Metrohm

CH-9101 Herisau/Switzerland
Phone ++41 71 353 85 85
Fax ++41 71 353 89 01
CompuServe 100031,3703
Internet http://www.metrohm.com

751 GPD Titrino

Applications

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99.06 ks/Ti

Introduction

The methods from the 6.6029.010 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 6.6029.010 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 combined ***Pt electrode*** a 6.0431.100 Pt Titrode can normally be used. The direction of the titration curve changes, e.g. if the curve goes from 100 to 400 mV with a combined Pt electrode, it will run from 400 to 100 mV with the Pt Titrode and vice versa.

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

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Strong Acid

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

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

Electrodes

6.0232.100 combined pH glass electrode; input 1

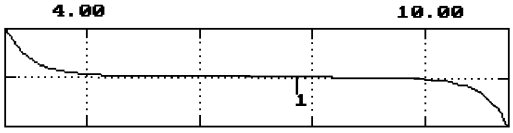
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-19      time  09:37        4
DET  pH                Acid
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   4 ml
  stop pH                 OFF
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-19      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-19      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
751 GPD Titrino       15215     751.0010
date  97-02-19      time  09:36        4
start V      0.000 ml  DET  pH      Acid
2.0 ml/div      dpH=2.0/div
```




```
            ============
```

Remarks

- **Calculations**:
  c(acid) = concentration of acid in mol/L
  C01 = concentration of titrating agent (0.1 mol/L)
  C37 = titer of titrating agent (1.0013 common variable of “Tit.NaOH“)

- Carbonate may be detected separately if present!

Literature

Titer of NaOH

Reagents

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

Sample

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

Electrodes

6.0232.100 combined pH glass electrode; input 1

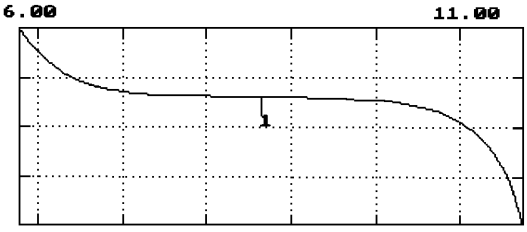
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-19      time  17:33        6
DET  pH            Tit.NaOH
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   20 ml
  stop pH                 OFF
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                  ON
  mean                n=     5
  res.tab:            original
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-19      time  17:33        6
DET  pH            Tit.NaOH
>calculations
Titer=C00*C01/C02/EP1;4;
C00=              0.3021
C01=               10000
C02=              204.23
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-19      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
751 GPD Titrino       15215     751.0010
date  97-02-19      time  17:30        6
start V     12.000 ml  DET  pH  Tit.NaOH
2.0 ml/div      dpH=1.0/div
```




```
          ============
```

Remarks

- **Calculations**:
  Titer = titer of NaOH
  C01 = theoretical consumption for 1 mol potassium hydrogen phthalate (for a solution with *c*=0.1 mol/L = 10000 mL/mol)
  C02 = molecular mass of potassium hydrogen phthalate (204.23 g/mol)

- **Common variables**
  C37 = MN1

- Mean from 5 determinations.

- For shorter titration times, a start volume may be used, see example curve.

Literature

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

Titer of HCl

Reagents

c(HCl) = 0.1 mol/L; D0

Sample

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

Electrodes

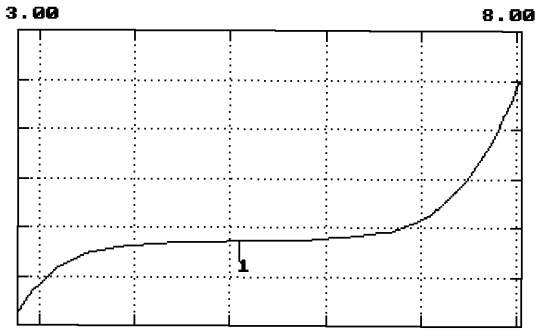
6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-20      time  10:03       10
DET  pH             Tit.HCl
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  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
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                99.99 ml
  stop pH                 2.8
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  ON
  mean                n=    10
  res.tab:            original
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-20      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( 5)    +/-s         s/%
Titer        0.9922  0.00044         0.04
stop meas.val.reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-20      time  10:02       10
start V      4.980 ml  DET  pH   Tit.HCl
1.0 ml/div      dpH=1.0/div
```




```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-20      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 5 determinations.

- This method may be used for the GLP validation, see Metrohm Bulletin 252.
  To shorten the titration times, a relative start volume is used for the GLP validation.

Literature

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

Oxalic Acid

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

2 mL $c(C_2H_2O_4) \cong 0.1$ mol/L; HOOC-COOH $pK_1 = 1.42$, $pK_2 = 4.31$
50 mL dist. water

Electrodes

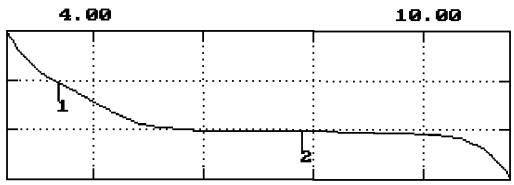
6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-20      time  11:07        8
DET  pH              Oxalic
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   6 ml
  stop pH                 OFF
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                  ON
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-20      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
751 GPD Titrino       15215     751.0010
date  97-02-20      time  11:04        8
start V      0.000 ml  DET  pH    Oxalic
2.0 ml/div      dpH=2.0/div
```




```
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-20      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; D0
TBAOH = Tetrabutyl ammonium hydroxide

Sample

2 mL $c(C_2H_2O_4) \cong 0.1$ mol/L; HOOC-COOH
25 mL ethanol

Electrodes

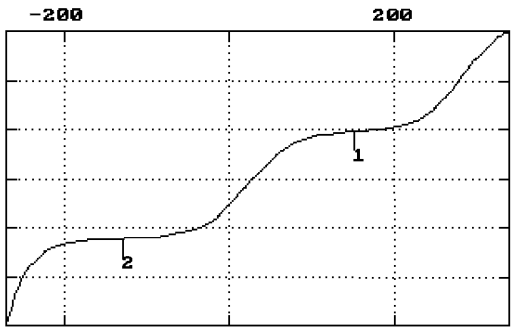
6.0103.100 pH glass electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode (LiCl sat. in ethanol)

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-20      time  13:43        9
DET  U             OxalicNA
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   6 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-20      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
751 GPD Titrino       15215     751.0010
date  97-02-20      time  13:38        9
start V      0.000 ml  DET  U   OxalicNA
1.0 ml/div      dU=200.0 mV/div
```




```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-20      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^{2+} with Amalgamated Ag Electrode

Reagents

$c(Na_2EDTA)$ = 0.1 mol/L; D0

Sample

2 mL $c(CaCl_2) \cong$ 0.1 mol/L
10 mL buffer pH = 10
40 mL dist. water

Electrodes

6.0430.100 Ag Titrode amalgamated; input 1

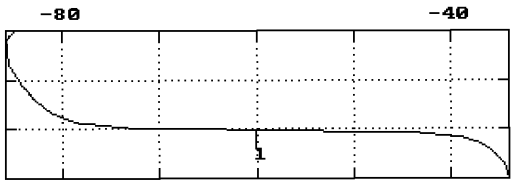
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-20      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
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                    3 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-20      time  14:53       15
MET  U                  Ca++
>calculations
c(Ca++)=EP1*C01/C00;3;mol/l
C00=                   2.0
C01=                   0.1
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-20      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
751 GPD Titrino       15215     751.0010
date  97-02-20      time  14:48       15
start V      0.000 ml  MET  U       Ca++
1.0 ml/div      dU=10.0 mV/div
```




```
          ============
```

Remarks

- **Calculations**:
  Ca++ = concentration of Ca^{2+} 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_3, then immerse it shortly in Hg.

- **Buffer pH = 10**:
  Dissolve 54 g NH_4Cl and 350 mL $w(NH_3)$ = 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(Na_2EDTA) = 0.05 mol/L in *c*(KOH) = 0.1 mol/L; D0

Sample

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

Electrodes

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

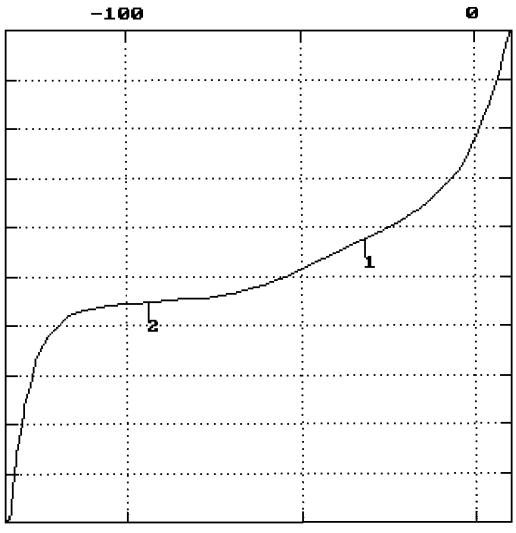
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-20      time  17:19       12
DET  U                Ca-Mg
parameters
>titration parameters
  meas.pt.density           1
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-20      time  17:19       12
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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-20      time  17:15       12
U(init)          10 mV DET U       Ca-Mg
smpl size       100 ml    id#1   Metrohm
id#2        Herisau       id#3  TapWater
EP1           4.245 ml            -32 mV
EP2           5.535 ml            -94 mV
Ca++           2.12 mmol/l
Mg++           0.64 mmol/l
Total          2.77 mmol/l
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-20      time  17:15       12
start V      0.000 ml  DET  U      Ca-Mg
1.0 ml/div      dU=50.0 mV/div
```




```
          ============
```

Remarks

- 1st break: Ca^{2+}
  2nd break: Mg^{2+}

- **Calculations**:
  Ca++ = calcium hardness in mmol/L
  Mg++ = magnesium hardness in mmol/L
  Total = total hardness in mmol/L
  C01 = concentration of titrating agent (0.05 mol/L)
  C02 = factor for the conversion mol ⇒ mmol (1000)

- **Electrode preparation**:
  Ca electrodes should be conditioned for 10 min. in $c(CaCl_2)$ = 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

$c(Na_2EDTA)$ = 0.1 mol/L; D0

Sample

2 mL $c(ZnSO_4) \cong 0.1$ mol/L
5 mL buffer pH = 10
1 mL c(CuEDTA) = 0.1 mol/L
40 mL dist. water

Electrodes

6.0502.140 Cu^{2+} sensitive indicator electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode (KNO_3 sat.)

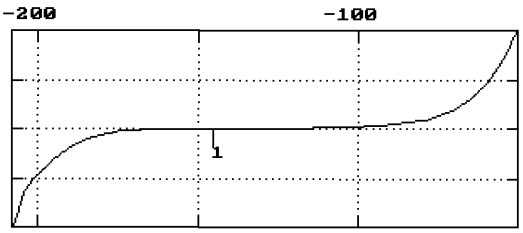
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-21      time  15:39        3
DET  U               Metals
parameters
>titration parameters
  meas.pt.density           2
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-21      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
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-21      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
751 GPD Titrino       15215     751.0010
date  97-02-21      time  15:30        3
start V      0.000 ml  DET  U     Metals
1.0 ml/div       dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Content = content of metal in g/L
  C01 = concentration of titrating agent (0.1 mol/L)
  C02 = molecular mass of metal (65.38 g/mol)

- **Buffer pH = 10**:
  Dissolve 54 g NH_4Cl and 350 mL $w(NH_3)$ = 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

$c(Na_2S_2O_3)$ = 0.1 mol/L; D0

Sample

3 mL $c(KI_3) \cong 0.05$ mol/L
5 mL $c(H_2SO_4)$ = 0.5 mol/L
20 mL dist. water

Electrodes

6.0431.100 Pt Titrode; input 1

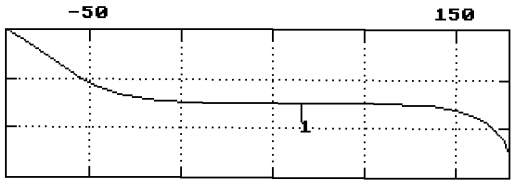
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-21      time  16:33        6
DET  U               Iodine
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   5 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-21      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
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-21      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
751 GPd Titrino       15215     751.0010
date  97-02-21      time  16:31        6
start V      0.000 ml  DET  U     Iodine
2.0 ml/div       dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Determination reaction**:

  $I_2 + 2\ S_2O_3^{2-} \Rightarrow 2\ I^- + S_4O_6^{2-}$

- **Calculations**:
  c(KI_3) = concentration of KI_3 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

$c(KMnO_4)$ = 0.02 mol/L; D0

Sample

2 mL $c[(NH_4)_2Fe(SO_4)_2] \cong 0.1$ mol/L, acidic solution
10 mL $c(H_2SO_4)$ = 0.5 mol/L
40 mL dist. water

Electrodes

6.0431.100 Pt Titrode; input 1

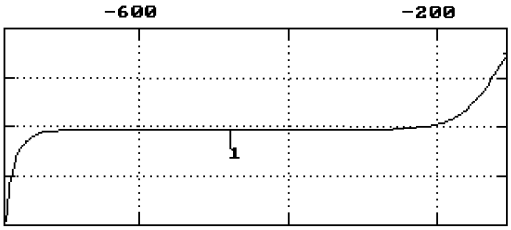
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-21      time  18:03        3
DET  U             Iron(II)
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  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
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                    4 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-21      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
         ------------
```

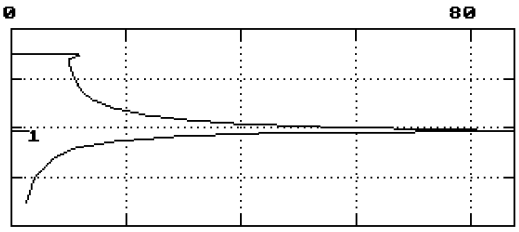
```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-21      time  18:02        3
U(init)        -136 mV DET U    Iron(II)
smpl size         2.0 ml
EP1            2.065 ml           -476 mV
Fe++           5.77 g/l
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-21      time  18:02        3
start V      0.500 ml  DET  U   Iron(II)
1.0 ml/div      dU=200.0 mV/div
```




```
            ============
```

```
 'dv
751 GPD Titrino       15215     751.0010
date  97-02-21      time  18:02        3
start V      0.500 ml  DET  U   Iron(II)
1.0 ml/div      ERC=20.0/div
```




```
            ============
```

Remarks

- **Determination reaction**:

  $5\ Fe^{2+} + MnO_4^- + 8\ H^+ \Rightarrow 5\ Fe^{3+} + Mn^{2+} + 4\ H_2O$

- **Calculations**:
  Fe++ = concentration of Fe^{2+} 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

$c(AgNO_3)$ = 0.1 mol/L; D0

Sample

5 mL c(NaCl) ≅ 0.1 mol/L
2 mL $c(HNO_3)$ = 2 mol/L
40 mL dist. water

Electrodes

6.0430.100 Ag Titrode; input 1

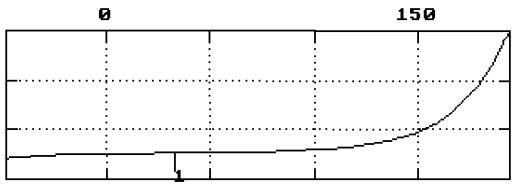
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      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
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                99.99 ml
  stop U                  OFF mV
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      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
751 GPD Titrino       15215     751.0010
date  97-02-24      time  09:40        3
start V      0.000 ml  DET  U   Chloride
2.0 ml/div      dU=50.0 mV/div
```




```
          ============
```

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^- (35.45 g/mol)
  C03 = factor for % (0.1)
  C04 = molecular mass of NaCl (58.44)

- Select the appropriate formula. The other may be deleted.
  Or change the formula according to your application.

