



CH-9101 Herisau/Switzerland

Phone            ++41 71 353 85 85

Fax                ++41 71 353 89 01

E-mail            sales@metrohm.ch

Internet         http://www.metrohm.ch

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## **798 MPT Titrino**

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Applications

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8.798.2003

02.5 ks/rh



**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	2-2
p and m Value	p+m val.	2-3
Oxidizability of Waste Water	Oxidiza.	2-4
<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
Peroxide Number	Perox.No	4-2
Saponification Number	Sapon.No	4-3
Acid Number	Acid.No	4-4
Iodine Number	Iod.No	4-5
Vitamine C	Vit.C	4-6

	<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
2-Aminophenol	Diazo	5-2
Vitamine C	Vit.C	5-3
<b>6. Detergents</b>		
EDTA/NTA in Detergents	EDTA-NTA	6-1
Non-ionic Surfactants	Tenside	6-2
Cationic Surfactants with two-phase titration	Cation2p	6-3
Anionic Surfactants in Shampoo	Tens.An	6-4
Anionic Surfactants with two-phase titration	Anion2p	6-5
<b>7. Galvanics</b>		
Pure Silver Content	Silver	7-1
Metals	Metals	7-2
<b>8. Basic.Mat</b>		
Epoxy Number	Epoxy.No	8-1
Nitrating Acid	NitrAcid	8-2
Hydrogen Peroxide	H2O2	8-3
Phosphate	Phosphat	8-4
2-Aminophenol	Diazo	8-5
<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
Analysis of Spinning Bath	Spinning	9-4
2-Aminophenol	Diazo	9-5
<b>10. KF Titr.</b>		
KF Titer Determination with water	H2OTiter	10-1
Blank Determination of Methanol	Blank_KF	10-2
Water Determination in Paper	KF-Blank	10-3
Water Determination in Honey	KF	10-4

The methods from the 6.6036.000 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 6.6036.000 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.
- Connect the printer to COM1. 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 6.0431.100 **Pt Titrode** a combined Pt electrode can normally be used. The direction of the titration curve changes, e.g. if the curves goes from 400 to 100 mV with a Pt Titrode, it will run from 100 to 400 mV with a combined Pt electrode and vice versa.
- Instead of a 6.0430.100 **Ag Titrode** a combined Ag electrode can normally be used. The direction of the titration curve changes, e.g. if the curves goes from 400 to 100 mV with a Ag Titrode, it will run from 100 to 400 mV with a combined Ag electrode and vice versa

# Strong Acid

## Reagents

c(NaOH) = 0.1 mol/L

## Sample

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

## Electrodes

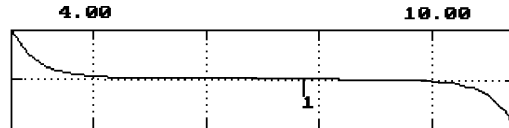
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 09:37      4
DET pH              Acid
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 09:36      4
pH(init)            2.55  DET pH      Acid
smpl size           2.00 ml
EP1                 1.996 ml          7.72
c(acid)             0.100 mol/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 09:36      4
start V             0.000 ml DET pH      Acid
2.0 ml/div          dpH=2.0/div
```



=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 09:38      4
DET pH              Acid
>calculations
c(acid)=EP1*C01*C37/C00;3;mol/l
C00=                 2.00
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  
free of carbonate

## Sample

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

## Electrodes

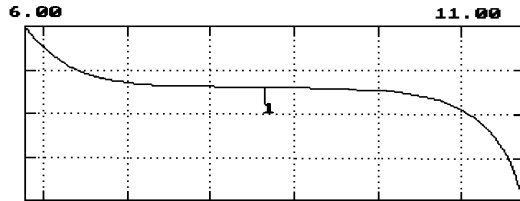
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 17:33      6
DET pH              Tit.NaOH
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 17:30      6
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
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 17:30      6
start V             12.000 ml  DET pH  Tit.NaOH
2.0 ml/div          dpH=1.0/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 17:33      6
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

## Sample

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

## Electrodes

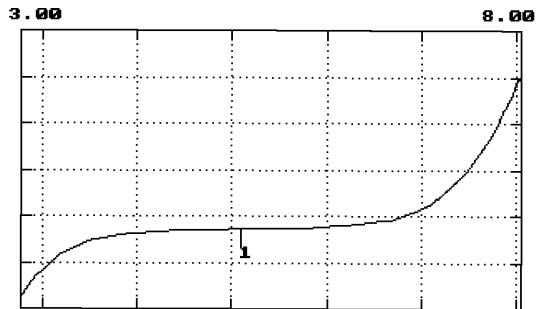
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 10:03      10
DET pH              Tit.HCl
parameters
>titration parameters
  meas.pt.density      4
  min.incr.           10.0 µl
  dos.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
  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
  limit smpl size:   OFF
  activate pulse:     OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 10:02      10
pH(init)            10.13  DET pH  Tit.HCl
smpl size           0.09959 g
EP1                 8.286 ml          5.09
Titer               0.9922
                    mean(10)  +/-s      s/%
Titer               0.9922  0.00044      0.04
stop meas.val.reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 10:02      10
start V             4.980 ml  DET pH  Tit.HCl
1.0 ml/div          dpH=1.0/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 10:04      10
DET pH              Tit.HCl
>calculations
Titer=C00*C01/C02/EP1;4;
C00=                 0.09959
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 10 determinations.
- This method may be used for the GLP validation, see Metrohm Bulletin 252.  
To shorten the titration time, 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

## 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

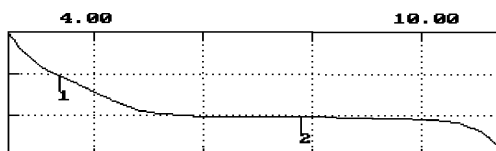
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 11:07      8
DET pH              Oxalic
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:     OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 11:04      8
pHc(init)           2.41      DET pH      Oxalic
smpl size            2.00 ml
EP1                  2.110 ml      3.63
EP2                  4.098 ml      7.77
pK1                  2.71
pK2                  4.17
Oxalic               0.103 mol/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 11:04      8
start V              0.000 ml  DET pH      Oxalic
2.0 ml/div           dpH=2.0/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-04      time 11:07      8
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  
 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 ethyl alcohol

## Electrodes

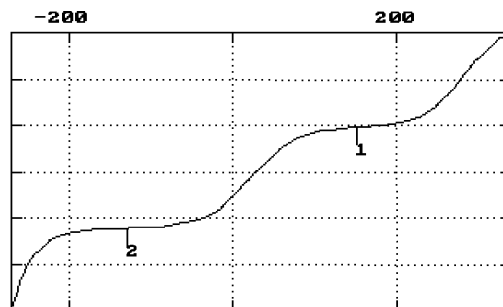
6.0103.100 pH glass electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (LiCl sat. in ethyl alcohol)

## Method documentation

```
'pa
798 MPT Titrino      15215      799.0010
date 2001-10-04     time 13:43      9
DET U               OxalicNA
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 13:38      9
U(init)             341 mV DET U   OxalicNA
smpl size           2.0 ml
EP1                 2.048 ml       152 mV
EP2                 4.221 ml       -130 mV
Oxalic              0.106 mol/l
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 13:38      9
start V             0.000 ml DET U   OxalicNA
1.0 ml/div          dU=200.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-04     time 13:44      9
DET U               OxalicNA
>calculations
Oxalic=EP2*C01/C02/C00;3;mol/l
C00=                2.0
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

## Sample

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

## Electrodes

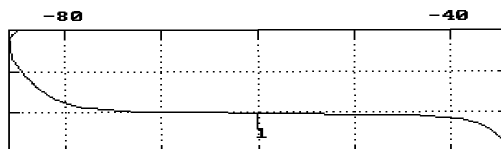
6.0430.100 Ag Titrode amalgamated; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-05     time 14:53      15
MET U                Ca++
parameters
>titration parameters
  V step              0.10 ml
  dos.rate            max. ml/min
  signal drift        OFF mV/min
  equilibr.time       10 s
  start V:            OFF
  pause               0 s
  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
  limit smpl size:  OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-05     time 14:48      15
U(init)              -82 mV MET U      Ca++
smpl size             2.0 ml
EP1                   2.043 ml          -60 mV
c(Ca++)               0.102 mol/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-05     time 14:48      15
start V              0.000 ml MET U      Ca++
1.0 ml/div           dU=10.0 mV/div
```



=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-05     time 14:53      15
MET U                Ca++
>calculations
c(Ca++)=EP1*C01/C00;3;mol/l
C00=                  2.0
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  $c(\text{KOH}) = 0.1 \text{ mol/L}$

## 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

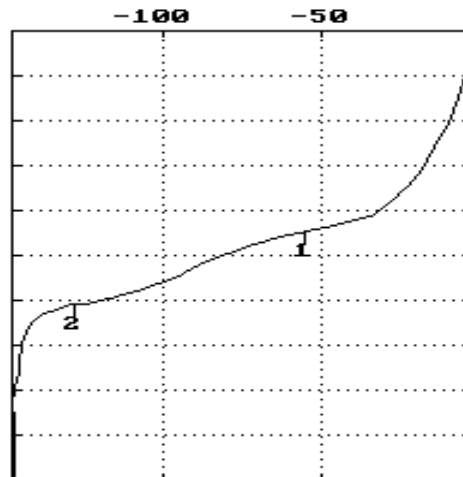
6.0504.100  $\text{Ca}^{2+}$  sensitive indicator electrode; input 1  
 6.0733.100 Ag/AgCl reference electrode ( $c(\text{KCl}) = 3 \text{ mol/L}$ )

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 10:37      2
DET U               Ca - Mg
parameters
>titration parameters
  meas.pt.density    1
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       20 mV/min
  equilibr.time      38 s
  start V:           OFF
  pause              0 s
  meas.input:        1
  temperature        25.0 C
>stop conditions
  stop V:            abs.
  stop V             10.0 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
  limit smpl size:  OFF
  activate pulse:    OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 10:37      2
U(init)             -13 mV DET U      Ca - Mg
smpl size           100 ml
EP1                 4.490 ml           -55 mV
EP2                 6.118 ml           -128 mV
Ca++                2.24 mmol/l
Mg++                0.81 mmol/l
Total               3.06 mmol/l
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 10:37      2
start V             0.000 ml DET U      Ca - Mg
1.0 ml/div          dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 10:37      2
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

## 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

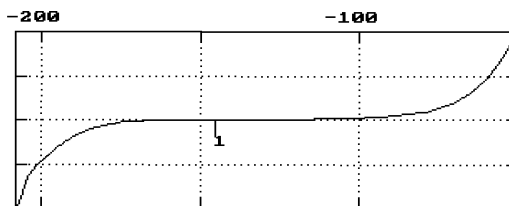
6.0502.140 Cu<sup>2+</sup> sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (KNO<sub>3</sub> sat.)

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 15:39      3
DET U                Metals
parameters
>titration parameters
  meas.pt.density    2
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       20 mV/min
  equilibr.time      38 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-1-03      time 15:30      3
U(init)              -50 mV DET U      Metals
smpl size            2.0 ml
EP1                  2.006 ml          -145 mV
Content              6.56 g/l
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 15:30      3
start V              0.000 ml DET U      Metals
1.0 ml/div           dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 15:40      3
DET U                Metals
>calculations
Content=EP1*C01*C02/C00;2;g/l
C00=                  2.0
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  $\text{NH}_4\text{Cl}$  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

# Iodine

## Reagents

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

## 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

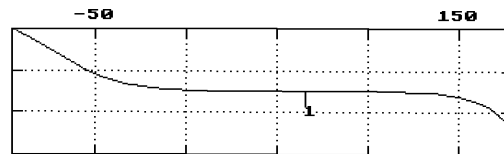
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-03      time 16:33      6
DET U                Iodine
parameters
>titration parameters
  meas.pt.density      4
  min.incr.           10.0 µl
  dos.rate             max. ml/min
  signal drift        20 mV/min
  equilibr.time       38 s
  start V:            OFF
  pause               0 s
  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
  limit smpl size:    OFF
  activate pulse:     OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-03      time 16:31      6
U(init)              -95 mV DET U      Iodine
smpl size             3.0 ml
EP1                   2.997 ml          65 mV
c(KI3)                0.0499 mol/l
stop V reached
=====

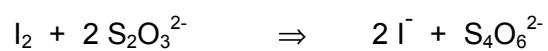
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-03      time 16:31      6
start V              0.000 ml DET U      Iodine
2.0 ml/div           dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-03      time 16:33      6
DET U                Iodine
>calculations
c(KI3)=EP1*C01/C02/C00;4;mol/l
C00=                  3.0
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

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

## 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

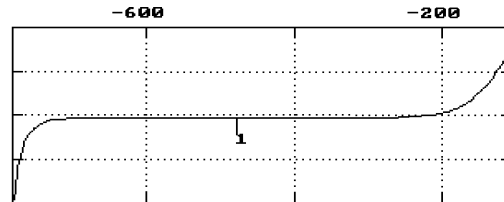
6.0431.100 Pt Titrode; input 1

## Method documentation

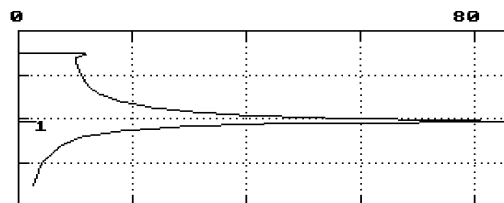
```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 18:03      3
DET U                Iron(II)
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         10 mV/min
  equilibr.time        52 s
  start V:             abs.
  start V              0.5 ml
  dos.rate             max. ml/min
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:     OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 18:02      3
U(init)              -136 mV DET U  Iron(II)
smpl size            2.0 ml
EP1                  2.065 ml          -476 mV
Fe++                  5.77 g/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 18:02      3
start V              0.500 ml DET U  Iron(II)
1.0 ml/div           dU=200.0 mV/div
```



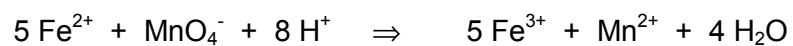
```
'dv
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 18:02      3
start V              0.500 ml DET U  Iron(II)
1.0 ml/div           ERC=20.0/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-03     time 18:03      3
DET U                Iron(II)
>calculations
Fe++=EP1*C01*C02/C00;2;g/l
C00=                  2.0
C01=                  0.1
C02=                  55.85
-----
```

**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

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

## 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

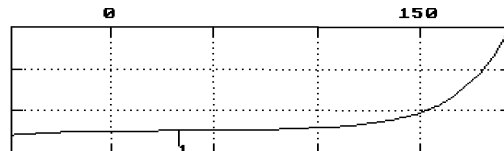
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-02     time 09:41      3
DET U               Chloride
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-02     time 09:40      3
U(init)             192 mV DET U   Chloride
smpl size           5.0 ml
EP1                 4.969 ml          33 mV
Chloride            0.35 %
NaCl                 5.81 g/l
stop #EP reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-02     time 09:40      3
start V             0.000 ml DET U   Chloride
2.0 ml/div          dU=50.0 mV/div
```



