thumb: Ratio Mg/Acetylacetone app. 0.05.

---

**Literature**

- Metrohm Application Bulletin No. 125: Complexometric simultaneous determination of calcium and magnesium in water samples and beverages with the aid of an ion-selective calcium electrode

# Metals

## Reagents

c(Na<sub>2</sub>EDTA) = 0.1 mol/L; D0

## Sample

2 mL c(ZnSO<sub>4</sub>) ≅ 0.1 mol/L  
 5 mL buffer pH = 10  
 1 mL c(CuEDTA) = 0.1 mol/L  
 40 mL dist. water

## Electrodes

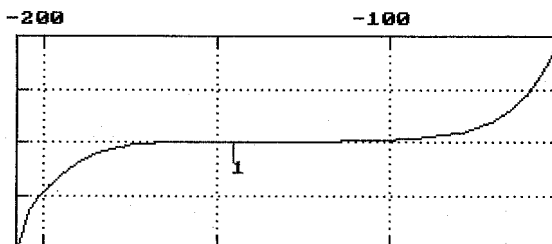
6.0502.140 Cu<sup>2+</sup> sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (KNO<sub>3</sub> sat.)

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-28      time 16:30   4
DET U              Metals
parameters
>titration parameters
  meas.pt.density  2
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     20 mV/min
  equilibr.time    38 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         10 ml
  stop U         OFF mV
  stop EP        9
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: all
  activate pulse: OFF
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-28      time 16:23   4
U(init)           -50 mV DET U   Metals
smpl size         2 ml
EP1               2.006 ml      -145 mV
Content           6.56 g/l
stop V reached
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-28      time 16:23   4
start V          0.000 ml DET U   Metals
1.0 ml/div      dU=50.0 mV/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-28      time 16:31   4
DET U              Metals
>calculations
Content=EP1*C01*C02/C00;2;g/l
C00=              2
C01=              0.1
C02=              65.38
```

### Remarks

- **Calculations:**  
 Content = content of metal in g/L  
 C01 = concentration of titrating agent (0.1 mol/L)  
 C02 = molecular mass of metal (65.38 g/mol)
- **Buffer pH = 10:**  
 Dissolve 54 g NH<sub>4</sub>Cl and 350 mL w(NH<sub>3</sub>) = 0.25 (25%) in dist. water and fill up to 1 litre.
- **Buffer pH = 4.7:**  
 Dissolve 123 g Na-acetate and 86 mL acetic acid in dist. water and fill up to 1 liter.

- The following metals can be determined according to this method:

		buffer solution	molar mass
Water, total hardness	(Ca + Mg)	pH = 10	64.40
Barium	Ba	pH = 10	137.36
Cadmium	Cd	pH = 10	112.41
Cobalt	Co	pH = 10	58.94
Nickel	Ni	pH = 10	58.71
Zinc	Zn	pH = 10	65.38
Lead	Pb	pH = 4.7	207.21

### Literature

- Metrohm Application Bulletin No. 101: Complexometric titrations with the Cu ISE

# Iodine

## Reagents

$c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol/L}; \text{D0}$

## Sample

3 mL  $c(\text{KI}_3) \cong 0.05 \text{ mol/L}$   
 5 mL  $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$   
 20 mL dist. water

## Electrodes

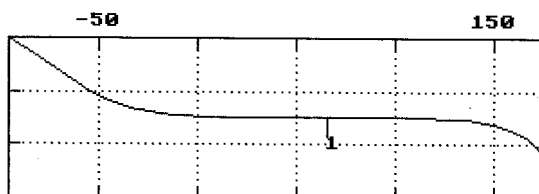
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 11:15   10
DET U              Jodine
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature           25.0 °C
>stop conditions
  stop V:              abs.
  stop V               5 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 11:12   10
U(init)             -95 mV DET U   Jodine
smpl size            3 ml
EP1                  2.997 ml
c(KI3)               0.0499 mol/l
stop V reached
=====
```

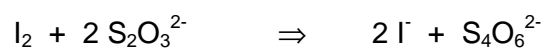
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 11:12   10
start V              0.000 ml DET U   Jodine
2.0 ml/div           dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 11:15   10
DET U              Jodine
>calculations
c(KI3)=EP1*C01/C02/C00;4;mol/l
C00=                 3
C01=                  0.1
C02=                  2
-----
```

**Remarks**

- **Determination reaction:**



- **Calculations:**

c(KI<sub>3</sub>) = concentration of KI<sub>3</sub> solution in mol/L

C01 = concentration of titrating agent (0.1 mol/L)

C02 = factor for "normality" (2)

- Titrate samples immediately.
- 

**Literature**

# Iron (II)

## Reagents

$c(\text{KMnO}_4) = 0.02 \text{ mol/L}; \text{D0}$

## Sample

2 mL  $c[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2] \cong 0.1 \text{ mol/L}$ , acidic solution  
 10 mL  $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$   
 40 mL dist. water

## Electrodes

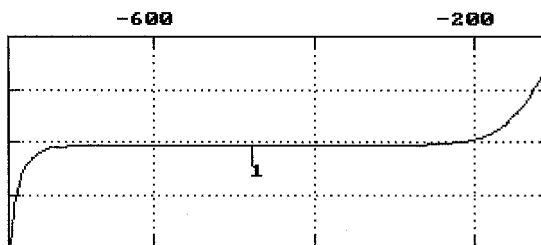
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-09      time 09:23    2
DET U              Iron(II)
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         10 mV/min
  equilibr.time        52 s
  start V:             abs.
  start V              0.5 ml
  dos.rate             max. ml/min
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               4 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:     OFF
-----
```

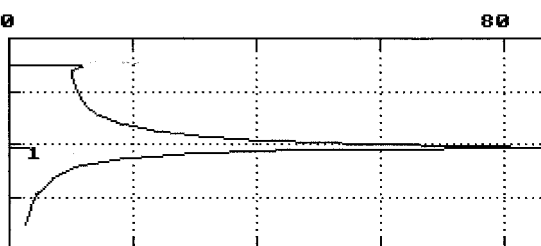
```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-09      time 09:33    3
U(init)            -136 mV DET U  Iron(II)
smpl size           2 ml
EP1                 2.065 ml      -476 mV
Fe++                5.77 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-09      time 09:34    3
start V            0.500 ml DET U  Iron(II)
1.0 ml/div         dU=200.0 mV/div
```



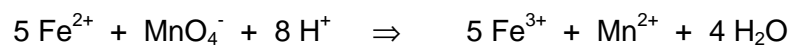
```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-09      time 09:23    2
DET U              Iron(II)
>calculations
Fe++=EP1*C01*C02/C00;2;g/l
C00=                2
C01=                 0.1
C02=                 55.85
-----
```

```
'dv
736 GP Titrino      OP1/103  736.0010
date 95-10-09      time 09:36    3
start V            0.500 ml DET U  Iron(II)
1.0 ml/div         ERC=20.0/div
```



**Remarks**

- **Determination reaction:**



- **Calculations:**

Fe<sup>++</sup> = concentration of Fe<sup>2+</sup> in g/L

C01 = concentration of titrating agent \* titer \* "normality"  
(0.02 \* 1.000 \* 5 = 0.1)

C02 = molecular mass of Iron (55.85 g/mol)

---

**Literature**

# Chloride

## Reagents

$c(\text{AgNO}_3) = 0.1 \text{ mol/L}$ ; D0

## Sample

5 mL  $c(\text{NaCl}) \cong 0.1 \text{ mol/L}$   
 2 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$   
 40 mL dist. water

## Electrodes

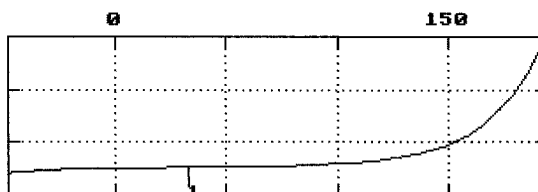
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 09:23   3
DET U              Chloride
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               99.99 ml
  stop U               OFF mV
  stop EP              1
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       all
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 09:19   3
U(init)             192 mV DET U Chloride
smpl size           5.00 ml
EP1                  4.969 ml           33 mV
Chloride             0.35 %
NaCl                  5.81 g/l
stop #EP reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 09:19   3
start V             0.000 ml DET U Chloride
2.0 ml/div          dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 09:23   3
DET U              Chloride
>calculations
Chloride=EP1*C01*C02*C03/C00;2;%
NaCl=EP1*C01*C04/C00;2;g/l
C00=                 5.00
C01=                  0.1
C02=                 35.45
C03=                  0.1
C04=                 58.44
-----
```

**Remarks**

- **Calculations:**  
Chloride = content of chloride in %  
NaCl = content of NaCl in g/L  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = molecular mass of Cl<sup>-</sup> (35.45 g/mol)  
C03 = factor for % (0.1)  
C04 = molecular mass of NaCl (58.44)
  - Select the appropriate formula. The other may be deleted.  
Or change the formula according to your application.
- 

**Literature**

- Metrohm Application Bulletin No. 130: Chloride titrations with potentiometric end-point indication.

# Phosphate

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

5 mL c(NaH<sub>2</sub>PO<sub>4</sub>) ≅ 0.1 mol/L  
 40 mL dist. water  
 adjust the pH value to 4.2 with dilute NaOH or H<sub>2</sub>SO<sub>4</sub>  
 10 mL c(La(NO<sub>3</sub>)<sub>3</sub>) = 0.1 mol/L, pH = 4.2

## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```

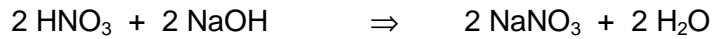
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-10-12      time 14:01    6
SET pH              Phosphat
parameters
>SET1
  EP at pH          4.20
  dynamics           1
  max.rate          10 ml/min
  min.rate          25 µl/min
  stop crit:        drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:   +
  pause 1           0 s
  start V:          OFF
  pause             0 s
  extr.time         0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    value
  activate pulse:   OFF
-----

'fr
736 GP Titrimo      OP1/103  736.0010
date 95-10-12      time 14:00    6
pHc(init)          1.79
smpl size           0.07798 g
EP1                 10.006 ml
P                   19.87 %
P2O5                 45.53 %
PO4                  60.93 %
=====

'fm
736 GP Titrimo      OP1/103  736.0010
date 95-10-12      time 14:01    6
SET pH              Phosphat
>calculations
P=EP1*C01*C02/C00;2;%
P2O5=EP1*C01*C03/C00;2;%
PO4=EP1*C01*C04/C00;2;%
C00=                 0.07798
C01=                 0.1
C02=                 1.5487
C03=                 3.5486
C04=                 4.7486
-----
    
```

### Remarks

- **Determination reaction:**



- **Calculations:**

P = content of P in %

P2O5 = content of P<sub>2</sub>O<sub>5</sub> in %

PO4 = content of PO<sub>4</sub><sup>3-</sup> in %

C01 = factor for %

C02 = 1 mL c(NaOH) = 0.1 mol/L = 1.5487 mg P

C03 = 1 mL c(NaOH) = 0.1 mol/L = 3.5486 mg P<sub>2</sub>O<sub>5</sub>

C04 = 1 mL c(NaOH) = 0.1 mol/L = 4.7486 mg PO<sub>4</sub>

- **Sample preparation:**

The pH of the sample aliquot has to be adjusted to pH = 4.2 with dil. NaOH or H<sub>2</sub>SO<sub>4</sub>. Add 10 mL c(La(NO<sub>3</sub>)<sub>3</sub>) = 0.1 mol/L (pH = 4.2) and titrate.

Calibrate the electrode for the SET titration.

- Select the appropriate formula. The others may be deleted.

### Literature

- Metrohm Application Bulletin No. 129: Potentiometric determination of ortho-, meta- and polyphosphates.

# Chloride in Tap Water

## Reagents

$c(\text{AgNO}_3) = 0.01 \text{ mol/L}$ ; D0

## Sample

100 mL tap water  
0.5 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$

## Electrodes

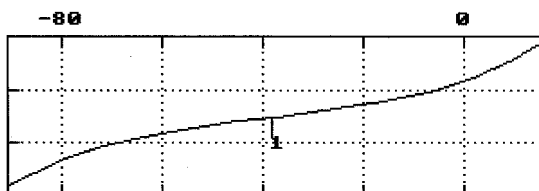
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-14      time 15:45   6
DET U              Chloride
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause            0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  stop U           OFF mV
  stop EP         1
  filling rate     max. ml/min
>statistics
  status:          OFF
>evaluation
  EPC              5
  EP recognition: all
  fix EP1 at U    OFF mV
  pK/HNP:         OFF
>preselections
  req.ident:       OFF
  req.smpl size:  OFF
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-14      time 15:44   6
U(init)            17 mV DET U Chloride
smpl size          100 ml
EP1                1.533 ml      -38 mV
Chloride           5.43 ppm
stop #EP reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-14      time 15:44   6
start V            0.000 ml DET U Chloride
1.0 ml/div         dU=20.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-14      time 15:45   6
DET U              Chloride
>calculations
Chloride=EP1*C01*C02*C03/C00;2;ppm
C00=               100
C01=               0.01
C02=               35.45
C03=               1000
-----
```

**Remarks**

- **Calculations:**  
Chloride = fraction of chloride in ppm  
C01 = concentration of titrating agent (0.01 mol/L)  
C02 = molecular mass of Cl<sup>-</sup> (35.45 g/mol)  
C03 = factor for ppm (1000)

---

**Literature**

- Metrohm Application Bulletin No. 130: Chloride titrations with potentiometric end-point indication.

# Boric Acid

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

5.00 mL c(H<sub>3</sub>BO<sub>3</sub>) ≅ 0.1 mol/L  
 10 mL d-mannitol solution, saturated  
 40 mL dist. water

## Electrodes

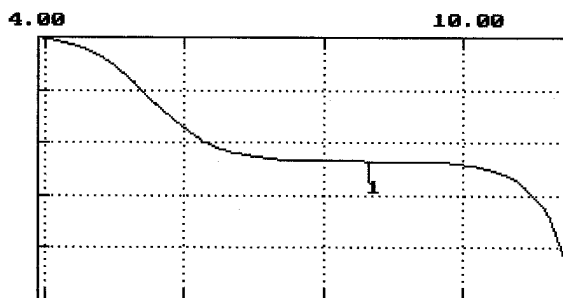
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-14      time 15:01  3
DET pH             Bor.AcId
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause            0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           10 ml
  stop pH          OFF
  stop EP          9
  filling rate     max. ml/min
>statistics
  status:          OFF
>evaluation
  EPC              5
  EP recognition:  greatest
  fix EP1 at pH   OFF
  pK/HNP:         OFF
>preselections
  req.ident:       OFF
  req.smpl size:  OFF
  activate pulse:  OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-14      time 14:57  3
pH(init)           3.90  DET pH  Bor.AcId
smpl size          5.00 ml
EP1                4.719 ml
Bor                1021.6 mg/l
stop V reached
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-14      time 14:57  3
start V            0.000 ml DET pH Bor.AcId
2.0 ml/div         dpH=2.0/div
```

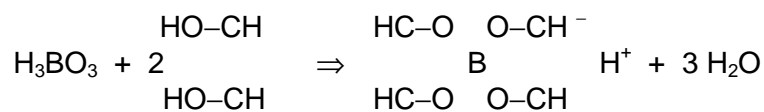


```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-14      time 15:01  3
DET pH             Bor.AcId
>calculations
Bor=EP1*C01*C02*C37/C00;1;mg/l
C00=                5.00
C01=                1.081
C02=                1000
C37=                1.0013
-----
```

### Remarks

- **Determination reaction:**

Boric acid forms an ester with mannitol:



- **Calculations:**

Bor = content of Bor in mg/L

C01 = Bor equivalent (1.081 mg/mL titrating agent)

C02 = factor for the conversion mL  $\Rightarrow$  L (1000)

C37 = titer of titrating agent (1.0013)

- d-mannitol solution, saturated:

App. 200 g d-mannitol dissolved in dist. water.

### Literature

- Metrohm Application Bulletin No. 66: Potentiometric determination of boric acid.

# p and m Value

## Reagents

c(HCl) = 0.1 mol/L; D1

## Sample

100 mL tap water

## Electrodes

6.0239.100 combined pH glass electrode; input 2

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 11:37    0
SET pH             p+m val.
parameters
>SET1
  EP at pH          8.20
  dynamics          2
  max.rate          10 ml/min
  min.rate          5 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at pH          4.30
  dynamics          3
  max.rate          10 ml/min
  min.rate          5 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>titration parameters
  titr.direction:   auto
  pause 1           0 s
  start V:          OFF
  pause            0 s
  extr.time        0 s
  dos.element:     external D1
  meas.input:      2
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:          OFF
>preselections
  conditioning:    OFF
  req.ident:       OFF
  req.smpl size:   OFF
  activate pulse:  OFF
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 11:35    18
pHc(init)          7.76   SET pH  p+m val.
smpl size          100 ml   id#1  Metrohm
id#2               Herisau id#3  TapWater
EP1                0.000 ml  7.77
EP2                5.544 ml  4.29
p value            0.00 mmol/l
m value            5.54 mmol/l
=====
```

```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 11:37    0
SET pH             p+m val.
>calculations
p value=EP1*C01;2;mmol/l
m value=EP2*C01;2;mmol/l
C01= 1.0
-----
```

**Remarks**

- Values for the acid capacity of water.  
p value = value of Phenolphthalein  
m value = value of Methyl orange  
C01= titer of titrating agent (1 mol/L)
- **Calculations:**  
p value = p value in mmol/L (if 0.00 mmol/L, the water has an initial pH below 8.2)  
m value = m value in mmol/L
- The dosing unit is external D1. This method can therefore be used directly in the TIP „Hardness“ (Application 2-4), an automated water analysis method.
- Calibrate the electrode for the SET titration.

---

**Literature**

- DIN 38 409, Teil 7 (1979)

# Water Hardness

## Reagents

$c(\text{Na}_2\text{EDTA}) = 0.05 \text{ mol/L}$  in  $c(\text{KOH}) = 0.1 \text{ mol/L}$ ; D0  
 $c(\text{HCl}) = 0.1 \text{ mol/L}$ ; D1  
 $c(\text{Acetylacetone}) = 0.1 \text{ mol/L}$  in  $c(\text{Trishydroxy methylaminomethane}) = 0.1 \text{ mol/L}$  (auxiliary complexing agent, pH app. 8.5); D2

## Sample

100 mL water

## Electrodes

6.0239.100 combined pH glass electrode; input 2  
 6.0504.100  $\text{Ca}^{2+}$  sensitive indicator electrode; input 1  
 reference system used from 6.0239.100

## Method documentation

```

'pa
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 10:56  11
TIP                    Hardness
parameters
>sequence
  1.method:             p+m val.
  2.method:             AddBufFe
  3.pause               15 s
  4.method:             Ca-Mg
>statistics
  status:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  meas.mode:           OFF
  temperature          25.0 °C
-----

'fr
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 10:54  11
TIP                    Hardness
smpl size              100 ml
id#2                   Herisau
id#3                   TapWater
C70                    7.15
C71                    0.000
C72                    5.609
C73                    4.237
C74                    5.748
pH                     7.15
p value                0.00 mmol/l
m value                5.61 mmol/l
Ca++                  2.12 mmol/l
Mg++                  0.76 mmol/l
Total                  2.87 mmol/l
TIP terminated
=====
    
```

```

'fm
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 10:56  11
TIP                    Hardness
>calculations
pH=C70;2;
p value=C71;2;mmol/l
m value=C72;2;mmol/l
Ca++=C73*C01*C02/C00;2;mmol/l
Mg++=(C74-C73)*C01*C02/C00;2;mmol/l
Total=C74*C01*C02/C00;2;mmol/l
C00=                   100
C01=                   0.05
C02=                   1000
C70=                   7.15
C71=                   0.000
C72=                   5.609
C73=                   4.237
C74=                   5.748
-----
    
```

**Submethods**

p+m value

p+m value

```
'pa
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 11:06      12
SET pH                  p+m val.
parameters
>SET1
  EP at pH              8.20
  dynamics              2
  max.rate              10 ml/min
  min.rate              5 µl/min
  stop crit:           drift
  stop drift            20 µl/min
>SET2
  EP at pH              4.30
  dynamics              3
  max.rate              10 ml/min
  min.rate              5 µl/min
  stop crit:           drift
  stop drift            20 µl/min
>titration parameters
  titr.direction:      auto
  pause 1              0 s
  start V:              OFF
  pause 2              0 s
  extr.time            0 s
  dos.element:         external D1
  meas.input:          2
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V                99.99 ml
  filling rate         max. ml/min
>statistics
  status:              OFF
>preselections
  conditioning:        OFF
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
```

```
'fr
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 10:46      11
pHc(init)              7.15   SET pH  p+m val.
smpl size              100 ml   id#1  Metrohm
id#2                   Herisau  id#3  TapWater
EP1                     0.000 ml
EP2                     5.609 ml
p value                 0.00 mmol/l
m value                 5.61 mmol/l
=====
```

Buffer addition

```
'de
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 11:00
SET pH                  p+m val.
def
>formula
  p value=EP1*C01
  RS1 text              p value
  RS1 decimal places   2
  RS1 unit:             mmol/l
  m value=EP2*C01
  RS2 text              m value
  RS2 decimal places   2
  RS2 unit:             mmol/l
>silco calculations
  match id:             OFF
>common variables
>report
  report:full;
>mean
  MN1=RS1
>temporary variables
  C70=C40
  C71=RS1
  C72=RS2
```

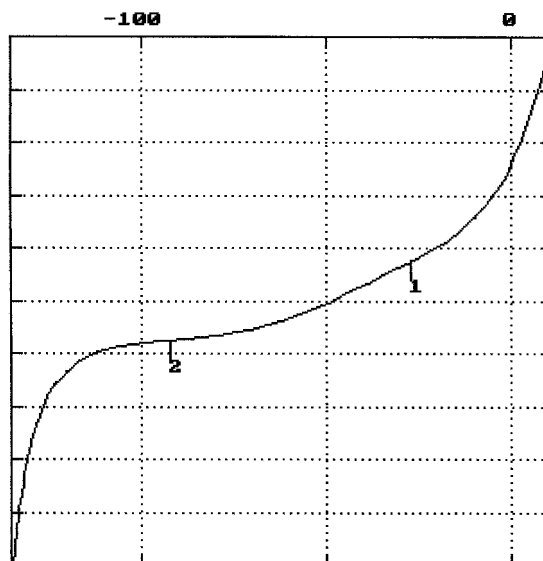
```
'pa
736 GP Titrimo          OP1/103  736.0010
date 95-09-15          time 11:08      12
DOS                     AddBuffe
parameters
>dosing parameters
  dispensing type:     volume
  volume               15 ml
  disp.crit:           rate
  rate                 max. ml/min
  pause                0 s
  time interval        10 s
  dos.element:         external D2
  temperature          25.0 °C
>stop conditions
  stop V:              OFF
  filling rate         max. ml/min
>statistics
  status:              OFF
>monitoring
  meas.mode:           OFF
  temperature:         OFF
  assign output:       none
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
```

**Submethods**

Ca-Mg

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-15      time 11:08   12
DET U              Ca-Mg
parameters
>titration parameters
  meas.pt.density  1
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     20 mV/min
  equilibr.time    38 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         10 ml
  stop U         OFF mV
  stop EP        9
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: OFF
  activate pulse: OFF
  -----
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-15      time 10:52   11
start V           0.000 ml DET U   Ca-Mg
1.0 ml/div        dU=50.0 mV/div
```



```
'de
736 GP Titrino      OP1/103  736.0010
date 95-09-15      time 11:13
DET U              Ca-Mg
def
>formula
  Ca++=EP1*C01*C02/C00
  RS1 text         Ca++
  RS1 decimal places 2
  RS1 unit:        mmol/l
  Mg++=(EP2-EP1)*C01*C02/C00
  RS2 text         Mg++
  RS2 decimal places 2
  RS2 unit:        mmol/l
  Total=EP2*C01*C02/C00
  RS3 text         Total
  RS3 decimal places 2
  RS3 unit:        mmol/l
>silco calculations
  match id:        OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C73=EP1
  C74=EP2
  -----
```

## Remarks

- Method p+m val.  
Values for the acid capacity of water.  
p value = value of Phenolphthalein  
m value = value of Methyl orange  
The method can also be used just for p+m values, see Application 2-3.
- Method Ca-Mg:  
1st break:  $\text{Ca}^{2+}$   
2nd break:  $\text{Mg}^{2+}$   
The method can also be used just for Ca/Mg determinations, see Application 1-7.
- **Calculations:**  
p value = p value in mmol/L (if 0.00 mmol/L, the water has an initial pH below 8.2)  
m value = m value in mmol/L (acid-binding-capability)  
Ca++ = calcium hardness in mmol/L  
Mg++ = magnesium hardness in mmol/L  
Total = total hardness in mmol/L  
C01 = concentration of titrating agent (0.05 mol/L)  
C02 = factor for conversion for mol  $\Rightarrow$  mmol (1000)  
C70 = initial pH of sample (p+m val.)  
C71 = EP1 at pH 8.2 (p+m val.)  
C72 = EP2 at pH 4.3 (p+m val.)  
C73 = EP1 from  $\text{Ca}^{2+}$  (Ca-Mg)  
C74 = EP2 from  $\text{Mg}^{2+}$  (Ca-Mg)
- **Electrode preparation:**  
Ca electrodes should be conditioned for 10 min. in  $c(\text{CaCl}_2) = 0.01$  mol/L before use.
- The volume of the auxiliary reagent can be optimised for the magnesium content. Rule of thumb: Ratio Mg/Acetylacetone app. 0.05.