Literature

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

Phosphate

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

5 mL *c*(NaH_2PO_4) ≅ 0.1 mol/L
40 mL dist. water
adjust the pH value to 4.2 with dilute NaOH or H_2SO_4
10 mL *c*($La(NO_3)_3$) = 0.1 mol/L, pH = 4.2

Electrodes

6.0232.100 combined pH glass electrode; input 1

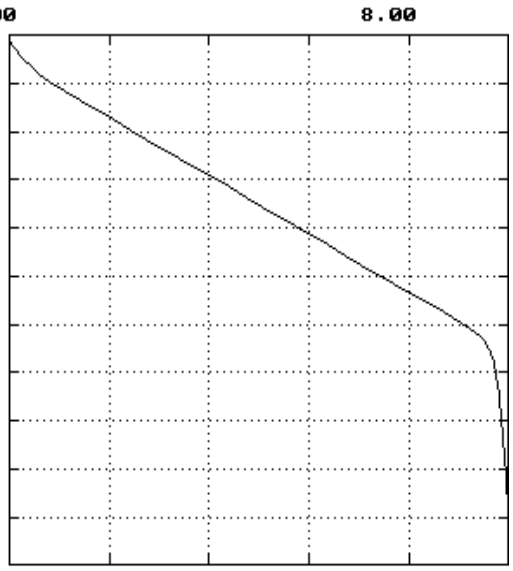
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-05      time  11:49        7
SET  pH            Phosphat
parameters
>SET1
  EP at pH               4.20
  dynamics                  1
  max.rate                 10 ml/min
  min.rate                 25 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  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
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-05      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
751 GPD Titrino       15215     751.0010
date  97-03-05      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
751 GPD Titrino       15215     751.0010
date  97-03-05      time  11:48        7
                      SET  pH  Phosphat
10.0  s/div      dV=2.0 ml/div
```




```
          ============
```

Remarks

- **Determination reaction**:

  $NaH_2PO_4 + La(NO_3)_3 \Rightarrow LaPO_4(\downarrow) + 2\ HNO_3 + NaNO_3$

  $2\ HNO_3 + 2\ NaOH \Rightarrow 2\ NaNO_3 + 2\ H_2O$

- **Calculations**:
  P = content of P in %
  P2O5 = content of P_2O_5 in %
  PO4 = content of PO_4^{3-} 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_2O_5
  C04 = 1 mL *c*(NaOH) = 0.1 mol/L = 4.7486 mg PO_4

- **Sample preparation**:
  The pH of the sample aliquot has to be adjusted to pH = 4.2 with dil. NaOH or H_2SO_4. Add 10 mL *c*($La(NO_3)_3$ = 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

$c(AgNO_3)$ = 0.01 mol/L; D0

Sample

100 mL tap water
0.5 mL $c(HNO_3)$ = 2 mol/L

Electrodes

6.0430.100 Ag Titrode; input 1

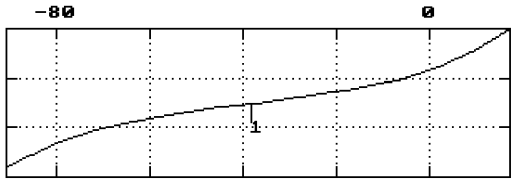
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      time  11:31        6
DET  U             Chloride
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop U                  OFF mV
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      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
751 GPD Titrino       15215     751.0010
date  97-02-24      time  11:30        6
start V      0.000 ml  DET  U   Chloride
1.0 ml/div      dU=20.0 mV/div
```




```
          ============
```

Remarks

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

Literature

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

Boric Acid

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

5.00 mL c(H_3BO_3) ≅ 0.1 mol/L
10 mL d-mannitol solution, saturated
40 mL dist. water

Electrodes

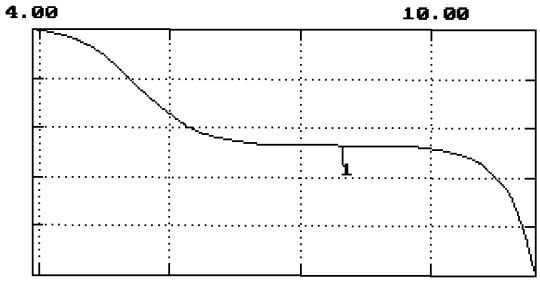
6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      time  13:59        9
DET  pH            Bor.Acid
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop pH                 OFF
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:    greatest
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      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
751 GPD Titrino       15215     751.0010
date  97-02-24      time  13:55        9
start V      0.000 ml  DET  pH  Bor.Acid
2.0 ml/div      dpH=2.0/div
```

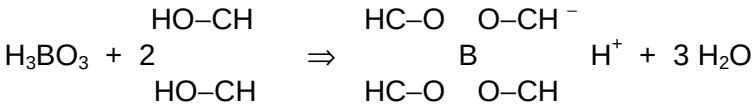



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      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 ⇒ L (1000)
  C37 = titer of titrating agent (1.0013)

- d-mannitol solution, saturated:
  App. 200 g d-mannitol dissolved in dist. water.

Literature

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

p and m Value

Reagents

c(HCl) = 0.1 mol/L; D1

Sample

100 mL tap water

Electrodes

6.0239.100 combined pH glass electrode; input 2

Method documentation

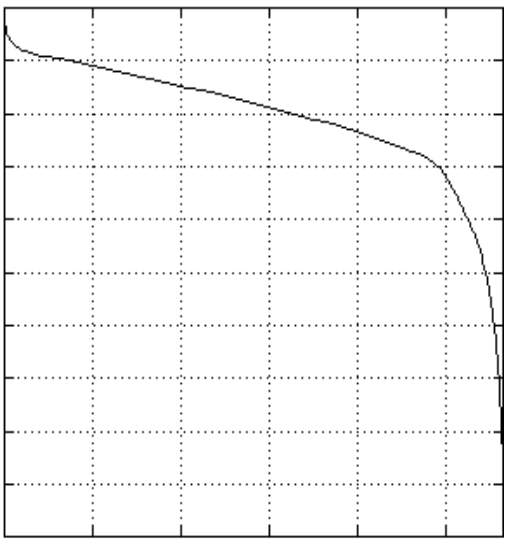
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  11:24        2
SET  pH            p+m val.
parameters
>SET1
  EP at pH               8.20
  dynamics                  2
  max.rate                 10 ml/min
  min.rate                  5 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>SET2
  EP at pH               4.30
  dynamics                  3
  max.rate                 10 ml/min
  min.rate                  5 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>titration parameters
  titr.direction:        auto
  pause 1                   0 s
  start V:                OFF
  pause 2                   0 s
  extr.time                 0 s
  dos.element:    external D1
  meas.input:               2
  temperature            25.0 °C
  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
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-10      time  11:24        2
SET  pH            p+m val.
>calculations
p value=EP1*C01;2;mmol/l
m value=EP2*C01;2;mmol/l
C01=                  1.0
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-10      time  11:07        2
pHc(init)      7.05    SET pH   p+m val.
smpl size       100 ml    id#1   Metrohm
id#2        Herisau       id#3  TapWater
EP1           0.000 ml              7.05
EP2           5.653 ml              4.28
p value        0.00 mmol/l
m value        5.65 mmol/l
           ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-10      time  11:07        2
                      SET  pH  p+m val.
20.0  s/div      dV=1.0 ml/div
```

```
0.00                                5.00
```



```
            ============
```

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
  C01= titer of titrating agent (1.0)

- 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)

Water Hardness

Reagents

c(Na_2EDTA) = 0.05 mol/L in c(KOH) = 0.1 mol/L; D0
c(HCl) = 0.1 mol/L; D1
c(Acetylacetone) = 0.1 mol/L in c(Trishydroxy methylaminomethane) = 0.1 mol/L (auxiliary complexing agent, pH app. 8.5); D2

Sample

100 mL water

Electrodes

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

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:47        1
TIP                Hardness
parameters
>sequence
  1.stirrer:              ON
  2.method:          p+m val.
  3.method:          AddBuffe
  4.pause                 15 s
  5.method:             Ca-Mg
>statistics
  status:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  meas.mode:              OFF
  temperature            25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:42        1
                       TIP      Hardness
smpl size       100 ml    id#1   Metrohm
id#2        Herisau       id#3  TapWater
C70            7.75
C71           0.000
C72           5.687
C73           4.471
C74           6.055
pH             7.75
p value        0.00 mmol/l
m value        5.69 mmol/l
Ca++           2.24 mmol/l
Mg++           0.79 mmol/l
Total          3.03 mmol/l
TIP terminated
           ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:47        1
TIP                Hardness
>calculations
pH=C70;2;
p value=C71;2;mmol/l
m value=C72;2;mmol/l
Ca++=C73*C01*C02/C00;2;mmol/l
Mg++=(C74-C73)*C01*C02/C00;2;mmol/l
Total=C74*C01*C02/C00;2;mmol/l
C00=                100
C01=               0.05
C02=               1000
C70=               7.75
C71=               0.00
C72=               5.69
C73=              4.471
C74=              6.055
         ------------
```

Submethods

p+m value

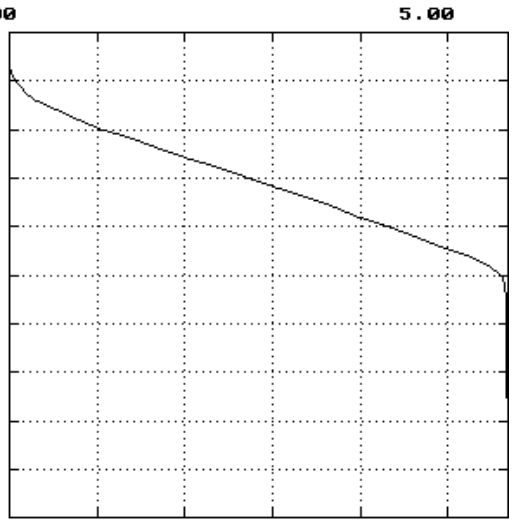
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:48        1
SET  pH            p+m val.
parameters
>SET1
  EP at pH               8.20
  dynamics                  2
  max.rate                 10 ml/min
  min.rate                  5 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>SET2
  EP at pH               4.30
  dynamics                  3
  max.rate                 10 ml/min
  min.rate                  5 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>titration parameters
  titr.direction:        auto
  pause 1                   0 s
  start V:                OFF
  pause 2                   0 s
  extr.time                 0 s
  dos.element:    external D1
  meas.input:               2
  temperature            25.0 °C
  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
  activate pulse:         OFF
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:50
SET  pH            p+m val.
def
>formula
  p value=EP1*C01
  RS1 text            p value
  RS1 decimal places        2
  RS1 unit:            mmol/l
  m value=EP2*C01
  RS2 text            m value
  RS2 decimal places        2
  RS2 unit:            mmol/l
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
  C70=C40
  C71=EP1
  C72=EP2
          ------------
```

p+m value

```
'fr
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:37        1
pHc(init)      7.75    SET pH   p+m val.
smpl size       100 ml    id#1   Metrohm
id#2        Herisau       id#3  TapWater
EP1           0.000 ml              7.75
EP2           5.687 ml              4.28
p value        0.00 mmol/l
m value        5.69 mmol/l
          ============
```

```
'cu
751 GF Titrino        Proto.1   751.000c
date  97-03-10      time  13:37        1
                      SET  pH  p+m val.
10.0  s/div      dV=1.0 ml/div
```




```
          ============
```

Buffer addition

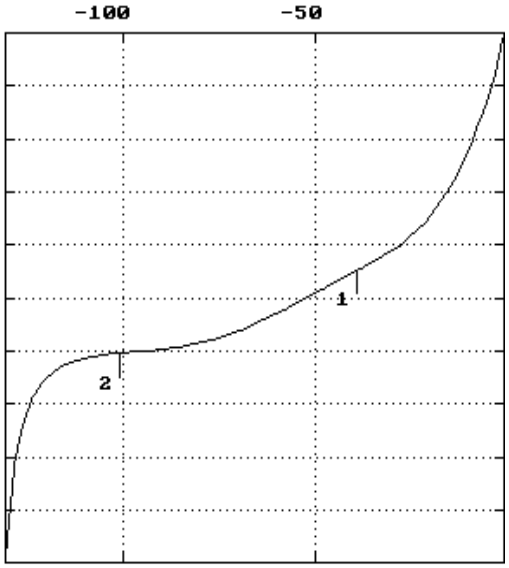
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:50        1
DOS                AddBuffe
parameters
>dosing parameters
  dispensing type:     volume
  volume                  15 ml
  disp.crit:            rate
  rate                  max. ml/min
  pause                    0 s
  time interval           10 s
  dos.element:    external D2
  temperature           25.0 °C
>stop conditions
  stop V:                 OFF
  filling rate          max. ml/min
>statistics
  status:                 OFF
>monitoring
  meas.mode:              OFF
  temperature:            OFF
  assign output L10:     none
  assign output L11:     none
  assign output L12:     none
  assign output L13:     none
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

Submethods

Ca-Mg

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:51        1
DET  U                Ca-Mg
parameters
>titration parameters
  meas.pt.density           1
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:42        1
start V      0.000 ml  DET  U      Ca-Mg
1.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-03-10      time  13:51
DET  U                Ca-Mg
def
>formula
  Ca++=EP1*C01*C02/C00
  RS1 text                Ca++
  RS1 decimal places         2
  RS1 unit:             mmol/l
  Mg++=(EP2-EP1)*C01*C02/C00
  RS2 text                Mg++
  RS2 decimal places         2
  RS2 unit:             mmol/l
  Total=EP2*C01*C02/C00
  RS3 text              Total
  RS3 decimal places         2
  RS3 unit:             mmol/l
>silo calculations
  match id:                OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C73=EP1
  C74=EP2
          ------------
```

Remarks

- Method p+m val.
  Values for the acid capacity of water.
  p value = value of Phenolphthalein
  m value = value of Methyl orange
  The method can also be used just for p+m values, see Application 2-3.

- Method Ca-Mg:
  1st break: Ca^{2+}
  2nd break: Mg^{2+}
  The method can also be used just for Ca/Mg determinations, see Application 1-7.

- **Calculations**:
  p value = p value in mmol/L (if 0.00 mmol/L, the water has an initial pH below 8.2)
  m value = m value in mmol/L (acid-binding-capability)
  Ca++ = calcium hardness in mmol/L
  Mg++ = magnesium hardness in mmol/L
  Total = total hardness in mmol/L
  C01 = concentration of titrating agent (0.05 mol/L)
  C02 = factor for conversion for mol ⇒ mmol (1000)
  C70 = initial pH of sample (p+m val.)
  C71 = EP1 at pH 8.2 (p+m val.)
  C72 = EP2 at pH 4.3 (p+m val.)
  C73 = EP1 from Ca^{2+} (Ca-Mg)
  C74 = EP2 from Mg^{2+} (Ca-Mg)

- **Electrode preparation**:
  Ca electrodes should be conditioned for 10 min. in $c(CaCl_2)$ = 0.01 mol/L before use.

- The volume of the auxiliary reagent can be optimised for the magnesium content. Rule of thumb: Ratio Mg/Acetylacetone app. 0.05.

Literature

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

Oxidizability of Waste Water

Reagents

$c(^1/_5 KMnO_4)$ = 0.01 mol/L; D0

Sample

25 mL waste water
5 mL $w(H_2SO_4)$ = 0.35 (35%)
75 mL dist. water
15 mL $c(^1/_5 KMnO_4)$ = 0.01 mol/L
15 mL $c(^1/_2$Oxalic acid) = 0.01 mol/L

Electrodes

6.0431.100 Pt Titrode; input 1

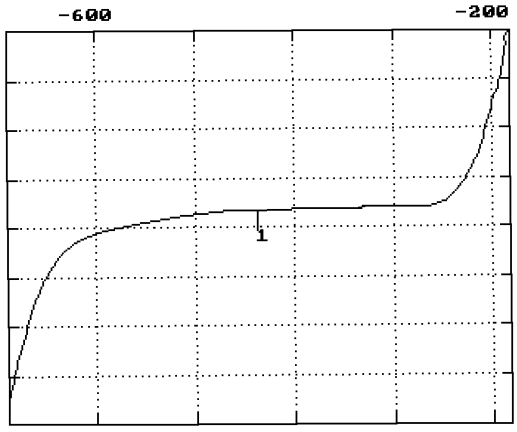
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      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
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                  7.5 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      time  16:00        2
MET  U             Oxidiza.
>calculations
Oxidiza.=EP1*C01*C02/C00;3;mg/L
C00=                    25
C01=                  0.316
C02=                   1000
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      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
751 GPD Titrino       15215     751.0010
date  97-02-24      time  15:58        2
start V       0.000 ml  MET  U   Oxidiza.
1.0 ml/div       dU=100.0 mV/div
```




```
            ============
```

Remarks

- **Prepare sample as follows**:
  Add 75 mL dist. water and 5 mL *w*(H_2SO_4) = 0.35 (35%) to 25 mL of sample and heat up to a boiling mixture. Add 15.00 mL of *c*($^1/_5KMnO_4$) = 0.01 mol/L and keep boiling for 10 minutes. Then add 15 mL *c*($^1/_2$Oxalic acid) =0.01 mol/L.
  Titrate with *c*($^1/_5KMnO_4$) = 0.01 mol/L according to parameters.

- **Calculations**:
  Oxidiza. = oxidizability in mg/L permanganate consumption
  C01 = molecular mass of $KMnO_4$ * concentration of titrating agent / normality (158 * 0.01 / 5 = 0.316 g/L)
  C02 = conversion g ⇒ mg for result expression in mg/L $KMnO_4$ (1000)

Literature

- Deutsche Einheitsverfahren zur Wasseruntersuchung
  Kapitel H4, Abschnitt 1

Total Acid Number (TAN)

Reagents

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

Sample

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

Electrodes

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

Method documentation

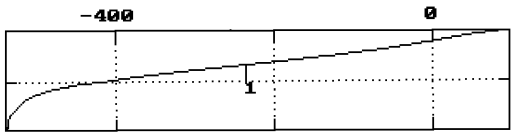
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      time  16:46        3
MET  U                  TAN
parameters
>titration parameters
  V step                 0.10 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time            50 s
  start V:                OFF
  pause                   100 s
  dos.element:    internal D0
  meas.input:           diff.
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      20 mV
  EP recognition:        last
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      time  16:46        3
MET  U                  TAN
>calculations
TAN=(EP1-C01)*C02*C03/C00;2;mg/g
C00=              1.6646
C01=                   0
C02=                 0.1
C03=              56.106

          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      time  16:45        3
U(init)          27 mV  MET U        TAN
smpl size    1.6646 g
EP1           0.680 ml          -236 mV
TAN            2.29 mg/g
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-24      time  16:45        3
start V      0.000 ml  MET  U        TAN
1.0 ml/div      dU=200.0 mV/div
```




```
          ============
```

Remarks

- **Calculations**:
  TAN = acid of sample, calculated as mg KOH per g of sample
  C01 = blank value, consumption of titrating agent by solvent mixture (0 mL)
  C02 = concentration of titrating agent (0.1 mol/L)
  C03 = molecular mass of KOH (56.106 g/mol)

- Store glass electrode in dist. water over night. Before titrating, precondition it in solvent during 10 - 30 min.

- The 6.0430.100 Ag Titrode may be used instead of the 6.0133.100 and 6.0331.000 electrodes. In this case, you make differential potentiometry with only two electrodes, the Ag Titrode and the 6.0729.100 reference electrode.

Literature

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

Total Base Number (TBN)

Reagents

$c(HClO_4)$ = 0.1 mol/L in acetic acid; D0

Sample

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

Electrodes

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

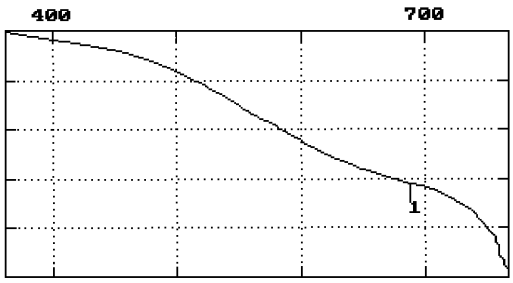
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-24      time  18:06        4
MET  U                  TBN
parameters
>titration parameters
  V step                 0.10 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time            50 s
  start V:                OFF
  pause                   100 s
  dos.element:    internal D0
  meas.input:           diff.
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      20 mV
  EP recognition:         last
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-24      time  18:06        4
MET  U                  TBN
>calculations
TBN=(EP1-C01)*C02*C03/C00;2;mg/g
C00=              1.6708
C01=                   0
C02=                 0.1
C03=              56.106
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-24      time  18:05        4
U(init)         351 mV  MET U         TBN
smpl size    1.6708 g
EP1           3.102 ml            688 mV
TBN           10.42 mg/g
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-24      time  18:05        4
start V      0.000 ml  MET U         TBN
1.0 ml/div      dU=100.0 mV/div
```




```
          ============
```

Remarks

- **Calculations**:
  TBN = base of sample, calculated as mg KOH per g of sample
  C01 = blank value, consumption of titrating agent by solvent mixture (0 mL)
  C02 = concentration of titrating agent (0.1 mol/L)
  C03 = molecular mass of KOH (56.106 g/mol)

- Store glass electrode in dist. water over night. Before titrating, precondition it in solvent during 10 - 30 min.