=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-02     time 09:41      3
DET U               Chloride
>calculations
Chloride=EP1*C01*C02*C03/C00;2;%
NaCl=EP1*C01*C04/C00;2;g/l
C00=                 5.0
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

## 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

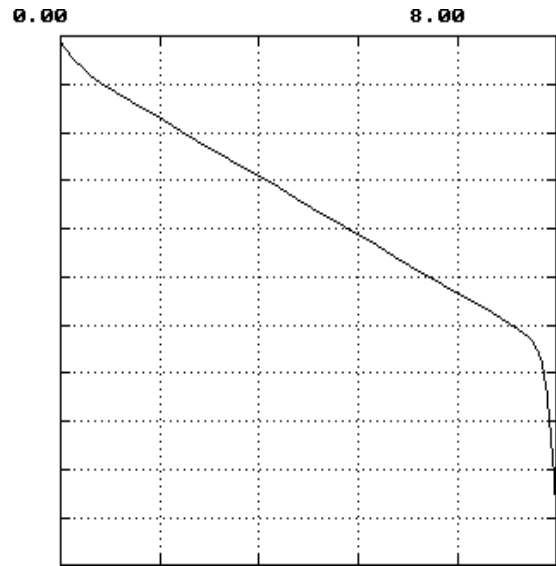
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-01     time 11:49      7
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 2          0 s
  extr.time        0 s
  meas.input:      1
  temperature      25.0 C
  time interval    2 s
>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
  limit smpl size: OFF
  activate pulse:  OFF
-----
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-01     time 11:49      7
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
-----
```

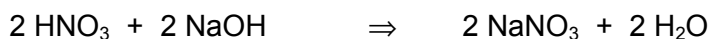
```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-01     time 11:48      7
pHc(init)          1.94      SET pH  Phosphat
smpl size          0.07798 g
EP1                9.992 ml      4.21
P                  19.84 %
P2O5               45.47 %
PO4                60.85 %
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-01     time 11:48      7
                    SET pH  Phosphat
10.0 s/div          dV=2.0 ml/div
```



### 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.
- For automatic curve output add in <DEF>, >report "curve".

### Literature

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

# Chloride in Tap Water

## Reagents

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

## Sample

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

## Electrodes

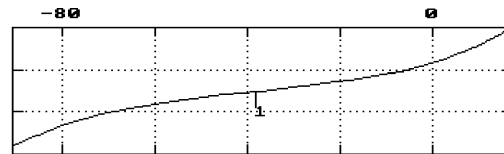
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-09-28      time 11:31      6
DET U                Chloride
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-09-28      time 11:30      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
798 MPT Titrino      15215      798.0010
date 2001-09-28      time 11:30      6
start V              0.000 ml DET U      Chloride
1.0 ml/div           dU=20.0 mV/div
```



=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-09-28      time 11:32      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)
- For the determination of samples with a lower content of chloride it is recommended to use an Ag Titrode with Ag<sub>2</sub>S coating.

---

**Literature**

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

# Boric Acid

## Reagents

c(NaOH) = 0.1 mol/L

## 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

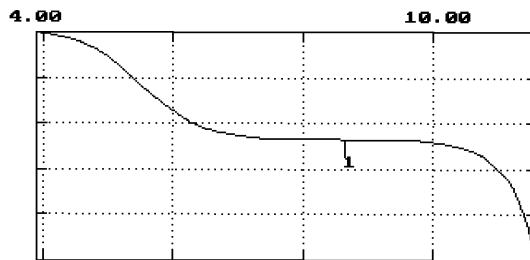
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-09-28     time 13:59      9
DET pH              Bor.AcId
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-09-28     time 13:55      9
pH(init)            3.90   DET pH   Bor.AcId
smpl size           5.0 ml
EP1                 4.719 ml           8.66
Bor                 1021.6 mg/l
stop V reached
=====

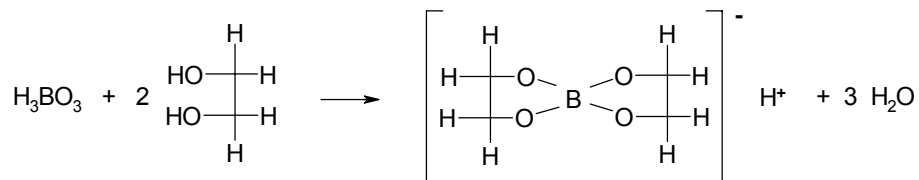
'cu
798 MPT Titrino      15215      798.0010
date 2001-09-28     time 13:55      9
start V             0.000 ml DET pH Bor.AcId
2.0 ml/div          dpH=2.0/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-09-28     time 13:59      9
DET pH              Bor.AcId
>calculations
Bor=EP1*C01*C02*C37/C00;1;mg/l
C00=                 5.0
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

## Sample

100 mL tap water

## Electrodes

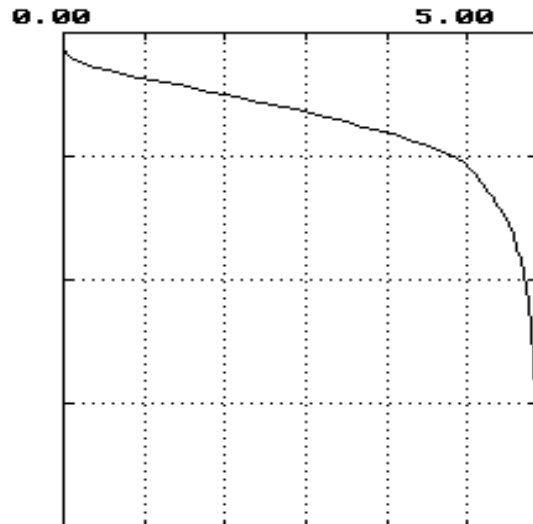
6.0253.000 Aquatrode Plus; input 2

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 16:36    11
SET pH              p+m val.
parameters
>SET1
  EP at pH          8.20
  dynamics          2
  max.rate          10.0 ml/min
  min.rate          5.0 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at pH          4.30
  dynamics          3
  max.rate          10.0 ml/min
  min.rate          5.0 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
  meas.input:       2
  temperature       25.0 C
  time interval     2 s
>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
  limit smpl size:  OFF
  activate pulse:   OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 16:36    11
pH(init)           7.22  SET pH    p+m val.
smpl size          100 ml  id 1   Metrohm
id 2               Herisau id 3   Tapwater
EP1                0.000 ml  7.22
EP2                5.885 ml  4.29
p value            0.00 mmol/l
m value            5.89 mmol/l
=====
```

```
'cu
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 16:36    11
                    SET pH    p+m val.
50.0 s/div          dV=1.0 ml/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2001-10-09     time 16:36    11
SET pH              p+m val.
>calculations
p value=EP1*C01*C36*C02/C00;2;mmol/l
m value=EP2*C01*C36*C02/C00;2;mmol/l
C00=                100
C01=                0.1
C02=                1000
C36=                1.0
=====
```

**Remarks**

- Values for the acid capacity of water.  
p value = value of Phenolphthalein  
m value = value of Methyl orange
- **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  
C00 = Sample size in (100 mL)  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = conversion factor from mol/L to mmol/L (1000)  
C36 = titer of the titrating agent (1.0)  
common variable of "Tit.HCl"
- 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.
- For automatic curve output add in <DEF>, >report "curve".

---

**Literature**

- DIN 38 409, Teil 7 (1979)

# Oxidizability of Waste Water

## Reagents

$c(\frac{1}{5}\text{KMnO}_4) = 0.01 \text{ mol/L}$

## Sample

25 mL waste water  
 5 mL  $w(\text{H}_2\text{SO}_4) = 0.35$  (35%)  
 75 mL dist. water  
 15 mL  $c(\frac{1}{5}\text{KMnO}_4) = 0.01 \text{ mol/L}$   
 15 mL  $c(\frac{1}{2}\text{Oxalic acid}) = 0.01 \text{ mol/L}$

## Electrodes

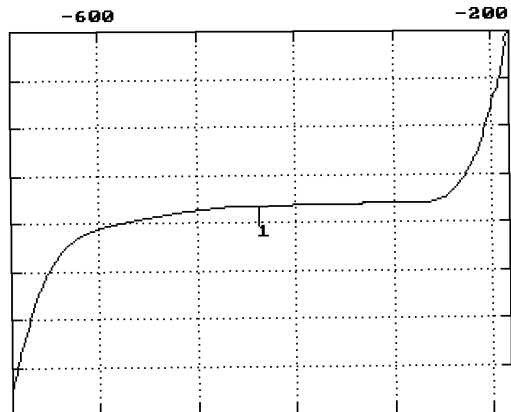
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-09-27     time 16:00      2
MET U                Oxidiza.
parameters
>titration parameters
V step               0.10 ml
dos.rate             max. ml/min
signal drift         20 mV/min
equilibr.time        38 s
start V:             OFF
pause                0 s
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
limit smpl size:     OFF
activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-09-27     time 15:58      2
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
798 MPT Titrino      15215      798.0010
date 2001-09-27     time 15:58      2
start V              0.000 ml MET U  Oxidiza.
1.0 ml/div           dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-09-27     time 16:00      2
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 = molecular mass of  $\text{KMnO}_4$  \* concentration of titrating agent / normality ( $158 * 0.01 / 5 = 0.316$  g/L)  
C02 = conversion from g to mg (1000)

---

**Literature**

- Deutsche Einheitsverfahren zur Wasseruntersuchung  
Kapitel H4, Abschnitt 1

# Total Acid Number (TAN)

## Reagents

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

## Sample

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

## Electrodes

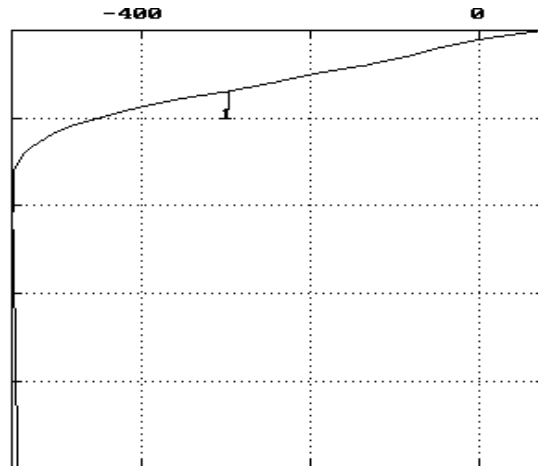
6.0229.100 Solvotrode; input 1  
 electrolyte c(TEABr) = 0.4 mol/L in ethylene glycol

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-01-28    time 16:44    9
MET U              TAN
parameters
>titration parameters
  V step            0.10 ml
  dos.rate          max. ml/min
  signal drift      20 mV/min
  equilibr.time     26 s
  start V:          OFF
  pause            0 s
  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: last
  fix EP1 at U   OFF mV
  pK/HNP:        OFF
>preselections
  req.ident:      id1
  req.smpl size:  all
  limit smpl size: OFF
  activate pulse: OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-01-28    time 16:44    9
U(init)            79 mV MET U      TAN
smpl size          1.6187 g      id#1  S3
EP1                0.693 ml      -298 mV
TAN                2.40 mg/g
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-01-28    time 16:44    9
start V           0.000 ml MET U      TAN
1.0 ml/div        dU=200.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-01-28    time 16:44    9
MET U              TAN
>calculations
TAN=(EP1-C31)*C01*C02*C30/C00;2;mg/g
C00=                1.6187
C01=                0.1
C02=                56.106
C30=                0.9958
C31=                0
=====
```

**Remarks**

- **Calculations:**

TAN = acid of sample in mg KOH per g of sample  
C01 = concentration of titrating agent in mol/L (0.1)  
C02 = molecular mass of KOH in g/mol (56.106)  
C30 = titer of titrating agent (0.9958)  
C31 = blank value in mL (0)

- **Standard for the titer determination:**

c(benzoic acid) = 0.100 mol/L in ethanol  
(1.220 g benzoic acid in 100 mL ethanol)

- **Handling of the electrode:**

It is recommended to use TEA-Br in ethylene glycol (c = 0.4 mol/L). The reason for this is that the alkali error increases a lot in non aqueous solvents. The curve flattens, the jump becomes lower and the reproducibility decreases when there are lithium or other alkali ions present.