---

## Literature

- Metrohm Application Bulletin No. 125: Complexometric simultaneous determination of calcium and magnesium in water samples and beverages with the aid of an ion-selective calcium electrode

# Oxidizability of Waste Water

## Reagents

$c(\frac{1}{5}KMnO_4) = 0.01 \text{ mol/L}; D0$

## Sample

25 mL waste water  
 5 mL  $w(H_2SO_4) = 0.35 (35\%)$   
 75 mL dist. water  
 15 mL  $c(\frac{1}{5}KMnO_4) = 0.01 \text{ mol/L}$   
 15 mL  $c(\frac{1}{2}Oxalic \text{ acid}) = 0.01 \text{ mol/L}$

## Electrodes

6.0431.100 Pt Titrode; input 1

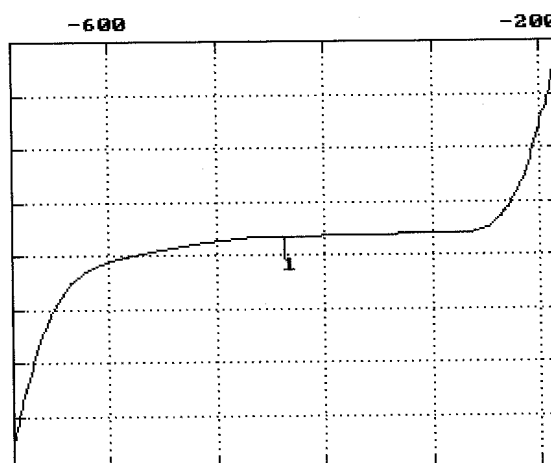
## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 11:25  1
MET U              Oxidiza.
parameters
>titration parameters
V step              0.10 ml
titr.rate           max. ml/min
signal drift        20 mV/min
equilibr.time       38 s
start V:            OFF
pause               0 s
dos.element:        internal D0
meas.input:         1
temperature         25.0 °C
>stop conditions
stop V:             abs.
stop V              7.5 ml
stop U              OFF mV
stop EP             9
filling rate        max. ml/min
>statistics
status:             OFF
>evaluation
EPC                 30 mV
EP recognition:     all
fix EP1 at U        OFF mV
pK/HNP:             OFF
>preselections
req.ident:          OFF
req.smpl size:      OFF
activate pulse:     OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 11:20  1
U(init)            -174 mV MET U Oxidiza.
smpl size           25 ml
EP1                 3.682 ml          -437 mV
Oxidiza.            46.540 mg/L
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 11:20  1
start V            0.000 ml MET U Oxidiza.
1.0 ml/div         dU=100.0 mV/div
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 11:25  1
MET U              Oxidiza.
>calculations
Oxidiza.=EP1*C01*C02/C00;3;mg/L
C00=                25
C01=                 0.316
C02=                 1000
-----
```



=====

### Remarks

- **Prepare sample as follows:**  
 Add 75 mL dist. water and 5 mL  $w(\text{H}_2\text{SO}_4) = 0.35$  (35%) to 25 mL of sample and heat up to a boiling mixture. Add 15.00 mL of  $c(\frac{1}{5}\text{KMnO}_4) = 0.01$  mol/L and keep boiling for 10 minutes. Then add 15 mL  $c(\frac{1}{2}\text{Oxalic acid}) = 0.01$  mol/L.  
 Titrate with  $c(\frac{1}{5}\text{KMnO}_4) = 0.01$  mol/L according to parameters.
- **Calculations:**  
 Oxidiza. = oxidizability in mg/L permanganate consumption  
 $C01 = \text{molecular mass of KMnO}_4 \cdot \text{concentration of titrating agent} / \text{normality}$  ( $158 \cdot 0.01 / 5 = 0.316$  g/L)  
 $C02 = \text{conversion g} \Rightarrow \text{mg for result expression in mg/L KMnO}_4$   
 (1000)

### Literature

- Deutsche Einheitsverfahren zur Wasseruntersuchung  
 Kapitel H4, Abschnitt 1

# Total Acid Number (TAN)

## Reagents

c(TBAOH) = 0.1 mol/L in isopropanol/methanol; D0  
TBAOH = Tetrabutyl ammonium hydroxide

## Sample

app. 1.5 g of used motor oil  
50 mL solvent: chlorobenzene : isopropanol (2 : 1)

## Electrodes

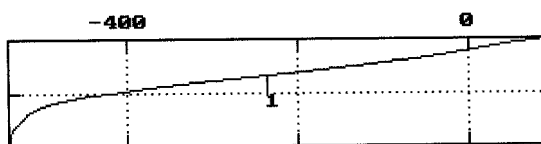
6.0133.100 pH glass electrode; input 1  
6.0729.100 Ag/AgCl reference electrode ( LiCl sat in ethanol); input 2  
6.0331.000 Pt auxiliary electrode; input Ref.  
Differential potentiometry

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 18:05   26
MET U              TAN
parameters
>titration parameters
V step             0.10 ml
titr.rate          max. ml/min
signal drift       OFF mV/min
equilibr.time      50 s
start V:           OFF
pause              100 s
dos.element:       internal D0
meas.input:        diff.
temperature        25.0 °C
>stop conditions
stop V:            abs.
stop V             10 ml
stop U             OFF mV
stop EP            9
filling rate       max. ml/min
>statistics
status:            OFF
>evaluation
EPC                20 mV
EP recognition:    last
fix EP1 at U      OFF mV
pK/HNP:           OFF
>preselections
req.ident:         OFF
req.smpl size:     value
activate pulse:    OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 18:00   26
U(init)            27 mV MET U      TAN
smpl size          1.6646 g
EP1                0.680 ml         -236 mV
TAN                2.29 mg/g
stop V reached
=====

'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 18:00   26
start V            0.000 ml MET U    TAN
1.0 ml/div         dU=200.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 18:05   26
MET U              TAN
>calculations
TAN=(EP1-C01)*C02*C03/C00;2;mg/g
C00=                1.6646
C01=                 0
C02=                 0.1
C03=                56.106
-----
```

**Remarks**

- **Calculations:**  
TAN = acid of sample, calculated as mg KOH per g of sample  
C01 = blank value, consumption of titrating agent by solvent mixture (0 mL)  
C02 = concentration of titrating agent (0.1 mol/L)  
C03 = molecular mass of KOH (56.106 g/mol)
- Store glass electrode in dist. water over night. Before titrating, precondition it in solvent during 10 - 30 min.
- The 6.0430.100 Ag Titrode may be used instead of the 6.0133.100 and 6.0331.000 electrodes. In this case, you make differential potentiometry with only two electrodes, the Ag Titrode and the 6.0729.100 reference electrode.

---

**Literature**

- Metrohm Application Bulletin No. 80: Determination of the acid / base number in petroleum products (TAN/TBN)
- ASTM D 2896 - 80
- DIN 51596

# Total Base Number (TBN)

## Reagents

$c(\text{HClO}_4) = 0.1 \text{ mol/L}$  in acetic acid; D0

## Sample

app. 1.5 g of used motor oil  
50 mL solvent  
chlorobenzene : acetic acid (2 : 1)

## Electrodes

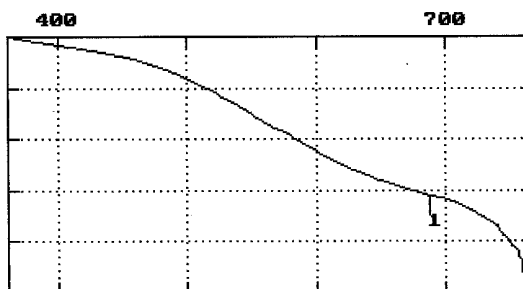
6.0133.100 pH glass electrode; input 1  
6.0729.100 Ag/AgCl reference electrode ( LiCl sat in ethanol); input 2  
6.0331.000 Pt auxiliary electrode; input Ref.  
Differential potentiometry

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:28  6
MET U              TBN
parameters
>titration parameters
V step             0.10 ml
titr.rate          max. ml/min
signal drift      OFF mV/min
equilibr.time     50 s
start V:          OFF
pause             100 s
dos.element:     internal D0
meas.input:      diff.
temperature       25.0 °C
>stop conditions
stop V:           abs.
stop V            10 ml
stop U            OFF mV
stop EP          9
filling rate     max. ml/min
>statistics
status:          OFF
>evaluation
EPC              20 mV
EP recognition:  last
fix EP1 at U    OFF mV
pK/HNP:         OFF
>preselections
req.ident:       OFF
req.smpl size:  value
activate pulse:  OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:04  6
U(init)          351 mV MET U      TBN
smpl size        1.6708 g
EP1              3.102 ml          688 mg
TBN              10.42 mg/g
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:04  6
start V          0.000 ml MET U      TBN
1.0 ml/div      dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:28  6
MET U              TBN
>calculations
TBN=(EP1-C01)*C02*C03/C00;2;mg/g
C00=              1.6708
C01=              0
C02=              0.1
C03=              56.106
-----
```

**Remarks**

- **Calculations:**  
TBN = base of sample, calculated as mg KOH per g of sample  
C01 = blank value, consumption of titrating agent by solvent mixture (0 mL)  
C02 = concentration of titrating agent (0.1 mol/L)  
C03 = molecular mass of KOH (56.106 g/mol)
- Store glass electrode in dist. water over night. Before titrating, precondition it in solvent during 10 - 30 min.
- The 6.0430.100 Ag Titrode may be used instead of the 6.0133.100 and 6.0331.000 electrodes. In this case, you make differential potentiometry with only two electrodes, the Ag Titrode and the 6.0729.100 reference electrode.

---

**Literature**

- Metrohm Application Bulletin No. 80: Determination of the acid / base number in petroleum products (TAN/TBN)
- ASTM D 2896 - 80
- DIN 51596

# Bromine Index

## Reagents

$c(\text{BrO}_3^-/\text{Br}^-) = 0.05 \text{ mol/L}; \text{D0}$

## Sample

50  $\mu\text{L}$   $c(\text{cyclohexene}) \cong 10 \%$  in solvent  
25 mL solvent

## Electrodes

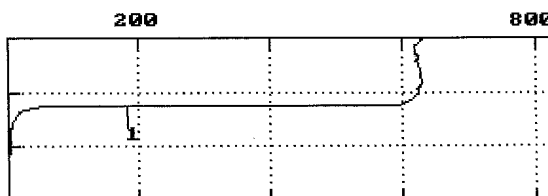
6.0308.100 double Pt electrode; input Pol  
polarized  $I_{\text{pol}} = 1 \mu\text{A}$   
Voltametric indication

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-10-04      time 11:40   3
MET Ipol           Br-Index
parameters
>titration parameters
  V step           0.05 ml
  titr.rate        max. ml/min
  signal drift     OFF mV/min
  equilibr.time    20 s
  start V:         OFF
  pause            0 s
  dos.element:    internal D0
  I(pol)           1  $\mu\text{A}$ 
  electrode test:  OFF
  temperature      25.0  $^\circ\text{C}$ 
>stop conditions
  stop V:          abs.
  stop V           10 ml
  stop U           1 mV
  stop EP         9
  filling rate     max. ml/min
>statistics
  status:         OFF
>evaluation
  EPC             30 mV
  EP recognition: greatest
  fix EP1 at U    OFF mV
>preselections
  req.ident:      OFF
  req.smpl size:  value
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-10-04      time 11:07   3
U(init)           295 mV MET IpolBr-Index
smpl size         0.050 g
EP1               1.287 ml           186 mV
Br-Index          10283.1 mg
manual stop
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-10-04      time 11:07   3
start V           0.000 ml MET IpolBr-Index
1.0 ml/div        dU=200.0 mV/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-10-04      time 11:40   3
MET Ipol           Br-Index
>calculations
Br-Index=(EP1-C01)*C02*C03/C00;1;mg
C00=               0.050
C01=               0.0
C02=               0.05
C03=               7990
-----
```

### Remarks

- Bromine index is the number of mg of bromine consumed per 100 g of sample
- **Solvent:**  
714 mL acetic acid  
134 mL CCl<sub>4</sub>  
134 mL CH<sub>3</sub>OH  
18 mL w(H<sub>2</sub>SO<sub>4</sub>) = 0.2 (20%)
- **Titration agent:**  
Dissolve 5.1 g KBr and 1.4 g KBrO<sub>4</sub> each in dist. water and add up to 1 liter.
- **Standardization of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution:**  
Determination reaction:  

$$3 \text{ Br}_2 + 6 \text{ S}_2\text{O}_3^{2-} \Rightarrow 6 \text{ Br}^- + 3 \text{ S}_4\text{O}_6^{2-}$$
  
Calculation:  
 RS1 = EP1\*C01/C00      normality of titrating agent (BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup>)  
 C01 = concentration of titrating agent (S<sub>2</sub>O<sub>3</sub><sup>2-</sup>)  
 C00 = mL of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution
- **Determination reaction for bromine index:**  

$$\text{BrO}_3^- + 5 \text{ Br}^- + 6 \text{ H}^+ \Rightarrow 3 \text{ Br}_2 + 3 \text{ H}_2\text{O}$$
  

$$3 \text{ Br}_2 + 3 \text{ Br}^- \Rightarrow 3 \text{ Br}_2 + 3 \text{ Br}^-$$
- **Calculations for bromine index:**  
 Br-Index = bromine index  
 C01 = consumption of blank sample (0 mL)  
 C02 = normality of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution as calculated above  
 ( 0.05 mol/L)  
 C03 = molecular mass of Br multiplied with 100 (7990 g/mol)

### Literature

- Metrohm Application Bulletin No. 177: Determination of the bromine index in petroleum products acc. to ASTM D 1491 - 60 and ASTM D 2710 - 72, resp. (and bromine number with ASTM D 1159 - 84)

# Bromine Number

## Reagents

$c(\text{BrO}_3^-/\text{Br}^-) = 0.5 \text{ mol/L}; \text{D0}$

## Sample

1 mL  $c(\text{cyclohexene}) \cong 6 \%$  in solvent  
25 mL solvent

## Electrodes

6.0309.100 double Pt electrode; input Pol  
polarized  $I_{\text{pol}} = 10 \mu\text{A}$   
Voltametric indication

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:33      8
KFT Ipol           BrNumber
parameters
>control parameters
  EP at U           500 mV
  dynamics          500 mV
  max.rate          5 ml/min
  min.volume incr.  min. µl
  stop crit:        time
  t(delay)          30 s
>titration parameters
  titr.direction:   -
  pause 1           0 s
  start V:          OFF
  pause             0 s
  extr.time         0 s
  dos.element:      internal D0
  I(pol)            10 µA
  electrode test:   OFF
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:31      8
KFT Ipol           BrNumber
smpl size          6.00 g
EP1                2.114 ml
BrNumber           141
=====
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 14:34      8
KFT Ipol           BrNumber
>calculations
BrNumber=(EP1-C01)*C02*C03*C04/C00;0;
C00=                6.00
C01=                0.0
C02=                0.5
C03=                7.99
C04=                100
-----
```

### Remarks

- Bromine number is the number of g of bromine consumed per 100 g of sample
- **Solvent:**  
714 mL acetic acid  
134 mL CCl<sub>4</sub>  
134 mL CH<sub>3</sub>OH  
18 mL w(H<sub>2</sub>SO<sub>4</sub>) = 0.2 (20%)
- **Titration agent:**  
Dissolve 51 g KBr and 13.92 g KBrO<sub>4</sub> each in dist. water and add up to 1 liter.
- **Standardization of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution:**  
Determination reaction:  

$$3 \text{ Br}_2 + 6 \text{ S}_2\text{O}_3^{2-} \Rightarrow 6 \text{ Br}^- + 3 \text{ S}_4\text{O}_6^{2-}$$
  
Calculation:  
 RS1 = EP1\*C01/C00      normality of titrating agent (BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup>)  
 C01 = concentration of titrating agent (S<sub>2</sub>O<sub>3</sub><sup>2-</sup>)  
 C00 = mL of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution
- **Determination reaction for bromine index:**  

$$\text{BrO}_3^- + 5 \text{ Br}^- + 6 \text{ H}^+ \Rightarrow 3 \text{ Br}_2 + 3 \text{ H}_2\text{O}$$
  

$$3 \text{ Br}_2 + 3 \text{ Br}^- \Rightarrow 3 \text{ Br}_2 + 3 \text{ Br}^-$$
- **Calculations for bromine number:**  
 BrNumber = bromine number  
 C01 = consumption of blank sample (0 mL)  
 C02 = normality of BrO<sub>3</sub><sup>-</sup>/Br<sup>-</sup> solution as calculated above  
 ( 0.05 mol/L)  
 C03 = molecular mass of Br<sub>2</sub> multiplied with 0.05 (7.99 g/mol)  
 C04 = dilution factor (100)

### Literature

- Metrohm Application Bulletin No. 177: Determination of the bromine index in petroleum products acc. to ASTM D 1491 - 60 and ASTM D 2710 - 72, resp. (and bromine number with ASTM D 1159 - 84)
- ASTM D 1159 - 84

# NaCl in Broth

## Reagents

$c(\text{AgNO}_3) = 0.1 \text{ mol/L}$ ; D0

## Sample

20 mL sample solution  
5 mL  $c(\text{HNO}_3) = 2 \text{ mol/L}$   
30 mL dist. water

## Electrodes

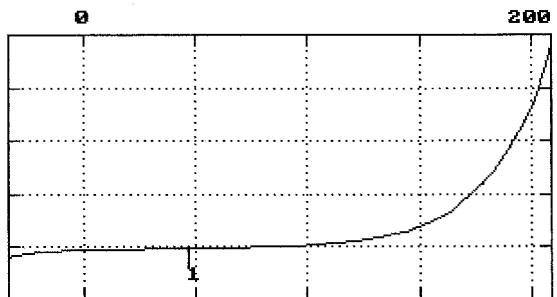
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 08:44    3
DET U              NaCl
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal.drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause             0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  stop U            OFF mV
  stop EP           1
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  EPC               5
  EP recognition:   all
  fix EP1 at U     OFF mV
  pK/HNP:           OFF
>preselections
  req.ident:        OFF
  req.smpl size:    all
  activate pulse:   OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 08:41    3
U(init)            208 mV DET U      NaCl
smpl size          0.1000 g id#1     Chicken
id#2               Broth
EP1                8.070 ml          46 mV
NaCl               47.16 %
stop #EP reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 08:41    3
start V            0.000 ml DET U      NaCl
2.0 ml/div         dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 08:44    3
DET U              NaCl
>calculations
NaCl=EP1*C01*C02*C03/C00;2;%
C00=                0.1000
C01=                58.44
C02=                0.1
C03=                0.1
-----
```

**Remarks**

- **Calculations:**  
NaCl = content of NaCl in broth in %  
C00 = sample size (10 g / 100 = 0.1 g)  
C01 = molecular mass of NaCl (58.44 g/mol)  
C02 = concentration of titrating agent (0.1 mol/L)  
C03 = factor for conversion mL  $\Rightarrow$  L and for % ( $0.001 \cdot 100 = 0.1$ )
- **Sample preparation:**  
Dissolve 10 g (1 cube) conc. broth in 800 mL boiling dist. water. Rinse this solution in a 2000 mL measuring flask. Allow to cool down and fill up to the mark. Filter with a folded filter. Take 20 mL aliquots.