- The 6.0430.100 Ag Titrode may be used instead of the 6.0133.100 and 6.0331.000 electrodes. In this case, you make differential potentiometry with only two electrodes, the Ag Titrode and the 6.0729.100 reference electrode.

Literature

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

Bromine Index

Reagents

$c(BrO_3^-/Br^-)$ = 0.05 mol/L; D0

Sample

50 µL cyclohexene ≅ 10 % in solvent
25 mL solvent

Electrodes

6.0308.100 double Pt electrode; input Pol
polarized I_{pol} = 1 µA
Voltametric indication

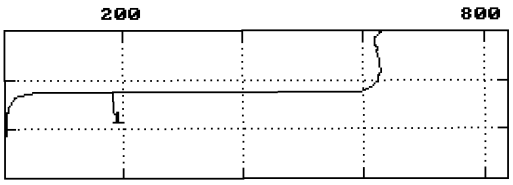
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  08:48        3
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:                OFF
  pause                     0 s
  dos.element:    internal D0
  I(pol)                    1 mA
  electrode test:         OFF
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop U                    1 mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:    greatest
  fix EP1 at U            OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  08:48        3
MET  Ipol          Br-Index
>calculations
Br-Index=(EP1-C01)*C02*C03/C00;1;mg
C00=               0.050
C01=                 0.0
C02=                0.05
C03=                7990
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  08:47        3
U(init)         295 mV MET Ipol Br-Index
smpl size     0.050 g
EP1           1.287 ml            186 mV
Br-Index    10283.1 mg
stop meas.val.reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  08:47        3
start V      0.000 ml  MET Ipol Br-Index
1.0 ml/div      dU=200.0 mV/div
```




```
            ============
```

Remarks

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

- **Solvent**:
  714 mL acetic acid
  134 mL CCl_4
  134 mL CH_3OH
  18 mL $w(H_2SO_4) = 0.2$ (20%)

- **Titrating agent**:
  Dissolve 5.1 g KBr and 1.4 g $KBrO_3$ each in dist. water and add up to 1 liter.

- **Standardization of BrO_3^-/Br^- solution**:
  Determination reaction:
  $3\ Br_2 + 6\ S_2O_3^{2-} \Rightarrow 6\ Br^- + 3\ S_4O_6^{2-}$

  Calculation:
  RS1 = EP1*C01/C00 normality of titrating agent (BrO_3^-/Br^-)
  C01 = concentration of titrating agent ($S_2O_3^{2-}$)
  C00 = mL of BrO_3^-/Br^- solution

- **Determination reaction for bromine index**:
  $BrO_3^- + 5\ Br^- + 6\ H^+ \Rightarrow 3\ Br_2 + 3\ H_2O$

  $3\ Br_2 + 3 \Rightarrow 3$ Br–Br

- **Calculations for bromine index**:
  Br-Index = bromine index
  C01 = consumption of blank sample (0 mL)
  C02 = normality of BrO_3^-/Br^- solution as calculated above
  (0.05 mol/L)
  C03 = molecular mass of Br multiplied with 100 (7990 g/mol)

Literature

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

Bromine Number

Reagents

$c(BrO_3^-/Br^-)$ = 0.5 mol/L; D0

Sample

1 mL cyclohexene ≅ 6 % in solvent
25 mL solvent

Electrodes

6.0309.100 double Pt electrode; input Pol
polarized I_{pol} = 10 µA
Voltametric indication

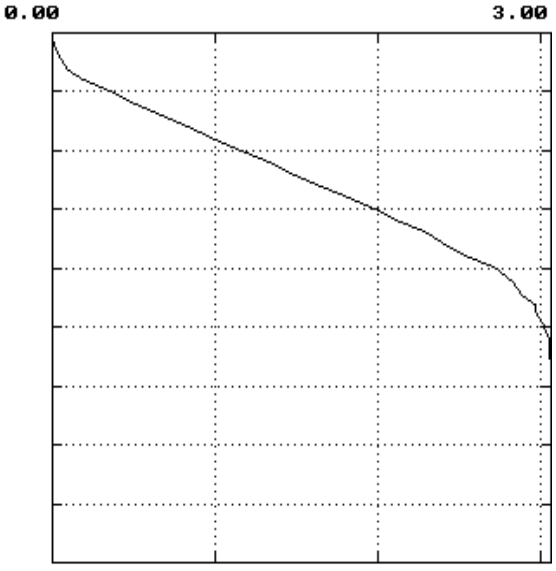
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-11      time  08:43        3
KFT  Ipol          BrNumber
parameters
>control parameters
  EP at U                 500 mV
  dynamics                500 mV
  max.rate                  5 ml/min
  min.volume incr.       min. ml
  stop crit:              time
  t(delay)                 30 s
>titration parameters
  titr.direction:           -
  pause 1                   0 s
  start V:                OFF
  pause 2                   0 s
  extr.time                 0 s
  dos.element:    internal D0
  I(pol)                   10 mA
  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:                 OFF
>preselections
  conditioning:           OFF
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-11      time  08:43        3
KFT  Ipol          BrNumber
>calculations
BrNumber=(EP1-C01)*C02*C03*C04/C00;0;
C00=                 6.00
C01=                  0.0
C02=                  0.5
C03=                 7.99
C04=                  100
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-11      time  08:42        3
                      KFT Ipol BrNumber
smpl size      6.00 g
EP1           3.060 ml
BrNumber        204
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-11      time  08:42        3
                      KFT  IpolBrNumber
10.0  s/div      dV=1.0 ml/div
```




```
          ============
```

Remarks

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

- **Solvent**:
  714 mL acetic acid
  134 mL CCl_4
  134 mL CH_3OH
  18 mL $w(H_2SO_4) = 0.2$ (20%)

- **Titrating agent**:
  Dissolve 51 g KBr and 13.92 g $KBrO_3$ each in dist. water and add up to 1 liter.

- **Standardization of BrO_3^-/Br^- solution**:
  Determination reaction:
  $3\ Br_2 + 6\ S_2O_3^{2-} \Rightarrow 6\ Br^- + 3\ S_4O_6^{2-}$

  Calculation:
  RS1 = EP1*C01/C00 normality of titrating agent (BrO_3^-/Br^-)
  C01 = concentration of titrating agent ($S_2O_3^{2-}$)
  C00 = mL of BrO_3^-/Br^- solution

- **Determination reaction for bromine index**:
  $BrO_3^- + 5\ Br^- + 6\ H^+ \Rightarrow 3\ Br_2 + 3\ H_2O$

  $3\ Br_2 + 3 \Rightarrow 3$ 

- **Calculations for bromine number**:
  BrNumber = bromine number
  C01 = consumption of blank sample (0 mL)
  C02 = normality of BrO_3^-/Br^- solution as calculated above
  (0.05 mol/L)
  C03 = molecular mass of Br_2 multiplied with 0.05 (7.99 g/mol)
  C04 = dilution factor (100)

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

Literature

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

NaCl in Broth

Reagents

$c(AgNO_3)$ = 0.1 mol/L; D0

Sample

20 mL sample solution
5 mL $c(HNO_3)$ = 2 mol/L
30 mL dist. water

Electrodes

6.0430.100 Ag Titrode; input 1

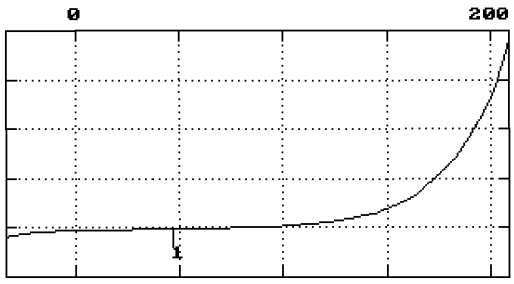
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      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
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                99.99 ml
  stop U                  OFF mV
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      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
751 GPD Titrino       15215     751.0010
date  97-02-25      time  09:59        2
start V      0.000 ml  DET  U       NaCl
1.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

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 ⇒ L and for % (0.001*100 = 0.1)

- **Sample preparation**:
  Dissolve 10 g (1 cube) conc. broth in 800 mL boiling dist. water. Rinse this solution in a 2000 mL measuring flask. Allow to cool down and fill up to the mark. Filter with a folded filter. Take 20 mL aliquots.

Literature

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

Formaldehyde Number in Fruit Juices

Reagents

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

Sample

25 mL sample (orange juice)

Electrodes

6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:06        4
TIP                FormolNo
parameters
>sequence
  1.stirrer:              ON
  2.method:          FormoPre
  3.method:          FormoDos
  4.pause                 60 s
  5.method:          FormoDet
>statistics
  status:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  meas.mode:              OFF
  temperature            25.0 °C
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:06        4
TIP                FormolNo
>calculations
FormolNo=C70*C01;1;
C01=                     4
C70=                 5.634
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:05        4
                      TIP      FormolNo
smpl size          25 ml
C70             5.634
FormolNo        22.5
TIP terminated
           ============
```

```
 'cr
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:06        4
meas.input:         1     CAL
cal.date      97-03-10
                       pH     U/mV
buffer  1           7.00       -5
buffer  2           9.00     -122
cal.temp           21.7 °C
slope(rel)       0.999       pH(as)  6.91
          ------------
```

Submethods

Preparation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:07        4
SET  pH            FormoPre
parameters
>SET1
  EP at pH               8.50
  dynamics               1.5
  max.rate              10.0 ml/min
  min.rate              25.0 ml/min
  stop crit:           drift
  stop drift              20 ml/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
  dos.element:    internal D0
  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
  activate pulse:       OFF
         ------------
```

Addition of Formaldehyde

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:08        4
DOS                FormoDos
parameters
>dosing parameters
  dispensing type:     volume
  volume                  15 ml
  disp.crit:            rate
  rate                  max. ml/min
  pause                    0 s
  time interval           10 s
  dos.element:    external D1
  temperature          25.0 °C
>stop conditions
  stop V:               OFF
  filling rate          max. ml/min
>statistics
  status:               OFF
>monitoring
  meas.mode:            OFF
  temperature:          OFF
  assign output L10:    none
  assign output L11:    none
  assign output L12:    none
  assign output L13:    none
>preselections
  req.ident:            OFF
  req.smpl size:        OFF
  activate pulse:       OFF
         ------------
```

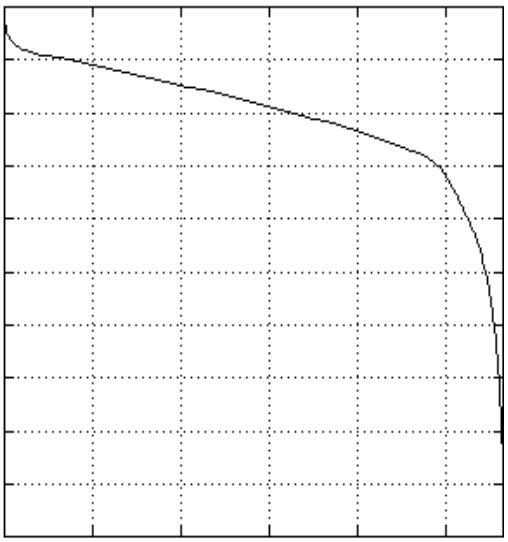
Submethods

Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:08        4
SET  pH            FormoDet
parameters
>SET1
  EP at pH               8.50
  dynamics                1.5
  max.rate               10.0 ml/min
  min.rate               25.0 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  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
  activate pulse:         OFF
          ------------
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:05        4
                      SET  pH  FormoDet
20.0  s/div      dV=1.0 ml/div

0.00                              5.00
```

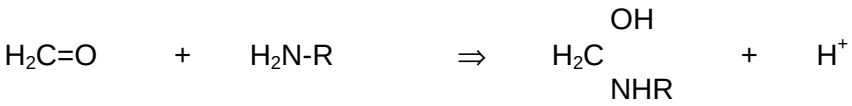


```
            ============
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-03-10      time  15:08
SET  pH            FormoDet
def
>formula
>silo calculations
  match id:               OFF
>common variables
>report
>mean
  MN1=RS1
>temporary variables
  C70=EP1
          ------------
```

Remarks

- **Determination reaction**:



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

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

- Calibrate the electrode for the SET titration.

Literature

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

Calcium in Milk Products

Reagents

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

Sample

app. 10 g milk

Electrodes

6.0502.140 Cu^{2+} sensitive indicator electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode (KNO_3 sat.)

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:51        3
TIP                 Milk-Ca
parameters
>sequence
  1.stirrer:              ON
  2.method:          Milk-Ca1
  3.pause                 40 s
  4.method:          Milk-Ca2
  5.pause                 10 s
  6.method:          Milk-Ca3
>statistics
  status:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  meas.mode:              OFF
  temperature            25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:50        3
                       TIP       Milk-Ca
smpl size   10.0516 g
C70           3.008
Calcium       0.120 %
TIP terminated
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:51        3
TIP                 Milk-Ca
>calculations
Calcium=C70*C01*C02*C03/C00;3;%
C00=             10.0516
C01=                 0.1
C02=               40.08
C03=                 0.1
C70=               3.008
         ------------
```

Submethods

Addition of Cu complex-solution

```
'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:52        3
DOS                Milk-Ca1
parameters
>dosing parameters
  dispensing type:     volume
  volume                    1 ml
  disp.crit:             rate
  rate                   max. ml/min
  pause                     0 s
  time interval            10 s
  dos.element:    external D1
  temperature            25.0 °C
>stop conditions
  stop V:                 OFF
  filling rate           max. ml/min
>statistics
  status:                 OFF
>monitoring
  meas.mode:              OFF
  temperature:            OFF
  assign output L10:     none
  assign output L11:     none
  assign output L12:     none
  assign output L13:     none
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

Addition of buffer

```
'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:52        3
DOS                Milk-Ca2
parameters
>dosing parameters
  dispensing type:     volume
  volume                10.000 ml
  disp.crit:             rate
  rate                   max. ml/min
  pause                     0 s
  time interval            10 s
  dos.element:    external D2
  temperature            25.0 °C
>stop conditions
  stop V:                 OFF
  filling rate           max. ml/min
>statistics
  status:                 OFF
>monitoring
  meas.mode:              OFF
  temperature:            OFF
  assign output L10:     none
  assign output L11:     none
  assign output L12:     none
  assign output L13:     none
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

Submethods

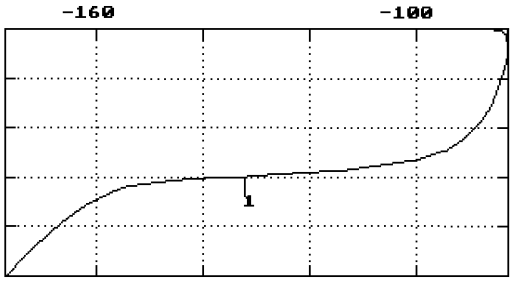
Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:52        3
DET  U             Milk-Ca3
parameters
>titration parameters
  meas.pt.density           1
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time             5 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                    5 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:52
DET  U             Milk-Ca3
def
>formula
  Calcium=EP1*C01*C02*C03/C00
  RS1 text            Calcium
  RS1 decimal places        3
  RS1 unit:                 %
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C70=EP1
          ------------
```

```
 'cf
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:53
DET  U             Milk-Ca3
C-fmla
  C01                   0.1
  C02                 40.08
  C03                   0.1
          ------------
```

```
'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  11:50        3
start V      0.000 ml  DET  U   Milk-Ca3
1.0 ml/div      dU=20.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Calcium = content of calcium in %
  C01 = concentration of titrating agent (0.1 mol/L)
  C02 = molecular mass of calcium (40.08 g/mol)
  C03 = factor for % (0.1)
  C70 = EP1 from submethod Milk-Ca3

- **Reagents**:
  c(EGTA) = 0.100 mol/L:
  38.04 g EGTA - ethylene glycol-0,0'-bis-(2-aminoethyl)-N,N,N',N'-tetraacetic acid are added to a 1 liter volumetric flask, dissolved in 250 mL *c*(NaOH) = 1 mol/L and the solution made up to the mark with dist. water.

  Cu complex:
  EGTA titrant (100 mL) is mixed with 100 mL of a solution containing 0.2 mol/L NH_4Cl and exactly 0.100 mol/L Cu(II)nitrate. Titration can used to check that this solution contains no excess of Cu(II) or EGTA.

  Buffer solution pH = 10:
  54 g NH_4Cl is dissolved in ca. 400 mL dist. water in a volumetric flask, 300 mL *w*(NH_3) = 0.25 (25%) added and the solution made up to 1 liter with dist. water.

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

Literature

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

Peroxide Number

Reagents

$c(Na_2S_2O_3)$ = 0.01 mol/L; D0
Prepared daily from c = 0.1 mol/L

Sample

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

Electrodes

6.0431.100 Pt Titrode; input 1

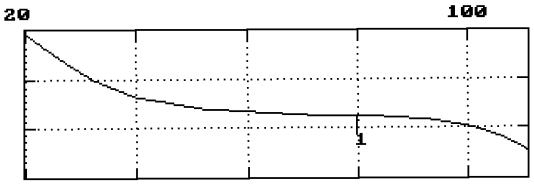
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:25        4
DET  U             Perox.No
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop U                  OFF mV
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:25        4
DET  U             Perox.No
>calculations
Perox.No=(EP1-C01)*C02/C00;2;meq/kg
C00=              5.0188
C01=                 0.0
C02=                  10
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:24        4
U(init)          20 mV DET U    Perox.No
smpl size    5.0188 g     id#1   Suprema
id#2            Oil
EP1          0.8874 ml             80 mV
Perox.No       1.77 meq/kg
stop #EP reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:24        4
start V     0.0000 ml  DET  U   Perox.No
0.5 ml/div      dU=20.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Perox.No = peroxide number in meq.O_2/kg
  C01 = consumption of blank sample (0 mL)
  C02 = factor (10)

- **Sample preparation**:
  Weigh out accurately 5 g sample in an Erlenmeyer flask and add 50 mL of the glacial acetic acid / chloroform mixture. Then add 1 mL of the KI solution and shake during 5 s. Now allow the mixture to stand for about 1 min. in a dark place. After this, rinse the contents of the Erlenmeyer flask out into a beaker with 100 mL dist. water and immediately back-titrate the iodine thus liberated with sodium thiosulphate. A blank control sample should be prepared and treated in the same way. Enter the blank value as C01.

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

Literature

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

Saponification Number

Reagents

c(HCl) = 1 mol/L; D0

Sample

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

Electrodes

6.0232.100 combined pH glass electrode; input 1

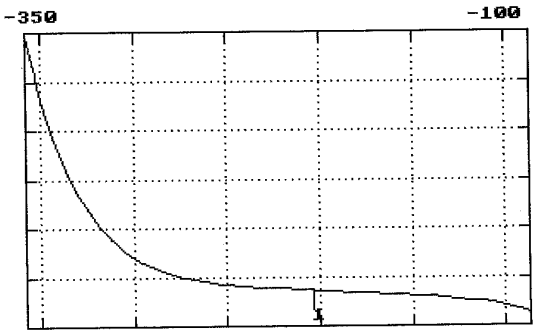
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:56        5
DET  U             Sapon.No
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop U                  OFF mV
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:56        5
DET  U             Sapon.No
>calculations
Sapon.No=(C01-EP1)*C02/C00;1;mg/g
C00=              2.0224
C01=              12.199
C02=                56.1
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:55        5
U(init)        -357 mV DET U    Sapon.No
smpl size    2.0224 g
EP1           5.271 ml           -203 mV
Sapon.No      192.2 mg/g
stop #EP reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  14:55        5
start V      0.000 ml  DET  U   Sapon.No
1.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Sapon.No = saponification number in mg KOH per g of sample
  C01 = consumption of blank sample (12.199 mL)
  C02 = mg KOH / 1 mL titrating agent (56.1)

- **Sample preparation**:
  Weigh out approx. 2 g sample in a round-bottomed flask. Add 25 mL alcoholic KOH solution plus a few boiling beads and allow to boil lightly for at least 30 min. Shake from time to time. Finally rinse the content of the round-bottomed flask into a beaker with a small quantity of dist. water and back titrate the excess of potassium hydroxide with HCl. A blank control sample should be prepared and treated identically. Enter the blank value as C01.

Literature

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

Acid Number

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

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

Electrodes

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

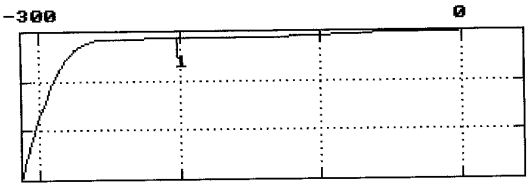
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  15:53        9
DET  U              Acid.No
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   3 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  15:53        9
DET  U              Acid.No
>calculations
Acid.No=EP1*C01/C00;2;mg/g
FFA=RS1*C02/C03;2;
C00=             10.3223
C01=                5.61
C02=                 282
C03=                 561
         ------------
```

```
'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  15:48        9
U(init)          35 mV DET U     Acid.No
smpl size   10.3223 g
EP1            0.150 ml           -201 mV
Acid.No         0.08 mg/g
FFA             0.04
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  15:48        9
start V      0.000 ml  DET  U    Acid.No
1.0 ml/div      dU=100.0 mV/div
```




```
            ============
```