**Preparation of the electrode:**

The Solvotrode is dipped in dist water up to the diaphragm over night, so that there can be build a new swell film on the glass membrane. After this 'conditioning' let some elektrolyte out of the electrode by opening the diaphragm for a short time and close it again. Rinse the electrode with ethanol (50...70%) and dry it with a soft towel. Between the titrations the electrode should be rinsed with ethanol (50...70%).

- If it is not possible to reach good curves using the solvotrode, the three electrodes technique can be very useful.

---

**Literature**

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

# Total Base Number (TBN)

## Reagents

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

## Sample

app. 1.0 g of used motor oil  
50 mL solvent  
toluene : acetic acid (1:1)

## Electrodes

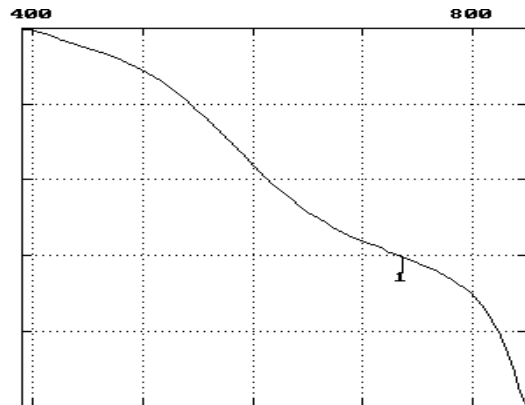
6.0229.100 Solvotrode; input 1  
electrolyte LiCl sat. in ethanol (6.2312.000)  
electrolyte : ethanol (1:1);  $\cong 2 \text{ mol/L}$  LiCl in ethanol

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-01-30    time 14:49      2
DET U              TBN
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10 µl
  dos.rate             max. ml/min
  signal drift         OFF mV/min
  equilibr.time        30 s
  start V:             OFF
  pause                60 s
  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:      last
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       value
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-01-30    time 14:49      2
U(init)              381 mV DET U      TBN
smpl size             1.5916 g      id#1  S3
EP1                   3.015 ml      737 mV
TBN                   10.67 mg/g
stop V reached
-----
```

```
'cu
798 MPT Titrino      01105  798.0010
date 2002-01-30    time 14:49      2
start V              0.000 ml DET U      TBN
1.0 ml/div           dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-01-30    time 14:49      2
DET U              TBN
>calculations
TBN=(EP1-C31)*C01*C02*C30/C00;2;mg/g
C00=                1.5916
C01=                  0.1
C02=                 56.106
C30=                 1.0041
C31=                  0
-----
```

### Remarks

- **Calculations:**

TBN = total base number of the sample in mg KOH per g of sample

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

C02 = molecular mass of KOH (56.106 g/mol)

C30 = titer of titrating agent (1.0041)

C31 = blank value in mL (0)

- **Standard for the titer determination:**

$c(\text{CH}_3\text{COONa}) = 0.100 \text{ mol/L}$  in glacial acetic acid

(0.820 g water free  $\text{CH}_3\text{COONa}$  in 100 mL glacial acetic acid)

- **Handling of the electrode:**

It is recommended to use LiCl in ethanol (6.2312.000) diluted to  $c = 2 \text{ mol/L}$  with ethanol (1:1) as electrolyte. This diluted solution is easier to handle, has a better electric conductivity and does not agglutinate the diaphragm.

The solvents decrease the amount of water in the membrane. This will cause flat, not reproducible curves with ghost end points.

**Preparation of the electrode:**

The Solvotrode is dipped in the solvent mixture up to the diaphragm over night, so that it get used to this kind of solution.

After this 'conditioning' let some electrolyte out of the electrode by opening the diaphragm for a short time and close it again.

For rinsing you should sway the electrode for a short time in glacial acetic acid.

Between the titrations the electrode is swayed in glacial acetic acid for cleaning. During the determination of some pharmaceutical products insoluble precipitations can occur. In this case the electrode will be rinsed first with water, then with ethanol and finally with glacial acetic acid.

- If it is not possible to reach good curves using the solvotrode, the three electrodes technique can be very useful.

### Literature

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

# Bromine Index

## Reagents

$c(\text{BrO}_3^-/\text{Br}^-) = 0.02 \text{ mol/L}$

## Sample

50  $\mu\text{L}$  cyclohexene  $\cong$  10 % in diethylcarbonate  
50 mL solvent mixture

## Electrodes

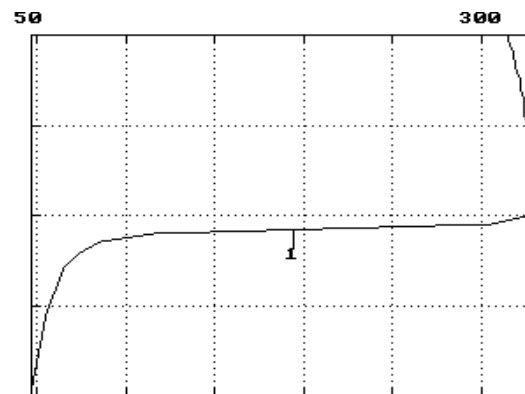
6.0308.100 double Pt electrode; input Pol  
polarized  $I_{\text{pol}} = 1 \mu\text{A}$   
Voltametric indication

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-02-12    time 17:12      5
MET Ipol           Br-Index
parameters
>titration parameters
  V step           0.05 ml
  dos.rate         max. ml/min
  signal drift     OFF mV/min
  equilibr.time    20 s
  start V:         abs.
  start V          1 ml
  dos.rate         3 ml/min
  pause           60 s
  I(pol)          1  $\mu\text{A}$ 
  electrode test:  OFF
  temperature     5.0  $^\circ\text{C}$ 
>stop conditions
  stop V:         abs.
  stop V          3 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:      id1
  req.smpl size:  all
  limit smpl size: OFF
  activate pulse: OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-02-12    time 17:13      5
U(init)            787 mV MET Ipol  Br-Index
smpl size          0.02 g
EP1                2.0790 ml          194 mV
Br-Index          16031.5
stop V reached
=====
```

```
'cu
798 MPT Titrino      01105  798.0010
date 2002-02-12    time 17:12      5
start V           1.0000 ml MET Ipol  Br-Index
0.5 ml/div        dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-02-12    time 17:13      5
MET Ipol           Br-Index
>calculations
Br-Index=(EP1-C31)*C01*C02*C30/C00;1;mg
C00=               0.02
C01=               0.02
C02=               7990
C30=               0.9651
C31=               0
=====
```

---

**Remarks**

- Bromine index is the number of mg of bromine consumed per 100 g of sample.

**Solvent mixture I (for aliphatic hydrocarbons):**

714 mL glacial acetic acid  
 134 mL carbon tetrachloride  
 134 mL methanol  
 18 mL  $w(\text{H}_2\text{SO}_4) = 0.2$  (20%)

**Solvent mixture II (for aromatic hydrocarbons):**

714 mL glacial acetic acid  
 134 mL 1-methyl-2-pyrrolidone  
 134 mL methanol  
 18 mL  $w(\text{H}_2\text{SO}_4) = 0.2$  (20%)

If possible, one should refrain from using chlorinated solvents. Our investigations have shown that carbon tetrachloride and 1,1,1-trichloroethane can be replaced by diethyl carbonate.

**Titration agent:**

0.02 N bromide-bromate solution [ $c(1/6 \text{KBrO}_3) = 0.02 \text{ mol/L}$ ]:  
 2.04 g KBr and 0.556 g  $\text{KBrO}_3$  are separately dissolved in dist. water.  
 Pour both solutions into a 1000 mL volumetric flask and fill to the mark with dist. water.

**Test solution:**

Cyclohexene can be used as a test substance. It has a theoretical bromine index of 194500 mg bromine/ 100 g. However, cyclohexene is never used in pure form, but a solution in 1,1,1-trichloroethane is prepared (e.g. 0.5 g cyclohexene per 50 mL 1,1,1-trichloroethane; 1 mL of this solution corresponds to 10 mg cyclohexene).

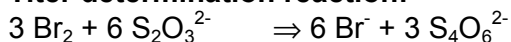
- **Sample preparation:**

Pour 110 mL solvent mixture into the titration vessel. Depending on the expected bromine index, add the corresponding amount of sample, cool down to 0...5°C and titrate with the 0.02 N bromide-bromate solution.

Samples with a higher bromine index should be diluted with carbon tetrachloride.

- **Titer determination of the  $\text{BrO}_3^-/\text{Br}^-$  solution:**

Pour 50 mL glacial acetic acid and 1 mL conc. HCl into an Erlenmeyer flask. Close the flask with a stopper and cool it in an ice bath for 10 min. Then, while tilting the flask back and forth, slowly add 1.00 mL 0.5 N bromide-bromate solution or 25 mL 0.02 N bromide-bromate solution. Now add 5 mL potassium iodide solution, stopper the flask, tilt it back and forth and place it in an ice bath for further 5 min. Afterwards, add 100 mL dist. Water and titrate with  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1000 \text{ mol/L}$  using the MET Ipol mode.

**Titer determination reaction:**


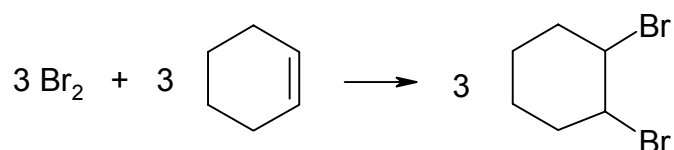
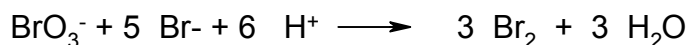
**Calculation:**

$$\text{Titer} = \text{EP1} / \text{C00}$$

- **Determination of the blank value:**

Pour 110 mL solvent mixture into the titration vessel and cool down to 0...5 °C, then titrate the blank with the titrant. The blank (titrant consumption [mL]) is stored as common variable C31.

- **Determination reaction for bromine index:**



- **Calculations for bromine index:**

Br index = brom index of sample in mg bromine per 100 g sample

EP1 = titrant consumption in mL

C00 = sample size in g

C01 = 'normality' of the titrating agent in mol/L (0.02)

C02 = 7990 (molecular mass of Br multiplied with 100 in g/mol)

C31 = blank value in mL (0)

C33 = titer of the titrating agent (0.9651)

---

**Literature**

- Metrohm Application Bulletin No. 177/4: Determination of the bromine index and/ or the bromine number
- Oil Pac (6.6040.001)

# Bromine Number

## Reagents

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

## Sample

50  $\mu\text{L}$  cyclohexene  $\cong$  10% in solvent  
50 mL solvent

## Electrodes

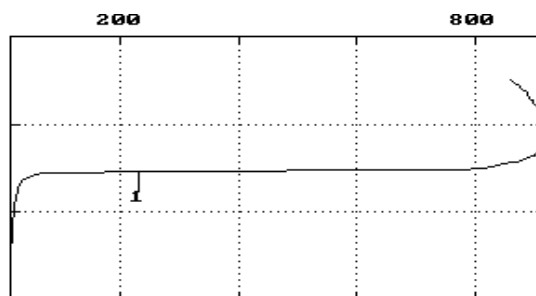
6.0308.100 double Pt electrode; input Pol  
polarized  $I_{\text{pol}} = 1 \mu\text{A}$   
Voltametric indication