---

**Literature**

- Metrohm Application Bulletin No. 130: Chloride titrations with potentiometric end-point indication.

# Formaldehyde Number in Fruit Juices

## Reagents

c(NaOH) = 0.1 mol/L; D0  
 w(HCHO) = 0.35 (35%), adjusted to pH = 8.5 with NaOH; D1

## Sample

25 mL sample (orange juice)

## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:28  4
TIP FormolNo
parameters
>sequence
  1.method:      FormoPre
  2.method:      FormoDos
  3.pause        60 s
  4.method:      FormoDet
>statistics
status:          OFF
>preselections
req.ident:       OFF
req.smpl size:   OFF
meas.mode:       OFF
temperature      25.0 °C
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:22  4
TIP FormolNo
C70                5.522
FormolNo           22.1
TIP terminated
=====
```

```
'cr
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:23  4
meas.input:        1      CAL
cal.date           95-09-21
                    pH      U/mV
buffer 1           7.00     -5
buffer 2           9.00     -122
cal.temp           21.7 °C
slope(rel)         0.999     pH(as) 6.91
-----
```

```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:28  4
TIP FormolNo
>calculations
FormolNo=C70*C01;1;
C01=                4
C70=                5.522
-----
```

**Submethods**

Preparation

Addition of Formaldehyde

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:29  4
SET pH              FormoPre
parameters
>SET1
  EP at pH          8.50
  dynamics          1.5
  max.rate          10.0 ml/min
  min.rate          25.0 µl/min
  stop crit:        drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:   +
  pause 1           0 s
  start V:          OFF
  pause            0 s
  extr.time         0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:29  4
DOS                 FormoDos
parameters
>dosing parameters
  dispensing type:  volume
  volume            15 ml
  disp.crit:        rate
  rate              max. ml/min
  pause            0 s
  time interval     10 s
  dos.element:      external D1
  temperature       25.0 °C
>stop conditions
  stop V:           OFF
  filling rate      max. ml/min
>statistics
  status:           OFF
>monitoring
  meas.mode:        OFF
  temperature:      OFF
  assign output:    none
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

## Submethods

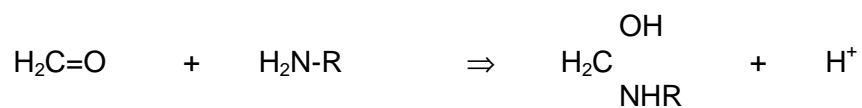
### Determination

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:30   4
SET pH              FormoDet
parameters
>SET1
  EP at pH          8.50
  dynamics          1.5
  max.rate          10.0 ml/min
  min.rate          25.0 µl/min
  stop crit:        drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:   +
  pause 1           0 s
  start V:          OFF
  pause             0 s
  extr.time         0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'de
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 15:30
SET pH              FormoDet
def
>formula
>silco calculations
  match id:         OFF
>common variables
>report
>mean
  MN1=RS1
>temporary variables
  C70=EP1
-----
```

### Remarks

- **Determination reaction:**



Amino groups of amino acid react according to the above reaction.

- **Calculations:**  
FormolNo = formol number as mL NaOH 0.1 mol/L for 100 mL sample solution  
C01 = factor for 100 mL sample solution (4)
- Calibrate the electrode for the SET titration.

### Literature

- Metrohm Application Bulletin No. 180: Automatic determination of the formol number in fruit and vegetable juices

# Calcium in Dairy Products

## Reagents

c(EGTA) = 0.1 mol/L; D0  
 c(Cu complex) = 0.100 mol/L; D1  
 Buffer solution pH = 10; D2

## Sample

app. 10 g milk

## Electrodes

6.0502.140 Cu<sup>2+</sup> sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (KNO<sub>3</sub> sat.)

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 17:22  6
TIP                Milk-Ca
parameters
>sequence
  1.method:         Milk-Ca1
  2.pause           40 s
  3.method:         Milk-Ca2
  4.pause           10 s
  5.method:         Milk-Ca3
>statistics
status:            OFF
>preselections
req.ident:         OFF
req.smpl size:     value
meas.mode:         OFF
temperature        25.0 °C
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 17:21  6
TIP                Milk-Ca
smpl size          10.0516 g
C70                3.008
Calcium            0.120 %
TIP terminated
=====
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 17:22  6
TIP                Milk-Ca
>calculations
Calcium=C70*C01*C02*C03/C00;3;%
C00=                10.0516
C01=                 0.1
C02=                 40.08
C03=                 0.1
C70=                 3.008
-----
```

**Submethods**

Addition of Cu complex-solution

Addition of buffer

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 17:23  6
DOS
Milk-Ca1
parameters
>dosing parameters
  dispensing type:  volume
  volume           1 ml
  disp.crit:       rate
  rate             max. ml/min
  pause           0 s
  time interval    10 s
  dos.element:     external D1
  temperature      25.0 °C
>stop conditions
  stop V:          OFF
  filling rate     max. ml/min
>statistics
  status:          OFF
>monitoring
  meas.mode:       OFF
  temperature:     OFF
  assign output:   none
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  activate pulse:  OFF
-----
```

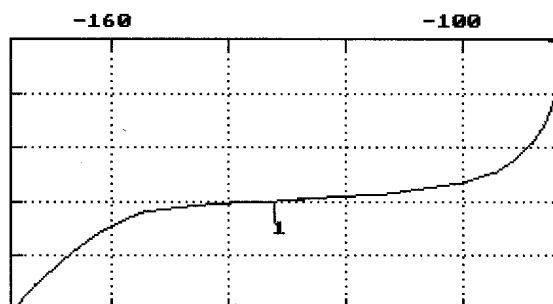
```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 17:23  6
DOS
Milk-Ca2
parameters
>dosing parameters
  dispensing type:  volume
  volume           10.000 ml
  disp.crit:       rate
  rate             max. ml/min
  pause           0 s
  time interval    10 s
  dos.element:     external D2
  temperature      25.0 °C
>stop conditions
  stop V:          OFF
  filling rate     max. ml/min
>statistics
  status:          OFF
>monitoring
  meas.mode:       OFF
  temperature:     OFF
  assign output:   none
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  activate pulse:  OFF
-----
```

**Submethods**

Determination

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 17:26   6
DET U              Milk-Ca3
parameters
>titration parameters
  meas.pt.density    1
  min.incr.          10.0 µl
  titr.rate          max. ml/min
  signal drift       OFF mV/min
  equilibr.time      5 s
  start V:           OFF
  pause              0 s
  dos.element:       internal D0
  meas.input:        1
  temperature        25.0 °C
>stop conditions
  stop V:            abs.
  stop V             5 ml
  stop U             OFF mV
  stop EP            9
  filling rate       max. ml/min
>statistics
  status:            OFF
>evaluation
  EPC                5
  EP recognition:    all
  fix EP1 at U      OFF mV
  pK/HNP:           OFF
>preselections
  req.ident:         OFF
  req.smpl size:     OFF
  activate pulse:    OFF
-----
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 17:20   6
start V            0.000 ml DET U Milk-Ca3
1.0 ml/div         dU=20.0 mV/div
```



```
'de
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 17:26
DET U              Milk-Ca3
def
>formula
  Calcium=EP1*C01*C02*C03/C00
  RS1 text          Calcium
  RS1 decimal places 3
  RS1 unit:         %
>silco calculations
  match id:         OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C70=EP1
-----
```

```
'cf
736 GP Titrimo      OP1/103  736.0010
date 95-09-21      time 17:26
DET U              Milk-Ca3
C-fmla
  C01                0.1
  C02                40.08
  C03                0.1
-----
```

## Remarks

- **Calculations:**

Calcium = content of calcium in %

C01 = concentration of titrating agent (0.1 mol/L)

C02 = molecular mass of calcium (40.08 g/mol)

C03 = factor for % (0.1)

C70 = EP1 from submethod Milk-Ca3

- **Reagents:**

$c(\text{EGTA}) = 0.100 \text{ mol/L}$ :

38.04 g EGTA - ethylene glycol-0,0'-bis-(2-aminoethyl)-N,N,N',N'-tetraacetic acid are added to a 1 liter volumetric flask, dissolved in 250 mL  $c(\text{NaOH}) = 1 \text{ mol/L}$  and the solution made up to the mark with dist. water.

Cu complex:

EGTA titrant (100 mL) is mixed with 100 mL of a solution containing 0.2 mol/L  $\text{NH}_4\text{Cl}$  and exactly 0.100 mol/L Cu(II)nitrate. Titration can be used to check that this solution contains no excess of Cu(II) or EGTA.

Buffer solution pH = 10:

54 g  $\text{NH}_4\text{Cl}$  is dissolved in ca. 400 mL dist. water in a volumetric flask, 300 mL  $w(\text{NH}_3) = 0.25$  (25%) added and the solution made up to 1 liter with dist. water.

- The method „Milk-Ca3“ can also be used as work-alone method (without TIP). Add the Cu complex-solution and the buffer solution manually.

---

## Literature

- Metrohm Application Bulletin No. 235: Potentiometric titration of Ca (Mg) in milk products

# Peroxide Number

## Reagents

$c(\text{Na}_2\text{S}_2\text{O}_3) = 0.01 \text{ mol/L}$ ; D0  
Prepared daily from  $c = 0.1 \text{ mol/L}$

## Sample

app. 5 g sunflower oil  
50 mL glacial acetic acid / chloroform (3 : 2)  
1 mL KI saturate  
100 mL dist. water

## Electrodes

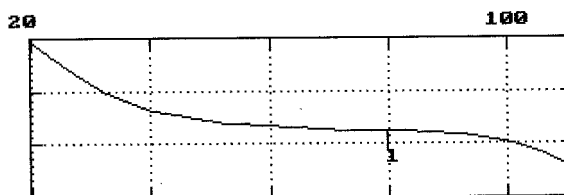
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 16:30   18
DET U              Perox.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            33.33 ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               99.99 ml
  stop U               OFF mV
  stop EP              1
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U         OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       value
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 16:27   18
U(init)             20 mV DET U Perox.No
smpl size           5.0188 g id#1  Suprema
id#2                 Oil
EP1                  0.8874 ml      80 mV
Perox.No             1.77 meq/kg
stop #EP reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 16:28   18
start V             0.0000 ml DET U Perox.No
0.5 ml/div          dU=20.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 16:30   18
DET U              Perox.No
>calculations
Perox.No=(EP1-C01)*C02/C00;2;meq/kg
C00=                5.0188
C01=                 0.0
C02=                 10
-----
```

**Remarks**

- **Calculations:**  
Perox.No = Peroxide number in meq.O<sub>2</sub>/kg  
C01 = consumption of blank sample (0 mL)  
C02 = factor (10)
- **Sample preparation:**  
Weigh out accurately 5 g sample in an Erlenmeyer flask and add 50 mL of the glacial acetic acid / chloroform mixture. Then add 1 mL of the KI solution and shake during 5 s. Now allow the mixture to stand for about 1 min. in a dark place. After this, rinse the contents of the Erlenmeyer flask out into a beaker with 100 mL dist. water and immediately back-titrate the iodine thus liberated with sodium thiosulphate. A blank control sample should be prepared and treated in the same way. Enter the blank value as C01.
- The sample must be stirred well during the titration, in order to obtain a good emulsion.

---

**Literature**

- Metrohm Application Bulletin No. 141: Analysis of edible oils and fats.

# Saponification Number

## Reagents

c(HCl) = 1 mol/L; D0

## Sample

app. 2 g sunflower oil  
 25 mL c(KOH) = 0.5 mol/L in ethanol  
 app. 10-20 mL dist. water

## Electrodes

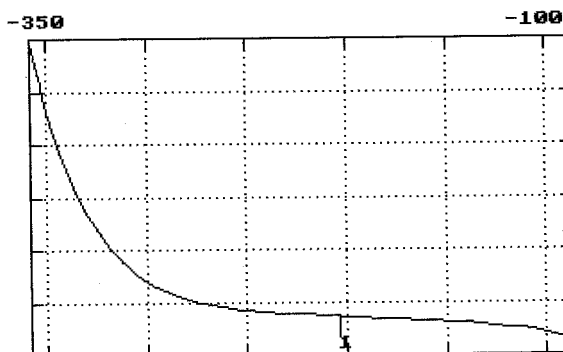
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 10:04    2
DET U              Sapon.No
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift     50 mV/min
  equilibr.time    26 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         99.99 ml
  stop U         OFF mV
  stop EP       1
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: value
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 09:58    2
U(init)           -357 mV DET U  Sapon.No
smpl size         2.0224 g
EP1              5.271 ml      -203 mV
Sapon.No         192.2 mg/g
stop #EP reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 09:58    2
start V          0.000 ml DET U  Sapon.No
1.0 ml/div       dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 10:04    2
DET U              Sapon.No
>calculations
Sapon.No=(C01-EP1)*C02/C00;1;mg/g
C00=                2.0224
C01=                12.199
C02=                56.1
-----
```

**Remarks**

- **Calculations:**  
Sapon.No = Saponification number in mg KOH per g of sample  
C01 = consumption of blank sample (12.199 mL)  
C02 = mg KOH / 1 mL titrating agent (56.1)
- **Sample preparation:**  
Weigh out approx. 2 g sample in a round-bottomed flask. Add 25 mL alcoholic KOH solution plus a few boiling beads and allow to boil lightly for at least 30 min. Shake from time to time. Finally rinse the content of the round-bottomed flask into a beaker with a small quantity of dist. water and back titrate the excess of potassium hydroxide with HCl. A blank control sample should be prepared and treated identically. Enter the blank value as C01.

---

**Literature**

- Metrohm Application Bulletin No. 141: Analysis of edible oils and fats.

# Acid Number

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

app. 5-10 g sunflower oil  
50 mL ethanol / diethylether (1 : 1), neutralized

## Electrodes

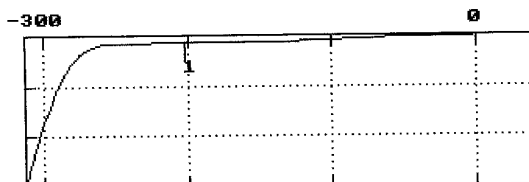
6.0133.100 pH glass electrode; input 1  
6.0726.100 Ag/AgCl double junction reference electrode  
(LiCl sat. in ethanol)

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 13:34    4
DET U              Acid.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               3 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U         OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       value
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 13:33    4
U(init)             35 mV DET U  Acid.No
smpl size           10.3223 g
EP1                  0.150 ml      -201 mV
Acid.No              0.08 mg/g
FFA                  0.04
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 13:33    4
start V             0.000 ml DET U  Acid.No
1.0 ml/div          dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 13:34    4
DET U              Acid.No
>calculations
Acid.No=EP1*C01/C00;2;mg/g
FFA=RS1*C02/C03;2;
C00=                6.9888
C01=                 5.61
C02=                 282
C03=                 561
-----
```

**Remarks**

- **Calculations:**  
Acid.No = Acid number in mg KOH per g of sample  
FFA = free fatty acid  
C01 = mg KOH / 1 mL titrating agent (5.61)  
C02 = relative molecular mass (282 for oleic acid)  
C03 = factor (561)

---

**Literature**

- Metrohm Application Bulletin No. 141: Analysis of edible oils and fats.

# Iodine Number

## Reagents

$c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol/L}; \text{D0}$

## Sample

app. 0.5 g sunflower oil  
 15 mL  $\text{CCl}_4$   
 25 mL  $c(\text{I}_2) = 0.1 \text{ mol/L}$  (according to Wijs in glacial acetic acid /  $\text{CCl}_4$ ,  
 e.g. Merck No. 9163)  
 10 mL  $w(\text{Hg acetate}) = 0.025$  (2.5%) in glacial acetic acid  
 15 mL  $w(\text{KI}) = 0.10$  (10%)  
 30-50 mL dist. water

## Electrodes

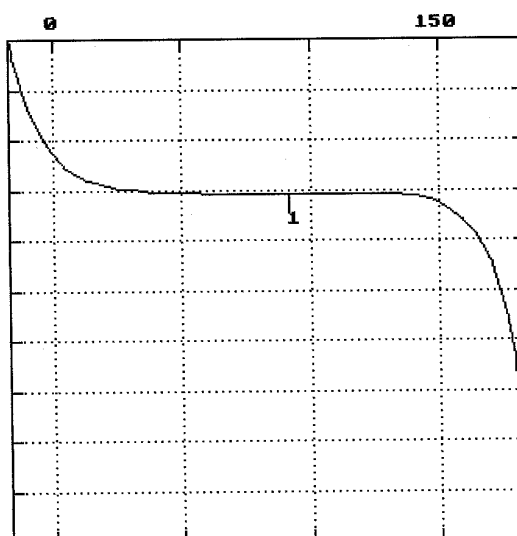
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 17:54   11
DET U              Iod.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         20.0 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V              20 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U         OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       value
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 17:42   11
U(init)             -16 mV DET U   Iod.No
smpl size            0.5204 g
EP1                  6.149 ml      92 mV
Iod.No               104.61 g/100g
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 17:42   11
start V              0.000 ml DET U   Iod.No
2.0 ml/div           dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 17:54   11
DET U              Iod.No
>calculations
Iod.No=(C01-EP1)*C02/C00;2;g/100g
C00=                1.0
C01=                 0
C02=                 1.269
-----
```

**Remarks**

- **Calculations:**  
Iod.No = Iodine number in g iodine / 100 g sample  
C01 = consumption of blank sample (0 mL)  
C02 = molecular mass of I \* concentration titrating agent \* 100g  
sample / factor mL -> L (126.9 \* 0.1 \* 100/1000 = 1.269)
- **Sample preparation:**  
According to the expected iodine number, weigh out 0.10 ... 1.00 g of the sample in an Erlenmeyer flask and add 15 mL CCl<sub>4</sub>. Add 25 mL iodine monochloride solution and add 10 mL mercuric acetate solution, mix and allow to stand for 5 min. in a dark place. Then add 15 mL KI solution, rinse into a beaker with dist. water and back-titrate the excess iodine with sodium thiosulphate. A blank control sample should be prepared and treated in the same way. Enter the blank value as C01.
- You may add a solution of w(Mg-acetate) = 0.03 (3%) instead of the Hg-acetate solution.
- The sample must be stirred well during the titration, in order to obtain a good emulsion.

---

**Literature**

- Metrohm Application Bulletin No. 141: Analysis of edible oils and fats.

---

# Vitamin C

---

**Reagents**

$c(\text{DPIP}) = 0.001 \text{ mol/L}$ ; D0  
2,6-Dichlorophenol indophenol

---

**Sample**

5 mL sample solution  
Dissolve 1 effervescent vitamin C tablet in 1 L dist. water  
15 mL oxalic acid solution 1 g/L  
1 mL w(sodium acetate) = 0.10 (10%)  
10 mL dist. water

---

**Electrodes**

6.0309.100 double Pt sheet electrode; input Pol  
polarized  $I_{\text{pol}} = 1 \mu\text{A}$   
Voltametric indication

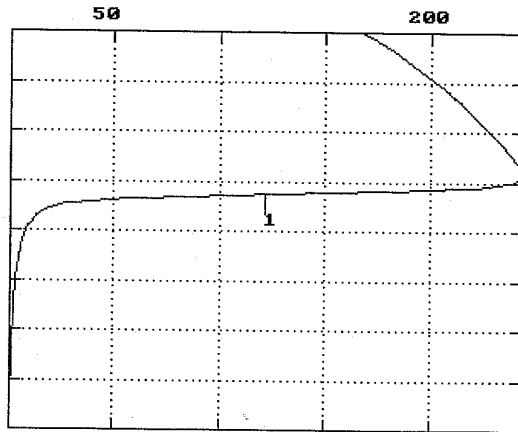
**Method documentation**

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:32  2
MET Ipol           Vit.C
parameters
>titration parameters
V step             0.10 ml
titr.rate          max. ml/min
signal drift       OFF mV/min
equilibr.time      15 s
start V:           OFF
pause              30 s
dos.element:       internal D0
I(pol)             1 µA
electrode test:    OFF
temperature        25.0 °C
>stop conditions
stop V:            abs.
stop V             20 ml
stop U             OFF mV
stop EP            9
filling rate       max. ml/min
>statistics
status:            OFF
>evaluation
EPC                30 mV
EP recognition:    all
fix EP1 at U      OFF mV
>preselections
req.ident:         OFF
req.smpl size:    OFF
activate pulse:    OFF
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:32  2
MET Ipol           Vit.C
>calculations
Vit.C=EP1*C01*C02*C03;0;mg/pc
Vit.C=EP1*C01*C02/C00;4;g/l
C00=                5.0
C01=                0.088
C02=                1.00
C03=                200
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:26  2
U(init)           121 mV MET Ipol  Vit.C
smpl size         5.0 ml
EP1               15.291 ml           122 mV
Vit.C             269 mg/pc
Vit.C             0.2691 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:26  2
start V           12.000 ml MET Ipol  Vit.C
1.0 ml/div        dU=50.0 mV/div
```



### Remarks

- **Calculations:**  
 Vit.C = content of Vitamin C in mg/pc (tablet)  
 Vit.C = content of Vitamin C in g/L  
 C01 = mg Vitamin C / 1 mL titrating agent (0.088)  
 C02 = factor of titrating agent (1.00)  
 C03 = dilution factor (200)
  
- **Titration agent:**  
 Dissolve 295 mg 2,6-Dichlorophenol indophenol with vigorous agitation in 1 L dist. water, then filter and mix with 100 mg sodium bicarbonate.  
 This solution can be stored in the refrigerator for about 1 month, the factor should be checked daily with standard ascorbic acid. (As a titrating agent, the more readily soluble sodium salt may be used instead.)
  
- **Standard solution:**  
 $r(\text{Vitamin C}) = 500 \text{ mg/L}$   
 Dissolve 50 mg ascorbic acid in oxalic acid solution (1 g/L) and make up to 100 mL. This solution should be freshly prepared daily.
  
- **Sample preparation:**  
 Place dist. water, oxalic acid solution and sodium acetate buffer in the titration vessel and deaerate by passing a stream of nitrogen for 3...5 min. Then add a quantity of sample or standard solution containing about 0.05...0.5 mg vitamin C. Now titrate under nitrogen with titrating agent.
  
- Store electrode in acidified  $\text{Na}_2\text{S}_2\text{O}_3$  solution.
- Select the appropriate formula. The other may be deleted.
- The 6.0431.100 Pt Titrode may be used together with mode MET U.