Remarks

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

Literature

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

Iodine Number

Reagents

$c(Na_2S_2O_3)$ = 0.1 mol/L; D0

Sample

app. 0.5 g sunflower oil
15 mL CCl_4
25 mL *c*(ICl) = 0.1 mol/L (according to Wijs in glacial acetic acid / CCl_4, e.g. Merck No. 9163)
10 mL *w*(Hg acetate) = 0.025 (2.5%) in glacial acetic acid
15 mL *w*(KI) = 0.10 (10%)
30-50 mL dist. water

Electrodes

6.0431.100 Pt Titrode; input 1

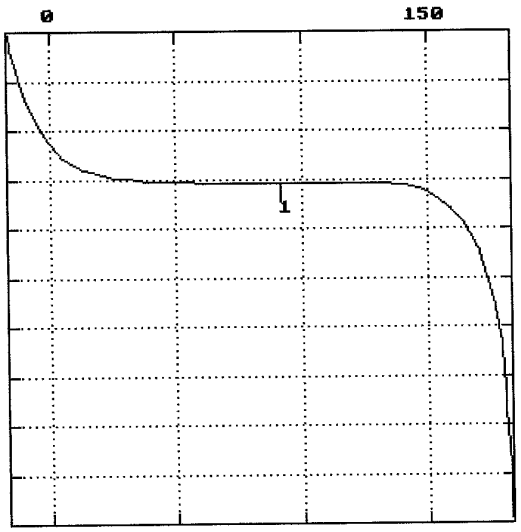
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  16:12       10
DET  U               Iod.No
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift           20.0 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   20 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      time  16:12       10
DET  U               Iod.No
>calculations
Iod.No=(C01-EP1)*C02/C00;2;g/100g
C00=              0.5204
C01=                   0
C02=              1.269
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      time  16:11       10
U(init)         -16 mV DET U      Iod.No
smpl size    0.5204 g
EP1           6.149 ml              92 mV
Iod.No       104.61 g/100g
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      time  16:11       10
start V      0.000 ml  DET  U     Iod.No
2.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Iod.No = iodine number in g iodine / 100 g sample
  C01 = consumption of blank sample (0 mL)
  C02 = molecular mass of I * concentration titrating agent * 100g sample / factor mL -> L (126.9 * 0.1 *100/1000 = 1.269)

- **Sample preparation**:
  According to the expected iodine number, weigh out 0.10 ... 1.00 g of the sample in an Erlenmeyer flask and add 15 mL CCl_4. Add 25 mL iodine monochloride solution and add 10 mL mercuric acetate solution, mix and allow to stand for 5 min. in a dark place. Then add 15 mL KI solution, rinse into a beaker with dist. water and back-titrate the excess iodine with sodium thiosulphate. A blank control sample should be prepared and treated in the same way. Enter the blank value as C01.

- You may add a solution of w(Mg-acetate) = 0.03 (3%) instead of the Hg-acetate solution.

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

Literature

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

Vitamin C

Reagents

c(DPIP) = 0.001 mol/L; D0
2,6-Dichlorophenol indophenol

Sample

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

Electrodes

6.0309.100 double Pt sheet electrode; input Pol
polarized I_{pol} = 1 µA
Voltametric indication

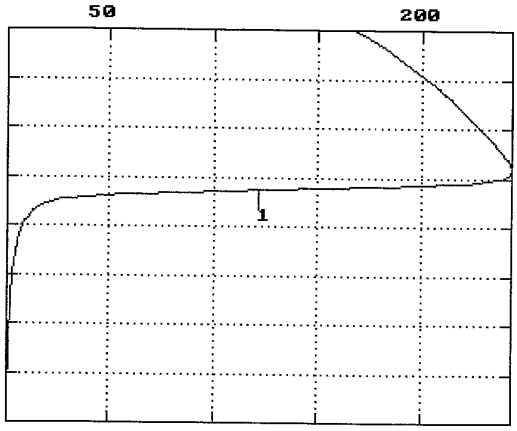
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      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
  dos.element:    internal D0
  I(pol)                    1 mA
  electrode test:         OFF
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   20 ml
  stop U                  OFF mV
  stop EP                    9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 at U            OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      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=                 0.088
C02=                  1.00
C03=                   200
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      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              269 mg/pc
Vit.C           0.2691 g/l
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      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 of titrating agent (1.00)
  C03 = dilution factor (200)

- **Titrating agent**:
  Dissolve 295 mg 2,6-Dichlorophenol indophenol with vigorous agitation in 1 L dist. water, then filter and mix with 100 mg sodium bicarbonate.
  This solution can be stored in the refrigerator for about 1 month, the factor should be checked daily with standard ascorbic acid. (As a titrating agent, the more readily soluble sodium salt may be used instead.)

- **Standard solution**:
  r (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 $Na_2S_2O_3$ solution.

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

- The 6.0431.100 Pt Titrode may be used together with mode MET U.

Literature

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

Na^+ / Cl^- in Isotonic Solution

Reagents

$c(AgNO_3)$ = 0.1 mol/L; D0

Sample

5 mL sample solution (153 mmol/L Na^+ / Cl^-)
2 mL $c(HNO_3)$ = 2 mol/L
30 mL dist. water

Electrodes

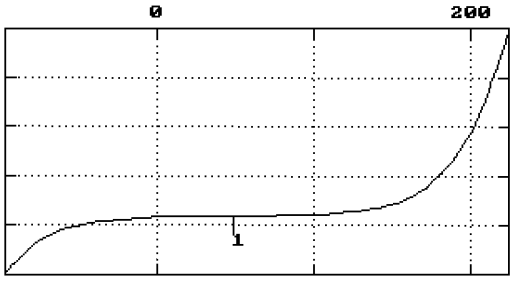
6.0430.100 Ag Titrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      time  17:52       14
DET  U               Na+Cl-
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      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
751 GPD Titrino       15215     751.0010
date  97-02-25      time  17:51       14
start V      0.000 ml  DET  U     Na+Cl-
2.0 ml/div      dU=100.0 mV/div
```




```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      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^+ / Cl^- in isotonic solution in mmol/L
  C01 = concentration of titrating agent (0.1 mol/L)
  C02 = factor for conversion mol/L ⇒ mmol/L (1000)

Literature

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

Antacid

Reagents

c(HCl) = 1.0 mol/L; D0

Sample

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

Electrodes

6.0233.100 combined pH glass electrode; input 1
6.1110.100 T sensor

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:16        3
TIP                 Antacid
parameters
>sequence
  1.stirrer:              ON
  2.method:          AntacPre
  3.info     Add smpl & start
  4.method:          AntacDet
>statistics
  status:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  meas.mode:                T
  temperature           25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:14        3
                       TIP       Antacid
smpl size     0.268 g     id#1  Trigast.
C70           3.218
acid cap     12.008 mmol/g
TIP terminated
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:16        3
TIP                 Antacid
>calculations
acid cap=C70*C01/C00;3;mmol/g
C00=               0.268
C01=                   1
C70=               3.218
         ------------
```

Submethods

Preparation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:17        3
SET  pH            AntacPre
parameters
>SET1
  EP at pH               3.00
  dynamics                  3
  max.rate                  1 ml/min
  min.rate                  1 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  meas.input:               1
  temperature            37.0 °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
  activate pulse:         OFF
          ------------
```

Submethods

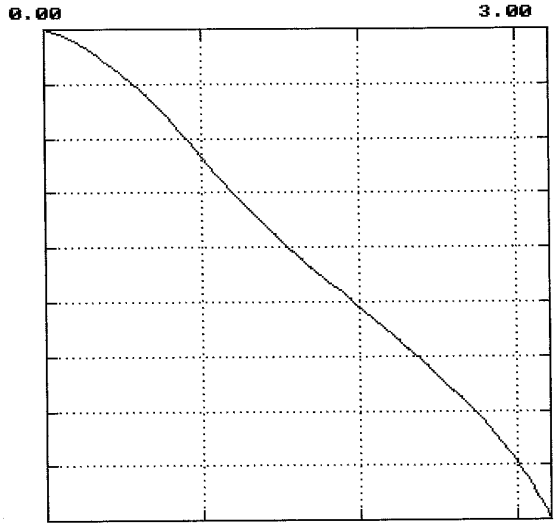
Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:17        3
STAT pH            AntacDet
parameters
>control parameters
  EP at pH                 3
  dynamics               1.2
  max.rate                15 ml/min
  min.rate                10 ml/min
>titration parameters
  start V:               OFF
  pause                    0 s
  start time               0 s
  start pH               OFF
  start rate             OFF ml/min
  time interval           30 s
  titr.direction:          -
  dos.element:    internal D0
  meas.input:              1
  temperature             37 °C
>stop conditions
  stop time:            abs.
  stop time             1800 s
  stop V:               abs.
  stop V               99.99 ml
  stop rate              OFF ml/min
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  low lim.1              OFF s
  fix V1                 450 s
  fix V2                 900 s
  fix V3                1620 s
  fix V4                1800 s
  fix V5                 OFF s
  fix time 1            0.25 V(tot)
  fix time 2             0.5 V(tot)
  fix time 3             0.9 V(tot)
  fix time 4               1 V(tot)
  fix time 5             OFF V(tot)
>monitoring
  meas.val:               ON
  low lim. pH          -20.00
  up lim. pH            20.00
  action:               none
  rate:                  OFF
  temperature:            ON
  low lim.                36 °C
  up lim.                 38 °C
  action:               none
  assign output L10:    none
  assign output L11:    none
  assign output L12:    none
  assign output L13:    none
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  display rate:          OFF
  activate pulse:        OFF
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:18
STAT pH            AntacDet
def
>formula
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:full;curve;
>mean
  MN1=RS1
>temporary variables
  C70=C41
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:13        3
pH(init)       3.48   STAT pH   AntacDet
smpl size     0.268 g     id#1  Trigast.
C80 m.rate   0.1070 +/-   0.00084 ml/min
fix V1        0.979 ml        450 s
fix V2        1.759 ml        900 s
fix V3        3.042 ml       1620 s
fix V4        3.218 ml       1800 s
fix time 1    332.9 s        0.25 V(tot)
fix time 2    825.0 s         0.5 V(tot)
fix time 3   1504.5 s         0.9 V(tot)
fix time 4   1800.0 s           1 V(tot)
stop time reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-26      time  09:13        3
start time       0 s   STAT pH  AntacDet
20 0.0  s/div     dV=1.0 ml/div
```




```
            ============
```

Remarks

- Method to determine the efficiency of tablets against an excess of acid in the stomach. Use a TIP method for the determination: The pH is adjusted to 3 with a pretitration (AntacPre), then the sample is added and the pH kept at 3 during 30 minutes (AntacDet). The determination is carried out in a thermostated titration vessel (e.g. 6.1418.220) at 37 °C.

- You may have to adjust the control parameters of the submethod “AntacDet“ according to your sample. The given parameters are optimized for products which release their base slowly (rate app. 100 µL/min).

- **Calculations**:
  acid cap = acid capacity in mmol/g
  C01 = concentration of titrating agent (1 mol/L)

- **Sample preparation**:
  Pulverize the tablet and weigh it exactly. Rinse the powder with 20 mL of pretitrated solution (pH=3) into the titration vessel during the request “Add smpl & start“ and titrate immediately.

Literature

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

Trypsin

Reagents

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

Sample

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

Electrodes

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

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:02        6
TIP                 Trypsin
parameters
>sequence
  1.stirrer:              ON
  2.method:          TrypsPre
  3.info     Add smpl & start
  4.method:          TrypsDet
>statistics
  status:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  meas.mode:                T
  temperature           25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:02        6
                        TIP      Trypsin
smpl size    0.0507 g
C70          0.1920
C71          0.0238
FIP            47.3 U/mg
FIP(reg)       46.9 U/mg
TIP terminated
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:02        6
TIP                 Trypsin
>calculations
FIP=C70*C01/C02/C00;1;U/mg
FIP(reg)=C71*C01/C00;1;U/mg
C00=              0.0507
C01=                 100
C02=                   8
C70=              0.1920
C71=              0.0238
         ------------
```

Submethods

Preparation

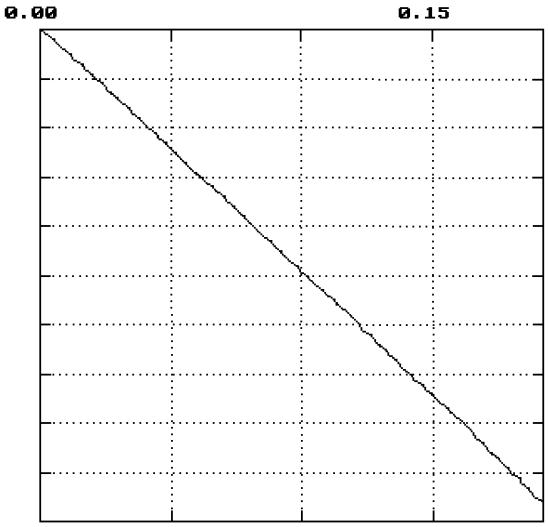
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:01        6
SET  pH            TrypsPre
parameters
>SET1
  EP at pH               8.00
  dynamics                  1
  max.rate                  1 ml/min
  min.rate                  1 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  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
  activate pulse:         OFF
         ------------
```

Submethods

Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:01        6
STAT pH            TrypsDet
parameters
>control parameters
  EP at pH                 8
  dynamics              1.00
  max.rate                 1 ml/min
  min.rate                 3 ml/min
>titration parameters
  start V:               OFF
  pause                    0 s
  start time               0 s
  start pH               OFF
  start rate             OFF ml/min
  time interval            5 s
  titr.direction:          +
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop time:            abs.
  stop time              480 s
  stop V:               abs.
  stop V               99.99 ml
  stop rate              OFF ml/min
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  low lim.1              OFF s
  fix V1                 OFF s
  fix time 1             OFF V(tot)
>monitoring
  meas.val:               ON
  low lim. pH           7.9
  up lim. pH            8.1
  action:               none
  rate:                  OFF
  temperature:            ON
  low lim.             24.9 °C
  up lim.              25.1 °C
  action:               none
  assign output L10:    none
  assign output L11:    none
  assign output L12:    none
  assign output L13:    none
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  display rate:          OFF
  activate pulse:        OFF
          ------------
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  14:01
STAT pH            TrypsDet
def
>formula
>silo calculations
  match id:              OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C70=C41
  C71=C80
          ------------
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-26      time  13:56        6
start time       0 s   STAT pH  TrypsDet
50.0  s/div      dV=0.05 ml/div
```




```
            ============
```

Remarks

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

- **Calculations**:
  FIP = calculation using the final volume, according to FIP. Result in FIP units/mg.
  FIP(reg) = calculation using the mean rate. Result in FIP units/mg. *)
  C01 = (factor for mL⇒µL) * (concentration of reagent)(100)
  C02 = determination time = 8 minutes
  C70 = final volume from TrypsDet
  C71 = mean rate from TrypsDet

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

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

- **Sample preparation**:
  Dissolve the enzyme in *c*(HCl) = 0.001 mol/L. The solution should not contain more than 50 FIP Units per mL.

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

- **Borate buffer solution pH = 8.00**:
  Dissolve 286.0 mg Disodiumtetraborate-decahydrate and 1.47 g Calciumchloride-dihydrate in 400 mL dist. water, set the pH with *c*(HCl) = 1 mol/L to 8.00 and fill up with dist. water to 500 mL.

- **Comments to parameters**:
  TrypsDet
  Monitoring of pH and temperature is ON: pH should be kept in the range of ±0.1, the temperature in ±0.1°C.
  Trypsin
  T may be measured with <MEAS/HOLD>.