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-02-01    time 14:06      4
MET Ipol           BrNumber
parameters
>titration parameters
  V step           0.025 ml
  dos.rate         max. ml/min
  signal drift     OFF mV/min
  equilibr.time    30 s
  start V:         abs.
  start V          1.50 ml
  dos.rate         3 ml/min
  pause           180 s
  I(pol)          1  $\mu\text{A}$ 
  electrode test:  OFF
  temperature     4.9  $^{\circ}\text{C}$ 
>stop conditions
  stop V:         abs.
  stop V          3.6 ml
  stop U         OFF mV
  stop EP        9
  filling rate   max. ml/min
>statistics
  status:        OFF
>evaluation
  EPC           400 mV
  EP recognition: all
  fix EP1 at U  OFF mV
>preselections
  req.ident:     id1
  req.smpl size: all
  limit smpl size: OFF
  activate pulse: OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-02-01    time 14:06      4
U(init)            1056 mV MET Ipol BrNumber
smpl size          0.0532 g
EP1                2.548 ml          231 mV
BrNumber           187.5
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-02-01    time 14:06      4
start V            1.500 ml MET Ipol BrNumber
1.0 ml/div         dU=200.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-02-01    time 14:06      4
MET Ipol           BrNumber
>calculations
BrNumber=(EP1-C31)*C01*C02*C30/C00;1;
C00=                0.0532
C01=                 0.5
C02=                 7.99
C31=                0.9651
C30=                 0
=====
```

### Remarks

- Bromine number is the number of g of bromine consumed per 100 g of sample

#### Solvent mixture :

714 mL glacial acetic acid  
 134 mL 1,1,1-trichloroethane  
 134 mL methanol  
 18 mL  $w(\text{H}_2\text{SO}_4) = 0.2$  (20%)

If possible, one should refrain from using chlorinated solvents. Our investigations have shown that 1,1,1-trichloroethane can be replaced by diethyl carbonate.

#### Titration agent:

0.5 N bromide-bromate solution [ $c(1/6 \text{ KBrO}_3) = 0.5 \text{ mol/L}$ ]:  
 51 g KBr and 13.92 g  $\text{KBrO}_3$  are separately dissolved in dist. water.  
 Pour both solutions into a 1000 mL volumetric flask and fill up to the mark with dist. water.

#### Test solution:

Cyclohexene can be used as a test substance. It has a theoretical bromine number of 194.5 g bromine/100 g. However, cyclohexene is never used in pure form, but a solution in 1,1,1-trichloroethane is prepared (e.g. 0.5 g cyclohexene per 50 mL 1,1,1-trichloroethane; 1 mL of this solution corresponds to 10 mg cyclohexene).

- **Sample preparation:**

Pour 10 mL 1,1,1-trichloroethane into a 50 mL volumetric flask. Depending on the expected bromine number, add 0.5 g ... 20 g sample, fill to the mark with 1,1,1-trichloroethane and mix well.

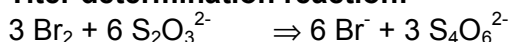
#### Analysis:

Pour 100 mL solvent mixture into the titration vessel. Add 5.00 mL sample solution, cool down to 0...5°C and titrate with the 0.5 N bromide-bromate solution.

- **Titer determination of the  $\text{BrO}_3^-/\text{Br}^-$  solution:**

Pour 50 mL glacial acetic acid and 1 mL conc. HCl into an Erlenmeyer flask. Close the flask with a stopper and cool it in an ice bath for 10 min. Then, while tilting the flask back and forth, slowly add 1.00 mL 0.5 N bromide-bromate solution. Now add 5 mL potassium iodide solution, stopper the flask, tilt it back and forth and place it in an ice bath for further 5 min. Afterwards, add 100 mL dist. Water and titrate with  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1000 \text{ mol/L}$  using the MET Ipol mode.

#### Titer determination reaction:



#### Calculation:

Titer = EP1/ C00

- **Determination of the blank value:**  
Pour 100 mL solvent mixture into the titration vessel and cool down to 0...5 °C, then titrate the blank with the titrant. The blank (titrant consumption [mL]) is stored as common variable C31.
- **Calculations for bromine number:**  
BrNumber = Bromine number in g bromine per 100 g of sample  
C00 = sample size in g  
C01 = 'normality' of the titrant in mol/L (0.5)  
C02 = 7.99 (molecular mass of Br<sub>2</sub> multiplied with 0.05 in g/mol)  
C31 = titrant consumption for the blank in mL (0)  
C33 = titer of the titrating agent (0.9651)

---

**Literature**

- Metrohm Application Bulletin No. 177/4: Determination of the bromine index and/ or the bromine number
- Oil Pac (6.6040.001)

# NaCl in Broth

## Reagents

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

## Sample

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

## Electrodes

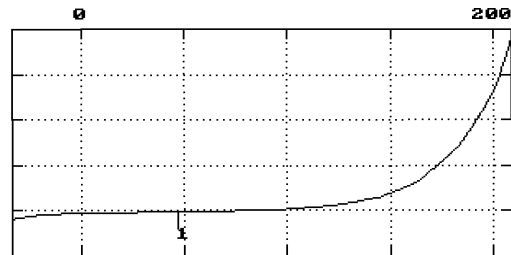
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 10:03      2
DET U               NaCl
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 09:59      2
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
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 09:59      2
start V              0.000 ml DET U   NaCl
1.0 ml/div           dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 10:03      2
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.

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**Literature**

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

# Peroxide Number

## Reagents

$c(\text{Na}_2\text{S}_2\text{O}_3) = 0.001 \text{ mol/L}$   
 Prepared daily from  $c = 0.1 \text{ mol/L}$

## Sample

1.2 g rape seed oil  
 50 mL glacial acetic acid / 1-Decanol (3 : 2), (v : v)  
 200  $\mu\text{L}$  KI saturate  
 50 mL dist. water

## Electrodes

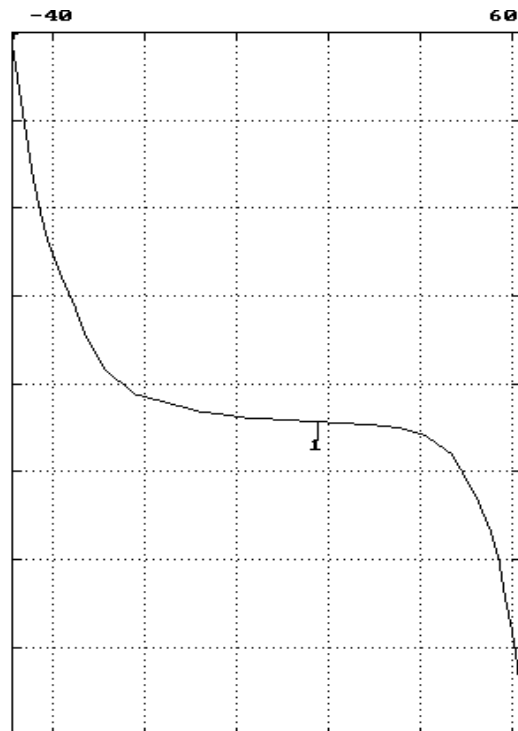
6.0431.100 Pt Titrade; input 1

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-02-18    time 17:21    13
DET U              Perox.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0  $\mu\text{l}$ 
  dos.rate             max. ml/min
  signal drift        50 mV/min
  equilibr.time       26 s
  start V:            OFF
  pause               0 s
  meas.input:         1
  temperature         25.0  $^{\circ}\text{C}$ 
>stop conditions
  stop V:             abs.
  stop V              15 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
  limit smpl size:   OFF
  activate pulse:     OFF
-----
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-02-18    time 17:21    13
U(init)             -46 mV DET U    Perox.No
smpl size            1.0721 g
EP1                  8.865 ml        18 mV
Perox.No             8.05 meq/kg
stop V reached
-----
```

```
'cu
798 MPT Titrino      01105  798.0010
date 2002-02-18    time 17:21    13
start V              0.000 ml DET U    Perox.No
2.0 ml/div           dU=20.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-02-18    time 17:21    13
DET U              Perox.No
>calculations
Perox.No= (EP1-C31)*C01*C30/C00;2;meq/kg
C00=                1.0721
C01=                 1
C30=                 1.000
C31=                 0.2326
-----
```

**Remarks**

- **Calculations:**

Perox.No. = Peroxide number in meq.O<sub>2</sub>/kg

C01 = for titrant c(Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) = 0.01 mol/L (**10**) and  
for titrant c(Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) = 0.001 mol/L (**1**)

C30 = titer of the titrating agent (1.000)

C31 = blank value in mL

- **Sample preparation:**

Weigh out 1...2 g sample in a beaker and add 10 mL of the glacial acetic acid / decanol mixture. Then add 200 µL 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, add 50 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 C31.

- The sample must be stirred well during the titration, in order to obtain a good emulsion.
- 

**Literature**

- Metrohm Application Bulletin No. 141/3: Analysis of edible oils and fats.

# Saponification Number

## Reagents

c(HCl) = 0.5 mol/L

## Sample

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

## Electrodes

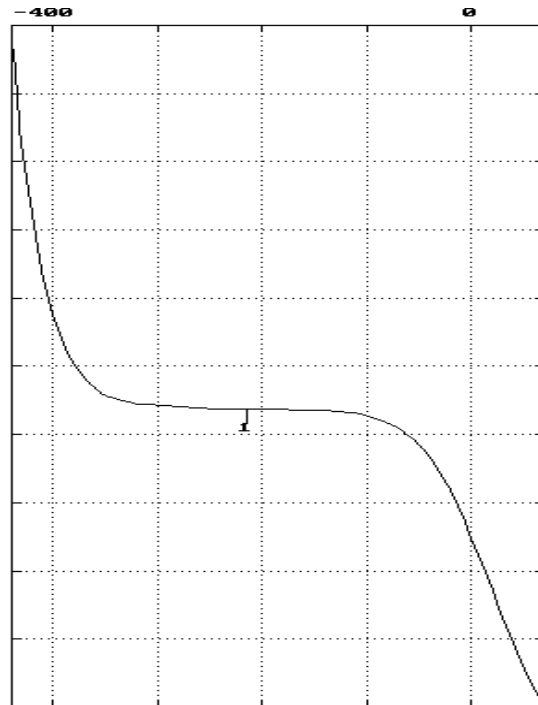
6.0229.100 Solvotrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-02-19     time 18:25      8
DET U               Sapon.No
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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:    greatest
  fix EP1 at U      OFF mV
  pK/HNP:           OFF
>preselections
  req.ident:         OFF
  req.smpl size:     all
  limit smpl size:  OFF
  activate pulse:    OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-02-19     time 18:25      8
U(init)              -436 mV DET U   Sapon.No
smpl size            1.5157 g
EP1                  11.256 ml       -213 mV
Sapon.No             187.53 mg/g
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-02-19     time 18:25      8
start V              0.000 ml DET U   Sapon.No
2.0 ml/div           dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-02-19     time 18:25      8
DET U               Sapon.No
>calculations
Sapon.No=(C31-EP1)*C01/C00;2;mg/g
C00=                 1.5157
C01=                 28.053
C31=                 21.388
=====
```

**Remarks**

- **Calculations:**  
Sapon.No = saponification number in mg KOH per g of sample  
C01 = mg KOH / 1 mL titrating agent (28.053)  
C31 = consumption of blank sample (21.388 mL)
- **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 C31.

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**Literature**

- Metrohm Application Bulletin No. 141/3: Analysis of edible oils and fats.

# Acid Number

## Reagents

c(KOH) = 0.1 mol/L in 2-Propanol

## Sample

app. 5-10 g sunflower oil  
50 mL ethanol / diethylether (1:1), neutralized with titrant solution to light pink using phenolphthaleine as color indicator

## Electrodes

6.0229.100 Solvotrode; input 1  
electrolyte c(TEABr) = 0.4 mol/L in ethylene glycol (6.2320.000)

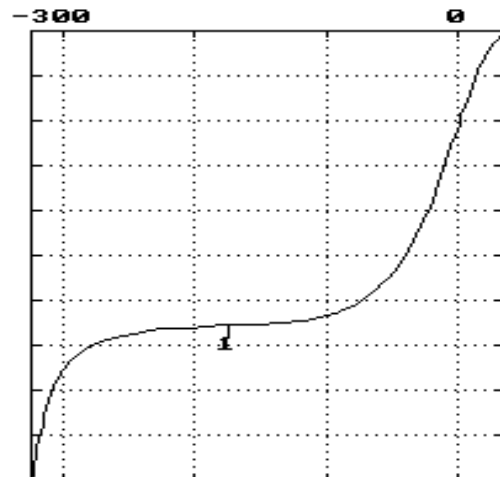
## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2001-10-30    time 18:29    12
DET U              Acid.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               5.0 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
  limit smpl size:    OFF
  activate pulse:     OFF
=====

'fm
798 MPT Titrino      01105  798.0010
date 2001-10-30    time 18:30    12
DET U              Acid.No
>calculations
Acid.No=EP1*C01/C00;2;mg
Ac.grade=EP1*C02/C00;2;ml
FFA=RS1*C03/C04;2;
C00=                9.0434
C01=                 5.61
C02=                 10
C03=                 282
C04=                 561
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2001-10-30    time 18:29    12
U(init)            32 mV DET U    Acid.No
smpl size          9.0434 g
EP1                3.2893 ml      -175 mV
Acid.No            2.04 mg
Ac.grade           3.64 ml
FFA                1.03
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2001-10-30    time 18:29    12
start V            0.0000 ml DET U    Acid.No
0.5 ml/div         dU=100.0 mV/div
```



**Remarks**

- **Calculations:**

Acid.No = acid number in mg KOH/g of sample

Ac.grade = acid grade mL c(KOH) = 1 mol/L per 100 g

FFA = free fatty acid

C01 = mg KOH/ 1 mL titrating agent (5.61)

C02 = factor for conversion from 0.1 to 1 mol/L (10)

C03 = relative molecular mass (282 for oleic acid)\*

C04 = factor (561)

$M_r$  (oleic acid) = 282 g/mol

$M_r$  (palmitic acid) = 256 g/mol [palm-oil]

$M_r$  (lauric acid) = 200 g/mol

$M_r$  (eruca acid) = 282 g/mol [rapic oil]

- The values for refined oils are much lower and the curves look different.
- 

**Literature**

- Metrohm Application Bulletin No. 141/3: Analysis of edible oils and fats.