### Literature

- Metrohm Application Bulletin No. 98: Determination of ascorbic acid (Vitamin C) and its compounds.

# Na<sup>+</sup> / Cl<sup>-</sup> in Isotonic Solution

## Reagents

c(AgNO<sub>3</sub>) = 0.1 mol/L; D0

## Sample

5 mL sample solution (153 mmol/L Na<sup>+</sup> / Cl<sup>-</sup>)  
 2 mL c(HNO<sub>3</sub>) = 2 mol/L  
 30 mL dist. water

## Electrodes

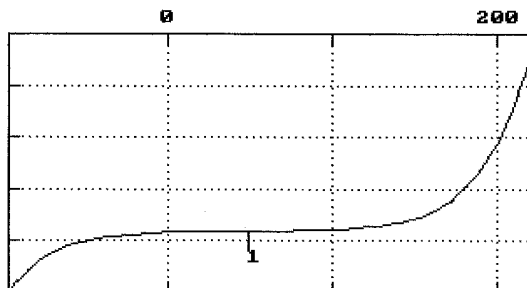
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 13:36    4
DET U              Na+Cl-
parameters
>titration parameters
meas.pt.density    4
min.incr.          10.0 µl
titr.rate          max. ml/min
signal drift       50 mV/min
equilibr.time      26 s
start V:           OFF
pause              0 s
dos.element:      internal D0
meas.input:        1
temperature        25.0 °C
>stop conditions
stop V:            abs.
stop V             10 ml
stop U             OFF mV
stop EP            9
filling rate       max. ml/min
>statistics
status:            OFF
>evaluation
EPC                5
EP recognition:    all
fix EP1 at U      OFF mV
pK/HNP:           OFF
>preselections
req.ident:         OFF
req.smpl size:    OFF
activate pulse:    OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 13:32    4
U(init)           224 mV DET U   Na+Cl-
smpl size         5 ml
EP1               7.652 ml      49 mV
Na+Cl-            153.04 mmol/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 13:33    4
start V           0.000 ml DET U   Na+Cl-
2.0 ml/div        dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-21      time 13:36    4
DET U              Na+Cl-
>calculations
Na+Cl-=EP1*C01*C02/C00;2;mmol/l
C00=                5
C01=                0.1
C02=                1000
-----
```

---

**Remarks**

- **Calculations:**  
Na+Cl<sup>-</sup> = concentration of Na<sup>+</sup> / Cl<sup>-</sup> in isotonic solution in mmol/L  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = factor for conversion mol/L ⇒ mmol/L (1000)

---

**Literature**

- Metrohm Application Bulletin No. 130: Chloride titrations with potentiometric end-point indication.

# Antacid

## Reagents

$c(\text{HCl}) = 1.0 \text{ mol/L}$ ; D0

## Sample

25 mL dist. water  
 1 drop  $c(\text{NaOH}) = 0.1 \text{ mol/L}$   
 0.268 g Antacid Trigastril (or 0.2-0.4 g)

## Electrodes

6.0233.100 combined pH glass electrode; input 1  
 6.1110.100 T sensor

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 12:59  5
TIP                Antacid
parameters
>sequence
  1.method:        AntacPre
  2.info           Add smpl & start
  3.method:        AntacDet
>statistics
  status:          OFF
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  meas.mode:       T
  temperature      25.0 °C
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 12:19  5
TIP                Antacid
smpl size          0.268 g   id#1  Trigast.
C70                3.218
acid cap           12.007 mmol/g
TIP terminated
=====
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 12:59  5
TIP                Antacid
>calculations
acid cap=C70*C01/C00;3;mmol/g
C00=                0.268
C01=                1
C70=                3.218
-----
```

## Submethods

### Preparation

```

'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-28      time 13:00   5
SET pH              AntacPre
parameters
>SET1
  EP at pH          3.00
  dynamics          3
  max.rate          1 ml/min
  min.rate          1 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:  -
  pause 1          0 s
  start V:         OFF
  pause            0 s
  extr.time        0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      37.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:         OFF
>preselections
  conditioning:   OFF
  display drift:  OFF
  req.ident:      OFF
  req.smpl size:  OFF
  activate pulse: OFF
  -----
    
```

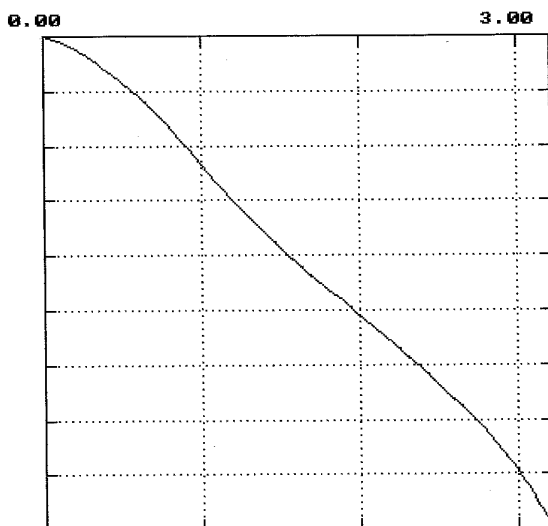
**Submethods**

**Determination**

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 13:02  5
STAT pH            AntacDet
parameters
>control parameters
  EP at pH          3
  dynamics          1.2
  max.rate          15 ml/min
  min.rate          10 µl/min
>titration parameters
  start V:          OFF
  pause             0 s
  start time        0 s
  start pH          OFF
  start rate        OFF ml/min
  time interval     30 s
  titr.direction:  -
  dos.element:     internal D0
  meas.input:       1
  temperature       37 °C
>stop conditions
  stop time:        abs.
  stop time         1800 s
  stop V:           abs.
  stop V            99.99 ml
  stop rate         OFF ml/min
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  low lim.1        OFF s
  fix V1           450 s
  fix V2           900 s
  fix V3           1620 s
  fix V4           1800 s
  fix V5           OFF s
  fix time 1       0.25 V(tot)
  fix time 2       0.5 V(tot)
  fix time 3       0.9 V(tot)
  fix time 4       1 V(tot)
  fix time 5       OFF V(tot)
>monitoring
  meas.val:        ON
  low lim. pH      -20.00
  up lim. pH       20.00
  action:          none
  rate:            OFF
  temperature:    ON
  low lim.         36 °C
  up lim.         38 °C
  action:          none
  assign output:  none
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  display rate:    OFF
  activate pulse:  OFF
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 12:14  5
pHc(init)         3.48  STAT pH AntacDet
smpl size         0.268 g  id#1 Trigast.
C80 m.rate        0.1070 +/- 0.00084 ml/min
fix V1            0.979 ml  450 s
fix V2            1.759 ml  900 s
fix V3            3.042 ml  1620 s
fix V4            3.218 ml  1800 s
fix time 1        332.9 s  0.25 V(tot)
fix time 2        825.0 s  0.5 V(tot)
fix time 3        1504.5 s 0.9 V(tot)
fix time 4        1800.0 s 1 V(tot)
stop time reached
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 12:15  5
start time        0 s  STAT pH AntacDet
200.0 s/div       dV=1.0 ml/div
```



```
'de
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 13:03
STAT pH            AntacDet
def
>formula
>silco calculations
  match id:        OFF
>common variables
>report
  report:full;curve;
>mean
  MN1=RS1
>temporary variables
  C70=C41
```

**Remarks**

- Method to determine the efficiency of tablets against an excess of acid in the stomach. Use a TIP method for the determination: The pH is adjusted to 3 with a pretitration (AntacPre), then the sample is added and the pH kept at 3 during 30 minutes (AntacDet). The determination is carried out in a thermostated titration vessel (e.g. 6.1418.220) at 37 °C.
- You may have to adjust the control parameters of the submethod "AntacDet" according to your sample. The given parameters are optimized for products which release their base slowly (rate app. 100 µL/min).
- **Calculations:**  
acid cap = acid capacity in mmol/g  
C01 = concentration of titrating agent (1 mol/L)
- **Sample preparation:**  
Pulverize the tablet and weigh it exactly. Rinse the powder with 20 mL of pretitrated solution (pH=3) into the titration vessel during the request "Add smpl & start" and titrate immediately.

---

**Literature**

- N.J. Kerkhoff et al., Journal of Pharmaceutical Sciences, 66, 1528-1535 (1977)

# Trypsin

## Reagents

c(NaOH) = 0.1 mol/L in a 1 mL exchange unit; D0

## Sample

10 mL borate buffer solution pH = 8.0  
 1 mL substrate solution  
 0.05 mL enzyme solution

## Electrodes

6.0234.100 combined micro pH glass electrode; input 1  
 6.1110.100 T sensor

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-25      time 13:36  7
TIP                Trypsin
parameters
>sequence
  1.method:        TrypsPre
  2.info           Add smpl & start
  3.method:        TrypsDet
>statistics
  status:          OFF
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  meas.mode:       T
  temperature      25.0 °C
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-25      time 13:28  7
TIP                Trypsin
smpl size          0.0507 mg
C70                0.1920
C71                0.0238
FIP                47.3 U/mg
FIP(reg)           46.9 U/mg
TIP terminated
=====
```

```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-25      time 13:37  7
TIP                Trypsin
>calculations
FIP=C70*C01/C02/C00;1;U/mg
FIP(reg)=C71*C01/C00;1;U/mg
C00=                0.0507
C01=                100
C02=                8
C70=                0.1920
C71=                0.0238
-----
```

---

## Submethods

### Preparation

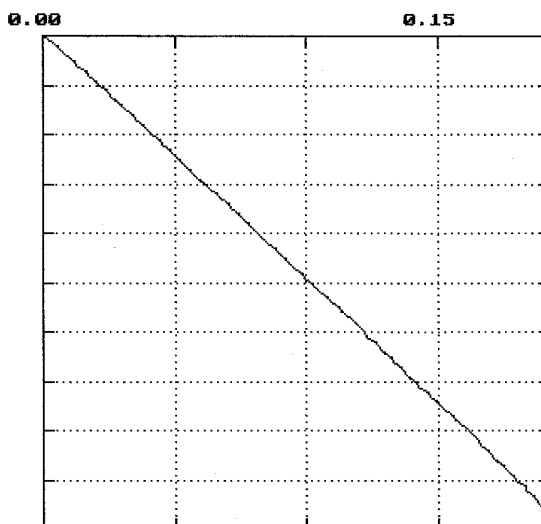
```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-25      time 13:43  7
SET pH              TrypsPre
parameters
>SET1
  EP at pH          8.00
  dynamics          1
  max.rate          1 ml/min
  min.rate          1 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:  +
  pause 1          0 s
  start V:         OFF
  pause            0 s
  extr.time        0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:         OFF
>preselections
  conditioning:   OFF
  req.ident:      OFF
  req.smpl size:  OFF
  activate pulse: OFF
-----
```

**Submethods**

Determination

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-25      time 13:41  7
STAT pH            TrypsDet
parameters
>control parameters
  EP at pH          8
  dynamics          1.00
  max.rate          1 ml/min
  min.rate          3 µl/min
>titration parameters
  start V:          OFF
  pause             0 s
  start time        0 s
  start pH          OFF
  start rate        OFF ml/min
  time interval     5 s
  titr.direction:  +
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop time:        abs.
  stop time         480 s
  stop V:           abs.
  stop V            99.99 ml
  stop rate         OFF ml/min
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  low lim.1         OFF s
  fix V1            OFF s
  fix time 1        OFF V(tot)
>monitoring
  meas.val:         ON
  low lim. pH       7.9
  up lim. pH        8.1
  action:           none
  rate:             OFF
  temperature:      ON
  low lim.          24.9 °C
  up lim.           25.1 °C
  action:           none
  assign output:    none
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  display rate:     OFF
  activate pulse:   OFF
-----
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-25      time 13:23  7
start time         0 s  STAT pH TrypsDet
50.0 s/div         dV=0.05 ml/div
```



```
'de
736 GP Titrino      OP1/103  736.0010
date 95-09-25      time 13:42
STAT pH            TrypsDet
def
>formula
>silo calculations
  match id:         OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C70=C41
  C71=C80
-----
```

## Remarks

- Trypsin is an enzyme which is determined using a TIP method: The pH is adjusted to 8.00 with a pretitration (TrypsPre), then the sample is added and the pH kept at 8 during 8 minutes (TrypsDet). The determination is carried out in a thermostated titration vessel (e.g. 6.9914.023 with 6.2036.000 holding ring) at 25 °C.

- **Calculations:**

FIP = Calculation using the final volume, according to FIP. Result in FIP units/mg.

FIP(reg) = Calculation using the mean rate. Result in FIP units/mg. \*)

C01 = (factor for mL $\Rightarrow$  $\mu$ L) \* (concentration of reagent)(100)

C02 = Determination time = 8 minutes

C70 = final volume from TrypsDet

C71 = mean rate from TrypsDet

\*) The calculation using the mean rate is fundamentally different from the FIP method (final volume / time). The mean rate is calculated by linear regression over all measuring points of the list. If you wish to use only the linear part of the curve for the regression, use a start time of app. 30 s in the method "TrypsDet".

If one of the formulas is not used, you may delete it.

- **Sample preparation:**

Dissolve the enzyme in  $c(\text{HCl}) = 0.001 \text{ mol/L}$ . The solution should not contain more than 50 FIP Units per mL.

- **Substrate solution:**

Dissolve 171.3 mg N-Benzoyl-L-arginine ethylester hydrochloride in dist. water and fill up to 25 mL.

- **Borate buffer solution pH = 8.00:**

Dissolve 286.0 mg Disodiumtetraborate-decahydrate and 1.47 g Calciumchloride-dihydrate in 400 mL dist. water, set the pH with  $c(\text{HCl}) = 1 \text{ mol/L}$  to 8.00 and fill up with dist. water to 500 mL.

- **Comments to parameters:**

*TrypsDet*

Monitoring of pH and temperature is ON: pH should be kept in the range of  $\pm 0.1$ , the temperature in  $\pm 0.1^\circ\text{C}$ .

*Trypsin*

T may be measured with <meas/hold>.

---

## Literature

- B. Stellmach, Bestimmungsmethoden Enzyme, Steinkopff Verlag Darmstadt, 1988, p. 263

# Lipase

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

29.5 mL substrate emulsion  
0.5 mL enzyme solution

## Electrodes

6.0233.100 combined pH glass electrode; input 1  
6.1110.100 T sensor

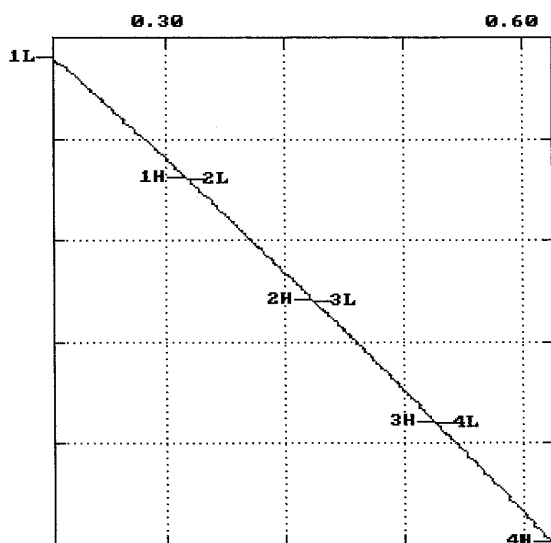
## Method documentation

### Results

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:01  14
pHc(init)          9.20    STAT pH  LipDeter
smpl size          0.3475 mg  id#1  Sample 2
C81 rate           0.1125 +/- 0.00033 ml/min
C82 rate           0.1064 +/- 0.00036 ml/min
C83 rate           0.1020 +/- 0.00029 ml/min
C84 rate           0.0980 +/- 0.00027 ml/min
C80 m.rate         0.1047 +/- 0.00017 ml/min
stop time reached
=====
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:06  14
                    TIP      LipSmpSC
smpl size          0.3475 mg  id#1  Sample 2
C70                0.1047
FIP                35.0 U/mg
                    mean( 2) +/-s      s/%
FIP                35.1      0.14 U/mg  0.40
TIP terminated
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:01  14
start time         60 s  STAT pH  LipDeter
50.0 s/div         dV=0.1 ml/div
```



=====

**Submethods**

Preparation

Determination

```
'pa
736 GP Titrimo          OP1/103  736.0010
date 95-10-03          time 08:32  0
SET pH                  LipPre
parameters
>SET1
  EP at pH              9.20
  dynamics              1
  max.rate              5 ml/min
  min.rate              5 µl/min
  stop crit:           drift
  stop drift            20 µl/min
>SET2
  EP at pH              OFF
>titration parameters
  titr.direction:      +
  pause 1              0 s
  start V:             OFF
  pause                0 s
  extr.time            0 s
  dos.element:         internal D0
  meas.input:          1
  temperature           37.0 °C
>stop conditions
  stop V:              abs.
  stop V               99.99 ml
  filling rate         max. ml/min
>statistics
  status:              OFF
>preselections
  conditioning:        OFF
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'pa
736 GP Titrimo          OP1/103  736.0010
date 95-10-03          time 08:33  0
STAT pH                LipDeter
parameters
>control parameters
  EP at pH              9.00
  dynamics              1
  max.rate              5 ml/min
  min.rate              10 µl/min
>titration parameters
  start V:             OFF
  pause                0 s
  start time           60 s
  start pH             OFF
  start rate           OFF ml/min
  time interval        1 s
  titr.direction:      +
  dos.element:         internal D0
  meas.input:          1
  temperature           37.0 °C
>stop conditions
  stop time:           abs.
  stop time            300 s
  stop V:              abs.
  stop V               99.99 ml
  stop rate            OFF ml/min
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  low lim.1            60 s
  up lim.1             119 s
  low lim.2            120 s
  up lim.2             179 s
  low lim.3            180 s
  up lim.3             239 s
  low lim.4            240 s
  up lim.4             299 s
  low lim.5            OFF s
  fix V1               OFF s
  fix time 1           OFF V(tot)
>monitoring
  meas.val:            OFF
  rate:                ON
  low lim.             0.08 ml/min
  up lim.              0.16 ml/min
  action:              none
  temperature:         ON
  low lim.             36.9 °C
  up lim.              37.1 °C
  action:              none
  assign output:       none
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  display rate:        OFF
  activate pulse:      OFF
-----
```

```
'de
736 GP Titrimo          OP1/103  736.0010
date 95-10-03          time 08:34
STAT pH                LipDeter
def
>formula
>silo calculations
  match id:           OFF
>common variables
>report
  report:full;curve;
>mean
  MN1=RS1
>temporary variables
  C70=C80
-----
```

**Determination of the Standard**

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:35  0
TIP                LipStd
parameters
>sequence
  1.method:         LipPre
  2.info           Add smpl & start
  3.method:         LipDeter
>statistics
  status:          ON
  mean             n= 3
  res.tab:         original
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  meas.mode:       T
  temperature      25.0 °C
-----
```

```
'de
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:36
TIP                LipStd
def
>sequence
  1.method:         LipPre
  2.info           Add smpl & start
  3.method:         LipDeter
>formula
  Factor=C00*C01/C70
  RS1 text         Factor
  RS1 decimal places 2
  RS1 unit:
  FIP=C70*C02/C00
  RS2 text         FIP
  RS2 decimal places 1
  RS2 unit:         U/mg
>silco calculations
  C24=RS1
  C25=RS2
  match id:        OFF
>common variables
  C30=MN1
  C31=C26
>report
  report:full;
>mean
  MN1=RS1
>temporary variables
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:37  0
TIP                LipStd
>calculations
Factor=C00*C01/C70;2;
FIP=C70*C02/C00;1;U/mg
C00=               1.0
C01=               36.2
C02=               100
C70=               not valid
-----
```

### Determination of Samples

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:41  0
TIP                LipSmpl
parameters
>sequence
  1.method:         LipPre
  2.info           Add smpl & start
  3.method:         LipDeter
>statistics
  status:          ON
  mean             n= 3
  res.tab:         original
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  meas.mode:       T
  temperature      25.0 °C
-----
```

```
'de
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:41
TIP                LipSmpl
def
>sequence
  1.method:         LipPre
  2.info           Add smpl & start
  3.method:         LipDeter
>formula
  FIP=C70*C30/C00
  RS1 text         FIP
  RS1 decimal places 1
  RS1 unit:        U/mg
>silco calculations
  match id:        OFF
>common variables
>report
  report:full;
>mean
  MN1=RS1
>temporary variables
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:42  0
TIP                LipSmpl
>calculations
FIP=C70*C30/C00;1;U/mg
C00=                1.0
C30=                115.46
C70=                not valid
-----
```

**Determination of Samples Using Silo Calculations**

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:28  0
TIP                LipSmpSC
parameters
>sequence
  1.method:        LipPre
  2.info           Add smpl & start
  3.method:        LipDeter
>statistics
  status:          ON
  mean             n=      3
  res.tab:         original
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  meas.mode:       T
  temperature      25.0 °C
-----
```

```
'de
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:31
TIP                LipSmpSC
def
>sequence
  1.method:        LipPre
  2.info           Add smpl & start
  3.method:        LipDeter
>formula
  FIP=C70*C31/C00
  RS1 text         FIP
  RS1 decimal places 1
  RS1 unit:        U/mg
>silo calculations
  C25=RS1
  match id:        OFF
>common variables
>report
  report:full;
>mean
  MN1=RS1
>temporary variables
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 08:29  0
TIP                LipSmpSC
>calculations
FIP=C70*C31/C00;1;U/mg
C00= 1.0
C31= 116.29
C70= 0.1047
-----
```

```
'si
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:07
>silo
  cycle lines:    OFF
  save lines:     ON
sl  method id#1/C21 id#2/C22 id#3/C23  C00      C24      C25
+ 1 LipStd FIP Std. 0.3486mg 116.20 31.2U/mg
+ 2 LipStd FIP Std. 0.3486mg 115.35 31.4U/mg
+ 3 LipStd FIP Std. 0.3486mg 118.16 30.6U/mg
+ 4 LipSmpSC Sample 1 0.3470mg NV 34.8U/mg
+ 5 LipSmpSC Sample 1 0.3470mg NV 35.1U/mg
+ 6 LipStd FIP Std. 0.3486mg 115.46 31.4U/mg
+ 7 LipSmpSC Sample 2 0.3475mg NV 35.2U/mg
/ 8 LipSmpSC Sample 2 0.3475mg NV 35.0U/mg
-----
```