Literature

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

Lipase

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

29.5 mL substrate emulsion
0.5 mL enzyme solution

Electrodes

6.0233.100 combined pH glass electrode; input 1
6.1110.100 T sensor

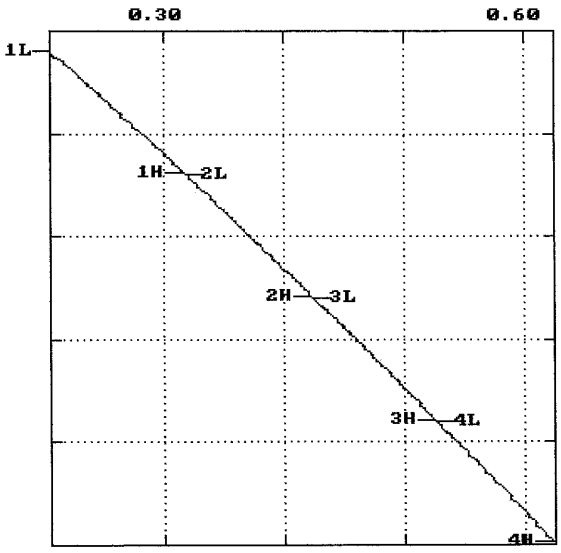
Method documentation

Results

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:53       10
pHc(init)      9.20   STAT pH   LipDeter
smpl size    0.3475 mg    id#1  Sample 2
C81 rate     0.1125 +/-   0.00033 ml/min
C82 rate     0.1064 +/-   0.00036 ml/min
C83 rate     0.1020 +/-   0.00029 ml/min
C84 rate     0.0980 +/-   0.00027 ml/min
C80 m.rate   0.1047 +/-   0.00017 ml/min
stop time reached
          ============
```

```
'fr
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:54       10
                       TIP      LipSmpSC
smpl size    0.3475 mg    id#1  Sample 2
C70          0.1047
FIP            35.0 U/mg
             mean( 2)    +/-s         s/%
FIP            35.1     0.14 U/mg   0.40
TIP terminated
           ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:53       10
start time      60 s   STAT pH  LipDeter
50.0  s/div      dV=0.1 ml/div
```




```
            ============
```

Submethods

Preparation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:55       10
SET  pH              LipPre
parameters
>SET1
  EP at pH               9.20
  dynamics                  1
  max.rate                  5 ml/min
  min.rate                  5 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  meas.input:               1
  temperature            37.0 °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
  activate pulse:         OFF
         ------------
```

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:56       10
STAT pH            LipDeter
parameters
>control parameters
  EP at pH               9.00
  dynamics                  1
  max.rate                  5 ml/min
  min.rate                 10 ml/min
>titration parameters
  start V:                OFF
  pause                     0 s
  start time               60 s
  start pH                OFF
  start rate              OFF ml/min
  time interval             1 s
  titr.direction:           +
  dos.element:    internal D0
  meas.input:               1
  temperature            37.0 °C
>stop conditions
  stop time:              abs.
  stop time                300 s
  stop V:                 abs.
  stop V                99.99 ml
  stop rate               OFF ml/min
  filling rate           max. ml/min
>statistics
  status:                 OFF
```

Determination

```
>evaluation
  low lim.1                60 s
  up lim.1                119 s
  low lim.2               120 s
  up lim.2                179 s
  low lim.3               180 s
  up lim.3                239 s
  low lim.4               240 s
  up lim.4                299 s
  low lim.5               OFF s
  fix V1                  OFF s
  fix time 1              OFF V(tot)
>monitoring
  meas.val:               OFF
  rate:                    ON
  low lim.              0.08 ml/min
  up lim.               0.16 ml/min
  action:               none
  temperature:             ON
  low lim.              36.9 °C
  up lim.               37.1 °C
  action:               none
  assign output L10:    none
  assign output L11:    none
  assign output L12:    none
  assign output L13:    none
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  display rate:            OFF
  activate pulse:          OFF
         ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:56       10
STAT pH            LipDeter
def
>formula
>silo calculation
  match id:               OFF
>common variables
>report
  report COM1:full;curve;
>mean
  MN1=RS1
>temporary variables
  C70=C80
           ------------
```

Determination of the Standard

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:57       10
TIP                  LipStd
parameters
>sequence
  1.stirrer:               ON
  2.method:             LipPre
  3.info     Add smpl & start
  4.method:           LipDeter
>statistics
  status:                  ON
  mean                n=     3
  res.tab:            original
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  meas.mode:                 T
  temperature            25.0 °C
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:57
TIP                  LipStd
def
>sequence
  1.stirrer:               ON
  2.method:             LipPre
  3.info     Add smpl & start
  4.method:           LipDeter
>formula
  Factor=C00*C01/C70
  RS1 text               Factor
  RS1 decimal places          2
  RS1 unit:
  FIP=C70*C02/C00
  RS2 text                  FIP
  RS2 decimal places          1
  RS2 unit:                U/mg
>silo calculations
  C24=RS1
  C25=RS2
  match id:                 OFF
>common variables
  C30=MN1
  C31=C26
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:57       10
TIP                  LipStd
>calculations
Factor=C00*C01/C70;2;
FIP=C70*C02/C00;1;U/mg
C00=                  1.0
C01=                 36.2
C02=                  100
C70=           not valid
          ------------
```

Determination of Samples

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:58       10
TIP                 LipSmpl
parameters
>sequence
  1.stirrer:              ON
  2.method:            LipPre
  3.info     Add smpl & start
  4.method:          LipDeter
>statistics
  status:                 ON
  mean               n=     3
  res.tab:           original
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  meas.mode:               T
  temperature           25.0 °C
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:58
TIP                 LipSmpl
def
>sequence
  1.stirrer:              ON
  2.method:            LipPre
  3.info     Add smpl & start
  4.method:          LipDeter
>formula
  FIP=C70*C30/C00
  RS1 text                FIP
  RS1 decimal places        1
  RS1 unit:              U/mg
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:58       10
TIP                 LipSmpl
>calculations
FIP=C70*C30/C00;1;U/mg
C00=                  1.0
C30=               115.46
C70=           not valid
          ------------
```

Determination of Samples Using Silo Calculations

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59       10
TIP                LipSmpSC
parameters
>sequence
  1.stirrer:               ON
  2.method:             LipPre
  3.info     Add smpl & start
  4.method:           LipDeter
>statistics
  status:                  ON
  mean               n=     3
  res.tab:           original
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  meas.mode:                 T
  temperature            25.0 °C
          ------------
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59
TIP                LipSmpSC
def
>sequence
  1.stirrer:               ON
  2.method:             LipPre
  3.info     Add smpl & start
  4.method:           LipDeter
>formula
  FIP=C70*C31/C00
  RS1 text                FIP
  RS1 decimal places        1
  RS1 unit:               U/mg
>silo calculations
  C25=RS1
  match id:               OFF
>common variables
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59       10
TIP                LipSmpSC
>calculations
FIP=C70*C31/C00;1;U/mg
C00=                  1.0
C31=               116.29
C70=               0.1047
          ------------
```

```
 'si
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59
>silo
  cycle lines:             OFF
  save line:                ON
 sl   method id#1/C21 id#2/C22 id#3/C23     C00          C24         C25
+ 1   LipStd FIP Std.                    0.3486mg     116.20        31.2U/mg
+ 2   LipStd FIP Std.                    0.3486mg     115.35        31.4U/mg
+ 3   LipStd FIP Std.                    0.3486mg     118.16        30.6U/mg
+ 4 LipSmpSC Sample 1                    0.3470mg         NV        34.8U/mg
+ 5 LipSmpSC Sample 1                    0.3470mg         NV        35.1U/mg
+ 6   LipStd FIP Std.                    0.3486mg     115.46        31.4U/mg
+ 7 LipSmpSC Sample 2                    0.3475mg         NV        35.2U/mg
/ 8 LipSmpSC Sample 2                    0.3475mg         NV        35.0U/mg
          ------------
```

```
 'ss
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59       10
  method id#1/C21 id#2/C22 id#3/C23               mean          +/-s    n
LipSmpSC Sample 2        *        *        FIP     35.1 U/mg      0.14    2
          ------------
```

```
 'sf
751 GPD Titrino       15215     751.0010
date  97-02-26      time  15:59       10
  method id#1/C21 id#2/C22 id#3/C23               mean          +/-s    n
  LipStd         *        *        *     Factor   116.29         1.301    4
                                            FIP     31.1 U/mg      0.38    4
LipSmpSC Sample 1        *        *        FIP     34.9 U/mg      0.21    2
LipSmpSC Sample 2        *        *        FIP     35.1 U/mg      0.14    2
          ------------
```

Remarks

- Pancreas lipase is determined using a TIP method: The pH is adjusted to 9.20 with a pretitration (LipPre), then the sample is added and the pH kept at 9 during 5 minutes (LipDeter). The determination is carried out in a thermostated titration vessel (e.g. 6.1418.220) at 37 °C.

- **Calculations**:
  LipStd
  Factor = calculation factor
  FIP = activity of the reference standard in FIP units/mg.
  C01 = declared activity of FIP standard (36.2)
  C02 = 1000*concentration of titrating agent (100)
  C70 = mean rate from LipDeter
  LipSmpl[1)]
  FIP = activity of the sample in FIP units/mg.
  C30 = calculation factor from LipStd
  C70 = mean rate from LipDeter
  LipSmpSC[2)]
  FIP = Aativity of the sample in FIP units/mg.
  C31 = calculation factor from silo calculation of LipStd
  C70 = mean rate from LipDeter

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

  2): Delete the following assignment in method „LipStd“:
  . in key <DEF>, >common variables, C30=MN1

- **Solution of Arabic gum**:
  Dissolve 100 g Arabic gum in 1000 mL dist. water. Centrifuge until the solution is clear (app. 10 minutes with 4000 r/min). This solution can be kept several weeks if frozen in portions.

- **Substrate standard solution**:
  Add 165 mL Arabic gum solution, 20 mL olive oil (BP 73) and 15 mL dist. water and cool it in ice to 5 °C. Homogenize with an electric mixer during 30 minutes. The temperature of the solution should never rise above 30 °C. This solution can be kept up to 14 days in a refrigerator. Always homogenize the emulsion before use.

- **Substrate emulsion**:
  Stir 100 mL substrate standard solution, 80 mL TRIS buffer solution, 20 mL sodium taurocholate and 95 mL dist. water. This solution must be prepared daily.

- **TRIS buffer solution**:
  Dissolve 60.6 mg Tris(hydroxymethyl)-amino methane ($C_4H_{11}NO_3$) and 234.0 mg sodium chloride in dist. water and fill up to 100 mL. This solution can be kept up to 3 days in a refrigerator.

- **Sodium taurocholate solution**:
  Dissolve 4.0 g sodium taurocholate (F.I.P. controlled) in dist. water and fill up to 50 mL.

- **Enzyme solvent**:
  Dissolve 10.0 g sodium chloride, 6.06 g Tris(hydroxymethyl)-amino methane and maleic acid anhydride ($C_4H_2O_3$) in 900 mL dist. water. Adjust pH to 7.0 with 4N NaOH (app. 13 mL) and fill up to 1000 mL. This solution can be kept up to 3 days in a refrigerator.

- **Sample preparation**:
  Dissolve the enzyme in the enzyme solvent. The solution should contain between 8 and 16 FIP Units per mL.

- **Comments to parameters**:
  LipDeter
  Time windows for rate evaluation.
  Monitoring of rate and temperature is ON: the rate should be kept in the range of 0.08...0.16 mL/min, the temperature in ±0.1°C.
  LipStd, LipSmpl, LipSmpSC
  T may be measured with <MEAS/HOLD>.

Literature

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

2-Aminophenol

Reagents

$c(NaNO_2)$ = 0.2 mol/L; D0

Sample

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

Electrodes

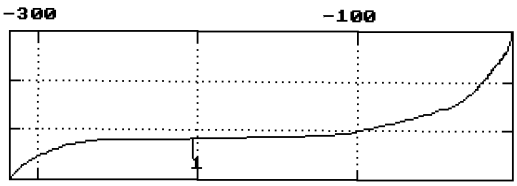
6.0431.100 Pt Titrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      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
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop V:               abs.
  stop V                  17 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 at U           OFF mV
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:       value
  activate pulse:        OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      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
751 GPD Titrino       15215     751.0010
date  97-02-27      time  11:32       18
start V     10.000 ml  MET  U      Diazo
2.0 ml/div      dU=100.0 mV/div
```

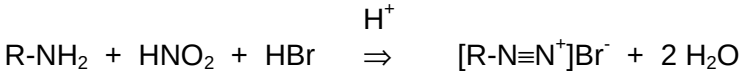



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      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**:



$$R\text{-}NH_2 + HNO_2 + HBr \xrightarrow{H^+} [R\text{-}N{\equiv}N^+]Br^- + 2\,H_2O$$

- **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; D0
2,6-Dichlorphenol indophenol

Sample

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

Electrodes

6.0309.100 double Pt sheet electrode; input Pol
polarized I_{pol} = 1 µA
Voltametric indication

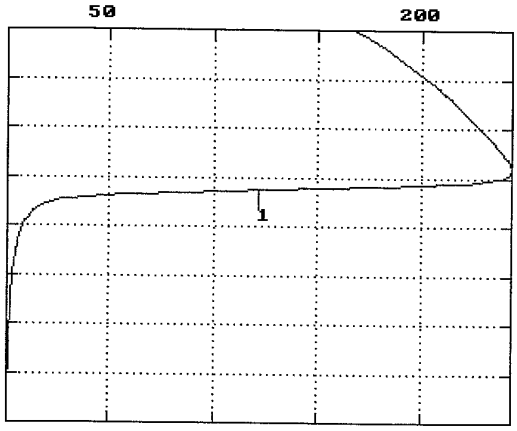
Method documentation

```
'pa
751 GPD Titrino       15215     751.0010
date  97-02-25      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
  dos.element:    internal D0
  I(pol)                    1 mA
  electrode test:         OFF
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   20 ml
  stop U                  OFF mV
  stop EP                    9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 at U            OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-25      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=                 0.088
C02=                  1.00
C03=                   200
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-25      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              269 mg/pc
Vit.C           0.2691 g/l
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-25      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)

- **Titrating agent**:
  Dissolve 295 mg 2,6-dichlorphenol indophenol with vigorous agitation in 1 L dist. water, then filter and mix with 100 mg sodium bicarbonate. This solution can be stored in the refrigerator for about 1 month, the factor should be checked daily with standard ascorbic acid. (As a titrating agent, the more readily soluble sodium salt may be used instead.)

- **Standard solution**:
  r (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 $Na_2S_2O_3$ solution.

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

- The 6.0431.100 Pt Titrode may be used together with mode MET U.

Literature

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

EDTA/NTA in Detergents

Reagents

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

Sample

10 mL sample solution
10 mL buffer pH = 9.6 [$c(NH_3/NH_4NO_3)$ = 1 mol/L]
2 mL $c(Na_2EDTA)$ = 0.01 mol/L
30 mL dist. water

Electrodes

6.0502.140 Cu^{2+} sensitive indicator electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode (KNO_3 sat.)

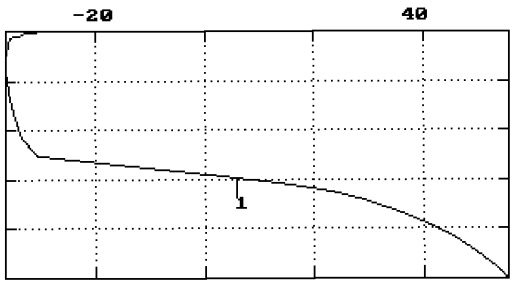
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      time  14:28       19
DET  U             EDTA-NTA
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      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
751 GPD Titrino       15215     751.0010
date  97-02-27      time  14:27       19
start V      0.000 ml  DET  U   EDTA-NTA
1.0 ml/div      dU=20.0 mV/div
```