# Iodine Number

## Reagents

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

## Sample

0.1...1.0 g rape oil  
 20 mL glacial acetic acid  
 25 mL  $c(\text{I}_2) = 0.1 \text{ mol/L}$  (according to Wijs in glacial acetic acid)  
 10 mL  $w(\text{Mg acetate}) = 3\%$  in glacial acetic acid  
 15 mL  $w(\text{KI}) = 10\%$  in dist. water  
 30-50 mL dist. water

## Electrodes

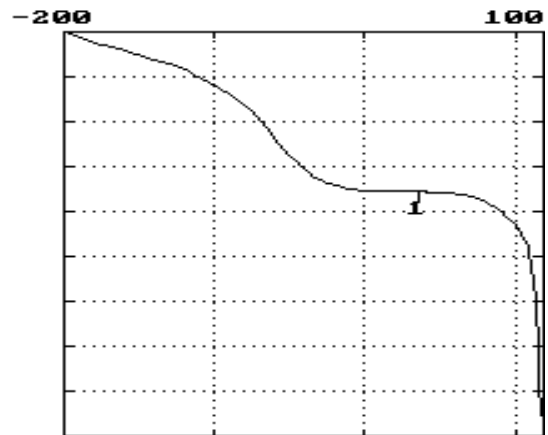
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrimo      01105  798.0010
date 2001-11-02    time 10:29    5
DET U              Iod.No
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift        20 mV/min
  equilibr.time       38 s
  start V:            abs.
  start V             5.0 ml
  dos.rate            max. ml/min
  pause               30 s
  meas.input:         1
  temperature         25.0 C
>stop conditions
  stop V:             abs.
  stop V              50 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
  limit smpl size:  OFF
  activate pulse:   OFF
=====
```

```
'fr
798 MPT Titrimo      01105  798.0010
date 2001-11-02    time 10:29    5
U(init)            -457 mV DET U    Iod.No
smpl size          0.3068 g    id 1  Metrohm
id 2               J.Smith      id 3  Rape oil
EP1                22.823 ml    36 mV
Iod No.            64.38 g
stop V reached
=====

'cu
798 MPT Titrimo      01105  798.0010
date 2001-11-02    time 10:29    5
start V            5.000 ml DET U    Iod.No
5.0 ml/div         dU=100.0 mV/div
```



```
'fm
798 MPT Titrimo      01105  798.0010
date 2001-11-02    time 10:29    5
DET U              Iod.No
>calculations
Iod.No=(C30-EP1)*C01/C00;2;g
C00=                0.3068
C01=                1.269
C31=                38.387
=====
```

**Remarks**

- **Calculations:**  
Iod.No = iodine number [g iodine / 100 g sample]  
C01 = molecular mass of I \* concentration titrating agent \* 100g  
sample / factor mL -> L (126.9 \* 0.1 \* 100/1000 = 1.269)  
C31 = consumption of blank sample
- **Sample preparation:**  
According to the expected iodine number, weigh out 0.10 ... 1.00 g of the sample in an Erlenmeyer flask and add 20 mL glacial acetic acid. Add 25 mL iodine monochloride solution and add 10 mL magnesium 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 C31.
- The sample must be stirred well during the titration, in order to obtain a good emulsion.
- The electrode and buret tip have to be rinsed properly with ethanol and dist. water after the titration to clean them from precipitations.

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**Literature**

- Metrohm Application Bulletin No. 141/3: Analysis of edible oils and fats.

# Vitamin C

## Reagents

c(DPIP) = 0.001 mol/L  
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  $I_{pol} = 1 \mu A$   
Voltametric indication

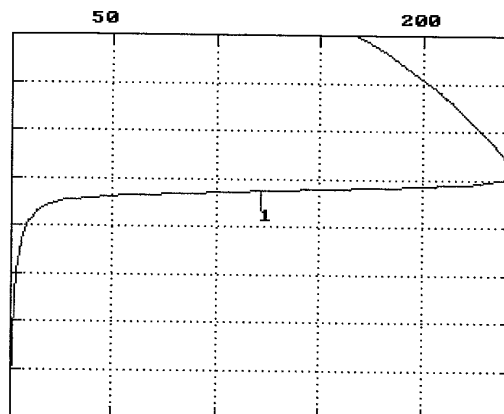
## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-10     time 16:46      11
MET Ipol            Vit.C
parameters
>titration parameters
  V step              0.10 ml
  dos.rate            max. ml/min
  signal drift        OFF mV/min
  equilibr.time       15 s
  start V:            OFF
  pause               30 s
  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
  limit smpl size:    OFF
  activate pulse:     OFF
-----

'fm
798 MPT Titrino      15215      798.0010
date 2001-10-10     time 16:46      11
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=                   0.176
C02=                   1.00
C03=                   200
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-10     time 16:45      11
U(init)              121 mV MET Ipol  Vit.C
smpl size            5.0 ml
EP1                  15.291 ml          122 mV
Vit.C                 538 mg/pc
Vit.C                 0.5382 g/l
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-10     time 16:45      11
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 (0.176)  
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:**  
 $\rho$  (Vitamin C) = 500 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.

---

**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

## 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

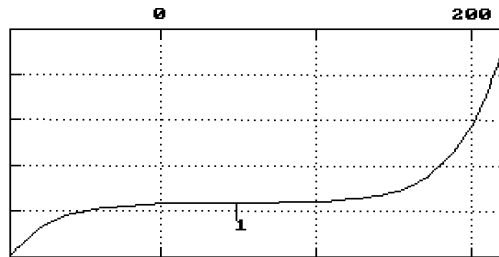
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-12     time 17:52      14
DET U               Na+Cl-
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpls size: OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-12     time 17:51      14
U(init)             224 mV DET U   Na+Cl-
smpl size           5.0 ml
EP1                 7.652 ml       49 mV
Na+Cl-              153.04 mmol/l
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-12     time 17:51      14
start V             0.000 ml DET U   Na+Cl-
2.0 ml/div          dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-12     time 17:53      14
DET U               Na+Cl-
>calculations
Na+Cl-=EP1*C01*C02/C00;2;mmol/l
C00=                 5.0
C01=                 0.1
C02=                 1000
-----
```

**Remarks**

- **Calculations:**

Na+Cl- = 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.

# 2-Aminophenol

## Reagents

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

## Sample

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

## Electrodes

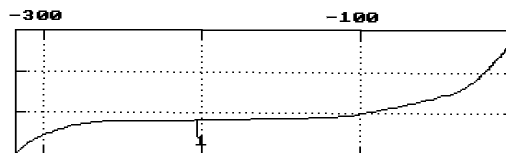
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 11:34      18
MET U               Diazo
parameters
>titration parameters
  V step             0.10 ml
  dos.rate           max. ml/min
  signal drift       OFF mV/min
  equilibr.time      15 s
  start V:           OFF
  pause              30 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 11:32      18
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
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 11:32      18
start V             10.000 ml MET U      Diazo
2.0 ml/div          dU=100.0 mV/div
```

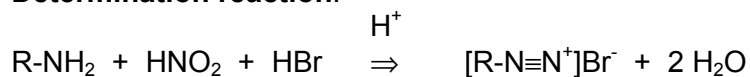


=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-08     time 11:34      18
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 g/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(DPIP) = 0.001 mol/L  
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  $I_{pol} = 1 \mu A$   
Voltametric indication

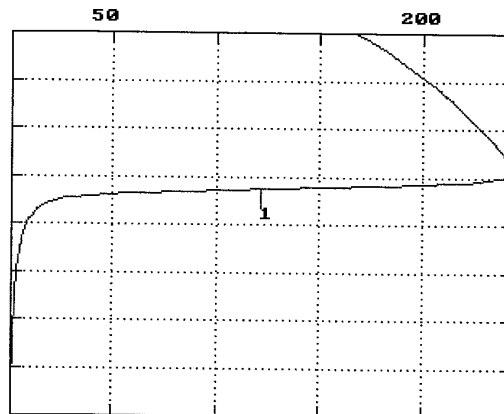
## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-09     time 16:46      11
MET Ipol            Vit.C
parameters
>titration parameters
  V step              0.10 ml
  dos.rate            max. ml/min
  signal drift        OFF mV/min
  equilibr.time       15 s
  start V:            OFF
  pause               30 s
  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
  limit smpl size:   OFF
  activate pulse:     OFF
  -----

'fm
798 MPT Titrino      15215      798.0010
date 2001-10-09     time 16:46      11
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=                   0.176
C02=                   1.00
C03=                   200
  -----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-09     time 16:45      11
U(init)              121 mV MET Ipol  Vit.C
smpl size            5.0 ml
EP1                  15.291 ml          122 mV
Vit.C                538 mg/pc
Vit.C                0.5382 g/l
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-09     time 16:45      11
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:**  
 $\rho$  (Vitamin C) = 500 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.

---

**Literature**

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

# EDTA/NTA in Detergents

## Reagents

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

## 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

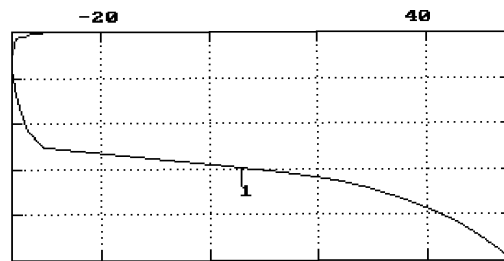
6.0502.140  $\text{Cu}^{2+}$  sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode ( $\text{KNO}_3 \text{ sat.}$ )

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-10    time 14:28      19
DET U              EDTA-NTA
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-10    time 14:27      19
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
798 MPT Titrino      15215      798.0010
date 2001-10-10    time 14:27      19
start V              0.000 ml DET U  EDTA-NTA
1.0 ml/div           dU=20.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-10    time 14:28      19
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  
 STPB = sodium tetraphenylborate

## Sample

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

## Electrodes

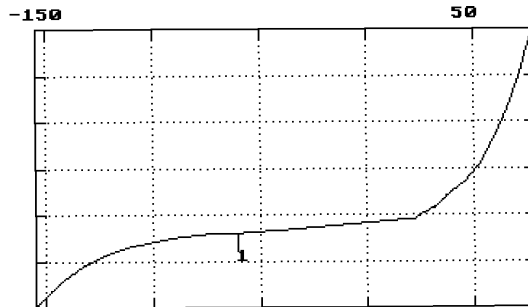
6.0507.010 NIO surfactant electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode  
 (inner electrolyte c(KCl) = 3 mol/L, outer electrolyte  
 c(NaCl) = 1 mol/L)

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:54      20
DET U               Tenside
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          100 µl
  dos.rate           5 ml/min
  signal drift       5 mV/min
  equilibr.time      60 s
  start V:           OFF
  pause              60 s
  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
  limit smpl size:  OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:47      20
U(init)             75 mV DET U   Tenside
smpl size           0.5145 g
EP1                 8.795 ml      -60 mV
Nonionic            3.4189 meq/g
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:47      20
start V             0.000 ml DET U Tenside
2.0 ml/div          dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:54      20
DET U               Tenside
>calculations
Nonionic=EP1*C01*C02/C00;4;meq/g
C00=                 0.5145
C01=                 0.01
C02=                 20
-----
```

**Remarks**

- **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, aliquote (20)
- **Titration agent:**  
Dissolve 3.4223 g sodium tetraphenylborate 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.

---

**Literature**

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

# Cationic Surfactants with two-phase titration

## Reagents

c(SDS) = 0.004 mol/L  
Sodium dodecylsulfate, Merck Nr. 112533

## Sample

app. 0.5 g sample (fabric softener)  
0.2 mLTEGO add, Metrohm Nr.6.2317.110  
70 mL dist. water  
20 mL MIBK:Ethanol =1:1 (v:v)

## Electrodes

6.0507.140 Surfactrode Refill; input 1  
6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/L)

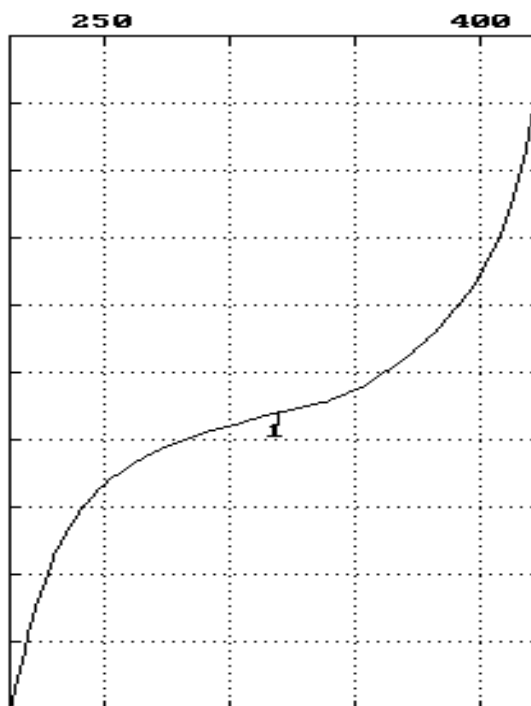
## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-01-16    time 15:34    27
DET U              Cation2p
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            100 µl
  dos.rate             5 ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  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                 20
  EP recognition:     all
  fix EP1 at U       OFF mV
  pK/HNP:            OFF
>preselections
  req.ident:          OFF
  req.smpl size:      value
  limit smpl size:   OFF
  activate pulse:    OFF
=====