```
'ss
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:07  14
method id#1/C21 id#2/C22 id#3/C23
LipSmpSC Sample 2 * * * FIP mean +/-s n
35.1 U/mg 0.14 2
-----
```

```
'sf
736 GP Titrino      OP1/103  736.0010
date 95-10-02      time 18:07  14
method id#1/C21 id#2/C22 id#3/C23
LipStd * * * Factor mean +/-s n
116.29 1.301 4
FIP 31.1 U/mg 0.38 4
LipSmpSC Sample 1 * * * FIP 34.9 U/mg 0.21 2
LipSmpSC Sample 2 * * * FIP 35.1 U/mg 0.14 2
-----
```

## Remarks

- Pancreas lipase is determined using a TIP method: The pH is adjusted to 9.20 with a pretitration (LipPre), then the sample is added and the pH kept at 9 during 5 minutes (LipDeter). The determination is carried out in a thermostated titration vessel (e.g. 6.1418.220) at 37 °C.
- **Calculations:**
  - LipStd*  
Factor = Calculation factor  
FIP = Activity of the reference standard in FIP units/mg.  
C01 = Declared activity of FIP standard (36.2)  
C02 = 1000\*concentration of titrating agent (100)  
C70 = mean rate from LipDeter
  - LipSmpl<sup>1)</sup>*  
FIP = Activity of the sample in FIP units/mg.  
C30 = Calculation factor from LipStd  
C70 = mean rate from LipDeter
  - LipSmpSC<sup>2)</sup>*  
FIP = Activity of the sample in FIP units/mg.  
C31 = Calculation factor from silo calculation of LipStd  
C70 = mean rate from LipDeter

1): Delete the following assignments in method „LipStd“:  
 . in key <def>, >silo calculations, C24=RS1 and C25=RS2  
 . in key <def>, >common variables, C31=C26

2): Delete the following assignment in method „LipStd“:  
 . in key <def>, >common variables, C30=MN1
- **Solution of Arabic gum:**  
Dissolve 100 g Arabic gum in 1000 mL dist. water. Centrifuge until the solution is clear (app. 10 minutes with 4000 r/min). This solution can be kept several weeks if frozen in portions.
- **Substrate standard solution:**  
Add 165 mL Arabic gum solution, 20 mL olive oil (BP 73) and 15 mL dist. water and cool it in ice to 5 °C. Homogenize with an electric mixer during 30 minutes. The temperature of the solution should never rise above 30 °C. This solution can be kept up to 14 days in a refrigerator. Always homogenize the emulsion before use.
- **Substrate emulsion:**  
Stir 100 mL substrate standard solution, 80 mL TRIS buffer solution, 20 mL sodium taurocholate and 95 mL dist. water. This solution must be prepared daily.
- **TRIS buffer solution:**  
Dissolve 60.6 mg Tris(hydroxymethyl)-amino methane (C<sub>4</sub>H<sub>11</sub>NO<sub>3</sub>) and 234.0 mg sodium chloride in dist. water and fill up to 100 mL. This solution can be kept up to 3 days in a refrigerator.

- **Sodium taurocholate solution:**  
Dissolve 4.0 g sodium taurocholate (F.I.P. controlled) in dist. water and fill up to 50 mL.
- **Enzyme solvent:**  
Dissolve 10.0 g sodium chloride, 6.06 g Tris(hydroxymethyl)-amino methane and maleic acid anhydride (C<sub>4</sub>H<sub>2</sub>O<sub>3</sub>) in 900 mL dist. water. Adjust pH to 7.0 with 4N NaOH (app. 13 mL) and fill up to 1000 mL. This solution can be kept up to 3 days in a refrigerator.
- **Sample preparation:**  
Dissolve the enzyme in the enzyme solvent. The solution should contain between 8 and 16 FIP Units per mL.
- **Comments to parameters:**  
*LipDeter*  
Time windows for rate evaluation.  
Monitoring of rate and temperature is ON: the rate should be kept in the range of 0.08...0.16 mL/min, the temperature in  $\pm 0.1^{\circ}\text{C}$ .  
*LipStd, LipSmpl, LipSmpSC*  
T may be measured with <meas/hold>.

---

**Literature**

- B. Stellmach, Bestimmungsmethoden Enzyme, Steinkopff Verlag Darmstadt, 1988, p. 263

# 2-Aminophenol

## Reagents

c(NaNO<sub>2</sub>) = 0.2 mol/L; D0

## Sample

app. 0.3 ... 0.35 g sample (2-aminophenol)  
 10 mL w(HBr) = 0.20 (20%)  
 30 mL dist. water

## Electrodes

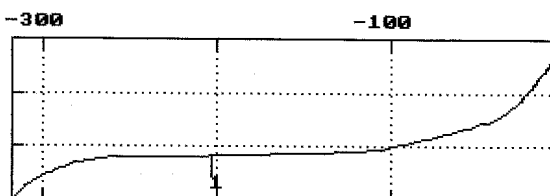
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
parameters
>titration parameters
  V step            0.10 ml
  titr.rate         max. ml/min
  signal drift      OFF mV/min
  equilibr.time     15 s
  start V:          OFF
  pause             30 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            17 ml
  stop U            OFF mV
  stop EP           9
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  EPC               30 mV
  EP recognition:   all
  fix EP1 at U     OFF mV
  pK/HNP:          OFF
>preselections
  req.ident:        OFF
  req.smpl size:    value
  activate pulse:   OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
U(init)            125 mV MET U      Diazo
smpl size          0.3188 g   id#1    09120
EP1                14.396 ml   -203 mV
Content             98.56 %
stop V reached
=====
```

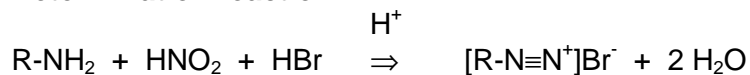
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
start V            10.000 ml MET U    Diazo
2.0 ml/div         dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
>calculations
Content=EP1*C01*C02*C03/C00;2;%
C00=                0.3188
C01=                109.13
C02=                 0.1
C03=                 0.2
-----
```

**Remarks**

- **Determination reaction:**



- **Calculations:**

Content = content of 2-aminophenol in %

C01 = molecular mass of 2-aminophenol (109.13 /mol)

C02 = factor for conversion mL to L, and for % (0.001\*100=0.1)

C03 = concentration of titrating agent (0.2 mol/L)

Enter the appropriate molecular mass for other amines (C01).

- Instead of the Pt Titrode the 6.0420.100 combined Pt electrode can be used.

**Literature**

- Metrohm Application Bulletin No. 228: Diazotisation titrations

---

# Vitamin C

---

**Reagents**

$c(\text{DPIP}) = 0.001 \text{ mol/L}$ ; D0  
2,6-Dichlorphenol indophenol

---

**Sample**

5 mL sample solution  
Dissolve 1 effervescent vitamin C tablet in 1 L dist. water  
15 mL oxalic acid solution 1 g/L  
1 mL w(sodium acetate) = 0.10 (10%)  
10 mL dist. water

---

**Electrodes**

6.0309.100 double Pt sheet electrode; input Pol  
polarized  $I_{\text{pol}} = 1 \mu\text{A}$   
Voltametric indication

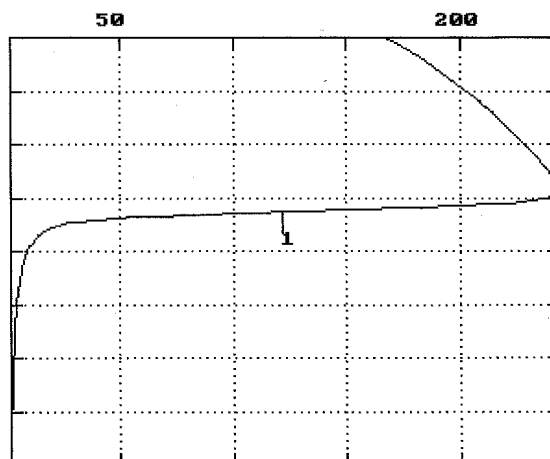
**Method documentation**

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:32  2
MET Ipol           Vit.C
parameters
>titration parameters
V step              0.10 ml
titr.rate           max. ml/min
signal drift        OFF mV/min
equilibr.time       15 s
start V:            OFF
pause               30 s
dos.element:        internal D0
I(pol)              1 µA
electrode test:     OFF
temperature         25.0 °C
>stop conditions
stop V:             abs.
stop V              20 ml
stop U              OFF mV
stop EP             9
filling rate        max. ml/min
>statistics
status:             OFF
>evaluation
EPC                 30 mV
EP recognition:     all
fix EP1 at U       OFF mV
>preselections
req.ident:          OFF
req.smpl size:      OFF
activate pulse:     OFF
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:33  2
MET Ipol           Vit.C
>calculations
Vit.C=EP1*C01*C02*C03;0;mg/pc
Vit.C=EP1*C01*C02/C00;4;g/l
C00=                5.0
C01=                0.088
C02=                1.00
C03=                200
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:26  2
U(init)            121 mV MET Ipol  Vit.C
smpl size           5.0 ml
EP1                 15.291 ml          122 mV
Vit.C               269 mg/pc
Vit.C               0.2691 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:26  2
start V            12.000 ml MET Ipol  Vit.C
1.0 ml/div         dU=50.0 mV/div
```



### Remarks

- **Calculations:**  
 Vit.C = content of Vitamin C in mg/pc (tablet)  
 Vit.C = content of Vitamin C in g/L  
 C01 = mg Vitamin C / 1 mL titrating agent (0.088)  
 C02 = factor titrating agent (1.00)  
 C03 = dilution factor (200)
- **Titration agent:**  
 Dissolve 295 mg 2,6-dichlorophenol indophenol with vigorous agitation in 1 L dist. water, then filter and mix with 100 mg sodium bicarbonate. This solution can be stored in the refrigerator for about 1 month, the factor should be checked daily with standard ascorbic acid. (As a titrating agent, the more readily soluble sodium salt may be used instead.)
- **Standard solution:**  
 $r(\text{Vitamin C}) = 500 \text{ mg/L}$   
 Dissolve 50 mg ascorbic acid in oxalic acid solution (1 g/L) and make up to 100 mL. This solution should be freshly prepared daily.
- **Sample preparation:**  
 Place dist. water, oxalic acid solution and sodium acetate buffer in the titration vessel and deaerate by passing a stream of nitrogen for 3...5 min. Then add a quantity of sample or standard solution containing about 0.05...0.5 mg vitamin C. Now titrate under nitrogen with titrating agent.
- Store electrode in acidified  $\text{Na}_2\text{S}_2\text{O}_3$  solution.
- Select the appropriate formula. The other may be deleted.
- The 6.0431.100 Pt Titrode may be used together with mode MET U.

### Literature

- Metrohm Application Bulletin No. 98: Determination of ascorbic acid (Vitamin C) and its compounds.

# EDTA/NTA in Detergents

## Reagents

$c(\text{Cu}^{2+}) = 0.01 \text{ mol/L}$ ; D0

## Sample

10 mL sample solution  
 10 mL buffer pH = 9.6 [ $c(\text{NH}_3/\text{NH}_4\text{NO}_3) = 1 \text{ mol/L}$ ]  
 2 mL  $c(\text{Na}_2\text{EDTA}) = 0.01 \text{ mol/L}$   
 30 mL dist. water

## Electrodes

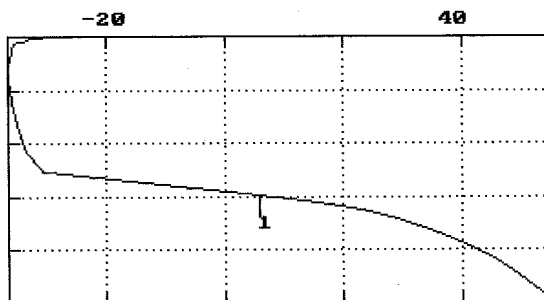
6.0502.140  $\text{Cu}^{2+}$  sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode ( $\text{KNO}_3$  sat.)

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 14:23   11
DET U              EDTA-NTA
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            345.0 ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               10 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:     all
  fix EP1 at U        OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:      all
  activate pulse:     OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 14:16   11
U(init)             -26 mV DET U EDTA-NTA
smpl size           82.63 mg id#1 Total
id#2                Futura id#3 MIFA
EP1                 3.004 ml      6 mV
NTA                 2.32 %
stop V reached
=====
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 14:16   11
start V             0.000 ml DET U EDTA-NTA
1.0 ml/div          dU=20.0 mV/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-15      time 14:24   11
DET U              EDTA-NTA
>calculations
EDTA  =(EP1-C01)*C02*C03/C00;2;%
NTA   =(EP1-C01)*C02*C04/C00;2;%
C00=   82.63
C01=    2
C02=  100
C03=  2.9225
C04=  1.9114
-----
```

**Remarks**

- **Calculations:**  
EDTA = content of EDTA in %  
NTA = content of NTA in %  
C01 = amount of EDTA/NTA added (2 mL)  
C02 = factor for % (100)  
C03 = 1 mL  $c(\text{Cu}^{2+}) = 0.01 \text{ mol/L} = 2.9225 \text{ mg EDTA}$   
C04 = 1 mL  $c(\text{Cu}^{2+}) = 0.01 \text{ mol/L} = 1.9114 \text{ mg NTA}$
- **Sample preparation:**  
Dissolve 0.5 ... 1 g detergent in 50 mL dist. water at 40 °C.  
Allow solution to cool and add up to 100 mL.
- Select the appropriate formula. The other may be deleted.
- $\text{Na}_2\text{EDTA}$  is added to get a greater break. Treat the amount of  $\text{Na}_2\text{EDTA}$  added like a blank value.

---

**Literature**

- Metrohm Application Bulletin No. 143: Potentiometric determination of nitrilotriacetic acid (NTA) and/or ethylenediaminetetracetic acid (EDTA) in detergents.

# Non-ionic Surfactants

## Reagents

c(STPB) = 0.01 mol/L; D0  
 STPB = sodium tetraphenylborate

## Sample

5 mL sample solution  
 3 mL c(BaCl<sub>2</sub>) = 0.1 mol/L  
 60 mL dist. water

## Electrodes

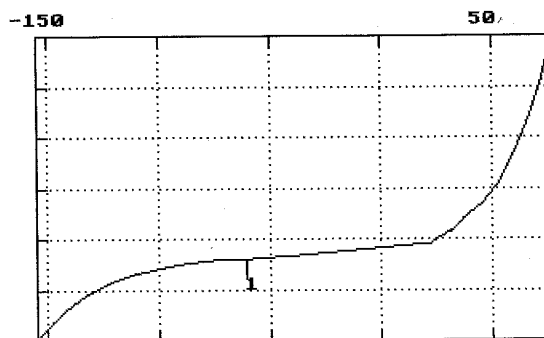
6.0507.010 NIO tenside electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode  
 (inner electrolyte c(KCl) = 3 mol/L, outer electrolyte  
 c(NaCl) = 1 mol/L)

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:30  9
DET U              Tenside
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            100 µl
  titr.rate            5 ml/min
  signal drift         5 mV/min
  equilibr.time        60 s
  start V:             OFF
  pause                60 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               12 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  10
  EP recognition:      all
  fix EP1 at U         OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:26  9
U(init)             75 mV DET U  Tenside
smpl size           0.5145 g
EP1                  8.795 ml
Nonionic             3.4189 meq/g
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:27  9
start V             0.000 ml DET U  Tenside
2.0 ml/div          dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:31  9
DET U              Tenside
>calculations
Nonionic=EP1*C01*C02/C00;4;meq/g
C00=                0.5145
C01=                 0.01
C02=                 20
-----
```

**Remarks**

- **Titration agent:**  
Dissolve 3.4223 g sodium tetrphenylborate in 300 mL dist. water. Dissolve separately 10 g polyvinyl alcohol in 300 mL dist. water and heat it slightly. Allow to cool and rinse both solutions in a 1000 mL flask. Add 10 mL buffer pH = 10 and fill up to 1 liter.
- **BaCl<sub>2</sub> solution:**  
Dissolve 21 g BaCl<sub>2</sub> or 25 g BaCl<sub>2</sub>·2H<sub>2</sub>O in dist. water, add 1 mL conc. HCl and fill up to 1 liter.
- **Buffer pH = 10:**  
Dissolve 1.24 g H<sub>3</sub>BO<sub>4</sub> in dist. water, add 10 mL c(NaOH) = 1 mol/L and fill up to 100 mL.
- **Sample preparation:**  
Dissolve app. 0.5 ... 1 g detergent (Renex 650 ICI; polyoxyethylene-(30)-nonylphenol, molecular mass ≈ 1541 g/mol) in 100 mL dist. water.
- **Calculations:**  
Nonionic = concentration of nonionic Tensides in meq/g  
C01 = concentration of titrating agent (0.01 mol/L)  
C02 = dilution factor for the sample solution, aliquot (20)

---

**Literature**

- Metrohm Application Bulletin No. 230: Titrimetric/potentiometric determination of non-ionic tensides (oxyethylates) with the NIO electrode

# Cationic Surfactants in Antiseptic Disinfectant

## Reagents

c(Na-laurylsulphate) = 0.004 mol/L; D0

## Sample

app. 0.1 g sample (Desogen)  
60 mL dist. water

## Electrodes

6.0504.150 high-sense surfactant electrode; input 1  
6.0733.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/L)

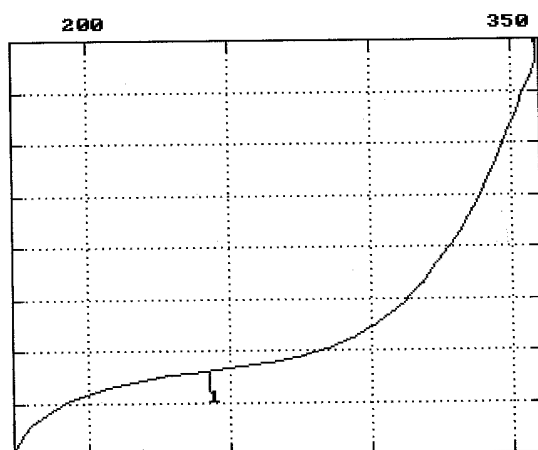
## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:00  8
DET U              Tens.Cat
parameters
>titration parameters
  meas.pt.density  3
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     OFF mV/min
  equilibr.time    10 s
  start V:         OFF
  pause           30 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V         10 ml
  stop U         OFF mV
  stop EP        OFF
  filling rate   max. ml/min
>statistics
  status:         OFF
>evaluation
  EPC            5
  EP recognition: greatest
  fix EP1 at U   OFF mV
  pK/HNP:        OFF
>preselections
  req.ident:      OFF
  req.smpl size:  OFF
  activate pulse: OFF
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 11:00  8
DET U              Tens.Cat
>calculations
Cationic=EP1*C01/C00;4;meq/g
C00=              0.1044
C01=              0.004
```

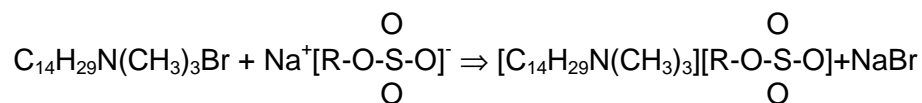
```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 10:55  8
U(init)            372 mV DET U Tens.Cat
smpl size          0.1044 g   id#1 DESOGENE
id#2               Solution   id#3 Geigy
EP1                6.391 ml   243 mV
Cationic           0.2449 meq/g
stop V reached
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 10:56  8
start V           0.000 ml DET U Tens.Cat
1.0 ml/div        dU=50.0 mV/div
```



### Remarks

- **Determination reaction:**



- **Calculations:**

Cationic = concentration of cationic Tensides in meq/g

C01 = concentration of titrating agent (0.004 mol/L)

- **Electrode preparation:**

The electrode is shaken vigorously downward (like a clinical thermometer) and is best conditioned by two to three titrations whose results are not taken into account. On completion of a titration or titration series (with a Sample Changer of up to 100 and more samples, no intermediate rinsing is required), the electrodes are rinsed briefly with methanol and then with dist. water. (If need be, carefully wipe with a soft paper towel soaked in methanol.) The electrode is stored dry.

- **Sample preparation:**

Cationic tensides are normally titrated at pH = 10, e.g. 5 mL buffer pH = 10 (boric acid/KCl/NaOH, e.g. Merck Titrisol 9890) is added. Sometimes an addition of 3 mL methanol may be helpful.

### Literature

- Metrohm Application Bulletin No. 233: Titrimetric/potentiometric determination of anionic and cationic detergents with the high-sense surfactant electrode.

# Anionic Surfactants in Liquid Soap

## Reagents

c(DDMICl) = 0.004 mol/L; D0  
 1,3-didecyl-2-methyl-imidazolium chloride (TEGO® trant A100)  
 Metrohm 6.2317.000

## Sample

app. 0.1 g sample (liquid soap)  
 60 mL dist. water

## Electrodes

6.0504.150 high-sense surfactant electrode; input 1  
 6.0733.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/L)

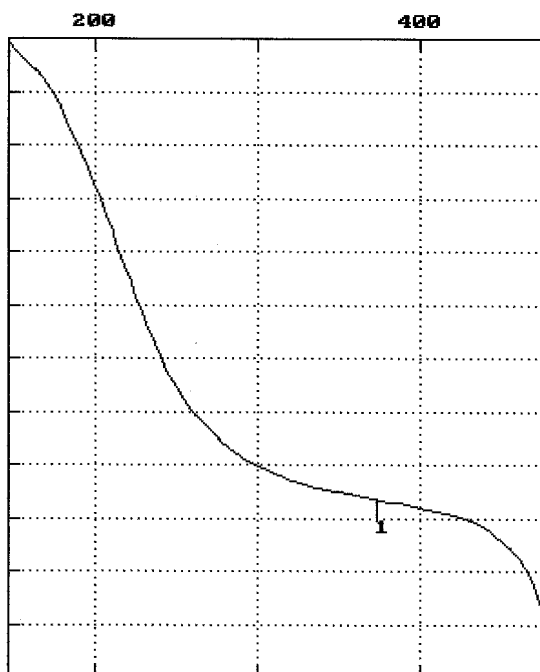
## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 15:18  17
DET U              Tens.An
parameters
>titration parameters
  meas.pt.density      3
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         OFF mV/min
  equilibr.time        10 s
  start V:             OFF
  pause                30 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               10 ml
  stop U               OFF mV
  stop EP              OFF
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      greatest
  fix EP1 at U         OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 15:07  17
U(init)            150 mV DET U  Tens.An
smpl size          0.1061 g   id#1   Hand
id#2               Soap      id#3   liquid
EP1                8.655 ml
Anionic            0.3263 meq/g
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 15:07  17
start V            0.000 ml DET U  Tens.An
1.0 ml/div         dU=100.0 mV/div
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-10      time 15:18  17
DET U              Tens.An
>calculations
Anionic=EP1*C01/C00;4;meq/g
C00=                0.1061
C01=                0.004
-----
```



**Remarks**

- **Calculations:**  
Anionic = concentration of anionic Tensides in meq/g  
C01 = concentration of titrating agent (0.004 mol/L)
- **Electrode preparation:**  
The electrode is shaken vigorously downward (like a clinical thermometer) and is best conditioned by two to three titrations whose results are not taken into account. On completion of a titration or titration series (with Sample Changer of up to 100 and more samples, no intermediate rinsing is required), the electrodes are rinsed briefly with methanol and then with dist. water. (If need be, carefully wipe with a soft paper towel soaked in methanol.)  
The electrode is stored dry.
- **Sample preparation:**  
Anionic tensides are normally titrated at pH = 3, e.g. 5 mL buffer pH = 3 (citrate/HCl, e.g. Merck Titrisol 9883) is added. Sometimes an addition of 3 mL methanol may be helpful.