```
            ============
```

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 $c(Cu^{2+})$ = 0.01 mol/L = 2.9225 mg EDTA
  C04 = 1 mL $c(Cu^{2+})$ = 0.01 mol/L = 1.9114 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.

- Na_2EDTA is added to get a greater break. Treat the amount of Na_2EDTA added like a blank value.

Literature

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

Non-ionic Surfactants

Reagents

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

Sample

5 mL sample solution
3 mL *c*($BaCl_2$) = 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)

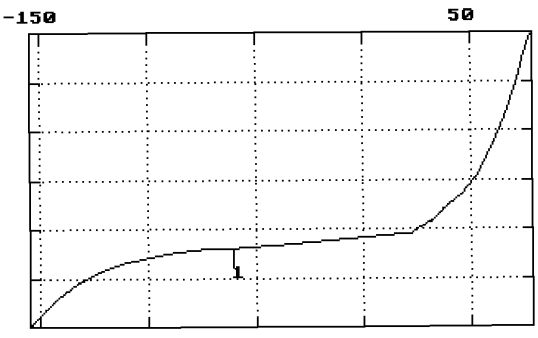
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      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
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   12 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      10
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      time  15:54       20
DET  U              Tenside
>calculations
Nonionic=EP1*C01*C02/C00;4;meq/g
C00=              0.5145
C01=                0.01
C02=                  20
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      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
751 GPD Titrino       15215     751.0010
date  97-02-27      time  15:47       20
start V      0.000 ml  DET  U    Tenside
2.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Titrating 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.

- **$BaCl_2$ solution**:
  Dissolve 21 g $BaCl_2$ or 25 g $BaCl_2$*$2H_2O$ in dist. water, add 1 mL conc. HCl and fill up to 1 liter.

- **Buffer pH = 10**:
  Dissolve 1.24 g H_3BO_4 in dist. water, add 10 mL *c*(NaOH) = 1 mol/L and fill up to 100 mL.

- **Sample preparation**:
  Dissolve app. 0.5 ... 1 g detergent (Renex 650 ICI; polyoxyethylene-(30)-nonylphenol, molecular mass ≈ 1541 g/mol) in 100 mL dist. water.

- **Calculations**:
  Nonionic = concentration of nonionic Tensides in meq/g
  C01 = concentration of titrating agent (0.01 mol/L)
  C02 = dilution factor for the sample solution, aliquot (20)

Literature

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

Cationic Surfactants in Fabric Softener

Reagents

c(DOS) = 0.01 mol/L; D0
Dioctylsodium sulfosuccinat M(DOS) = 444.57 g/mol, Fluka No. 86139

Sample

app. 1 g sample (fabric softener)
5 mL Methanol puriss p.a.
5 mL Buffer solution pH = 3, citrate/HCl, e.g. Merck Titrisol No. 9883
1 mL *c*(HCl) = 2 mol/L
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)

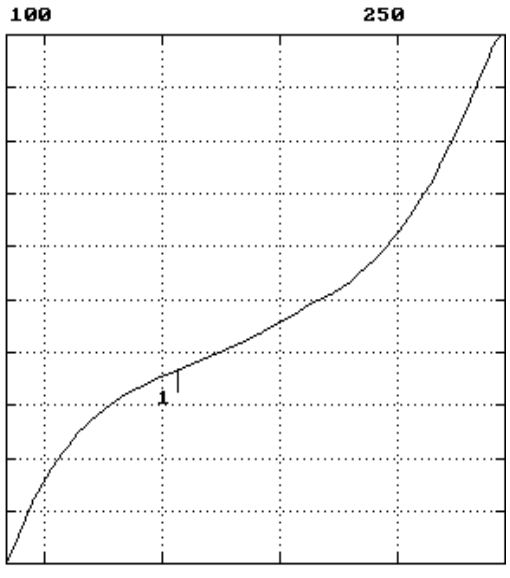
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:54        8
DET  U             Tens.Cat
parameters
>titration parameters
  meas.pt.density           3
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time            10 s
  start V:                OFF
  pause                    30 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                    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
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:54        8
DET  U             Tens.Cat
>calculations
Cationic=EP1*C01/C00;4;meq/g
C00=              1.0631
C01=                0.01
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:51        8
U(init)          300 mV DET U    Tens.Cat
smpl size     1.0631 g
EP1           12.631 ml            157 mV
Cationic      0.1188 meq/g
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:51        8
start V       0.000 ml  DET  U   Tens.Cat
2.0 ml/div       dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Cationic = concentration of cationic surfactants in meq/g
  C01 = concentration of titrating agent (0.01 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 woth 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 or the titrating agent 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 in Shampoo

Reagents

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

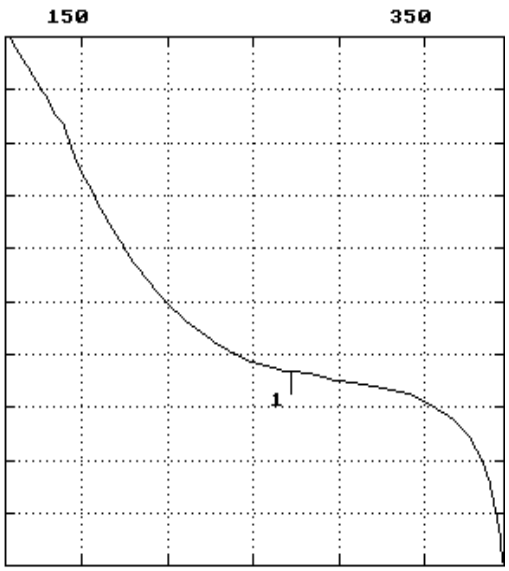
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:20        6
DET  U              Tens.An
parameters
>titration parameters
  meas.pt.density           3
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time            10 s
  start V:                OFF
  pause                    30 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   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
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      time  16:21        6
DET  U              Tens.An
>calculations
Anionic=EP1*C01/C00;4;meq/g
C00=              0.2128
C01=               0.004
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      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
751 GPD Titrino       15215     751.0010
date  97-03-04      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 woth 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.

Perborates in Detergents

Reagents

$c(KMnO_4)$ = 0.02 mol/L; D0

Sample

5 mL sample solution
20 mL $w(H_2SO_4)$ = 0.30 (30%)

Electrodes

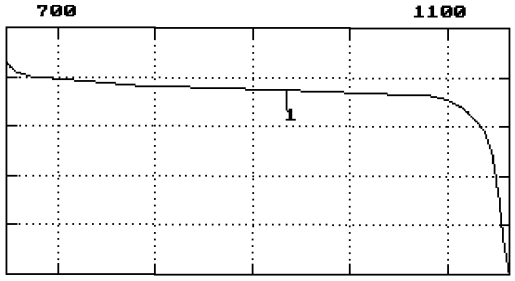
6.0420.100 LL combined Pt electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      time  17:33       21
MET  U             Perborat
parameters
>titration parameters
  V step                0.10 ml
  dos.rate              max. ml/min
  signal drift           OFF mV/min
  equilibr.time           10 s
  start V:              abs.
  start V                0.5 ml
  dos.rate              max. ml/min
  pause                   30 s
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop V:               abs.
  stop V                   5 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 at U           OFF mV
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      time  17:29       21
U(init)         697 mV MET U    Perborat
smpl size    2.5023 g
EP1           1.289 ml            933 mV
Perborat       7.93 %
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-27      time  17:29       21
start V      0.500 ml  MET  U   Perborat
1.0 ml/div      dU=100.0 mV/div
```




```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      time  17:33       21
MET  U             Perborat
>calculations
Perborat=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=              2.5023
C01=                0.02
C02=                 2.5
C03=              153.86
C04=                 0.1
C05=                  20
         ------------
```

Remarks

- **Determination reaction**:

  $5\ NaBO_2{*}H_2O_2 + 2\ MnO_4^- + 6\ H^+ \Rightarrow 2\ Mn^{2+} + 5\ O_2 + 8\ H_2O + 5NaBO_2$

- **Calculations**:
  Perborat = fraction of perborates in %
  C01 = concentration of titrating agent (0.02 mol/L)
  C02 = factor for "normality" (5/2=2.5)
  C03 = molecular mass of $NaBO_2{*}H_2O_2{*}3H_2O$ (153.86 g/mol)
  C04 = conversion mL $\rightarrow$ L * factor for % (0.001*100 = 0.1)
  C05 = factor for dilution (20)

- **Sample preparation**:
  Weigh 2.5 g in a measuring flask of 100 mL. Dissolve sample with $c(H_2SO_4)$ = 1 mol/L and wait for the reaction of the carbonate. When gas evolution has stopped, fill measuring flask to the mark. Pipette 5 mL of solution, add 20 mL of $w(H_2SO_4)$ = 0.30 (30%) and titrate.

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

Literature

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

Pure Silver Content

Reagents

c(KBr) = 0.1 mol/L; D0

Sample

app. 250 mg Ag
10 mL HNO_3 65 %
100 mL dist. water

Electrodes

6.0404.100 combined massive Ag electrode with AgBr coating; input 1

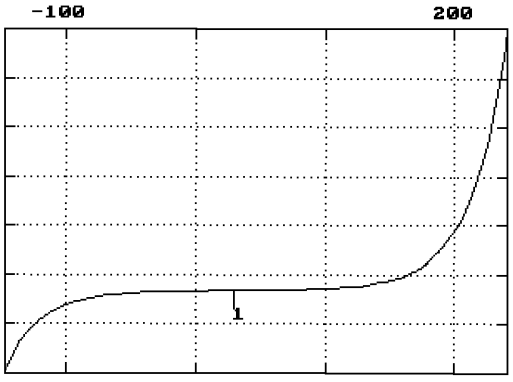
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  14:16        6
DET  U               Silver
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:               abs.
  start V                  17 ml
  dos.rate               max. ml/min
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   24 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-28      time  14:16        6
DET  U               Silver
>calculations
Silver=EP1*C01*C02*C03*C04/C00;3;%
C00=               257.7
C01=                 0.1
C02=                 1.0
C03=              107.87
C04=                 100
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-28      time  14:14        6
U(init)          280 mV DET U      Silver
smpl size      257.7 mg
EP1           22.332 ml             30 mV
Silver        93.479 %
stop V reached
          ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-02-28      time  14:14        6
start V     17.000 ml  DET  U     Silver
1.0 ml/div      dU=100.0 mV/div
```




```
            ============
```

Remarks

- **Calculations**:
  Silver = content of silver in % (purity)
  C01 = concentration of titrating agent (0.1 mol/L)
  C02 = titer (1.0)
  C03 = molecular mass of Ag (107.87 g/mol)
  C04 = factor for % (100)

- **Sample preparation**:
  Heat silver in HNO_3. Allow nitrous fumes to evaporate.

- **AgBr coating of electrode**:
  Clean Ag electrode with scouring agent and electrolyse in *w*(HBr) = 0.1 (10%) during 2 h with 5 mA using a 6.0305.000 Pt electrode as cathode. Stir during electrolysis.

Literature

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

Metals

Reagents

$c(Na_2EDTA)$ = 0.1 mol/L; D0

Sample

2 mL $c(ZnSO_4) \cong$ 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^{2+} sensitive indicator electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode (KNO_3 sat.)

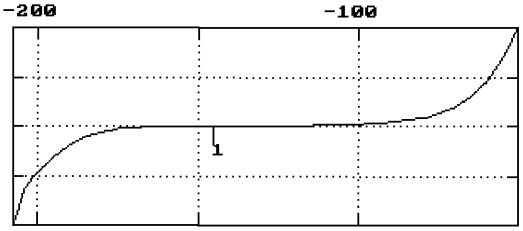
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  10:44        3
DET  U               Metals
parameters
>titration parameters
  meas.pt.density           2
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          all
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-28      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
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-28      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
751 GPD Titrino       15215     751.0010
date  97-02-28      time  10:43        3
start V      0.000 ml  DET  U     Metals
1.0 ml/div       dU=50.0 mV/div
```




```
            ============
```

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_4Cl and 350 mL $w(NH_3)$ = 0.25 (25%) in dist. water and fill up to 1 litre.

- **Buffer pH = 4.7**:
  Dissolve 123 g Na-acetate and 86 mL acetic acid in dist. water and fill up to 1 liter.

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

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

Literature

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

Aluminium

Reagents

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

Sample

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

Electrodes

6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:00        3
TIP                Aluminum
parameters
>sequence
  1.stirrer:              ON
  2.method:             Al 1
  3.method:             Al 2
  4.method:             Al 3
  5.pause                180 s
  6.method:             Al 4
>statistics
  status:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:       value
  meas.mode:              pH
  meas.input:              1
  temperature           25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-28      time  16:59        3
                       TIP      Aluminum
smpl size    0.2986 g
C70           0.193
C71           2.684
Aluminum      8.666 %
TIP terminated
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:00        3
TIP                Aluminum
>calculations
Aluminum=(C70+C71)*C01*C02*C03/C04/C00;3
;%
C00=              0.2986
C01=                   1
C02=               8.994
C03=                 100
C04=                1000
C70=               0.193
C71=               2.684
         ------------
```

Submethods

pH adjustment

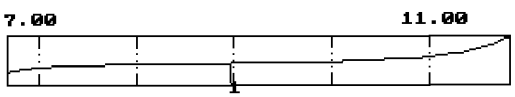
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:00        3
SET  pH                Al 1
parameters
>SET1
  EP at pH              11.50
  dynamics                 2
  max.rate              10.0 ml/min
  min.rate              25.0 ml/min
  stop crit:           drift
  stop drift               20 ml/min
>SET2
  EP at pH               OFF
>titration parameters
  titr.direction:       auto
  pause 1                  0 s
  start V:               OFF
  pause 2                  0 s
  extr.time                0 s
  dos.element:    external D1
  meas.input:              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
  activate pulse:        OFF
          ------------
```

Titration of excess of NaOH

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:01        3
DET  pH                Al 2
parameters
>titration parameters
  meas.pt.density          4
  min.incr.             10.0 ml
  dos.rate              max. ml/min
  signal drift            50 mV/min
  equilibr.time           26 s
  start V:               OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop V:                abs.
  stop V                99.99 ml
  stop pH                OFF
  stop EP                  1
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                      5
  EP recognition:        all
  fix EP1 at pH          OFF
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
          ------------

 'de
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:01
DET  pH                Al 2
def
>formula
  excess=C41-EP1
  RS1 text             excess
  RS1 decimal places        3
  RS1 unit:                ml
>silo calculations
  match id:              OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C70=RS1
          ------------


'cu
751 GPD Titrino       15215     751.0010
date  97-02-28      time  16:53        3
start V      0.000 ml  DET  pH      Al 2
1.0 ml/div      dpH=1.0/div
```




```
            ============
```

Submethods

Addition of KF

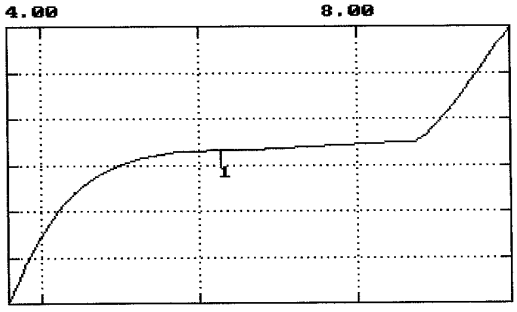
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:02        3
DOS                    Al 3
parameters
>dosing parameters
  dispensing type:     volume
  volume                    5 ml
  disp.crit:             rate
  rate                   max. ml/min
  pause                     0 s
  time interval            10 s
  dos.element:    external D2
  temperature            25.0 °C
>stop conditions
  stop V:                 OFF
  filling rate           max. ml/min
>statistics
  status:                 OFF
>monitoring
  meas.mode:              OFF
  temperature:            OFF
  assign output L10:     none
  assign output L11:     none
  assign output L12:     none
  assign output L13:     none
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:03        3
DET  pH                Al 4
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                    6 ml
  stop pH                 OFF
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------

 'de
751 GPD Titrino       15215     751.0010
date  97-02-28      time  17:03
DET  pH                Al 4
def
>formula
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C71=EP1
         ------------

 'cu
751 GPD Titrino       15215     751.0010
date  97-02-28      time  16:58        3
start V      0.000 ml  DET  pH      Al 4
1.0 ml/div      dpH=2.0/div
```




```
            ============
```

Remarks

- **Determination reaction**:

  $Al(OH)_3 + 6\ KF \Rightarrow K_3AlF_6 + 3\ OH^-$

- **Calculations**:
  Aluminum = content of Al in %
  C01 = concentration of titrating agent (1 mol/L)
  C02 = molecular mass / "normality" (26.982 / 3 = 8.994)
  C03 = factor for % (100)
  C04 = factor for mL → L (1000)
  C70 = excess of the method "Al 2" (stop V - EP1)
  C71 = consumption in method "Al 4" (EP1)

Literature

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

Phosphorous in Fertilizer

Reagents

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

Sample

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

Electrodes

6.0232.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:12        3
TIP                P2O5Fert
parameters
>sequence
  1.stirrer:              ON
  1.method:           P2O5-1
  2.method:           P2O5-2
  3.pause                 30 s
  4.method:           P2O5-3
>statistics
  status:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  meas.mode:             OFF
  temperature           25.0 °C
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:11        3
                       TIP      P2O5Fert
smpl size        10 ml
C70           1.031
C71          10.614
P2O5           8.27 %
TIP terminated
          ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:12        3
TIP                P2O5Fert
>calculations
P2O5=(C70+C71)*C01*C02/C00;2;%
C00=                  10
C01=                   1
C02=                 7.1
C70=               1.031
C71=              10.614
         ------------
```

Submethods

Titration of acid

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:13        3
DET  pH              P2O5-1
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop pH                 OFF
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

Addition of Na-oxalate

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:14        3
DOS                  P2O5-2
parameters
>dosing parameters
  dispensing type:     volume
  volume                   15 ml
  disp.crit:             rate
  rate                   max. ml/min
  pause                     0 s
  time interval            10 s
  dos.element:    external D1
  temperature            25.0 °C
>stop conditions
  stop V:                 OFF
  filling rate           max. ml/min
>statistics
  status:                 OFF
>monitoring
  meas.mode:              OFF
  temperature:            OFF
  assign output L10:     none
  assign output L11:     none
  assign output L12:     none
  assign output L13:     none
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

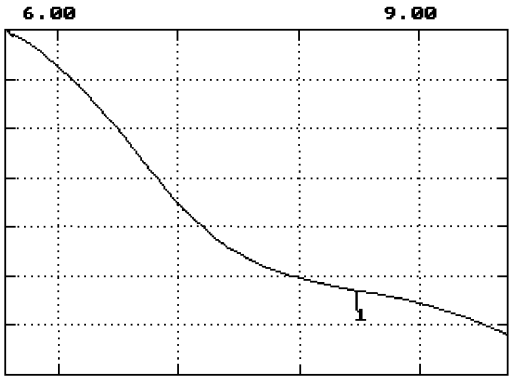
```
 'de
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:13
DET  pH              P2O5-1
def
>formula
  excess=C41-EP1
  RS1 text             excess
  RS1 decimal places        3
  RS1 unit:                ml
>silo calculations
  match id:               OFF
>common variables
>report
>mean
  MN1=RS1
>temporary variables
  C70=RS1
          ------------
```

Submethods

Determination

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:14        3
DET  pH              P2O5-3
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop pH                 OFF
  stop EP                   1
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:      window
  low lim.1 pH              7
  up lim.1 pH               9
  low lim.2 pH            OFF
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:11        3
start V      0.000 ml  DET  pH    P2O5-3
2.0 ml/div      dpH=1.0/div
```




```
            ============
```

```
 'de
751 GPD Titrino       15215     751.0010
date  97-03-03      time  09:14
DET  pH              P2O5-3
def
>formula
>silo calculations
  match id:               OFF
>common variables
>report
  report COM1:curve;
>mean
  MN1=RS1
>temporary variables
  C71=EP1
          ------------
```

Remarks

- **Determination reaction**:

HCl	+	NaOH	⇒	NaCl	+	H_2O (1)
H_3PO_4	+	NaOH	⇒	NaH_2PO_4	+	H_2O (1)
Ca^{2+}	+	$(COO)_2^{2-}$	⇒	$Ca(COO)_2\downarrow$		(2)
NaH_2PO_4	+	NaOH	⇒	Na_2HPO_4	+	H_2O (3)

- **Calculations**:
  P2O5 = content of P_2O_5 in %
  C01 = concentration of titrating agent (1 mol/L)
  C02 = equivalent weight P_2O_5 in g/mol(71*100/1000=7.1)
  C70 = volume in mL of excess titrant in the 1st titration (P2O5-1)
  C71 = volume in mL of the titrant consumed in the 2nd titration (P2O5-3)

Literature

- Metrohm Application Bulletin No. 240: Fully automatic determination of the phosphorous content (P_2O_5) in fertilizers

Epoxy Number

Reagents

$c(HClO_4)$ = 0.1 mol/L; D0

Sample

app. 0.2 g UHU resin
25 mL *c*(TBA) = 0.2 mol/L in acetic acid
TBA = Tetrabutyl ammonium bromide