'fm
798 GPT Titrino      01105  798.0010
date 2002-01-16    time 15:34    27
DET U              Cation2p
>calculations
Cat.Surf=EP1*C01*C02*C30/C00;4;mmol
C00=                 0.5723
C01=                 0.004
C02=                 100
C30=                 0.9943
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-01-16    time 15:34    27
U(init)             424 mV DET U    Cation2p
smpl size           0.5723 g
EP1                 11.222 ml      319 mV
Cat.Surf            7.7987 mmol
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-01-16    time 15:34    27
start V            0.000 ml DET U    Cation2p
2.0 ml/div         dU=50.0 mV/div
```



**Remarks**

- **Calculations:**  
Cat.Surf. = concentration of cationic surfactants in mmol/100g  
C01 = concentration of titrating agent (0.004 mol/L)  
C02 = factor for 100g (100)  
C30 = titer of the titratng agent (0.9943)
- **Electrode preparation, maintenance and storage:**  
Store the Surfactant electrode dry. Refill it before a new series of determinations. It is conditioned by two to three titrations. Rinse the electrode after several titrations with methanol or wipe it with a cloth moistend woth methanol and rinse with dist. water.
- **Sample preparation:**  
Weigh 0.5 to 2.0 g sample in a titration vessel and add 70 mL dist. water as well as 0.2 mL TEGOadd. Adjust the pH to 2...3 with  $c(\text{HCl}) = 1.0 \text{ mol/L}$ . Add 20 mL solvent and start the titration.

---

**Literature**

- Metrohm Surf Pac 6.6041.001

# Anionic Surfactants in Shampoo

## Reagents

c(TEGO<sup>®</sup> trant A100) = 0.004 mol/L  
 1,3-didecyl-2-methyl-imidazolium chloride (DDMICI)  
 Metrohm 6.2317.000

## Sample

app. 0.2 g sample (shampoo)  
 5 mL Methanol puriss p.a.  
 10 mL Buffer solution pH = 3.0, citrate/HCl, e.g. Merck Tirisol No. 9883  
 60 mL dist. water

## Electrodes

6.0507.120 Ionic surfactant electrode; input 1  
 6.0733.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/L)

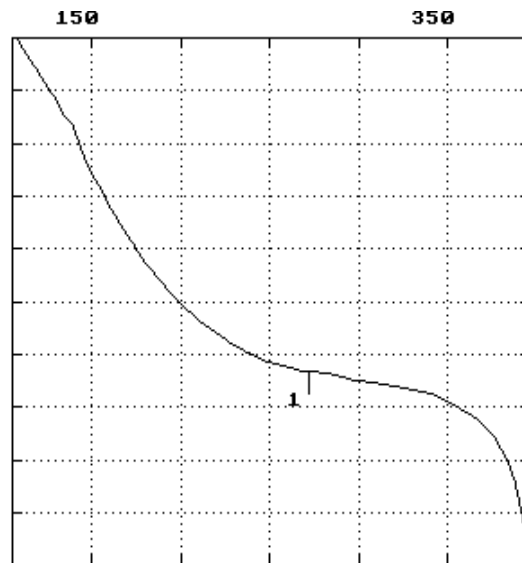
## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-16      time 16:20      6
DET U                Tens.An
parameters
>titration parameters
  meas.pt.density      3
  min.incr.            10.0 µl
  dos.rate              max. ml/min
  signal drift         OFF mV/min
  equilibr.time        10 s
  start V:             OFF
  pause                30 s
  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:      greatest
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       value
  limit smpl size:    OFF
  activate pulse:      OFF
-----

'fm
798 MPT Titrino      15215      798.0010
date 2001-10-16      time 16:21      6
DET U                Tens.An
>calculations
Anionic=EP1*C01/C00;4;meq/g
C00=                  0.2128
C01=                  0.004
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-1      time 16:19      6
U(init)              103 mV DET U      Tens.An
smpl size             0.2128 g
EP1                   12.638 ml          271 mV
Anionic               0.2376 meq/g
stop V reached
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-16      time 16:19      6
start V              0.000 ml DET U      Tens.An
2.0 ml/div           dU=50.0 mV/div
```



**Remarks**

- **Calculations:**  
Anionic = concentration of anionic Tensides in meq/g  
C01 = concentration of titrating agent (0.004 mol/L)
- **Electrode preparation, maintenance and storage:**  
Store the Ionic Surfactant electrode dry. They are conditioned by two to three titrations. Rinse the electrode after several titrations with methanol or wipe it with a cloth moistend with methanol and rinse with dist. water.  
If you use the electrode frequently, it may also be stored in a solution of 1% polyethyleneglycol 1000. In this case, the electrode is always ready to use.
- **Sample preparation:**  
Adjust the sample size in order to have a titrating agent consumption of at least 10 mL.
- The pH of buffer solution, the amount of added methanol may change according to the type of surfactant. See also Application Bulletin No. 233.

---

**Literature**

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

# Anionic Surfactants with two-phase titration

## Reagents

c(TEGO trant A100) = 0.004 mol/L

## Sample

approx. 0.5 g sample  
70 mL dist. Water  
20 mL MIBK/ Ethanol (1:1)

## Electrodes

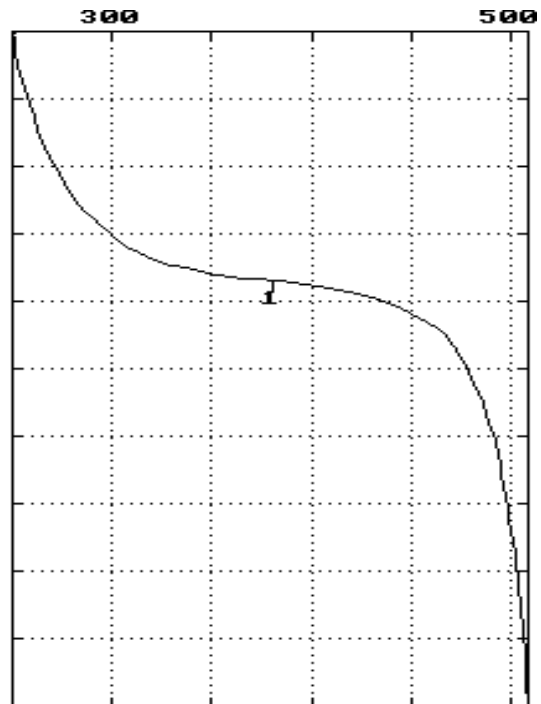
6.0507.130 Surfactrode Resistant; input 1  
6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3mol/L)

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-01-11    time 16:10    23
DET U              Anion2p
parameters
>titration parameters
  meas.pt.density      2
  min.incr.           50 µl
  dos.rate             max. ml/min
  signal drift        OFF mV/min
  equilibr.time       15 s
  start V:            OFF
  pause               30 s
  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                 2
  EP recognition:     greatest
  fix EP1 at U       OFF mV
  pK/HNP:            OFF
>preselections
  req.ident:          OFF
  req.smpl size:      value
  limit smpl size:   OFF
  activate pulse:     OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-01-11    time 16:10    23
U(init)            251 mV DET U    Anion2p
smpl size           0.54 g
EP1                 7.418 ml        381 mV
A.Surf.             5.535 mmol
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-01-11    time 16:10    23
start V            0.000 ml DET U    Anion2p
2.0 ml/div         dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-01-11    time 16:10    23
DET U              Anion2p
>calculations
A.Surf.=EP1*C01*C02*C30/C00;3;mmol
C00=                0.54
C01=                0.004
C02=                100
C30=                1.0073
=====
```

**Remarks**

- **Calculations:**
  - A.Surf. = concentration of anionic surfactants in mmol/ 100g
  - C01 = concentration of the titrating agent (0.004 mol/L)
  - C02 = factor for 100 g (100)
  - C30 = titer of the titrant (1.0073)  
saved as common variable
- **Electrode preparation, maintenance and storage:**

Store the Surfactant Resistant electrode dry. It is conditioned by two to three titrations. Rinse the electrode after several titrations with methanol or wipe it with a cloth moistend with methanol and rinse with dist. water.
- **Sample preparation:**

Adjust the sample size in order to have a titrating agent consumption between 5 mL and 15 mL.

---

**Literature**

- Metrohm Surf Pac 6.6041.001

# Pure Silver Content

## Reagents

c(KBr) = 0.1 mol/L

## Sample

app. 150 mg Ag  
10 mL HNO<sub>3</sub> 65 %  
100 mL dist. water

## Electrodes

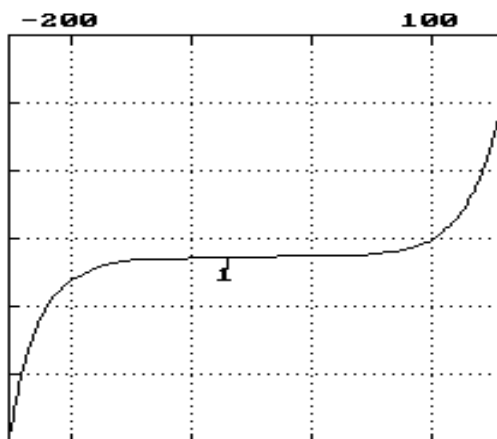
6.0450.100 Ag Titrode with Br coating; input 1

## Method documentation

```
'pa
798 MPT Titrino      01105  798.0010
date 2002-01-29    time 14:54    6
DET U              Silver
parameters
>titration parameters
  meas.pt.density      4
  min.incr.           10 µl
  dos.rate             max ml/min
  signal drift        50 mV/min
  equilibr.time       26 s
  start V:            abs.
  start V             9 ml
  dos.rate            max. ml/min
  pause               20 s
  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 mV
  EP recognition:     greatest
  fix EP1 at U       OFF mV
  pK/HNP:             OFF
>preselections
  req.ident:          OFF
  req.smpl size:      value
  limit smpl size:    OFF
  activate pulse:     OFF
=====
```

```
'fr
798 MPT Titrino      01105  798.0010
date 2002-01-29    time 14:54    6
U(init)             185 mV DET U    Silver
smpl size           157.14 mg
EP1                 14.576 ml      -71 mV
Ag                  99.758 %
stop V reached
=====

'cu
798 MPT Titrino      01105  798.0010
date 2002-01-29    time 14:54    6
start V             9.000 ml DET U    Silver
2.0 ml/div          dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      01105  798.0010
date 2002-01-29    time 14:54    6
DET U              Silver
>calculations
Ag=EP1*C01*C02*C03*C30/C00;3;%
C00=                157.14
C01=                 0.1
C02=                107.87
C03=                 100
C30=                 0.9970
=====
```

**Remarks**

- **Calculations:**  
Silver = content of silver in % ( purity )  
C01 = concentration of titrating agent (0.1 mol/L)  
C02 = molecular mass of Ag (107.87 g/mol)  
C03 = factor for % (100)  
C30 = titer of titrating agent(0.9970)
  - **Sample preparation:**  
Heat silver in HNO<sub>3</sub>. Allow nitrous fumes to evaporate.
- 

**Literature**

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

# Metals

## Reagents

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

## 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

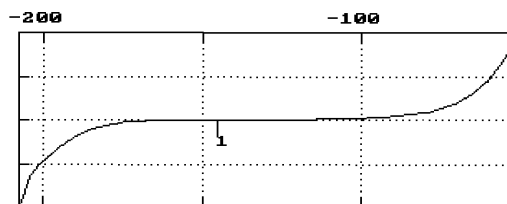
6.0502.140 Cu<sup>2+</sup> sensitive indicator electrode; input 1  
 6.0726.100 Ag/AgCl double junction reference electrode (KNO<sub>3</sub> sat.)

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-18     time 10:44      3
DET U               Metals
parameters
>titration parameters
  meas.pt.density    2
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       20 mV/min
  equilibr.time      38 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size   OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-18     time 10:43      3
U(init)             -50 mV DET U      Metals
smpl size           2.0 ml
EP1                 2.006 ml          -145 mV
Content              6.56 g/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-18     time 10:43      3
start V             0.000 ml DET U      Metals
1.0 ml/div          dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-18     time 10:44      3
DET U               Metals
>calculations
Content=EP1*C01*C02/C00;2;g/l
C00=                 2.0
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 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

# Epoxy Number

## Reagents

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

## 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

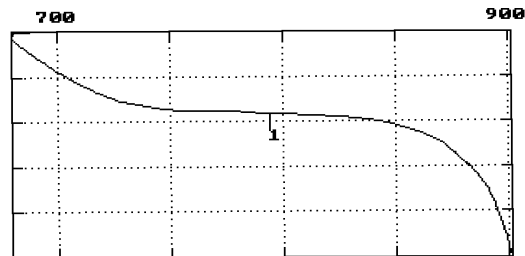
6.0430.100 Ag Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-19     time 13:28      2
DET U                Epoxy.No
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       OFF mV/min
  equilibr.time      20 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:   OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-19     time 13:27      2
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
=====
```

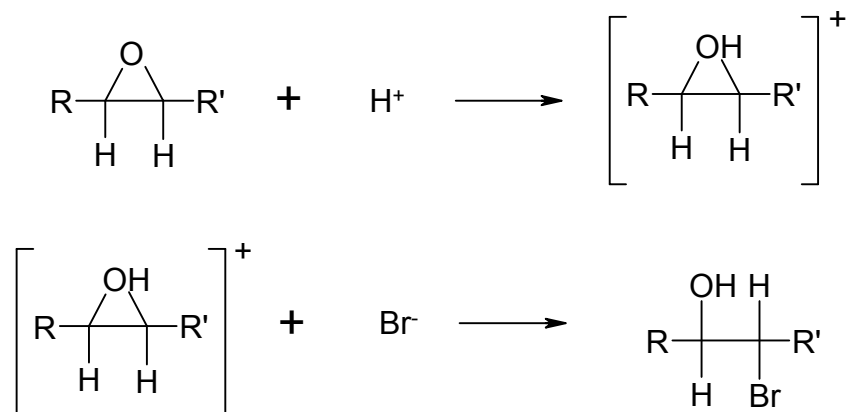
```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-19     time 13:27      2
start V              7.000 ml DET U   Epoxy.No
1.0 ml/div           dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-19     time 13:28      2
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, "Titrationsen in nichtwässrigen Medien" Ferdinand Enke Verlag, Stuttgart (1970), p. 598

# Nitrating Acid

## Reagents

c(Cyclohexylamine) = 0.5 mol/L in methanol

## Sample

3 mL nitrating acid  
25 mL methanol

## Electrodes

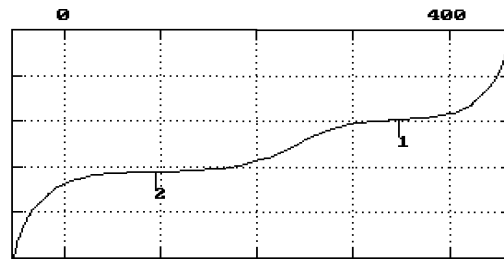
6.0133.100 pH glass electrode; input 1  
6.0726.100 Ag/AgCl double junction reference electrode  
(c(LiClO<sub>4</sub>) = 1 mol/L in acetic acid)

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:52      3
DET U              NitrAcid
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       50 mV/min
  equilibr.time      26 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:48      3
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
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:48      3
start V             0.000 ml DET U  NitrAcid
1.0 ml/div          dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-15     time 15:52      3
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:**
  - H2SO4 = sulphuric acid in g/L of nitrating acid
  - HNO3 = 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

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

## 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

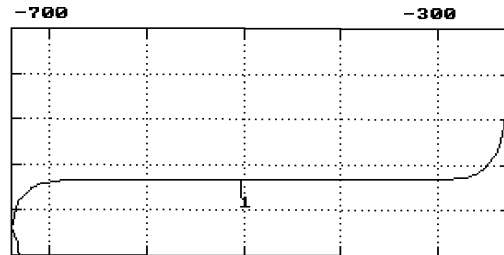
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-16     time 16:39      4
DET U                H2O2
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.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
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-16     time 16:33      4
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
798 MPT Titrino      15215      798.0010
date 2001-10-16     time 16:33      4
start V              1.000 ml DET U      H2O2
1.0 ml/div           dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-16     time 16:39      4
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  $\rightarrow$  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

## 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

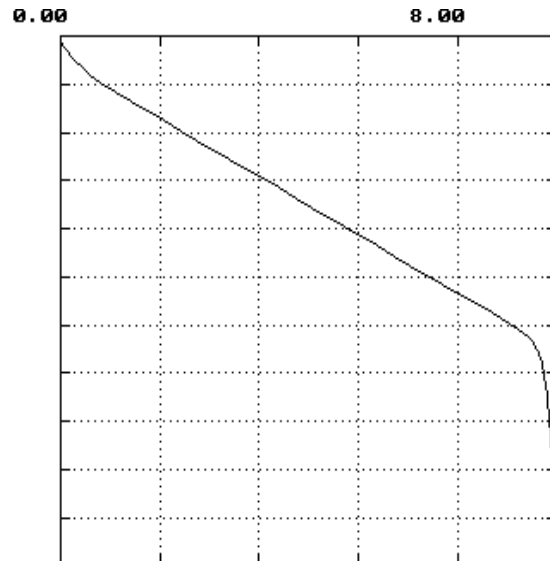
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-19      time 11:49      7
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 2           0 s
  extr.time         0 s
  meas.input:       1
  temperature       25.0 °C
  time interval     2 s
>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
  limit smpl size: OFF
  activate pulse:   OFF
-----
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-19      time 11:49      7
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
-----
```

