702 SM Titrino

Applications

8.702.0003/ks, Ti

1. Aqueous pH Titrations

2. Non Aqueous Titrations

3. Precipitation Titrations

4. Complexometric Titrations

5. Redox Titrations

6. SET Titrations

Index according to the substance to be determined

Index according to the type of the titration reaction

1. Aqueous pH titrations

2. Non aqueous titrations

3. Precipitation titrations

4. Complexometric titrations

Substance(s) to be determined	Titrant	Interested industry	Page
Calcium/Magnesium in Tap Water	EDTA	Water analytics	4-1
EDTA/NTA in Detergents	$CuSO_4$	Detergents	4-2
Nickel (back titration)	$CuSO_4$	Galvanic	4-3
Ca^{2+} with Amalgamated Ag Electrode	EDTA	General	4-4

5. Redox titrations

Substance(s) to be determined	Titrant	Interested industry	Page
Diazotation of 1-Naphtylamine-5-sulfonic acid	$NaNO_2$	General	5-1
Diazotation of Cyclamate	$NaNO_2$	Food	5-2
Hydrogen Peroxide	MnO_4^-	General	5-3
Perborates in Detergents	MnO_4^-	Detergents	5-4
Iron (II)	MnO_4^-	General	5-5
COD Determination	$(NH_4)_2Fe(SO_4)_2$	Water analytics	5-6
Iodine	$Na_2S_2O_3$	General	5-7
Ascorbic Acid	KI_3	Pharmaceutical, foods	5-8
Tin (II) in Plating Bath	KI_3	Galvanic	5-9
Oxidizability of Waste Water	MnO_4^-	Water analytics	5-10

6. SET titrations

Substance(s) to be determined	Titrant	Interested industry	Page
Chloride	$AgNO_3$	General	6-1
Analysis of Rayon Spinning Bath	NaOH	Synthetic fiber	6-2
p and m Value	HCl	Water analytics	6-3
Bromine Number	BrO_3^-	Petroleum products	6-4
pH Stat Titration	HCl	General	6-5
Hydrogen Peroxide	MnO_4^-	General	6-6
KF Water Determination	Composite 5	General	6-7

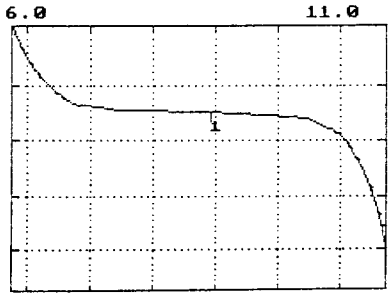
Example 1-1

Determination:	**Titer of NaOH**	
Reagent:	c(NaOH) = 0.1 mol/l	free of carbonate
Sample:	app. 300 mg potassium hydrogen phthalate 50 ml H_2O	Potassium hydrogen phthalate dried 2 h at 105°C.
Electrodes:	6.0203.100 combined pH glass electrode	

```
MET  pH                Titer
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            50 mV/min
  equilibr.time           26 s
  start V:              abs.
  start V                 12 ml
  dos.rate              max. ml/min
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V               20.00 ml
  stop pH                OFF
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                 ON
  mean              n=     5
  res.tab:          original
>evaluation
  EPC                   0.50
  EP recognition:        all
  fix EP1 pH             OFF
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
          ------------
```

```
fr
date  91-07-11       time  07:09        1
pHc(init)      4.02      MET pH      Titer
smpl size    0.3055 g
EP1          15.028 ml                8.43
Titer         0.9954
                    mean( 5)    +/-s         s/%
Titer          0.9961  0.00082          0.08
stop V reached
          ============
```

```
cu
date  91-07-11       time  07:09        1
start V     12.000 ml  MET pH       Titer
2.0 ml/div       dpH=1.0/div
```




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

```
MET  pH                Titer
>calculations
Titer=C00*C01/C02/EP1;4;
C00=                0.3055
C01=                 10000
C02=                204.23
          ------------
```

Remarks:

- Calculations:

  Titer = Titer of NaOH
  CO1 = Theoretical consumption for 1 mol potassium hydrogen phthalate (for a solution with c=0.1 mol/l = 10000 ml/mol)
  CO2 = molecular mass of potassium hydrogen phthalate (204.23 g/mol)

- Mean from 5 determinations.