Electrodes

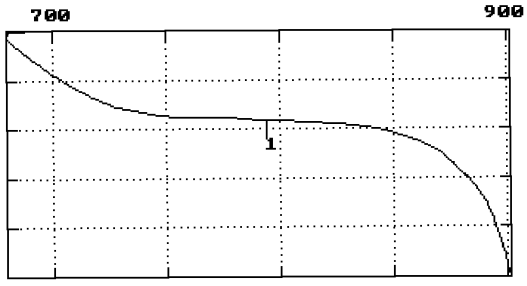
6.0430.100 Ag Titrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  13:28        2
DET  U             Epoxy.No
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift            OFF mV/min
  equilibr.time            20 s
  start V:                OFF
  pause                     0 s
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   12 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-03      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
751 GPD Titrino       15215     751.0010
date  97-03-03      time  13:27        2
start V      7.000 ml  DET  U   Epoxy.No
1.0 ml/div      dU=50.0 mV/div
```

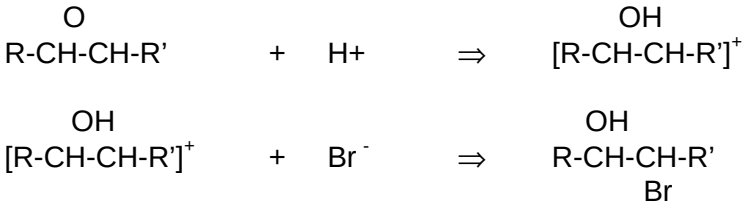



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-03      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 ⇒ 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, "Titrationen in nichtwässrigen Medien"
  Ferdinand Enke Verlag, Stuttgart (1970), p. 598

Nitrating Acid

Reagents

c(Cyclohexylamine) = 0.5 mol/L; D0

Sample

3 mL nitrating acid
25 mL methanol

Electrodes

6.0133.100 pH glass electrode; input 1
6.0726.100 Ag/AgCl double junction reference electrode
(*c*($LiClO_4$) = 1 mol/L in acetic acid)

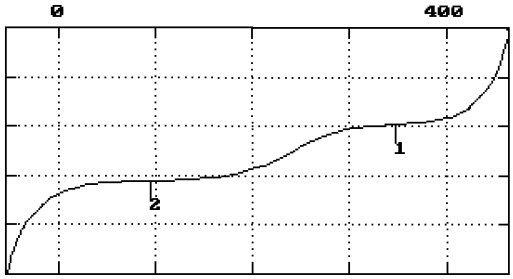
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  15:52        3
DET  U             NitrAcid
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   10 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-03      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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-03      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
751 GPD Titrino       15215     751.0010
date  97-03-03      time  15:48        3
start V      0.000 ml  DET  U   NitrAcid
1.0 ml/div      dU=100.0 mV/div
```




```
            ============
```

Remarks

- **Nitrating acid**:
  1 mL H_2SO_4 (96 %, δ = 1.84 g/L)
  1 mL HNO_3 (app. 65 %)
  20 mL dist. water
  fill up with methanol to 100 mL

- 1st break: $HNO_3 + H_2SO_4 + 2\ CHA \Rightarrow NO_3^- + HSO_4^- + 2\ HCHA^+$

  2nd break: $HSO_4^- + CHA \Rightarrow SO_4^{--} + HCHA^+$

  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_2SO_4 (98.08 g/mol)
  C03 = molecular mass of HNO_3 (63.01 g/mol)

Literature

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

Hydrogen Peroxide

Reagents

$c(KMnO_4)$ = 0.02 mol/L; D0

Sample

2 mL sample
2 mL $c(H_2SO_4)$ = 0.5 mol/L
0.1 g $MnSO_4*H_2O$ (catalyst)
100 mL dist. water

Electrodes

6.0431.100 Pt Titrode; input 1

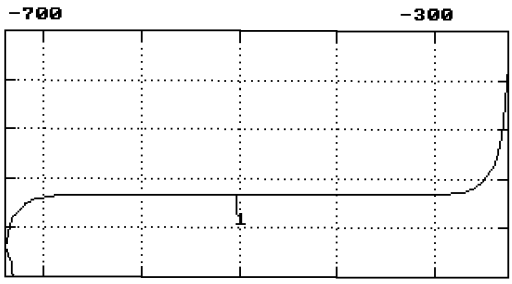
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-03      time  16:39        4
DET  U                 H2O2
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  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
  dos.element:    internal D0
  meas.input:               1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                    6 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-03      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
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-03      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
751 GPD Titrino       15215     751.0010
date  97-03-03      time  16:33        4
start V      1.000 ml  DET  U       H2O2
1.0 ml/div      dU=100.0 mV/div
```




```
          ============
```

Remarks

- **Determination reaction**:

  $5\ H_2O_2\ +\ 2\ MnO_4^-\ +\ 6\ H^+ \quad \Rightarrow \quad 2\ Mn^{2+}\ +\ 5\ O_2\ +\ 8\ H_2O$

- **Sample preparation**:
  app. 1.2 g $w(H_2O_2) \cong 0.30$ (30%) in 100 mL $c(H_2SO_4) = 0.5$ mol/L

- **Calculations**:
  H2O2 = fraction of H_2O_2 in %
  C01 = concentration of titrating agent * titer (0.02 mol/L)
  C02 = factor for “normality“ (5/2 = 2.5)
  C03 = molecular mass of H_2O_2 (34.04 g/mol)
  C04 = factor for conversion 1 mL→L* factor for % (0.001 * 100 = 0.1)
  C05 = dilution factor (50)

- The start V in this method meets two purposes:
  1. It speeds up the titration.
  2. Mn^{2+} is generated which serves as catalyst for the reaction so there is no need to add Mn^{2+} separately.

Literature

Phosphate

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

5 mL *c*(NaH_2PO_4) ≅ 0.1 mol/L
40 mL dist. water
adjust the pH value to 4.2 with dilute NaOH or H_2SO_4
10 mL *c*($La(NO_3)_3$) = 0.1 mol/L, pH = 4.2

Electrodes

6.0232.100 combined pH glass electrode; input 1

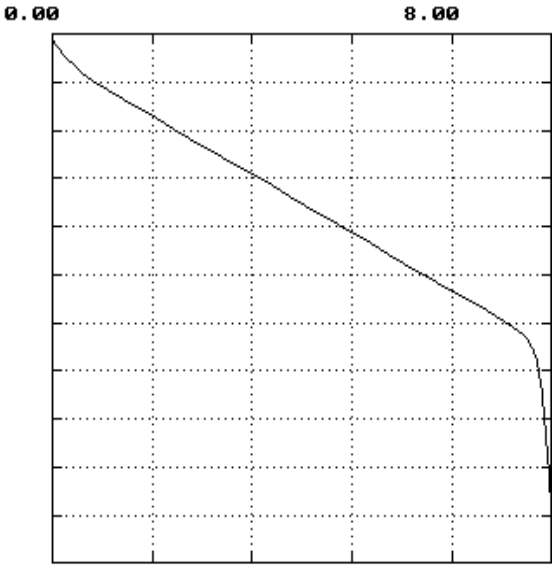
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-05      time  11:49        7
SET  pH            Phosphat
parameters
>SET1
  EP at pH               4.20
  dynamics                  1
  max.rate                 10 ml/min
  min.rate                 25 ml/min
  stop crit:            drift
  stop drift               20 ml/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
  dos.element:    internal D0
  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
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-05      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
751 GPD Titrino       15215     751.0010
date  97-03-05      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
751 GPD Titrino       15215     751.0010
date  97-03-05      time  11:48        7
                       SET  pH  Phosphat
10.0  s/div      dV=2.0 ml/div
```




```
            ============
```

Remarks

- **Determination reaction**:

  $NaH_2PO_4 + La(NO_3)_3 \Rightarrow LaPO_4(\downarrow) + 2\ HNO_3 + NaNO_3$

  $2\ HNO_3 + 2\ NaOH \Rightarrow 2\ NaNO_3 + 2\ H_2O$

- **Calculations**:
  P = content of P in %
  P2O5 = content of P_2O_5 in %
  PO4 = content of PO_4 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_2O_5
  C04 = 1 mL *c*(NaOH) = 0.1 mol/L = 4.7486 mg PO_4

- **Sample preparation**:
  Adjust the pH of your sample aliquot to 4.2 by adding either NaOH or H_2SO_4. Add 10 mL *c*($La(NO_3)_3$ = 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

$c(NaNO_2)$ = 0.2 mol/L; D0

Sample

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

Electrodes

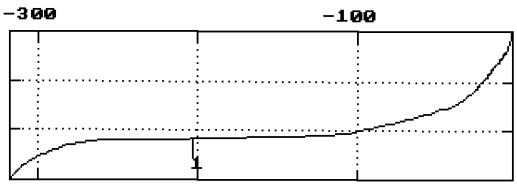
6.0431.100 Pt Titrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      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
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop V:               abs.
  stop V                  17 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 at U           OFF mV
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:       value
  activate pulse:        OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      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
751 GPD Titrino       15215     751.0010
date  97-02-27      time  11:32       18
start V     10.000 ml  MET  U      Diazo
2.0 ml/div      dU=100.0 mV/div
```

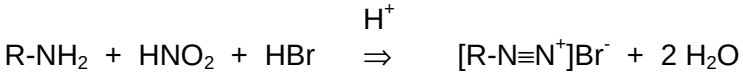



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      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; D0

Sample

2.00 mL white liquor
white liquor is a mixture of NaOH, Na_2S (pK_1=7.04, pK_2=11.96), Na_2CO_3 (pK_1=6.37, pK_2=10.25)
50 mL dist. water

Electrodes

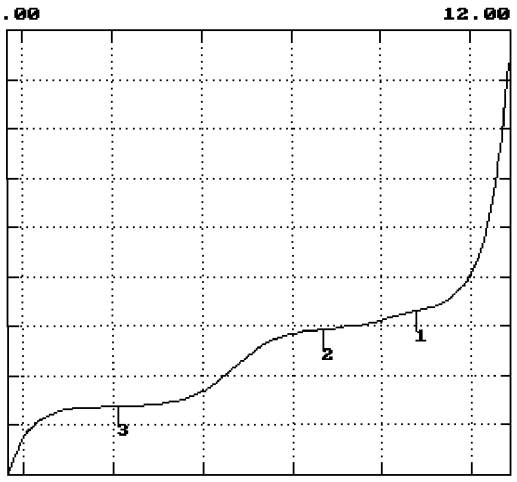
6.0239.100 combined pH glass electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  09:12        3
DET  pH            W-Liquor
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   9 ml
  stop pH                 OFF
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at pH           OFF
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      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/C
00;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
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      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
751 GPD Titrino       15215     751.0010
date  97-03-04      time  09:11        3
start V      0.000 ml  DET  pH  W-Liquor
1.0 ml/div      dpH=2.0/div
```




```
            ============
```

Remarks

- Pass a stream of nitrogen through the solution during titration.

- 1st break: $OH^- + S^{--} + 2\ H^+ \Rightarrow H_2O + HS^-$
  2nd break: $CO_3^{--} + H^+ \Rightarrow HCO_3^-$
  3rd break: $HCO_3^- + HS^- + 2\ H^+ \Rightarrow H_2CO_3 + H_2S$

- **Calculations**:
  Total = total alkali (NaOH + Na_2S + Na_2CO_3) as g NaOH per liter
  Active = active alkali (NaOH + Na_2S) as g NaOH per liter
  Effecti. = effective alkali (NaOH + ½ Na_2S) as g NaOH per liter
  NaOH = concentration of NaOH in g/L
  Na2S = concentration of Na_2S in g/L
  Na2CO3 = concentration of Na_2CO_3 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 ½ Na_2S (39 g/mol)
  C05 = molecular mass of ½ Na_2CO_3 (53 g/mol)

Literature

- SCAN - N2:63 (1963)

Silver in Fixing Baths

Reagents

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

Sample

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

Electrodes

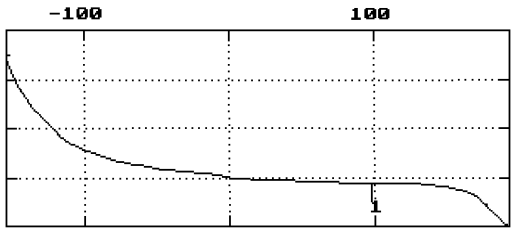
6.0430.100 Ag Titrode with Ag_2S coating; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  10:35        2
DET  U             FixBaths
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   12 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:    greatest
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      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
751 GPD Titrino       15215     751.0010
date  97-03-04      time  10:34        2
start V      5.000 ml  DET  U   FixBaths
2.0 ml/div      dU=100.0 mV/div
```

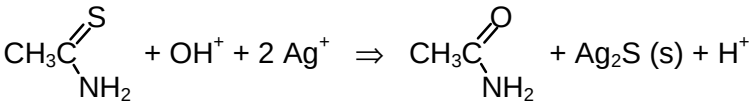



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      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_2S 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_3PO_4 solution, *c* = 0.05 mol/L. Ratio 50:24. Dissolve 0.5 g thymol per liter buffer solution.

- **Titrating 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^2 Ilford black and white film in 100 mL fixing agent $Na_2S_2O_3$ 300 g/L)
20 mL *c*(NaOH) = 2 mol/L
20 mL *c*(EDTA) = 0.1 mol/L
10 mL *w*(gelatine) = 0.12 (12%)

Electrodes

6.0430.100 Ag Titrode with Ag_2S coating; input 1

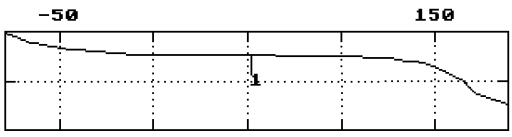
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:03        3
DET  U              Film Ag
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                   3 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      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=              169.87
C03=                 100
C04=                0.01
C05=                1000
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      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          4.192 g/m2
stop V reached
          ============
```

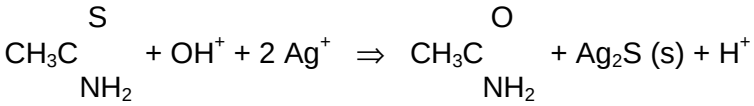
```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:02        3
start V      0.000 ml  DET  U    Film Ag
2.0 ml/div      dU=50.0 mV/div
```




```
            ============
```

Remarks

- **Determination reaction**:



- **Calculations**:
  AgNO3 = amount of $AgNO_3$ in g/m^2
  C01 = concentration of titrating agent (0.025 mol/L)
  C02 = molecular mass of $AgNO_3$ (169.87 g/mol)
  C03 = factor for 100 mL sample (100)
  C04 = size of sample (0.01 m^2)
  C05 = conversion mL ⇒ L (1000)

- EDTA keeps silver in solution and gelatine prevents the precipitation from massing together.

- **Ag_2S 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_3PO_4 solution, *c* = 0.05 mol/L. Ratio 50:24. Dissolve 0.5 g thymol per liter buffer solution.

- **Titrating agent**:
  Dissolve 1.9 g Thioacetamide in 1 L buffer pH = 5 solution.

- **Gelatine solution**:
  Dissolve 12 g gelatine in hot water, add 0.5 g thymol, allow to cool and add up to 1 L.

Literature

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

Kappa Number of Paper Pulp

Reagents

$c(^1/_2Na_2S_2O_3)$ = 0.2 mol/L; D0

Sample

13 g cellulose pulp
780 mL dist. water
100 mL $c(^1/_5KMnO_4)$ = 0.1 mol/L
100 mL $c(^1/_2H_2SO_4)$ = 4 mol/L
20 mL dist. water
35 mL w(KI) = 0.10 (10%)

Electrodes

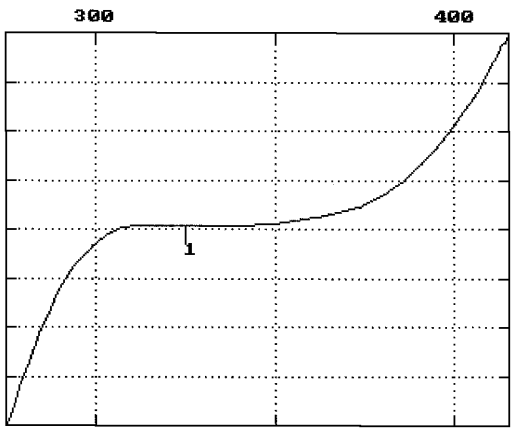
6.0418.120 combined massive Pt electrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:32        6
DET  U              KappaNo
parameters
>titration parameters
  meas.pt.density           4
  min.incr.              10.0 ml
  dos.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                OFF
  pause                    0 s
  dos.element:    internal D0
  meas.input:              1
  temperature            25.0 °C
>stop conditions
  stop V:                 abs.
  stop V                   40 ml
  stop U                  OFF mV
  stop EP                  9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      5
  EP recognition:         all
  fix EP1 at U            OFF mV
  pK/HNP:                 OFF
>preselections
  req.ident:              OFF
  req.smpl size:        value
  activate pulse:         OFF
         ------------
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:32        6
DET  U              KappaNo
>calculations
p=(C02-EP1)*C04/C05;1;
KappaNo=RS1*C01*C06/C00/C03;1;
C00=             13.0051
C01=               1.020
C02=              49.339
C03=              21.519
C04=                 0.2
C05=                 0.1
C06=                 100
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:28        6
U(init)         416 mV DET U     KappaNo
smpl size   13.0051 g
EP1          19.672 ml            325 mV
p              59.3
KappaNo         0.0
stop V reached
          ============

 'cu
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:28        6
start V      0.000 ml  DET  U    KappaNo
5.0 ml/div      dU=50.0 mV/div
```