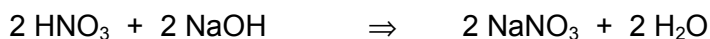
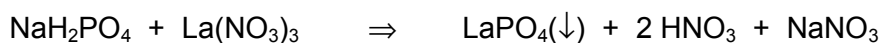
```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-19      time 11:48      7
pHc(init)           1.94      SET pH  Phosphat
smpl size            0.07798 g
EP1                  9.992 ml      4.21
P                    19.84 %
P2O5                 45.47 %
PO4                  60.85 %
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-19      time 11:48      7
SET pH              Phosphat
10.0 s/div          dV=2.0 ml/div
```



### 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> 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.
- For automatic curve output add in <DEF>, >report "curve".

### 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

## Sample

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

## Electrodes

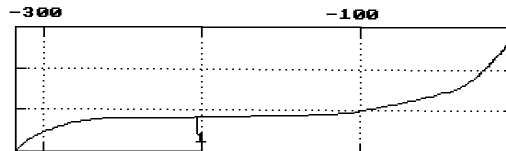
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-22     time 11:34      18
MET U               Diazo
parameters
>titration parameters
  V step            0.10 ml
  dos.rate          max. ml/min
  signal drift      OFF mV/min
  equilibr.time     15 s
  start V:          OFF
  pause            30 s
  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
  limit smpl size: OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-22     time 11:32      18
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
798 MPT Titrino      15215      798.0010
date 2001-10-22     time 11:32      18
start V            10.000 ml MET U      Diazo
2.0 ml/div         dU=100.0 mV/div
```

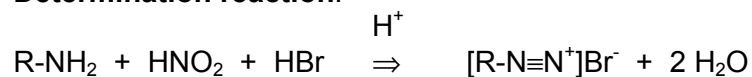


=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-22     time 11:34      18
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 g/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

## 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

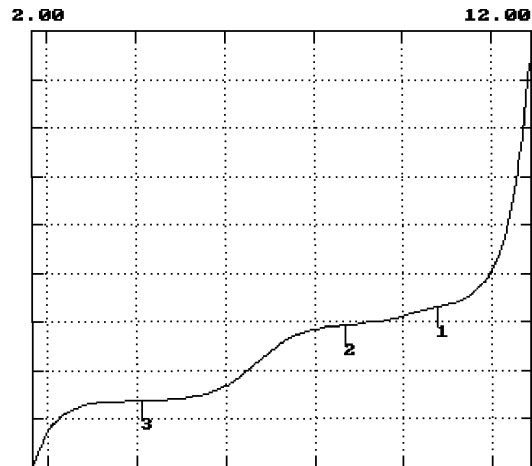
6.0239.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-23    time 09:12      3
DET pH              W-Liquor
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate              max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:              OFF
  pause                 0 s
  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
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-23    time 09:11      3
pHc(init)           12.85
DET pH              W-Liquor
smpl size            2.0 ml
EP1                  5.714 ml      10.76
EP2                  6.081 ml      8.67
EP3                  7.625 ml      4.09
Total                152.50 g/l
Active               137.82 g/l
Effecti.             114.28 g/l
NaOH                 90.74 g/l
Na2S                 45.91 g/l
Na2CO3              19.45 g/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-23    time 09:11      3
start V              0.000 ml
1.0 ml/div           dpH=2.0/div
DET pH              W-Liquor
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-23    time 09:12      3
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=                2.0
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) = 0.012 (1.2%)

## Electrodes

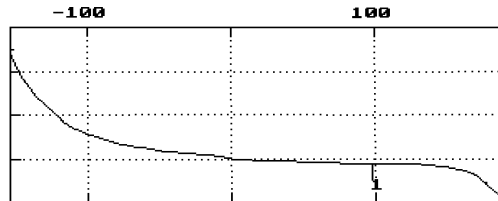
6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-24     time 10:35      2
DET U               FixBaths
parameters
>titration parameters
  meas.pt.density    4
  min.incr.          10.0 µl
  dos.rate           max. ml/min
  signal drift       20 mV/min
  equilibr.time      38 s
  start V:           OFF
  pause              0 s
  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
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-24     time 10:34      2
U(init)             -168 mV DET U   FixBaths
smpl size           5.0 ml
EP1                 10.220 ml          99 mV
Silver              11.025 g/l
stop V reached
=====
```

```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-24     time 10:34      2
start V             5.000 ml DET U   FixBaths
2.0 ml/div          dU=100.0 mV/div
```

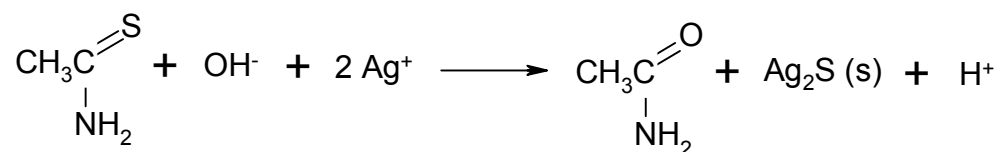


=====

```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-24     time 10:35      2
DET U               FixBaths
>calculations
Silver=EP1*C01*C02/C00;3;g/l
C00=                 5.0
C01=                 5.394
C02=                 1.0000
-----
```

### 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) = 0.012 (1.2%)

## Electrodes

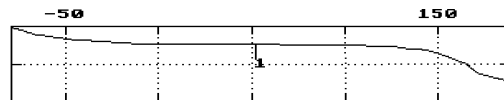
6.0430.100 Ag Titrode with Ag<sub>2</sub>S coating; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-24    time 11:03      3
DET U              Film Ag
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate              max. ml/min
  signal drift         20 mV/min
  equilibr.time        38 s
  start V:             OFF
  pause                0 s
  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
  limit smpl size:    OFF
  activate pulse:      OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-24    time 11:02      3
U(init)             -17 mV DET U      Film Ag
smpl size           10.0 ml      id#1      ILFORD
EP1                 0.987 ml      52 mV
AgNO3               8.383 g/m2
stop V reached
=====
```

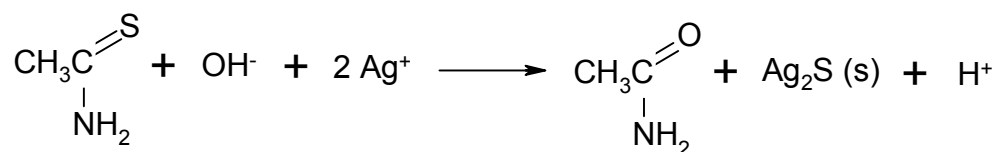
```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-24    time 11:02      3
start V             0.000 ml DET U      Film Ag
2.0 ml/div          dU=50.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-24    time 11:03      3
DET U              Film Ag
>calculations
AgNO3=EP1*C01*C02/C00*C03/C04/C05;3;g/m2
C00=                10.0
C01=                 0.025
C02=                339.74
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 2 AgNO<sub>3</sub> (339.74 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.

# Analysis of Spinning Bath

## Reagents

c(NaOH) = 0.1 mol/L

## 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

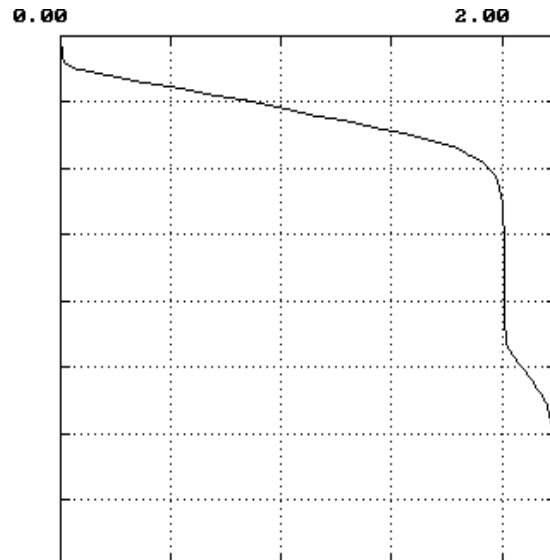
6.0232.100 combined pH glass electrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-25      time 10:28      3
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 2           0 s
  extr.time         0 s
  meas.input:       1
  temperature       25.0 °C
  time interval     2 s
>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
  limit smpl size: OFF
  activate pulse:   OFF
-----
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-25      time 10:28      3
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
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-25      time 10:21      3
pHc(init)          2.22   SET pH   Spinning
smpl size           0.1 ml
EP1                 2.011 ml          5.61
EP2                 2.245 ml          9.53
H2SO4               99.47 g/l
ZnSO4               18.86 g/l
=====

'cu
798 MPT Titrino      15215      798.0010
date 2001-10-25      time 10:21      3
                    SET pH   Spinning
20.0 s/div          dV=0.5 ml/div
```



**Remarks**

- EP1:  $\text{H}_2\text{SO}_4 + 2 \text{OH}^- \Rightarrow \text{SO}_4^{2-} + 2 \text{H}_2\text{O}$   
EP2:  $\text{Zn}^{2+} + 2 \text{OH}^- \Rightarrow \text{Zn(OH)}_2 (\text{s})$
  - **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)
  - For automatic curve output add in <DEF>, >report "curve".
- 

**Literature**

# 2-Aminophenol

## Reagents

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

## Sample

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

## Electrodes

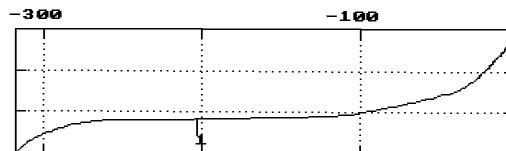
6.0431.100 Pt Titrode; input 1

## Method documentation

```
'pa
798 MPT Titrino      15215      798.0010
date 2001-10-26     time 11:34      18
MET U               Diazo
parameters
>titration parameters
  V step            0.10 ml
  dos.rate          max. ml/min
  signal drift      OFF mV/min
  equilibr.time     15 s
  start V:          OFF
  pause            30 s
  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
  limit smpl size: OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      15215      798.0010
date 2001-10-26     time 11:32      18
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
=====
```

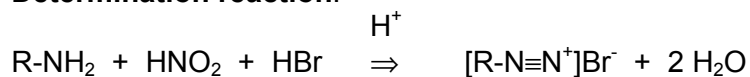
```
'cu
798 MPT Titrino      15215      798.0010
date 2001-10-26     time 11:32      18
start V             10.000 ml MET U      Diazo
2.0 ml/div          dU=100.0 mV/div
```



```
'fm
798 MPT Titrino      15215      798.0010
date 2001-10-26     time 11:34      18
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 g/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 Water

## Reagents

HYDRANAL® Composite 5

## Sample

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

## Electrodes

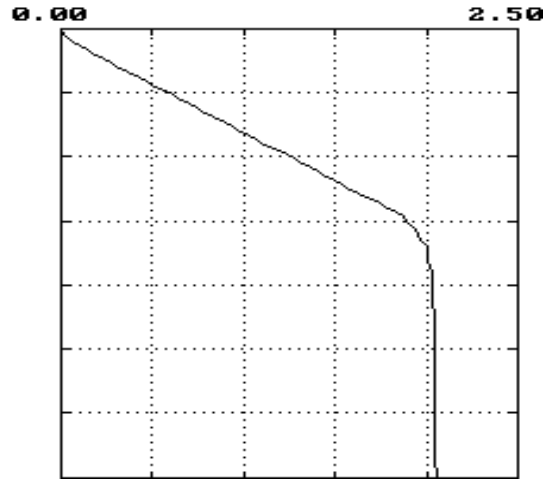
6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 09:24   49
SET Ipol            H2OTiter
parameters
>SET1
  EP at U           250 mV
  dynamics          250 mV
  max.rate          2 ml/min
  min.rate          5 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at U           OFF mV
>titration parameters
  titr.direction:  -
  pause 1           0 s
  start V:          OFF
  pause 2           0 s
  extr.time         0 s
  I(pol)            50 µA
  electrode test:   OFF
  temperature       25.0 °C
  time interval     2 s
>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
  limit smpl size: OFF
  activate pulse:   OFF
-----
```