---

**Literature**

- Metrohm Application Bulletin No. 233: Titrimetric/potentiometric determination of anionic and cationic detergents with the high-sense surfactant electrode.

# Perborates in Detergents

## Reagents

$c(\text{KMnO}_4) = 0.02 \text{ mol/L}; \text{D0}$

## Sample

5 mL sample solution  
20 mL  $w(\text{H}_2\text{SO}_4) = 0.30$  (30%)

## Electrodes

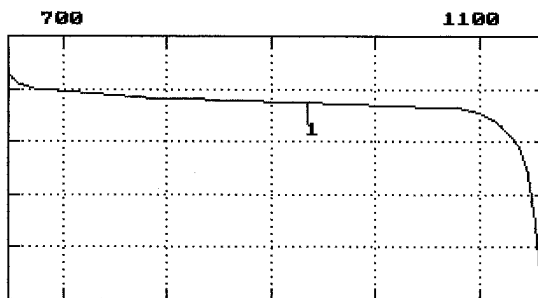
6.0420.100 LL combined Pt electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 16:21  15
MET U              Perborat
parameters
>titration parameters
  V step           0.10 ml
  titr.rate        max. ml/min
  signal drift     OFF mV/min
  equilibr.time    10 s
  start V:         abs.
  start V          0.5 ml
  dos.rate         max. ml/min
  pause           30 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V          5 ml
  stop U          OFF mV
  stop EP        9
  filling rate    max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           30 mV
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: OFF
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 16:18  15
U(init)           697 mV MET U Perborat
smpl size         2.5023 g
EP1               1.289 ml          933 mV
Perborat          7.93 %
stop V reached
=====
```

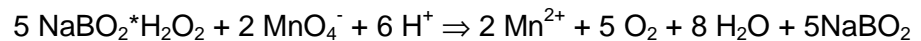
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 16:18  15
start V          0.500 ml MET U Perborat
1.0 ml/div       dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 16:21  15
MET U              Perborat
>calculations
Perborat=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                2.5023
C01=                 0.02
C02=                 2.5
C03=                153.86
C04=                 0.1
C05=                 20
-----
```

### Remarks

- **Determination reaction:**



- **Calculations:**

Perborat = fraction of perborates in %

C01 = concentration of titrating agent (0.02 mol/L)

C02 = factor for "normality" ( $5/2=2.5$ )

C03 = molecular mass of  $\text{NaBO}_2 \cdot \text{H}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$  (153.86 g/mol)

C04 = conversion mL  $\rightarrow$  L \* factor for % ( $0.001 \cdot 100 = 0.1$ )

C05 = factor for dilution (20)

- **Sample preparation:**

Weigh 2.5 g in a measuring flask of 100 mL. Dissolve sample with  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/L}$  and wait for the reaction of the carbonate. When gas evolution has stopped, fill measuring flask to the mark. Pipette 5 mL of solution, add 20 mL of  $w(\text{H}_2\text{SO}_4) = 0.30$  (30%) and titrate.

- The 6.0431.100 Pt Titrode may be used instead of the 6.0420.100 LL Pt electrode.

### Literature

- G. Jander, K.F.Jahr, "Massanalyse", Sammlung Gröschen de Gruyter, Berlin, New York (1973), p. 67

# Pure Silver Content

## Reagents

c(KBr) = 0.1 mol/L; D0

## Sample

app. 250 mg Ag  
10 mL HNO<sub>3</sub> 65 %  
100 mL dist. water

## Electrodes

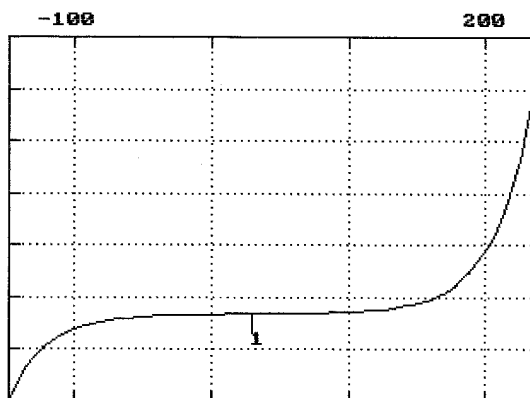
6.0404.100 combined massive Ag electrode with AgBr coating; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 15:28    8
DET U              Silver
parameters
>titration parameters
meas.pt.density    4
min.incr.          10.0 µl
titr.rate          max. ml/min
signal drift       50 mV/min
equilibr.time      26 s
start V:           abs.
start V            17 ml
dos.rate           max. ml/min
pause              0 s
dos.element:       internal D0
meas.input:        1
temperature        25.0 °C
>stop conditions
stop V:            abs.
stop V             24 ml
stop U             OFF mV
stop EP            9
filling rate      max. ml/min
>statistics
status:            OFF
>evaluation
EPC                5
EP recognition:    all
fix EP1 at U      OFF mV
pK/HNP:           OFF
>preselections
req.ident:         OFF
req.smpl size:     all
activate pulse:    OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 15:24    8
U(init)            280 mV DET U   Silver
smpl size          257.7 mg
EPI                22.332 ml      30 mV
Silver             93.479 %
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 15:24    8
start V            17.000 ml DET U   Silver
1.0 ml/div         dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-18      time 15:29    8
DET U              Silver
>calculations
Silver =EP1*C01*C02*C03*C04/C00;3;%
C00=                257.7
C01=                 0.1
C02=                 1.0
C03=                107.87
C04=                 100
-----
```

**Remarks**

- **Calculations:**  
Silver = content of silver in % ( purity)  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = titer (1.0)  
C03 = molecular mass of Ag (107.87 g/mol)  
C04 = factor for % (100)
- **Sample preparation:**  
Heat silver in HNO<sub>3</sub>. Allow nitrous fumes to evaporate.
- **AgBr coating of electrode:**  
Clean Ag electrode with scouring agent and electrolyse in w(HBr) = 0.1 (10%) during 2 h with 5 mA using a 6.0305.000 Pt electrode as cathode. Stir during electrolysis.

---

**Literature**

- Metrohm Application Bulletin No. 61: Potentiometric determination of silver.

# Metals

## Reagents

c(Na<sub>2</sub>EDTA) = 0.1 mol/L; D0

## Sample

2 mL c(ZnSO<sub>4</sub>) ≅ 0.1 mol/L  
 5 mL buffer pH = 10  
 1 mL c(CuEDTA) = 0.1 mol/L  
 40 mL dist. water

## Electrodes

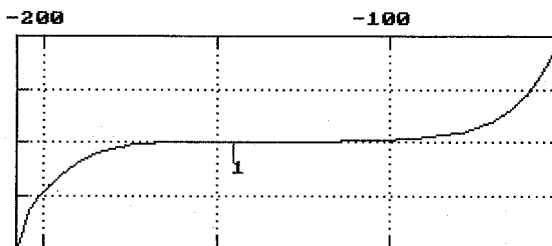
6.0502.140 Cu<sup>2+</sup> sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (KNO<sub>3</sub> sat.)

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 16:30   4
DET U              Metals
parameters
>titration parameters
  meas.pt.density  2
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     20 mV/min
  equilibr.time    38 s
  start V:         OFF
  pause           0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V          10 ml
  stop U          OFF mV
  stop EP        9
  filling rate    max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           5
  EP recognition: all
  fix EP1 at U  OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:     OFF
  req.smpl size: all
  activate pulse: OFF
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 16:23   4
U(init)           -50 mV DET U   Metals
smpl size         2 ml
EP1               2.006 ml      -145 mV
Content           6.56 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 16:23   4
start V          0.000 ml DET U   Metals
1.0 ml/div      dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-28      time 16:31   4
DET U              Metals
>calculations
Content=EP1*C01*C02/C00;2;g/l
C00=              2
C01=              0.1
C02=              65.38
```

### Remarks

- Calculations:**  
 Content = content of metal in g/L  
 C01 = concentration of titrating agent (0.1 mol/L)  
 C02 = molecular mass of metal
- Buffer pH = 10:**  
 Dissolve 54 g  $\text{NH}_4\text{Cl}$  and 350 mL  $w(\text{NH}_3) = 0.25$  (25%) in dist. water and fill up to 1 litre.
- Buffer pH = 4.7:**  
 Dissolve 123 g Na-acetate and 86 mL acetic acid in dist. water and fill up to 1 liter.
- The following metals can be determined according to this method:

		buffer solution	molar mass
Water, total hardness	(Ca + Mg)	pH = 10	64.40
Barium	Ba	pH = 10	137.36
Cadmium	Cd	pH = 10	112.41
Cobalt	Co	pH = 10	58.94
Nickel	Ni	pH = 10	58.71
Zinc	Zn	pH = 10	65.38
Lead	Pb	pH = 4.7	207.21

### Literature

- Metrohm Application Bulletin No. 101: Complexometric titrations with the Cu ISE

# Aluminium

## Reagents

c(HCl) = 1 mol/L; D0  
 c(NaOH) = 1 mol/L; D1  
 w(KF) = 0.10 (10%); D2

## Sample

a sample with 40...50 mg Al content  
 50 mL dist. water  
 5 mL w(sodium gluconate) = 0.25 (25%)

## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:17  1
TIP                Aluminum
parameters
>sequence
  1.method:        Al 1
  2.method:        Al 2
  3.method:        Al 3
  4.pause         180 s
  5.method:        Al 4
>statistics
  status:         OFF
>preselections
  req.ident:      OFF
  req.smpl size:  value
  meas.mode:     pH
  meas.input:    1
  temperature    25.0 °C
-----

'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 08:41  1
TIP                Aluminum
smpl size         0.2986 g
C70               0.193
C71               2.684
Aluminum          8.666 %
TIP terminated
=====

'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:17  1
TIP                Aluminum
>calculations
Aluminum=(C70+C71)*C01*C02*C03/C04/C00;3
;%
C00=              0.2986
C01=              1
C02=              8.994
C03=              100
C04=              1000
C70=              not valid
C71=              not valid
-----
```

**Submethods**

pH adjustment

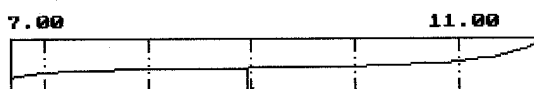
Titration of excess of NaOH

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-10-16      time 09:07  1
SET pH              Al 1
parameters
>SET1
  EP at pH          11.50
  dynamics           2
  max.rate          10.0 ml/min
  min.rate          25.0 µl/min
  stop crit:        drift
  stop drift        20 µl/min
>SET2
  EP at pH          OFF
>titration parameters
  titr.direction:   auto
  pause 1           0 s
  start V:          OFF
  pause             0 s
  extr.time         0 s
  dos.element:      external D1
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-10-16      time 09:08  1
DET pH              Al 2
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause             0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  stop pH           OFF
  stop EP           1
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  EPC               5
  EP recognition:   all
  fix EP1 at pH    OFF
  pK/HNP:          OFF
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'de
736 GP Titrimo      OP1/103  736.0010
date 95-10-16      time 09:08
DET pH              Al 2
def
>formula
  excess=C41-EP1
  RS1 text          excess
  RS1 decimal places 3
  RS1 unit:         ml
>sil calculations
  match id:         OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C70=RS1
-----
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-10-16      time 08:32  1
start V            0.000 ml DET pH      Al 2
1.0 ml/div         dpH=1.0/div
```



**Submethods**

Addition of KF

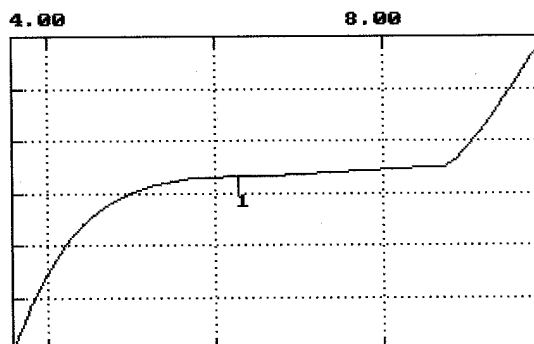
Determination

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:09    1
DOS                Al 3
parameters
>dosing parameters
  dispensing type:  volume
  volume           5 ml
  disp.crit:       rate
  rate             max. ml/min
  pause           0 s
  time interval    10 s
  dos.element:     external D2
  temperature      25.0 °C
>stop conditions
  stop V:          OFF
  filling rate     max. ml/min
>statistics
  status:          OFF
>monitoring
  meas.mode:       OFF
  temperature:     OFF
  assign output:   none
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  activate pulse:  OFF
  -----
```

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:10    1
DET pH             Al 4
parameters
>titration parameters
  meas.pt.density  4
  min.incr.        10.0 µl
  titr.rate        max. ml/min
  signal drift     50 mV/min
  equilibr.time    26 s
  start V:         OFF
  pause           0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           6 ml
  stop pH          OFF
  stop EP          9
  filling rate     max. ml/min
>statistics
  status:          OFF
>evaluation
  EPC              5
  EP recognition:  all
  fix EP1 at pH   OFF
  pK/HNP:          OFF
>preselections
  req.ident:       OFF
  req.smpl size:   OFF
  activate pulse:  OFF
  -----
```

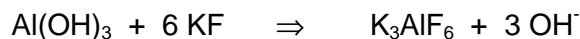
```
'de
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 09:10
DET pH             Al 4
def
>formula
>silo calculations
  match id:        OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C71=EP1
  -----
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-16      time 08:40    1
start V           0.000 ml DET pH    Al 4
1.0 ml/div        dpH=2.0/div
```



**Remarks**

- **Determination reaction:**



- **Calculations:**

Aluminum = content of Al in %

C01 = concentration of titrating agent (1 mol/L)

C02 = molecular mass / "normality" (26.982 / 3 = 8.994)

C03 = factor for % (100)

C04 = factor for mL → L (1000)

C70 = excess of the method "Al 2" (stop V - EP1)

C71 = consumption in method "Al 4" (EP1)

---

**Literature**

- Metrohm Application Bulletin No. 24: Potentiometric determination of aluminium

# Phosphorous in Fertilizer

## Reagents

c(NaOH) = 1.0 mol/L; D0  
Sodium oxalate, saturated; D1

## Sample

10 mL liquid fertilizer  
5 mL c(HCl) = 1 mol/L  
40 mL dist. water

## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```

'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:51  13
TIP                 P205Fert
parameters
>sequence
  1.method:         P205-1
  2.method:         P205-2
  3.pause:          30 s
  4.method:         P205-3
>statistics
  status:           OFF
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  meas.mode:        OFF
  temperature       25.0 °C
-----

'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:50  13
TIP                 TIP      P205Fert
smpl size          10 ml
C70                1.031
C71                10.614
P205               8.27 %
TIP terminated
=====

```

```

'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:51  13
TIP                 P205Fert
>calculations
P205=(C70+C71)*C01*C02/C00;2;%
C00=                10
C01=                1
C02=                7.1
C70=                1.031
C71=                10.614
-----

```

**Submethods**

Titration of acid

Addition of Na-oxalate

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:52  13
DET pH              P205-1
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature           25.0 °C
>stop conditions
  stop V:              abs.
  stop V               99.99 ml
  stop pH              OFF
  stop EP              1
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at pH       OFF
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:52  13
DOS                  P205-2
parameters
>dosing parameters
  dispensing type:     volume
  volume               15 ml
  disp.crit:           rate
  rate                 max. ml/min
  pause                0 s
  time interval        10 s
  dos.element:         external D1
  temperature           25.0 °C
>stop conditions
  stop V:              OFF
  filling rate         max. ml/min
>statistics
  status:              OFF
>monitoring
  meas.mode:           OFF
  temperature:         OFF
  assign output:       none
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

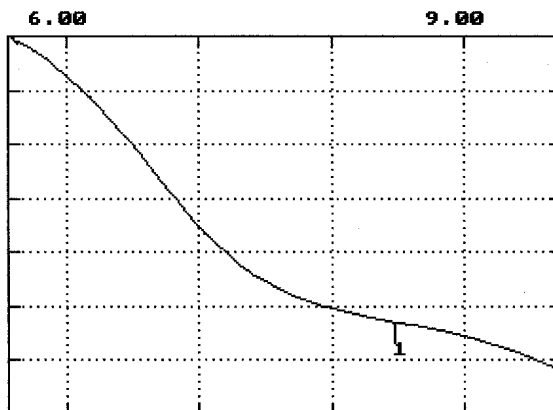
```
'de
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:52
DET pH              P205-1
def
>formula
  excess=C41-EP1
  RS1 text             excess
  RS1 decimal places   3
  RS1 unit:            ml
>silo calculations
  match id:            OFF
>common variables
>report
>mean
  MN1=RS1
>temporary variables
  C70=RS1
-----
```

**Submethods**

Determination

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:52  13
DET pH             P205-3
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause             0 s
  dos.element:      internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  stop pH           OFF
  stop EP           1
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  EPC               5
  EP recognition:   window
  low lim.1 pH     7
  up lim.1 pH      9
  low lim.2 pH     OFF
  fix EP1 at pH    OFF
  pK/HNP:          OFF
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:49  13
start V            0.000 ml DET pH  P205-3
2.0 ml/div         dpH=1.0/div
```



```
'de
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 15:53
DET pH             P205-3
def
>formula
>silo calculations
  match id:         OFF
>common variables
>report
  report:curve;
>mean
  MN1=RS1
>temporary variables
  C71=EP1
-----
```

### Remarks

- **Determination reaction:**



- **Calculations:**

P2O5 = content of P<sub>2</sub>O<sub>5</sub> in %

C01 = concentration of titrating agent (1 mol/L)

C02 = Equivalent weight P<sub>2</sub>O<sub>5</sub> in g/mol (71\*100/1000=7.1)

C70 = Volume in mL of excess titrant in the 1<sup>st</sup> titration (P2O5-1)

C71 = Volume in mL of the titrant consumed in the 2<sup>nd</sup> titration  
(P2O5-3)

---

### Literature

- Metrohm Application Bulletin No. 240: Fully automatic determination of the phosphorous content (P<sub>2</sub>O<sub>5</sub>) in fertilizers

# Epoxy Number

## Reagents

$c(\text{HClO}_4) = 0.1 \text{ mol/L}; \text{D0}$

## Sample

app. 0.2 g UHU resin  
 25 mL  $c(\text{TBA}) = 0.2 \text{ mol/L}$  in acetic acid  
 TBA = Tetrabutyl ammonium bromide

## Electrodes

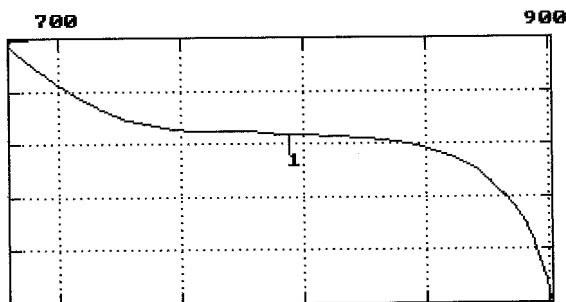
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 18:11  13
DET U              Epoxy.No
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      OFF mV/min
  equilibr.time     20 s
  start V:          OFF
  pause            0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           12 ml
  stop U           OFF mV
  stop EP         9
  filling rate     max. ml/min
>statistics
  status:          OFF
>evaluation
  EPC             5
  EP recognition: all
  fix EP1 at U   OFF mV
  pK/HNP:        OFF
>preselections
  req.ident:      OFF
  req.smpl size: value
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 18:06  13
U(init)            646 mV DET U Epoxy.No
smpl size          0.2261 g id#1  UHU
id#2               plus
EP1                8.828 ml      795 mV
Epoxy.No           0.390
stop V reached
=====
```

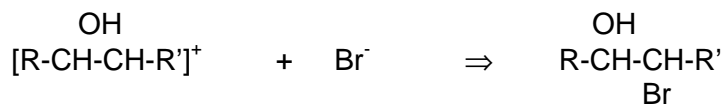
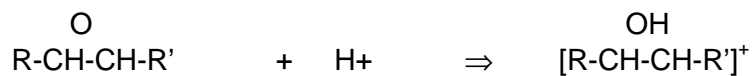
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 18:06  13
start V           7.000 ml DET U Epoxy.No
1.0 ml/div        dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-04      time 18:11  13
DET U              Epoxy.No
>calculations
Epoxy.No=EP1*C01*C02*C03/C00*3;
C00=               0.2261
C01=               0.001
C02=               0.1
C03=               100
-----
```

### Remarks

- **Determination reaction:**



- **Calculations:**

Epoxy.No = epoxy number, number of epoxide groups per 100 g of sample

C01 = factor for conversion mL  $\Rightarrow$  L (0.001)

C02 = concentration of titrating agent (0.1 mol/L)

C03 = factor for 100 g of sample (1000)

- Stir sample in solvent during 3 minutes before titration

### Literature

- I. Gyenes, "Titrations in nichtwässrigen Medien"  
Ferdinand Enke Verlag, Stuttgart (1970), p. 598