References:

Metrohm Application Bulletin No. 206

Example 1-2

Determination: **Strong Acid**

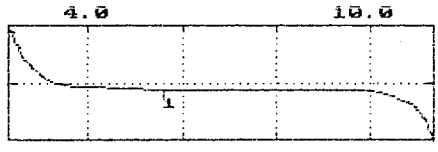
Reagent: c(NaOH) = 0.1 mol/l

Sample: 2 ml c(HCl) $\tilde{=}$ 0.1 mol/l
50 ml H_2O

Electrodes: 6.0203.100 combined pH glass electrode

```
MET  pH                  1-2
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    4 ml
  stop pH                  OFF
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                    0.50
  EP recognition:         all
  fix EP1 pH               OFF
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------

MET  pH                  1-2
>calculations
RS1=EP1*C01*C02/C00;3;mol/l
C00=                       2
C01=                     0.1
C02=                  0.9961
          ------------
```

```
fr
date  91-07-11       time  16:29        36
pHc(init)       2.30      MET pH        1-2
smpl size          2 ml
EP1            2.208 ml                 5.59
RS1            0.110 mol/l
stop V reached
          ============

cu
date  91-07-11       time  16:29        36
start V       0.000 ml  MET pH         1-2
2.0 ml/div       dpH=2.0/div
```




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

Remarks:

- Calculations:

  RS1 = concentration of acid in mol/l
  C01 = concentration of titrating agent (0.1 mol/l)
  C02 = titer of titrating agent (0.9961)

- Carbonate may be dedected separately if present!

References:

Example 1-3

Determination: **Ammonium Chloride**

Reagent: c(NaOH) = 0.1 mol/l

Sample: 2 ml $c(NH_4Cl) \cong 0.1$ mol/l $pK(NH_4Cl) = 9.21$
50 ml H_2O

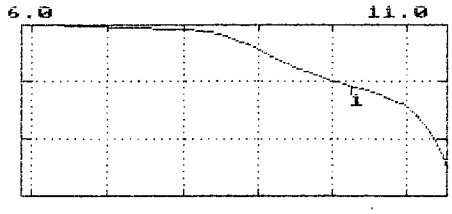
Electrodes: 6.0203.100 combined pH glass electrode

```
MET  pH                  1-3
parameters
>titration parameters
  V step                  0.2 ml
  titr.rate               max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                    5 ml
  stop pH                  OFF
  stop EP                   9
  filling rate            max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                     0.50
  EP recognition:          all
  fix EP1 pH               OFF
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
         ------------
```

```
MET  pH                  1-3
>calculations
RS1=EP1*C01/C00;2;g/l
C00=                        2
C01=                     5.35
         ------------
```

```
fr
date  91-07-12       time  10:48        18
pHc(init)       5.86      MET pH        1-3
smpl size          2 ml
EP1            2.179 ml                10.24
RS1             5.83 g/l
stop V reached
          ============
```

```
cu
date  91-07-12       time  10:48        18
start V       0.000 ml  MET pH         1-3
2.0 ml/div       dpH=1.0/div
```




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

Remarks:

- Calculations:

RS1 = concentration of NH_4Cl in g/l

CO1 = concentration of titrating agent x molecular mass of NH_4Cl (5.35)

References:

Example 1-4

Determination: **Oxalic Acid**

Reagent: c(NaOH) = 0.1 mol/l

Sample: 2 ml $c(C_2H_2O_4) \widetilde{=} 0.1$ mol/l
50 ml H_2O

HOOC-COOH
pK_1 = 1.42
pK_2 = 4.31

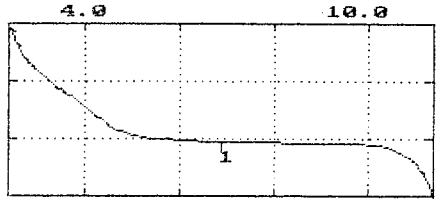
Electrodes: 6.0203.100 combined pH glass electrode

```
MET  pH                  1-4
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    6 ml
  stop pH                  OFF
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                    0.50
  EP recognition:     greatest
  fix EP1 pH               OFF
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
MET  pH                  1-4
>calculations
RS1=EP1*C01/C02/C00;3;mol/l
C00=                       2
C01=                     0.1
C02=                       2
          ------------
```

```
fr
date  91-07-16       time  16:46        15
pHc(init)       2.43      MET pH        1-4
smpl size          2 ml
EP1            4.113 ml                 6.92
RS1            0.103 mol/l
stop V reached
            ============
```

```
cu
date  91-07-16       time  16:46        15
start V       0.000 ml  MET pH         1-4
2.0 ml/div       dpH=2.0/div
```




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

Remarks:

- Calculations:

  RS1 = concentration of acid in mol/l
  C01 = concentration of titrating agent (0.1 mol/l)
  C02 = factor for "normality" (2)

- Compare also titration in non aqueous medium, page 2-5

References:

Example 1-5

Determination: **Mixture of 4 Acids**

Reagent: c(NaOH) = 0.1 mol/l

Sample: 2 ml acid mixture
50 ml H_2O

Acid mixture:	pK
c(HCl) ≅ 0.1 mol/l	-1.74
c(CH_3COOH) ≅ 0.1 mol/l	4.74
c(4-Nitrophenol) ≅ 0.1 mol/l	7.14
c(NH_4Cl) ≅ 0.1 mol/l	9.21