```
'fr
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 09:22   49
U(init)             643 mV SET Ipol H2OTiter
smpl size           0.010 g
EP1                 2.0515 ml      242 mV
Titer               4.8745 mg/ml
                   mean( 5)  +/-s      s/%
Titer              4.8681  0.00972 mg/ml  0.20
=====

'cu
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 09:22   49
                        SET Ipol H2OTiter
20.0 s/div           dV=0.5 ml/div
```



```
'fm
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 09:24   49
SET Ipol            H2OTiter
>calculations
Titer=C00/EP1*C01;4;mg/ml
C00=                 0.010
C01=                 1000
-----
```

```
'st
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 09:23
statistics
  Titer
  1  4.8520
  2  4.8757
  3  4.8662
  4  4.8721
  5  4.8745
                   mean( 5)  +/-s      s/%
Titer              4.8681  0.00972 mg/ml  0.20
-----
```

**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  $\mu$ L    C01=Density of H<sub>2</sub>O [mg/mL]  $\approx$  1  
*Methanol standard*  
C00 in mL    C01=Content of methanol [mg/mL]  
C00 in  $\mu$ L    C01=0.001 \* content of methanol [mg/mL]
- For automatic curve output add in <DEF>, >report "curve".

---

**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

## Sample

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

## Electrodes

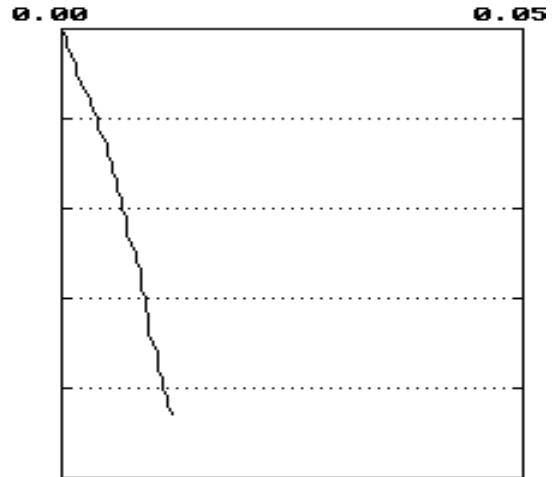
6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 10:34  9
SET Ipol           Blank_KF
parameters
>SET1
  EP at U           250 mV
  dynamics          250 mV
  max.rate          2 ml/min
  min.rate          5 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at U           OFF mV
>titration parameters
  titr.direction:  -
  pause 1           0 s
  start V:          OFF
  pause 2           0 s
  extr.time         0 s
  I(pol)            50 µA
  electrode test:   OFF
  temperature       25.0 °C
  time interval     2 s
>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:    value
  limit smpl size: OFF
  activate pulse:   OFF
-----
'de
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 10:34
SET Ipol           Blank_KF
def
>formula
  Blank=EP1
  RS1 text          Blank
  RS1 decimal places 4
  RS1 unit:         ml
  RS1 limit control: OFF
>silco calculations
  match id:         OFF
>common variables
  C38=MN1
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
-----
```

```
'fr
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 10:33  9
U(init)            328 mV SET Ipol Blank_KF
smpl size          1.00 ml
EP1                0.0120 ml      243 mV
Blank              0.0120 ml
                  mean( 3)  +/-s      s/%
Blank              0.0122  0.00029 ml  2.37
=====
```

```
'cu
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 10:33  9
                SET Ipol Blank_KF
20.0 s/div        dV=0.05 ml/div
```



```
'st
798 MPT Titrino      01102  798.0010
date 2001-10-3-26  time 10:33
statistics
  Blank
  1  0.0125
  2  0.0120
  3  0.0120
                  mean( 3)  +/-s      s/%
Blank              0.0122  0.00029 ml  2.37
-----
```

**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 <PARAM>, >titration parameters.
- Use the same control parameters for the blank determination as for the sample titrations.
- For automatic curve output add in <DEF>, >report "curve".

---

**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

## 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

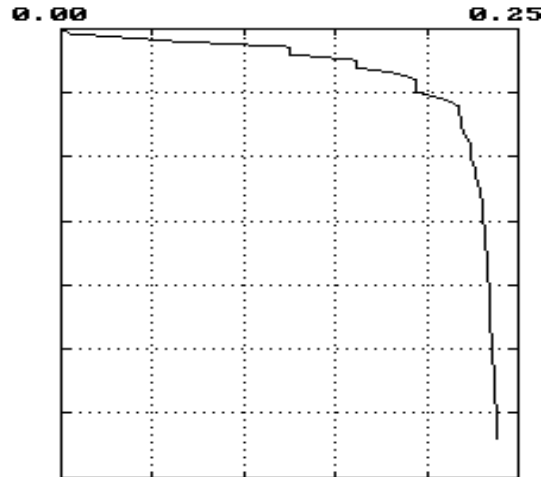
## Method documentation

```
'pa
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 11:12    18
SET Ipol           KF-Blank
parameters
>SET1
  EP at U           250 mV
  dynamics          250 mV
  max.rate          2 ml/min
  min.rate          5 µl/min
  stop crit:       drift
  stop drift        20 µl/min
>SET2
  EP at U           OFF mV
>titration parameters
  titr.direction:  -
  pause 1          0 s
  start V:         OFF
  pause 2          0 s
  extr.time        0 s
  I(pol)           50 µA
  electrode test:  OFF
  temperature      25.0 °C
  time interval    2 s
>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:   value
  limit smpl size: OFF
  activate pulse:  OFF
-----
```

```
'fm
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 11:12    18
SET Ipol           KF-Blank
>calculations
Water=(EP1-C38)*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
Blank=C38;4;ml
C00=                0.01992
C01=                 0.1
C02=                 1
C38=                 0.0122
C39=                 4.8681
-----
```

```
'fr
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 11:11    18
U(init)            630 mV SET Ipol KF-Blank
smpl size          0.01992 g
EP1                0.2385 ml          239 mV
Water              5.53 %
Titer              4.8681 mg/ml
Blank              0.0122 ml
                  mean( 3)    +/-s    s/%
Water              5.52    0.026 %    0.46
=====
```

```
'cu
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 11:12    18
                SET Ipol KF-Blank
20.0 s/div        dV=0.05 ml/div
```



```
'st
798 MPT Titrino      01102  798.0010
date 2001-10-26    time 11:11
statistics
  Water
  1    5.54
  2    5.49
  3    5.53
                  mean( 3)    +/-s    s/%
Water              5.52    0.026 %    0.46
-----
```

---

**Remarks**

- **Calculations:**  
 Water = content of water in paper in %  
 C00 = sample size / aliquot (1.0015 g / 50 mL = 0.02003 g/mL)  
 C01 = factor for % (0.1)  
 C02 = divisor (1)  
 C38 = blank value in mL of "Blank\_KF" method (0.0145 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 <PARAM>, >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	µL	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

- **Sample preparation:**  
 Stir sample solution for app. ½ h to extract the water and take aliquots (see calculation).
- For automatic curve output add in <DEF>, >report "curve".

---

**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

## Sample

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

## Electrodes

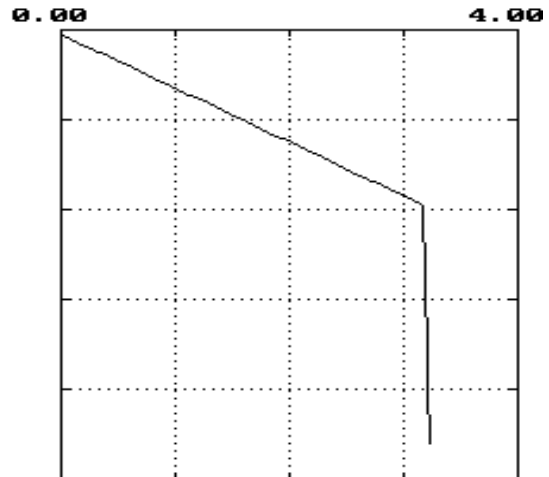
6.0338.100 double Pt electrode; input Pol

## Method documentation

```
'pa
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 14:17   23
SET Ipol            KF
parameters
>SET1
  EP at U            250 mV
  dynamics           250 mV
  max.rate           2 ml/min
  min.rate           5 µl/min
  stop crit:         drift
  stop drift         20 µl/min
>SET2
  EP at U            OFF mV
>titration parameters
  titr.direction:    -
  pause 1            0 s
  start V:           OFF
  pause 2            0 s
  extr.time          0 s
  I(pol)             50 µA
  electrode test:    OFF
  temperature        25.0 °C
  time interval      2 s
>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:     value
  limit smpl size:  OFF
  activate pulse:    OFF
-----
```

```
'fr
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 14:17   23
U(init)             663 mV SET Ipol  KF
smpl size           0.0864 g   id 1   Honey
EP1                 3.2235 ml   249 mV
Water               18.16 %
Titer               4.8681 mg/ml
                    mean( 3)   +/-s   s/%
Water              18.11    0.047 %   0.26
=====

'cu
798 MPT Titrino      01102  798.0010
date 1999-10-26     time 14:18   23
                    SET Ipol      KF
50.0 s/div          dV=1.0 ml/div
```



```
'fm
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 14:18   23
SET Ipol            KF
>calculations
Water=EP1*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
C00=                0.0864
C01=                0.1
C02=                1
C39=                4.8681
-----
```

```
'st
798 MPT Titrino      01102  798.0010
date 2001-10-26     time 14:17
statistics
  Water
  1  18.07
  2  18.09
  3  18.16
  mean( 3)   +/-s   s/%
Water       18.11    0.047 %   0.26
-----
```

### 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 <PARAM>, >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	µL	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

- For automatic curve output add in <DEF>, >report "curve".

### 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

2-Aminophenol.....	5-2, 8-5, 9-5	NaCl .....	4-1, 5-1
Acid .....	1-1	NaOH .....	1-2
Acid capacity.....	2-3	Ni <sup>2+</sup> .....	1-8
Acid number.....	4-4	Nitrating acid .....	8-2
Ag <sup>+</sup> .....	7-1, 9-2, 9-3	Non-ionic surfactants .....	6-2
Agro chemistry .....	9-ff	NTA.....	6-1
Anionic surfactants .....	6-4, 6-5		
		Oxalic acid .....	1-4, 1-5
Ba <sup>2+</sup> .....	1-8	Oxidizability .....	2-4
Basic materials .....	8-ff		
Beverages.....	4-ff	p value .....	2-3
Blank (KF).....	10-3	Paper .....	9-3, 10-3
Boric acid.....	2-2	Pb <sup>2+</sup> .....	1-8
Bromine index.....	3-3	Peroxide number.....	4-2
Bromine number .....	3-4	Petro chemistry .....	3-ff
Broth .....	4-1	Pharmaceutics .....	5-ff
		Phosphate .....	1-12, 8-4
Ca <sup>2+</sup> .....	1-6, 1-7	Photo.....	9-ff
Cationic surfactants .....	6-3	PO <sub>4</sub> <sup>3-</sup> .....	1-12, 8-5
Cd <sup>2+</sup> .....	1-8	Pure silver .....	7-1
Chloride .....	1-11, 2-1		
Cl <sup>-</sup> .....	1-11, 2-1	Saponification number .....	4-3
Co <sup>2+</sup> .....	1-8	Shampoo.....	6-4
		Silver .....	7-1, 9-2, 9-3
Detergents .....	6-ff	Spinning Bath.....	9-4
Diazotisation .....	5-2, 8-5, 9-5	Strong acid.....	1-1
		Surfactants.....	6-2, 6-3, 6-4, 6-5
EDTA .....	6-1		
Epoxy number.....	8-1	TAN.....	3-1
		Tap water .....	1-7, 2-1
Fabric softener.....	6-3	TBN.....	3-2
Fe <sup>2+</sup> .....	1-10	Tensides .....	6-2, 6-3, 6-4, 6-5
Film .....	9-3	Titer	
Fixing baths .....	9-2	HCl .....	1-3
Food.....	4-ff	KF .....	10-1, 10-2
		NaOH .....	1-2
Galvanics .....	7-ff	Total acid number .....	3-1
General .....	1-ff	Total base number .....	3-2
H <sub>2</sub> O <sub>2</sub> .....	8-3	Vitamin C .....	4-6, 5-3
H <sub>2</sub> SO <sub>4</sub> .....	1-1, 8-3		
H <sub>3</sub> BO <sub>3</sub> .....	2-2	Water .....	1-7, 2-ff,
HCl.....	1-1, 1-3	Water determination .....	10-ff
Honey .....	10-4	Whith liquor.....	9-1
HNO <sub>3</sub> .....	1-1, 8-3		
		Zn <sup>2+</sup> .....	1-8
I <sub>2</sub> .....	1-9		
Iodine.....	1-9		
Iodine number.....	4-5		
Iron (II) .....	1-10		
Isotonic solution .....	5-1		
Juices.....	4-2		
KF titrations.....	10-ff		
m value .....	2-3		
Metals .....	1-8, 7-2		
Mg <sup>2+</sup> .....	1-7, 1-8		