# Nitrating Acid

## Reagents

c(Cyclohexylamine) = 0.5 mol/L; D0

## Sample

3 mL nitrating acid  
25 mL methanol

## Electrodes

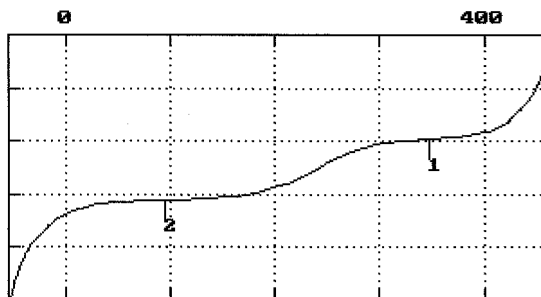
6.0133.100 pH glass electrode; input 1  
6.0726.100 Ag/AgCl double junction reference electrode  
(c(LiClO<sub>4</sub>) = 1 mol/L in acetic acid)

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 18:03   9
DET U              NitrAcid
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               10 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 17:57   9
U(init)             463 mV DET U  NitrAcid
smpl size           3.0 ml
EP1                 1.965 ml          349 mV
EP2                 3.113 ml          96 mV
H2SO4                18.77 g/l
HNO3                 8.58 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 17:57   9
start V             0.000 ml DET U  NitrAcid
1.0 ml/div          dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-20      time 18:03   9
DET U              NitrAcid
>calculations
H2SO4  =(EP2-EP1)*C01*C02/C00;2;g/l
HNO3   =(EP1-(EP2-EP1))*C01*C03/C00;2;g
/l
C00=    3.0
C01=    0.5
C02=    98.08
C03=    63.01
-----
```

### Remarks

- **Nitrating acid:**
  - 1 mL H<sub>2</sub>SO<sub>4</sub> ( 96 %,  $\delta = 1.84$  g/L)
  - 1 mL HNO<sub>3</sub> (app. 65 %)
  - 20 mL dist. water
  - fill up with methanol to 100 mL
  
- 1st break:  $\text{HNO}_3 + \text{H}_2\text{SO}_4 + 2 \text{CHA} \Rightarrow \text{NO}_3^- + \text{HSO}_4^- + 2 \text{HCHA}^+$
  
- 2nd break:  $\text{HSO}_4^- + \text{CHA} \Rightarrow \text{SO}_4^{2-} + \text{HCHA}^+$
  
- CHA = Cyclohexylamine
  
- **Calculations:**
  - H<sub>2</sub>SO<sub>4</sub> = sulphuric acid in g/L of nitrating acid
  - HNO<sub>3</sub> = nitric acid in g/L of nitrating acid
  - C01 = concentration of titrating agent (0.5 mol/L)
  - C02 = molecular mass of H<sub>2</sub>SO<sub>4</sub> (98.08 g/mol)
  - C03 = molecular mass of HNO<sub>3</sub> (63.01 g/mol)

### Literature

- Metrohm Application Bulletin No. 39: Potentiometric analysis of nitrating acid

# Hydrogen Peroxide

## Reagents

$c(\text{KMnO}_4) = 0.02 \text{ mol/L}; \text{D0}$

## Sample

2 mL sample  
 2 mL  $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$   
 0.1 g  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  (catalyst)  
 100 mL dist. water

## Electrodes

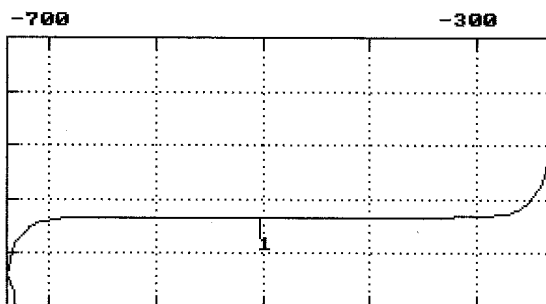
6.0431.100 Ag Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 13:39   13
DET U              H2O2
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  titr.rate          max. ml/min
  signal drift       10 mV/min
  equilibr.time      52 s
  start V:           abs.
  start V            1 ml
  dos.rate           max. ml/min
  pause              0 s
  dos.element:      internal D0
  meas.input:       1
  temperature        25.0 °C
>stop conditions
  stop V:           abs.
  stop V            6 ml
  stop U            OFF mV
  stop EP           9
  filling rate      max. ml/min
>statistics
  status:           OFF
>evaluation
  EPC               5
  EP recognition:   all
  fix EP1 at U     OFF mV
  pK/HNP:          OFF
>preselections
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 13:22   13
U(init)            -225 mV DET U      H2O2
smpl size          1.2005 g
EP1                4.348 ml          -501 mV
H2O2               30.80 %
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 13:22   13
start V            1.000 ml DET U      H2O2
1.0 ml/div         dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-06      time 13:40   13
DET U              H2O2
>calculations
H2O2=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                1.2005
C01=                 0.02
C02=                 2.5
C03=                 34.02
C04=                 0.1
C05=                 50
-----
```

### Remarks

- **Determination reaction:**



- **Sample preparation:**

app. 1.2 g  $w(\text{H}_2\text{O}_2) \cong 0.30$  (30%) in 100 mL  $c(\text{H}_2\text{SO}_4) = 0.5$  mol/L

- **Calculations:**

H2O2 = fraction of  $\text{H}_2\text{O}_2$  in %

C01 = concentration of titrating agent \* titer (0.02 mol/L)

C02 = factor for "normality" ( $5/2 = 2.5$ )

C03 = molecular mass of  $\text{H}_2\text{O}_2$  (34.04 g/mol)

C04 = factor for conversion 1 mL→L \* factor for % ( $0.001 * 100 = 0.1$ )

C05 = dilution factor (50)

- The start V in this method meets two purposes:

1. It speeds up the titration.

2.  $\text{Mn}^{2+}$  is generated which serves as catalyst for the reaction so there is no need to add  $\text{Mn}^{2+}$  separately.

### Literature

# Phosphate

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

5 mL c(NaH<sub>2</sub>PO<sub>4</sub>) ≅ 0.1 mol/L  
 40 mL dist. water  
 adjust the pH value to 4.2 with dilute NaOH or H<sub>2</sub>SO<sub>4</sub>  
 10 mL c(La(NO<sub>3</sub>)<sub>3</sub>) = 0.1 mol/L, pH = 4.2

## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```

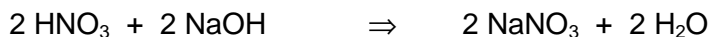
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 14:15      8
SET pH             Phosphat
parameters
>SET1
  EP at pH         4.20
  dynamics         1
  max.rate        10 ml/min
  min.rate        25 µl/min
  stop crit:      drift
  stop drift      20 µl/min
>SET2
  EP at pH         OFF
>titration parameters
  titr.direction: +
  pause 1         0 s
  start V:        OFF
  pause          0 s
  extr.time       0 s
  dos.element:    internal D0
  meas.input:     1
  temperature     25.0 °C
>stop conditions
  stop V:         abs.
  stop V          99.99 ml
  filling rate    max. ml/min
>statistics
  status:         OFF
>preselections
  conditioning:   OFF
  req.ident:      OFF
  req.smpl size:  value
  activate pulse: OFF
-----

'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 14:15      8
pHc(init)         1.78
smpl size         0.07798 g
EP1               10.022 ml
P                 19.90 %
P205              45.61 %
PO4               61.03 %
=====

'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 14:16      8
SET pH             Phosphat
>calculations
P=EP1*C01*C02/C00;2;%
P205=EP1*C01*C03/C00;2;%
PO4=EP1*C01*C04/C00;2;%
C00=               0.07798
C01=               0.1
C02=              1.5487
C03=              3.5486
C04=              4.7486
-----
    
```

### Remarks

- **Determination reaction:**



- **Calculations:**

P = content of P in %

P<sub>2</sub>O<sub>5</sub> = content of P<sub>2</sub>O<sub>5</sub> in %

PO<sub>4</sub> = content of PO<sub>4</sub> in %

C01 = factor for %

C02 = 1 mL c(NaOH) = 0.1 mol/L = 1.5487 mg P

C03 = 1 mL c(NaOH) = 0.1 mol/L = 3.5486 mg P<sub>2</sub>O<sub>5</sub>

C04 = 1 mL c(NaOH) = 0.1 mol/L = 4.7486 mg PO<sub>4</sub>

- **Sample preparation:**

Adjust the pH of your sample aliquot to 4.2 by adding either NaOH or H<sub>2</sub>SO<sub>4</sub>. Add 10 mL c(La(NO<sub>3</sub>)<sub>3</sub>) = 0.1 mol/L, pH = 4.2, to this sample solution and titrate.

- Select the appropriate formula. The others may be deleted.

### Literature

- Metrohm Application Bulletin No. 129: Potentiometric determination of ortho-, meta- and polyphosphates.

# 2-Aminophenol

## Reagents

c(NaNO<sub>2</sub>) = 0.2 mol/L; D0

## Sample

app. 0.3 ... 0.35 g sample (2-aminophenol)  
 10 mL w(HBr) = 0.20 (20%)  
 30 mL dist. water

## Electrodes

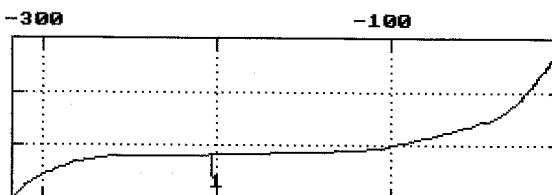
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
parameters
>titration parameters
  V step           0.10 ml
  titr.rate        max. ml/min
  signal drift     OFF mV/min
  equilibr.time    15 s
  start V:         OFF
  pause            30 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           17 ml
  stop U           OFF mV
  stop EP         9
  filling rate     max. ml/min
>statistics
  status:          OFF
>evaluation
  EPC              30 mV
  EP recognition:  all
  fix EP1 at U    OFF mV
  pK/HNP:         OFF
>preselections
  req.ident:       OFF
  req.smpl size:  value
  activate pulse:  OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
U(init)            125 mV MET U    Diazo
smpl size          0.3188 g      id#1    09120
EP1                14.396 ml     -203 mV
Content            98.56 %
stop V reached
=====

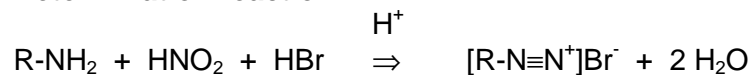
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
start V           10.000 ml MET U    Diazo
2.0 ml/div        dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
>calculations
Content=EP1*C01*C02*C03/C00;2;%
C00=               0.3188
C01=               109.13
C02=               0.1
C03=               0.2
-----
```

**Remarks**

- **Determination reaction:**



- **Calculations:**

Content = content of 2-aminophenol in %

C01 = molecular mass of 2-aminophenol (109.13 /mol)

C02 = factor for conversion mL → L, and for % (0.001\*100=0.1)

C03 = concentration of titrating agent (0.2 mol/L)

- Instead of the Pt Titrode a combined Pt electrode 6.0420.100 can be used.
- 

**Literature**

- Metrohm Application Bulletin No. 228: Diazotisation titrations

# White Liquor

## Reagents

c(HCl) = 1 mol/L; D0

## Sample

2.00 mL white liquor  
 white liquor is a mixture of NaOH, Na<sub>2</sub>S (pK<sub>1</sub>=7.04, pK<sub>2</sub>=11.96), Na<sub>2</sub>CO<sub>3</sub> (pK<sub>1</sub>=6.37, pK<sub>2</sub>=10.25)  
 50 mL dist. water

## Electrodes

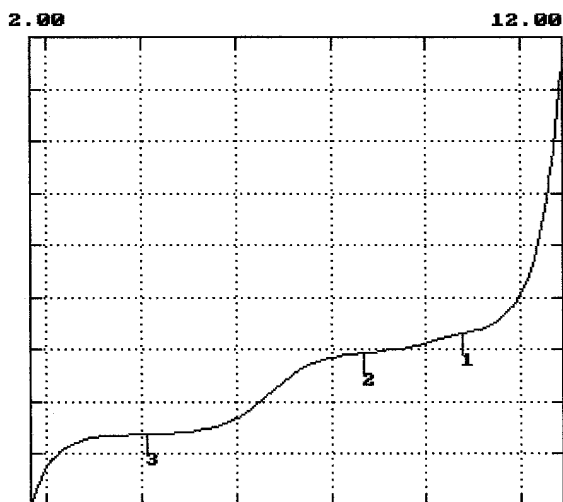
6.0239.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 17:07    4
DET pH             W-Liquor
parameters
>titration parameters
  meas.pt.density   4
  min.incr.         10.0 µl
  titr.rate         max. ml/min
  signal drift      50 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause            0 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           9 ml
  stop pH         OFF
  stop EP         9
  filling rate    max. ml/min
>statistics
  status:         OFF
>evaluation
  EPC             5
  EP recognition: all
  fix EP1 at pH  OFF
  pK/HNP:        OFF
>preselections
  req.ident:      OFF
  req.smpl size: OFF
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 17:05    4
pHc(init)         12.85   DET pH  W-Liquor
EP1                5.714 ml  10.76
EP2                6.081 ml   8.67
EP3                7.625 ml   4.09
Total              305.00 g/l
Active             275.64 g/l
Effecti.           228.56 g/l
NaOH               181.48 g/l
Na2S                91.81 g/l
Na2CO3             38.90 g/l
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 17:05    4
start V           0.000 ml DET pH  W-Liquor
1.0 ml/div        dpH=2.0/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-11      time 17:07    4
DET pH             W-Liquor
>calculations
Total=EP3*C01*C02/C00;2;g/l
Active=(EP3-C03*(EP2-EP1))*C01*C02/C00;2;g/l
Effecti.=EP1*C01*C02/C00;2;g/l
NaOH=(EP1-((EP3-EP2)-(EP2-EP1))*C01*C02/C00;2;g/l
Na2S=((EP3-EP2)-(EP2-EP1))*C03*C02*C04/C00;2;g/l
Na2CO3=(EP2-EP1)*C03*C02*C05/C00;2;g/l
C00= 1.0
C01= 40
C02= 1
C03= 2
C04= 39
C05= 53
-----
```

### Remarks

- Pass a stream of nitrogen through the solution during titration.
- 1st break:  $\text{OH}^- + \text{S}^{2-} + 2 \text{H}^+ \Rightarrow \text{H}_2\text{O} + \text{HS}^-$   
 2nd break:  $\text{CO}_3^{2-} + \text{H}^+ \Rightarrow \text{HCO}_3^-$   
 3rd break:  $\text{HCO}_3^- + \text{HS}^- + 2 \text{H}^+ \Rightarrow \text{H}_2\text{CO}_3 + \text{H}_2\text{S}$
- **Calculations:**  
 Total = total alkali ( $\text{NaOH} + \text{Na}_2\text{S} + \text{Na}_2\text{CO}_3$ ) as g NaOH per liter  
 Active = active alkali ( $\text{NaOH} + \text{Na}_2\text{S}$ ) as g NaOH per liter  
 Effecti. = effective alkali ( $\text{NaOH} + \frac{1}{2} \text{Na}_2\text{S}$ ) as g NaOH per liter  
 NaOH = concentration of NaOH in g/L  
 Na<sub>2</sub>S = concentration of Na<sub>2</sub>S in g/L  
 Na<sub>2</sub>CO<sub>3</sub> = concentration of Na<sub>2</sub>CO<sub>3</sub> in g/L  
 C01 = molecular mass of NaOH (40 g/mol)  
 C02 = concentration of titrating agent (1 mol/L)  
 C03 = factor (2)  
 C04 = molecular mass of  $\frac{1}{2} \text{Na}_2\text{S}$  (39 g/mol)  
 C05 = molecular mass of  $\frac{1}{2} \text{Na}_2\text{CO}_3$  (53 g/mol)

### Literature

- SCAN - N2:63 (1963)

# Silver in Fixing Baths

## Reagents

c(Thioacetamide) = 0.025 mol/L in buffer pH = 5

## Sample

5 mL of sample  
 20 mL c(NaOH) = 2 mol/L  
 20 mL c(EDTA) = 0.1 mol/L  
 10 mL w(gelatine) = 0.12 (12%)

## Electrodes

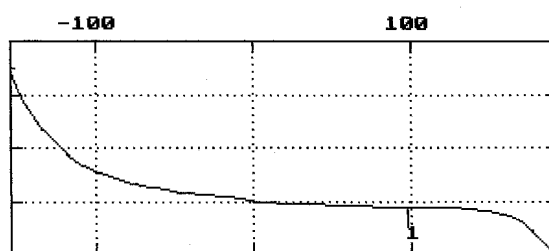
6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating; input 1

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 13:44   11
DET U              FixBaths
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               12 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      greatest
  fix EP1 at U        OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 13:36   11
U(init)            -168 mV DET U  FixBaths
smpl size          5 ml
EP1                 10.220 ml           99 mV
Silver              11.025 g/l
stop V reached
=====
```

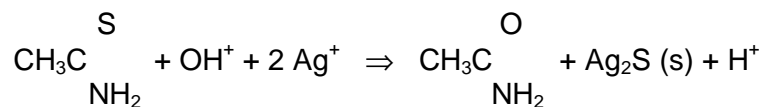
```
'cu
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 13:36   11
start V            5.000 ml DET U  FixBaths
2.0 ml/div         dU=100.0 mV/div
```



```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-19      time 13:44   11
DET U              FixBaths
>calculations
Silver=EP1*C01*C02/C00;3;g/l
C00=                5
C01=                 5.394
C02=                 1.000
-----
```

### Remarks

- **Determination reaction:**



- **Calculations:**

Silver = Ag content in g/L

C01 = Ag equivalent (5.394 mg/mL titrating agent)

C02 = titer of titrating agent (1.000)

- EDTA keeps silver in solution and gelatine prevents the precipitation from massing together.
- **Ag<sub>2</sub>S coating of electrode:**  
Keep Ag Titrode in alkaline solution of thioacetamide during 15 min.
- **Buffer pH = 5:**  
Mix potassium biphthalate solution,  $c = 0.1 \text{ mol/L}$  with  $\text{Na}_3\text{PO}_4$  solution,  $c = 0.05 \text{ mol/L}$ . Ratio 50:24. Dissolve 0.5 g thymol per liter buffer solution.
- **Titration agent:**  
Dissolve 1.9 g Thioacetamide in 1 L buffer pH = 5 solution.
- **Gelatine solution:**  
Dissolve 12 g gelatine in hot water, add 0.5 g thymol, allow to cool and add up to 1 L.

### Literature

- Metrohm Application Bulletin No. 72: Potentiometric determination of mercury or silver in the presence of halide ions.

# Silver in Film Emulsions

## Reagents

c(Thioacetamide) = 0.025 mol/L in buffer pH = 5

## Sample

10 mL solution (100 cm<sup>2</sup> Ilford black and white film in 100 mL fixing agent Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 300 g/L)  
 20 mL c(NaOH) = 2 mol/L  
 20 mL c(EDTA) = 0.1 mol/L  
 10 mL w(gelatine) = 0.12 (12%)

## Electrodes

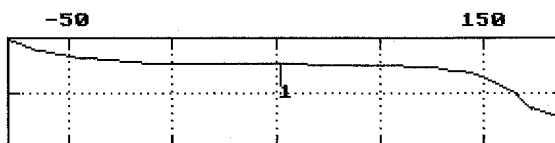
6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-19      time 15:23   15
DET U              Film Ag
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  titr.rate            max. ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  dos.element:         internal D0
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               3 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-19      time 15:22   15
U(init)             -17 mV DET U   Film Ag
smpl size           10 ml id#1  ILFORD
EP1                  0.987 ml
AgNO3                4.192 g/m2
stop V reached
=====
```

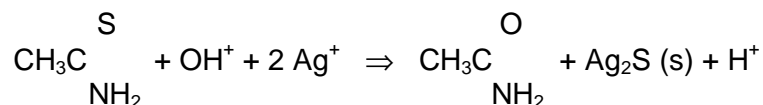
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-09-19      time 15:22   15
start V             0.000 ml DET U   Film Ag
2.0 ml/div          dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-19      time 15:23   15
DET U              Film Ag
>calculations
AgNO3=EP1*C01*C02/C00*C03/C04/C05;3;g/m2
C00=                10
C01=                 0.025
C02=                169.87
C03=                 100
C04=                 0.01
C05=                1000
-----
```

### Remarks

- **Determination reaction:**



- **Calculations:**

AgNO<sub>3</sub> = amount of AgNO<sub>3</sub> in g/m<sup>2</sup>

C01 = concentration of titrating agent (0.025 mol/L)

C02 = molecular mass of AgNO<sub>3</sub> (169.87 g/mol)

C03 = factor for 100 mL sample (100)

C04 = size of sample (0.01 m<sup>2</sup>)

C05 = conversion mL ⇒ L (1000)

- EDTA keeps silver in solution and gelatine prevents the precipitation from massing together.
- **Ag<sub>2</sub>S coating of electrode:**  
Keep Ag Titrode in alkaline solution of thioacetamide during 15 min.
- **Buffer pH = 5:**  
Mix potassium biphthalate solution, *c* = 0.1 mol/L with Na<sub>3</sub>PO<sub>4</sub> solution, *c* = 0.05 mol/L. Ratio 50:24. Dissolve 0.5 g thymol per liter buffer solution.
- **Titration agent:**  
Dissolve 1.9 g Thioacetamide in 1 L buffer pH = 5 solution.
- **Gelatine solution:**  
Dissolve 12 g gelatine in hot water, add 0.5 g thymol, allow to cool and add up to 1 L.

### Literature

- Metrohm Application Bulletin No. 72: Potentiometric determination of mercury or silver in the presence of halide ions.