```
          ============

 'fr
751 GPD Titrino       15215     751.0010
date  97-03-04      time  11:31        6
U(init)         416 mV DET U     KappaNo
smpl size   13.0051 g
EP1          19.672 ml            325 mV
p              59.3
KappaNo        21.6
stop V reached
          ------------
```

Remarks

- The Kappa number is the consumption of $c(^1/_5KMnO_4)$ = 0.1 mol/L in mL. The results are corrected to the consumption of 50% of the permanganate solution added.

- Determine the dry content of the sample: Dry sample at 105 °C for app. 90 minutes. Calculate the dry content in % and enter it as calculation value C03.

- Weigh in an appropriate amount of sample. Add 780 mL of dist.water and defibrate it with the stirrer. Add the mixture of 100 mL $c(^1/_5KMnO_4)$ = 0.1 mol/L und100 mL $c(^1/_2H_2SO_4)$ = 4 mol/L as quickly as possible and rinse with 20 mL dist. water. Add 35 mL *w*(KI) = 0.10 (10%) after a waiting time of exactly 10 minutes to stop the reaction. Titrate immediately.
  Treat the blank sample in the same way as above.
  The waiting time may be programmed in the Titrino; the additions can be carried out automatically using a TIP.

- **Calculations**:
  p = consumption of $c(^1/_5KMnO_4)$ = 0.1 mol/L in mL
  KappaNo = Kappa number
  C01 = factor for correction to a 50 % permanaganate consumption, see table below (p.e. 59.3 ⇒ 1.020)
  C02 = mL of $c(^1/_2Na_2S_2O_3)$ = 0.2 mol/L consumption of blank sample
  C03 = dry content in % (p.e. 21.519)
  C04 = concentration of $c(^1/_2Na_2S_2O_3)$ = 0.2 mol/L (0.2)
  C05 = factor for percent of permanaganate consumption (0.1)
  C06 = factor to compensate for entry of dry content in % (100)

- C01 can only be entered after the titration, because the p value is necessary. Recalculate and print another full report.

- Table for correction of 50% of the permanganate solution:

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

Literature

- TAPPI T236 os-76

Analysis of Spinning Bath

Reagents

c(NaOH) = 0.1 mol/L; D0

Sample

0.1 mL $c(ZnSO_4) \cong 0.1$ mol/L in $c(H_2SO_4) \cong 1$ mol/L
25 mL dist. water

Electrodes

6.0232.100 combined pH glass electrode; input 1

Method documentation

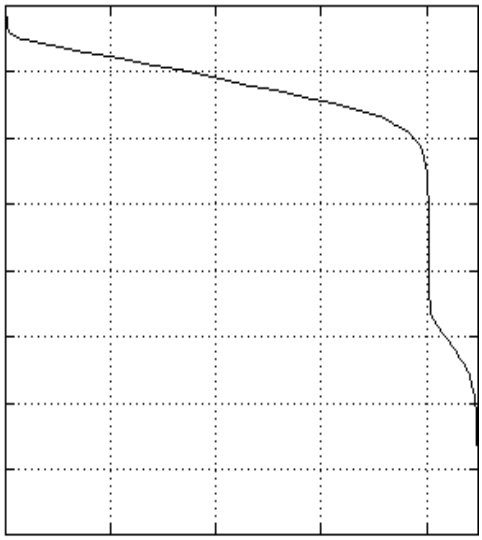
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-10      time  10:28        3
SET  pH            Spinning
parameters
>SET1
  EP at pH               5.50
  dynamics                  3
  max.rate                  5 ml/min
  min.rate                  1 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>SET2
  EP at pH               9.50
  dynamics                  4
  max.rate                  1 ml/min
  min.rate                  1 ml/min
  stop crit:            drift
  stop drift               20 ml/min
>titration parameters
  titr.direction:         auto
  pause 1                   0 s
  start V:                OFF
  pause 2                   0 s
  extr.time                 0 s
  dos.element:    internal D0
  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
  activate pulse:         OFF
         ------------

 'fm
751 GPD Titrino       15215     751.0010
date  97-03-10      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
751 GPD Titrino       15215     751.0010
date  97-03-10      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
751 GPD Titrino       15215     751.0010
date  97-03-10      time  10:21        3
                       SET  pH  Spinning
20.0  s/div      dV=0.5 ml/div
```

```
0.00                                2.00
```



```
            ============
```

Remarks

- EP1: H_2SO_4 + 2 OH^- ⇒ SO_4^{2-} + 2 H_2O
  EP2: Zn^{2+} + 2 OH^- ⇒ $Zn(OH)_2$ (s)

- **Calculations**:
  H2SO4 = concentration of H_2SO_4 in g/L
  ZnSO4 = concentration of $ZnSO_4$ in g/L
  C01 = concentration of titrating agent (0.1 mol/L)
  C02 = molecular mass of H_2SO_4 (98.08 g/mol)
  C03 = molecular mass of $ZnSO_4$ (161.23 g/mol)
  C04 = factor for “normality“ (2)

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

Literature

2-Aminophenol

Reagents

$c(NaNO_2)$ = 0.2 mol/L; D0

Sample

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

Electrodes

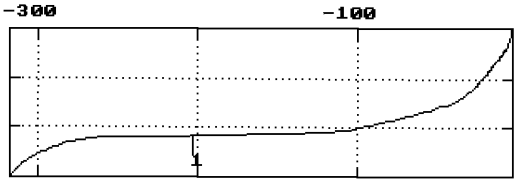
6.0431.100 Pt Titrode; input 1

Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-02-27      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
  dos.element:    internal D0
  meas.input:              1
  temperature           25.0 °C
>stop conditions
  stop V:               abs.
  stop V                  17 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 at U           OFF mV
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:       value
  activate pulse:        OFF
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-02-27      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
751 GPD Titrino       15215     751.0010
date  97-02-27      time  11:32       18
start V     10.000 ml  MET  U      Diazo
2.0 ml/div      dU=100.0 mV/div
```

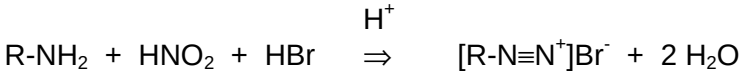



```
            ============
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-02-27      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 Sodium Tartrate

Reagents

HYDRANAL® Titrant 5; D0

Sample

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

Electrodes

6.0338.100 double Pt electrode; input Pol

Method documentation

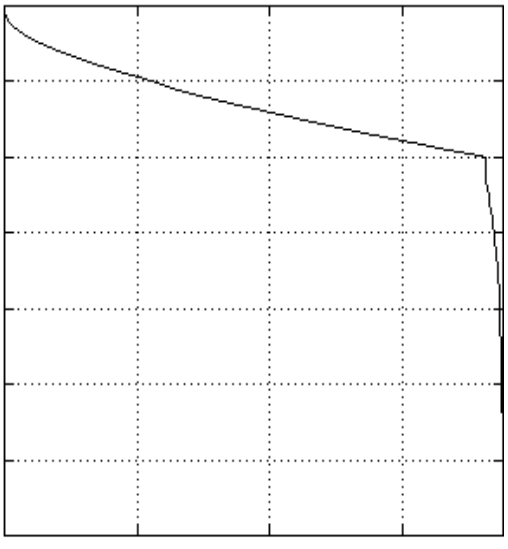
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-13      time  17:37       26
KFT  Ipol          TarTiter
parameters
>control parameters
  EP at U                 250 mV
  dynamics                100 mV
  max.rate               max. ml/min
  min.volume incr.       min. ml
  stop crit:            drift
  stop drift               20 ml/min
>titration parameters
  titr.direction:           -
  pause 1                   0 s
  start V:                OFF
  pause 2                   0 s
  extr.time                 0 s
  dos.element:    internal D0
  I(pol)                   50 mA
  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
  activate pulse:           OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-13      time  17:37       26
KFT  Ipol          TarTiter
>calculations
Titer=C00/EP1*C01;4;mg/ml
C00=              0.2294
C01=               156.6
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-13      time  17:34       26
                       KFT Ipol TarTiter
smpl size    0.2294 g
EP1           7.508 ml
Titer        4.7848 mg/ml
            mean( 5)    +/-s         s/%
Titer        4.7893  0.00506 mg/ml  0.11
        ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-13      time  17:34       26
                        KFT  IpolTarTiter
20.0  s/div      dV=2.0 ml/div

0.00                              6.00
```



```
            ============
```

```
'st
751 GPD Titrino       15215     751.0010
date  97-03-13      time  17:36
statistics
##    Titer
 1   4.7936
 2   4.7939
 3   4.7830
 4   4.7910
 5   4.7848
            mean( 5)    +/-s         s/%
Titer        4.7893  0.00506 mg/ml  0.11
         ------------
```

Remarks

- **Calculations**:
  Titer = titer of HYDRANAL® Titrant 5
  C01 = water content * 10 (156,6)

- **Common variables**
  C39 = MN1

- Mean from 5 determinations.

- Reweigh the sodium tartrate in a weighing boat.

- Adjust the calculation value C01 according to the unit of your sample weight:
  C00 in g C01=156.6
  C00 in mg C01=0.1566

- 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

KF Titer Determination with Water

Reagents

HYDRANAL® Composite 5; D0

Sample

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

Electrodes

6.0338.100 double Pt electrode; input Pol

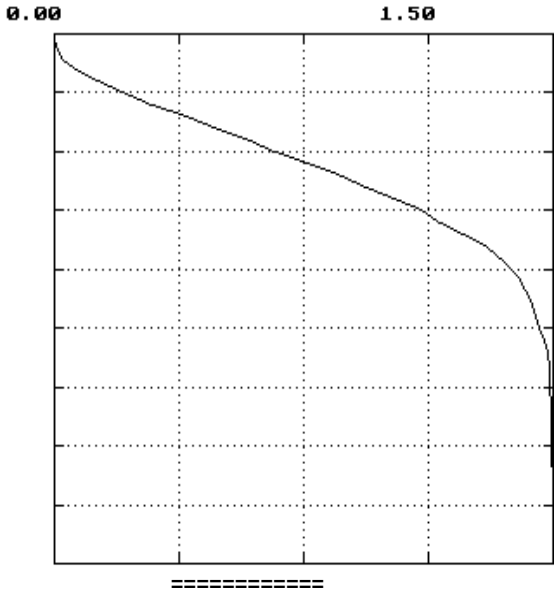
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-11      time  13:42       24
KFT  Ipol          H2OTiter
parameters
>control parameters
  EP at U                250 mV
  dynamics               100 mV
  max.rate              max. ml/min
  min.volume incr.      min. ml
  stop crit:           drift
  stop drift              20 ml/min
>titration parameters
  titr.direction:          -
  pause 1                  0 s
  start V:               OFF
  pause 2                  0 s
  extr.time                0 s
  dos.element:    internal D0
  I(pol)                  50 mA
  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
  activate pulse:        OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-11      time  13:42       24
KFT  Ipol          H2OTiter
>calculations
Titer=C00/EP1*C01;4;mg/ml
C00=               0.010
C01=                1000
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-11      time  13:40       24
                      KFT Ipol H2OTiter
smpl size     0.010 g
EP1          1.9975 ml
Titer        5.0063 mg/ml
            mean( 5)    +/-s         s/%
Titer        5.0058  0.00287 mg/ml  0.06
        ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-11      time  13:40       24
                      KFT  IpolH2OTiter
10.0  s/div      dV=0.5 ml/div
```




```
           ============
```

```
 'st
751 GPD Titrino       15215     751.0010
date  97-03-11      time  13:40
statistics
##    Titer
 1   5.0050
 2   5.0075
 3   5.0013
 4   5.0088
 5   5.0063
           mean( 5)    +/-s         s/%
Titer        5.0058  0.00287 mg/ml  0.06
          ------------
```

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 µL C01=Density of H_2O [mg/mL] ≈ 1
  Methanol standard
  C00 in mL C01=Content of methanol [mg/mL]
  C00 in µL 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; D0

Sample

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

Electrodes

6.0338.100 double Pt electrode; input Pol

Method documentation

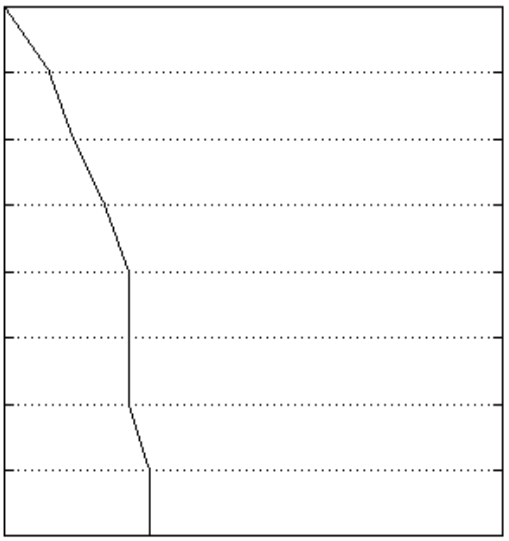
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:46       11
KFT  Ipol          Blank_KF
parameters
>control parameters
  EP at U                250 mV
  dynamics               100 mV
  max.rate              max. ml/min
  min.volume incr.      min. ml
  stop crit:           drift
  stop drift              20 ml/min
>titration parameters
  titr.direction:          -
  pause 1                  0 s
  start V:               OFF
  pause 2                  0 s
  extr.time                0 s
  dos.element:    internal D0
  I(pol)                  50 mA
  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:          OFF
  activate pulse:         OFF
         ------------

 'de
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:46
KFT  Ipol          Blank_KF
def
>formula
  Blank=EP1
  RS1 text             Blank
  RS1 decimal places       4
  RS1 unit:               ml
>silo calculations
  match id:              OFF
>common variables
  C38=MN1
>report
  report COM1:full;
>mean
  MN1=RS1
>temporary variables
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:45       11
                        KFT Ipol Blank_KF
EP1          0.0145 ml
Blank        0.0145 ml
            mean( 2)    +/-s         s/%
Blank        0.0145  0.00000 ml     0.00
           ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:45       11
                        KFT  IpolBlank_KF
2.0  s/div      dV=0.05 ml/div

0.00                                0.05
```



```
            ============
```

```
 'st
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:46
statistics
##    Blank
 1   0.0145
 2   0.0145
            mean( 2)    +/-s         s/%
Blank        0.0145  0.00000 ml     0.00
           ------------
```

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; D0

Sample

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

Electrodes

6.0338.100 double Pt electrode; input Pol

Method documentation

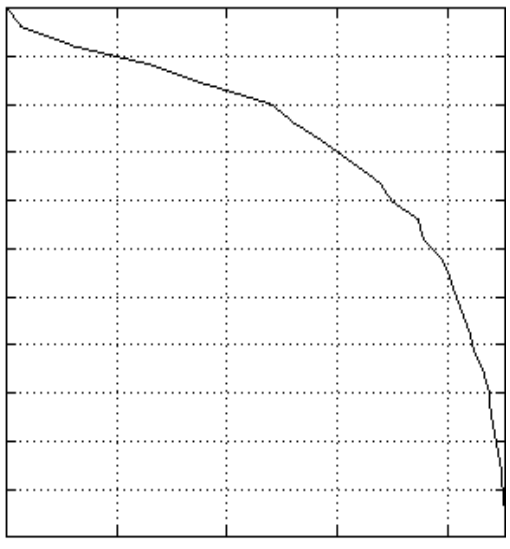
```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-12      time  14:07       29
KFT  Ipol          KF-Blank
parameters
>control parameters
  EP at U                250 mV
  dynamics               100 mV
  max.rate              max. ml/min
  min.volume incr.      min. ml
  stop crit:           drift
  stop drift              20 ml/min
>titration parameters
  titr.direction:          -
  pause 1                  0 s
  start V:               OFF
  pause 2                  0 s
  extr.time                0 s
  dos.element:    internal D0
  I(pol)                  50 mA
  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:         all
  activate pulse:        OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-12      time  14:07       29
KFT  Ipol          KF-Blank
>calculations
Water=(EP1-C38)*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
Blank=C38;4;ml
C00=             0.02003
C01=                 0.1
C02=                   1
C38=              0.0145
C39=              5.0058
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-12      time  14:05       29
                       KFT Ipol KF-Blank
smpl size   0.02003 g
EP1          0.2260 ml
Water          5.29 %
Titer         5.0058 mg/ml
Blank         0.0145 ml
              mean( 3)    +/-s         s/%
Water            5.30    0.010 %      0.19
         ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-12      time  14:05       29
                       KFT  IpolKF-Blank
5.0  s/div      dV=0.05 ml/div

0.00                              0.20
```



```
         ============
```

```
 'st
751 GPD Titrino       15215     751.0010
date  97-03-12      time  14:06
statistics
##    Water
 1     5.30
 2     5.31
 3     5.29
              mean( 3)    +/-s         s/%
Water            5.30    0.010 %      0.19
         ------------
```

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_2O
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; D0

Sample

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

Electrodes

6.0338.100 double Pt electrode; input Pol

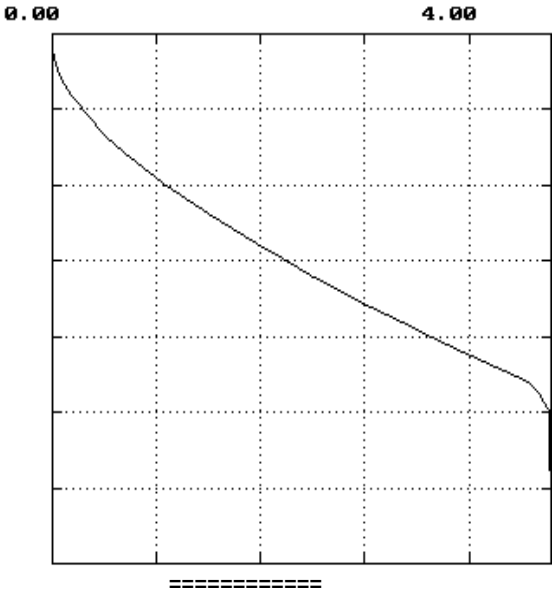
Method documentation

```
 'pa
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:08        9
KFT  Ipol               KF
parameters
>control parameters
  EP at U                250 mV
  dynamics               100 mV
  max.rate              max. ml/min
  min.volume incr.      min. ml
  stop crit:           drift
  stop drift              20 ml/min
>titration parameters
  titr.direction:          -
  pause 1                  0 s
  start V:               OFF
  pause 2                  0 s
  extr.time                0 s
  dos.element:    internal D0
  I(pol)                  50 mA
  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:          all
  activate pulse:         OFF
         ------------
```

```
 'fm
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:08        9
KFT  Ipol               KF
>calculations
Water=EP1*C39*C01/C00/C02;2;%
Titer=C39;4;mg/ml
C00=              0.1402
C01=                 0.1
C02=                   1
C39=              5.0058
         ------------
```

```
 'fr
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:05        9
                      KFT Ipol       KF
smpl size    0.1402 g
EP1          4.7895 ml
Water         17.10 %
Titer        5.0058 mg/ml
                mean( 3)    +/-s         s/%
Water           17.09    0.010 %      0.06
            ============
```

```
 'cu
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:05        9
                      KFT  Ipol      KF
10.0  s/div      dV=1.0 ml/div
```




```
            ============
```

```
 'st
751 GPD Titrino       15215     751.0010
date  97-03-12      time  10:05
statistics
##    Water
 1    17.09
 2    17.08
 3    17.10
                mean( 3)    +/-s         s/%
Water           17.09    0.010 %      0.06
            ------------
```

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_2O
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