# Kappa Number of Paper Pulp

## Reagents

$c(\frac{1}{2}\text{Na}_2\text{S}_2\text{O}_3) = 0.2 \text{ mol/L}; \text{D0}$

## Sample

50 g cellulose pulp  
 780 mL dist. water  
 100 mL  $c(\frac{1}{5}\text{KMnO}_4) = 0.1 \text{ mol/L}$   
 100 mL  $c(\frac{1}{2}\text{H}_2\text{SO}_4) = 4 \text{ mol/L}$   
 20 mL dist. water  
 35 mL  $w(\text{KI}) = 0.10 (10\%)$

## Electrodes

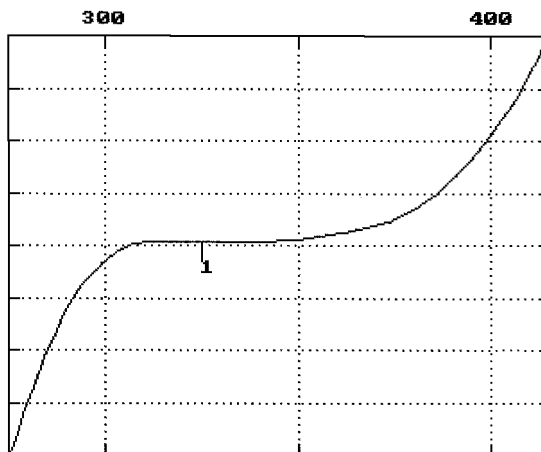
6.0418.120 combined massive Pt electrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 16:23    3
DET U              KappaNo
parameters
>titration parameters
meas.pt.density    4
min.incr.          10.0 µl
titr.rate          max. ml/min
signal drift      50 mV/min
equilibr.time     26 s
start V:          OFF
pause             0 s
dos.element:      internal D0
meas.input:       1
temperature       25.0 °C
>stop conditions
stop V:           abs.
stop V            40 ml
stop U            OFF mV
stop EP          9
filling rate     max. ml/min
>statistics
status:          OFF
>evaluation
EPC              5
EP recognition:  all
fix EP1 at U    OFF mV
pK/HNP:         OFF
>preselections
req.ident:       OFF
req.smpl size:  value
activate pulse:  OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 16:13    3
U(init)           416 mV DET U   KappaNo
EP1               19.672 ml      325 mV
p                 59.3
KappaNo           0.0
stop V reached
=====
```

```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 16:13    3
start V           0.000 ml DET U   KappaNo
5.0 ml/div        dU=50.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 16:26    3
DET U              KappaNo
>calculations
p=(C02-EP1)*C04/C05;1;
KappaNo=RS1*C01*C06/C00/C03;1;
C00=              13.0051
C01=              1.020
C02=              49.339
C03=              21.519
C04=              0.2
C05=              0.1
C06=              100
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-03      time 16:27    3
U(init)           416 mV DET U   KappaNo
smpl size         13.0051 g
EP1               19.672 ml      325 mV
p                 59.3
KappaNo           21.6
stop V reached
-----
```

### Remarks

- The Kappa number is the consumption of  $c(^{1/5}\text{KMnO}_4) = 0.1 \text{ mol/L}$  in mL. The results are corrected to the consumption of 50% of the permanganate solution added.
- Determine the dry content of the sample: Dry sample at 105 °C for app. 90 minutes. Calculate the dry content in % and enter it as calculation value C03.
- Weigh in an appropriate amount of sample. Add 780 mL of dist.water and defibrate it with the stirrer. Add the mixture of 100 mL  $c(^{1/5}\text{KMnO}_4) = 0.1 \text{ mol/L}$  und 100 mL  $c(^{1/2}\text{H}_2\text{SO}_4) = 4 \text{ mol/L}$  as quickly as possible and rinse with 20 mL dist. water. Add 35 mL  $w(\text{KI}) = 0.10$  (10%) after a waiting time of exactly 10 minutes to stop the reaction. Titrate immediately.  
Treat the blank sample in the same way as above.  
The waiting time may be programmed in the Titrimo; the additions can be carried out automatically using a TIP.
- **Calculations:**  
 $p$  = consumption of  $c(^{1/5}\text{KMnO}_4) = 0.1 \text{ mol/L}$  in mL  
 KappaNo = Kappa number  
 C01 = factor for correction to a 50 % permanaganate consumption, see table below (p.e. 59.3  $\Rightarrow$  1.020)  
 C02 = mL of  $c(^{1/2}\text{Na}_2\text{S}_2\text{O}_3) = 0.2 \text{ mol/L}$  consumption of blank sample  
 C03 = dry content in % (p.e. 21.519)  
 C04 = concentration of  $c(^{1/2}\text{Na}_2\text{S}_2\text{O}_3) = 0.2 \text{ mol/L}$  (0.2)  
 C05 = factor for percent of permanaganate consumption (0.1)  
 C06 = factor to compensate for entry of dry content in % (100)
- C01 can only be entered after the titration, because the  $p$  value is necessary. Recalculate and print another full report.
- Table for correction of 50% of the permanganate solution:

p	0	1	2	3	4	5	6	7	8	9
30	0.958	0.960	0.962	0.964	0.966	0.968	0.970	0.973	0.975	0.977
40	0.979	0.981	0.983	0.985	0.987	0.989	0.991	0.994	0.996	0.998
50	1.000	1.002	1.004	1.006	1.009	1.011	1.013	1.015	1.017	1.019
60	1.022	1.024	1.026	1.028	1.030	1.033	1.035	1.037	1.039	1.042
70	1.044									

### Literature

- TAPPI T236 os-76

# Analysis of Spinning Bath

## Reagents

c(NaOH) = 0.1 mol/L; D0

## Sample

0.1 mL c(ZnSO<sub>4</sub>) = 0.1 mol/L in c(H<sub>2</sub>SO<sub>4</sub>) = 1 mol/L  
25 mL dist. water

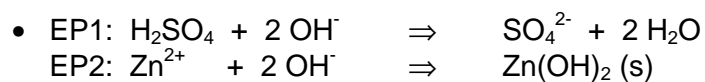
## Electrodes

6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-10-05      time 17:45  10
SET pH              Spinning
parameters
>SET1
  EP at pH          5.50
  dynamics          3
  max.rate          5 ml/min
  min.rate          1 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at pH          9.50
  dynamics          4
  max.rate          1 ml/min
  min.rate          1 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>titration parameters
  titr.direction:   auto
  pause 1           0 s
  start V:          OFF
  pause             0 s
  extr.time         0 s
  dos.element:     internal D0
  meas.input:       1
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V            99.99 ml
  filling rate      max. ml/min
>statistics
  status:           OFF
>preselections
  conditioning:     OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----

'fm
736 GP Titrimo      OP1/103  736.0010
date 95-10-05      time 17:45  10
SET pH              Spinning
>calculations
H2SO4=EP1*C01*C02/C04/C00;2;g/l
ZnSO4=(EP2-EP1)*C01*C03/C04/C00;2;g/l
C00=                0.1
C01=                0.1
C02=                98.93
C03=                161.23
C04=                2
-----
```

**Remarks**

- **Calculations:**

H2SO4 = concentration of  $\text{H}_2\text{SO}_4$  in g/L

ZnSO4 = concentration of  $\text{ZnSO}_4$  in g/L

C01 = concentration of titrating agent (0.1 mol/L)

C02 = molecular mass of  $\text{H}_2\text{SO}_4$  (98.08 g/mol)

C03 = molecular mass of  $\text{ZnSO}_4$  (161.23 g/mol)

C04 = factor for "normality" (2)

---

**Literature**

# 2-Aminophenol

## Reagents

c(NaNO<sub>2</sub>) = 0.2 mol/L; D0

## Sample

app. 0.3 ... 0.35 g sample (2-aminophenol)  
 10 mL w(HBr) = 0.20 (20%)  
 30 mL dist. water

## Electrodes

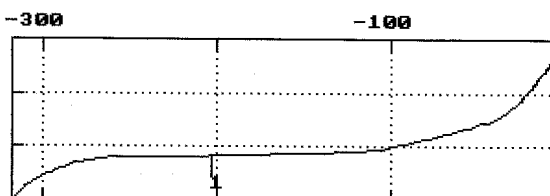
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
parameters
>titration parameters
  V step            0.10 ml
  titr.rate         max. ml/min
  signal drift      OFF mV/min
  equilibr.time     15 s
  start V:         OFF
  pause            30 s
  dos.element:     internal D0
  meas.input:      1
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           17 ml
  stop U           OFF mV
  stop EP         9
  filling rate    max. ml/min
>statistics
  status:         OFF
>evaluation
  EPC            30 mV
  EP recognition: all
  fix EP1 at U   OFF mV
  pK/HNP:       OFF
>preselections
  req.ident:      OFF
  req.smpl size: value
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
U(init)           125 mV MET U      Diazo
smpl size         0.3188 g   id#1    09120
EP1              14.396 ml   -203 mV
Content           98.56 %
stop V reached
=====
```

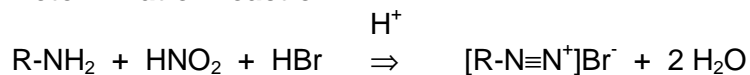
```
'cu
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:20    2
start V          10.000 ml MET U      Diazo
2.0 ml/div       dU=100.0 mV/div
```



```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-10-12      time 18:26    2
MET U              Diazo
>calculations
Content=EP1*C01*C02*C03/C00;2;%
C00=              0.3188
C01=              109.13
C02=              0.1
C03=              0.2
-----
```

**Remarks**

- **Determination reaction:**



- **Calculations:**

Content = content of 2-aminophenol in %

C01 = molecular mass of 2-aminophenol (109.13 /mol)

C02 = factor for conversion mL to L, and for % (0.001\*100=0.1)

C03 = concentration of titrating agent (0.2 mol/L)

- Instead of the Pt Titrode a 6.0420.100 combined Pt electrode can be used.
- 

**Literature**

- Metrohm Application Bulletin No. 228: Diazotisation titrations

# KF Titer Determination with Sodium Tartrate

## Reagents

HYDRANAL® Titrant 5; D0

## Sample

20 -30 mL HYDRANAL® Solvent, conditioned to complete dryness  
 app. 200 mg di-Sodium tartrate dihydrate  
 Water 15.66 ±0.05%

## Electrodes

6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-11      time 15:13   30
KFT Ipol           TarTiter
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr. min. µl
  stop crit:       drift
  stop drift        20 µl/min
>titration parameters
  titr.direction:  -
  pause 1          0 s
  start V:         OFF
  pause           0 s
  extr.time        0 s
  dos.element:     internal D0
  I(pol)           50 µA
  electrode test:  OFF
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:         ON
  mean            n= 5
  res.tab:        original
>preselections
  conditioning:   ON
  display drift:  ON
  drift corr:     OFF
  req.ident:      OFF
  req.smpl size:  value
  activate pulse: OFF
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-11      time 15:13   30
KFT IpolTarTiter
smpl size          0.19731 g
EP1                7.021 ml
Titer              4.4009 mg/ml
                  mean( 5)  +/-s      s/%
Titer              4.4037  0.00368 mg/ml  0.08
=====
```

```
'st
736 GP Titrimo      OP1/103  736.0010
date 95-09-11      time 15:13
statistics
## Titer
1 4.4035
2 4.4024
3 4.4100
4 4.4015
5 4.4009
mean( 5)  +/-s      s/%
Titer     4.4037  0.00368 mg/ml  0.08
-----
```

```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-11      time 15:13   30
KFT Ipol           TarTiter
>calculations
Titer=C00/EP1*C01;4;mg/ml
C00=                0.19731
C01=                156.6
-----
```

**Remarks**

- **Calculations:**  
Titer = Titer of HYDRANAL® Titrant 5  
 $C01 = \text{Water content} * 10 (156,6)$
- **Common variables**  
 $C39 = MN1$
- Mean from 5 determinations.
- Reweigh the sodium tartrate in a weighing boat.
- Adjust the calculation value C01 according to the unit of your sample weight:  
 $C00 \text{ in g} \quad C01=156.6$   
 $C00 \text{ in mg} \quad C01=0.1566$

---

**Literature**

- Water determination by Karl Fisher titration, G. Wieland, GIT Verlag, Darmstadt (Germany)
- HYDRANAL®, practical course, Water reagents according to Eugen Scholz, Riedel de Haën, Seelze (Germany)
- Metrohm Application Bulletin No. 77: KF water determination

# KF Titer Determination with Water

## Reagents

HYDRANAL® Composite 5; D0

## Sample

20 -30 mL methanol, conditioned to complete dryness  
10 µL dist. water

## Electrodes

6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 11:29   25
KFT Ipol           H2OTiter
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr.  min. µl
  stop crit:       drift
  stop drift       20 µl/min
>titration parameters
  titr.direction:  -
  pause 1         0 s
  start V:        OFF
  pause          0 s
  extr.time      0 s
  dos.element:   internal D0
  I(pol)         50 µA
  electrode test: OFF
  temperature    25.0 °C
>stop conditions
  stop V:         abs.
  stop V         99.99 ml
  filling rate    max. ml/min
>statistics
  status:        ON
  mean          n= 5
  res.tab:      original
>preselections
  conditioning:  ON
  display drift: ON
  drift corr:   OFF
  req.ident:    OFF
  req.smpl size: value
  activate pulse: OFF
-----
```

```
'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 11:29   25
KFT IpolH2OTiter
smpl size         0.010 g
EP1               2.047 ml
Titer             4.8852 mg/ml
                 mean( 5) +/-s      s/%
Titer             4.8881 0.00573 mg/ml 0.12
=====
```

```
'st
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 11:29
statistics
##   Titer
1   4.8876
2   4.8924
3   4.8804
4   4.8948
5   4.8852
                 mean( 5) +/-s      s/%
Titer             4.8881 0.00573 mg/ml 0.12
-----
```

```
'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 11:29   25
KFT Ipol           H2OTiter
>calculations
Titer=C00/EP1*C01;4;mg/ml
C00=                0.010
C01=                1000
-----
```

**Remarks**

- **Calculations:**  
Titer = Titer of HYDRANAL® Composite 5  
C01 = Factor (1000 if water is used as standard; if methanol is used, enter its water content in mg/mL)
- **Common variables**  
C39 = MN1
- Mean from 5 determinations.
- Water or methanol standard can be injected either with a microliter syringe or with any syringe and reweighed.
- Adjust the calculation value C01 according to your sample size:  
*Water*  
C00 in g    C01=1000  
C00 in uL    C01=Density of H<sub>2</sub>O [mg/mL] ≈ 1  
*Methanol standard*  
C00 in mL    C01=Content of methanol [mg/mL]  
C00 in ul    C01=0.001 \* content of methanol [mg/mL]

---

**Literature**

- Water determination by Karl Fisher titration, G. Wieland, GIT Verlag, Darmstadt (Germany)
- HYDRANAL®, practical course, Water reagents according to Eugen Scholz, Riedel de Haën, Seelze (Germany)
- Metrohm Application Bulletin No. 77: KF water determination

# Blank Determination of Methanol

## Reagents

HYDRANAL® Composite 5; D0

## Sample

20 -30 mL methanol, conditioned to complete dryness  
1.000 mL methanol (extraction medium)

## Electrodes

6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 14:00   41
KFT Ipol           Blank_KF
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr.  min. µl
  stop crit:       drift
  stop drift        20 µl/min
>titration parameters
  titr.direction:   -
  pause 1           0 s
  start V:          OFF
  pause            0 s
  extr.time         0 s
  dos.element:     internal D0
  I(pol)           50 µA
  electrode test:   OFF
  temperature       25.0 °C
>stop conditions
  stop V:           abs.
  stop V           99.99 ml
  filling rate      max. ml/min
>statistics
  status:           ON
  mean              n= 3
  res.tab:          original
>preselections
  conditioning:     ON
  display drift:    ON
  drift corr:       OFF
  req.ident:        OFF
  req.smpl size:    OFF
  activate pulse:   OFF
-----

'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 13:56   40
KFT IpolBlank_KF
EP1                 0.034 ml
Blank               0.0340 ml
                   mean( 2)  +/-s      s/%
Blank               0.0340  0.00000 ml  0.00
=====

'st
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 13:56
statistics
## Blank
1 0.0340
2 0.0340
                   mean( 2)  +/-s      s/%
Blank               0.0340  0.00000 ml  0.00
-----

'de
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 14:00
KFT Ipol           Blank_KF
def
>formula
  Blank=EP1
  RS1 text          Blank
  RS1 decimal places 4
  RS1 unit:         ml
>silco calculations
  match id:         OFF
>common variables
  C38=MN1
>report
  report:full;
>mean
  MN1=RS1
>temporary variables
-----
```

**Remarks**

- **Calculations:**  
Blank = Blank of methanol (extraction medium)
- **Common variables**  
C38 = MN1
- Mean from 2-3 determinations.
- This method can be generally used for KF blank determinations. For work with a KF Oven, you need an extraction time. Enter it in key <parameters>, >titration parameters.
- Use the same control parameters for the blank determination as for the sample titrations.

---

**Literature**

- Water determination by Karl Fisher titration, G. Wieland, GIT Verlag, Darmstadt (Germany)
- HYDRANAL®, practical course, Water reagents according to Eugen Scholz, Riedel de Haën, Seelze (Germany)
- Metrohm Application Bulletin No. 77: KF water determination

# Water Determination in Paper

## Reagents

HYDRANAL® Composite 5; D0

## Sample

20 -30 mL methanol, conditioned to complete dryness  
 1.000 mL sample solution  
 extract app. 1.0g of paper (cut in small pieces) in  
 50 mL methanol (extraction medium)

## Electrodes

6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
736 GP Titrino      OP1/103  736.0010
date 95-09-12      time 14:43  50
KFT Ipol           KF-Blank
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr.  min. µl
  stop crit:       drift
  stop drift        20 µl/min
>titration parameters
  titr.direction:  -
  pause 1          0 s
  start V:         OFF
  pause            0 s
  extr.time        0 s
  dos.element:    internal D0
  I(pol)           50 µA
  electrode test:  OFF
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:          ON
  mean             n= 3
  res.tab:         original
>preselections
  conditioning:    ON
  display drift:   ON
  drift corr:      OFF
  req.ident:       OFF
  req.smpl size:   all
  activate pulse:  OFF
```

```
'fr
736 GP Titrino      OP1/103  736.0010
date 95-09-12      time 14:42  50
KFT IpolKF-Blank
smpl size          0.02112 g
EP1                0.332 ml
Water              6.90 %
Titer              4.8881 mg/ml
Blank              0.0340 ml
                  mean( 3)  +/-s      s/%
Water              6.88      0.029 %    0.42
=====
```

```
'st
736 GP Titrino      OP1/103  736.0010
date 95-09-12      time 14:42
statistics
## Water
1 6.90
2 6.85
3 6.90
                  mean( 3)  +/-s      s/%
Water              6.88      0.029 %    0.42
-----
```

```
'fm
736 GP Titrino      OP1/103  736.0010
date 95-09-12      time 14:43  50
KFT Ipol           KF-Blank
>calculations
Water=(EP1-C38)*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
Blank=C38;4;ml
C00=                0.02112
C01=                0.1
C02=                1
C38=                0.0340
C39=                4.8881
-----
```

**Remarks**

- **Calculations:**  
 Water = Content of water in paper in %  
 C00 = sample size / aliquot (1.0559 g / 50 mL = 0.021118 g/mL)  
 C01 = Factor for % (0.1)  
 C02 = Divisor (1)  
 C38 = Blank value in mL of "Blank\_KF" method (0.0340 mL ??)  
 C39 = Titer of HYDRANAL® Composite 5
- Mean from 3 determinations.
- This method can be generally used for KF titrations with blank values. For work with a KF Oven, you need an extraction time. Enter it in key <parameters>, >titration parameters.
- Adjust the calculation values C01 and C02 according the desired result unit and your sample size:

Unit RS	Smpl size in..	C01	C02
%	g	0.1	1
%	mg	100	1
%	mL	0.1	Dens. of sample
ppm	g	1000	1
ppm	mL	1000	Dens. of sample
ppm	uL	1000 000	Dens. of sample
mg/mL	g	Dens. of sample	1
mg/mL	mL	1	1
g/L	g	Dens. of sample	1
g/L	mL	1	1
mg	1	1	1
mL	1	1	1000*Dens. H <sub>2</sub> O
mg/pc	pc	1	1

- Stir sample solution for app. ½ h to extract the water and take aliquots (see calculation).

**Literature**

- Water determination by Karl Fisher titration, G. Wieland, GIT Verlag, Darmstadt (Germany)
- HYDRANAL®, practical course, Water reagents according to Eugen Scholz, Riedel de Haën, Seelze (Germany)
- Metrohm Application Bulletin No. 77: KF water determination

# Water Determination in Honey

## Reagents

HYDRANAL® Composite 5; D0

## Sample

20 -30 mL methanol, conditioned to complete dryness  
0.1 -0.2 g Honey

## Electrodes

6.0338.100 double Pt electrode; input Pol

## Method documentation

```

'pa
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 16:39   67
KFT Ipol           KF
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr.  min. µl
  stop crit:       drift
  stop drift       20 µl/min
>titration parameters
  titr.direction:   -
  pause 1           0 s
  start V:          OFF
  pause            0 s
  extr.time        0 s
  dos.element:     internal D0
  I(pol)           50 µA
  electrode test:  OFF
  temperature      25.0 °C
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:          ON
  mean             n= 3
  res.tab:         original
>preselections
  conditioning:    ON
  display drift:   ON
  drift corr:      OFF
  req.ident:       OFF
  req.smpl size:   all
  activate pulse:  OFF
-----

'fr
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 16:38   67
KFT Ipol           KF
smpl size          0.1883 g
EP1                7.099 ml
Water              18.43 %
Titer              4.8881 mg/ml
                  mean( 3)  +/-s      s/%
Water              18.43    0.006 %    0.03
=====

'fm
736 GP Titrimo      OP1/103  736.0010
date 95-09-12      time 16:39   67
KFT Ipol           KF
>calculations
Water=EP1*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
C00=                0.1883
C01=                0.1
C02=                1
C39=                4.8881
-----

```

**Remarks**

- **Calculations:**  
 Water = Content of water in honey in %  
 C01 = Factor for % (0.1)  
 C02 = Divisor (1)  
 C39 = Titer of HYDRANAL® Composite 5
- Mean from 3 determinations.
- This method can be generally used for KF titrations without blank values. For work with a KF Oven, you need an extraction time. Enter it in key <parameters>, >titration parameters.
- Adjust the calculation values C01 and C02 according the desired result unit and your sample size:

Unit RS	Smpl size in..	C01	C02
%	g	0.1	1
%	mg	100	1
%	mL	0.1	Dens. of sample
ppm	g	1000	1
ppm	mL	1000	Dens. of sample
ppm	uL	1000 000	Dens. of sample
mg/mL	g	Dens. of sample	1
mg/mL	mL	1	1
g/L	g	Dens. of sample	1
g/L	mL	1	1
mg	1	1	1
mL	1	1	1000*Dens. H <sub>2</sub> O
mg/pc	pc	1	1

**Literature**

- Water determination by Karl Fisher titration, G. Wieland, GIT Verlag, Darmstadt (Germany)
- HYDRANAL®, practical course, Water reagents according to Eugen Scholz, Riedel de Haën, Seelze (Germany)
- Metrohm Application Bulletin No. 208: Volumetric determination of water in honey with Karl Fisher reagent.
- Metrohm Application Bulletin No. 77: KF water determination