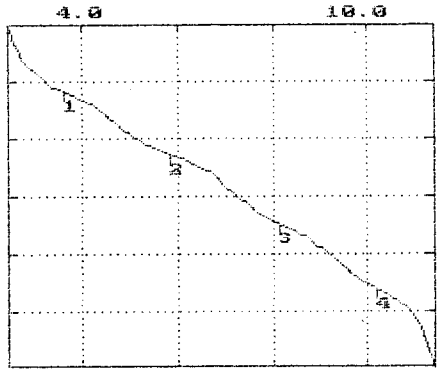
Electrodes: 6.0203.100 combined pH glass electrode

```
MET  pH                    1-9
parameters
>titration parameters
  V step                  0.2 ml
  titr.rate              max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                    0 s
  meas.input:              1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                   12 ml
  stop pH                 OFF
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                    0.50
  EP recognition:         all
  fix EP1 pH              OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
MET  pH                    1-9
>calculations
RS1=EP1*C01*C02;2;mg
RS2=(EP2-EP1)*C01*C03;2;mg
RS3=(EP3-EP2)*C01*C04;2;mg
RS4=(EP4-EP3)*C01*C05;2;mg
C01=                     0.1
C02=                    36.5
C03=                      60
C04=                     139
C05=                    53.5
          ------------
```

```
fr
date  91-07-12        time  12:06        24
pHc(init)       2.44       MET pH        1-9
EP1           2.346 ml                   3.63
EP2           4.557 ml                   5.82
EP3           6.931 ml                   8.11
EP4           9.238 ml                  10.20
RS1            8.56 mg
RS2           13.27 mg
RS3           32.99 mg
RS4           12.34 mg
stop V reached
            ============
```

```
cu
date  91-07-12        time  12:06        24
start V       0.000 ml  MET pH          1-9
2.0 ml/div       dpH=2.0/div
```




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

Remarks:

- 1st break: HCl
  2nd break: CH_3COOH
  3rd break: 4-Nitrophenol
  4th break: NH_4Cl

- Calculations:
  RS1 = amount of HCl in mg
  RS2 = amount of CH_3COOH in mg
  RS3 = amount of 4-Nitrophenol in mg
  RS4 = amount of NH_4Cl in mg
  C01 = concentration of titrating agent (0.1 mol/l)
  C02 = molecular mass of HCl (36.5 g/mol)
  C03 = molecular mass of CH_3COOH (60.0 g/mol)
  C04 = molecular mass of 4-Nitrophenol (139 g/mol)
  C05 = molecular mass of NH_4Cl (53.5 g/mol)

References:

Example 1-6

Determination: **White Liquor**

(Paper industry)

Reagent: c(HCl) = 1 mol/l

Sample: 2 ml white liquor
50 ml H_2O

White liquor is mixture of
NaOH,
Na_2S (pK_1=7.04, pK_2=11.96),
Na_2CO_3 (pK_1=6.37, pK_2=10.25)

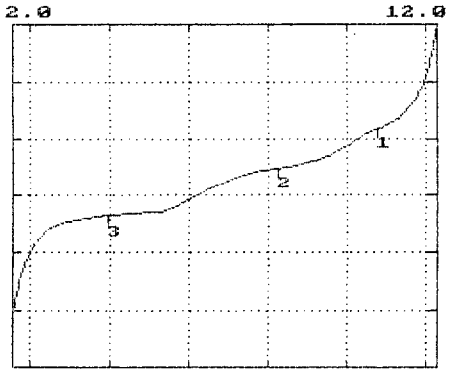
Electrodes: 6.0203.100 combined pH glass electrode

```
MET  pH                  1-8
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:               abs.
  start V                   4 ml
  dos.rate               max. ml/min
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    9 ml
  stop pH                 OFF
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       1
  EP recognition:         all
  fix EP1 pH              OFF
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
>MET  pH                  1-8
 >calculations
 RS1=EP3*C01*C02/C00;2;g/l
 RS2=(EP3-C03*(EP2-EP1))*C01*C02/C00;2;g/
 l
 RS3=EP1*C01*C02/C00;2;g/l
 RS4=(EP1-((EP3-EP2)-(EP2-EP1)))*C01*C02/
 C00;2;g/l
 RS5=((EP3-EP2)-(EP2-EP1))*C03*C02*C04/C0
 0;2;g/l
 RS6=(EP2-EP1)*C03*C02*C05/C00;2;g/l
 C00=                     2
 C01=                    40
 C02=                     1
 C03=                     2
 C04=                    39
 C05=                    53
          ------------
```

```
fr
date  91-07-16       time  15:25        10
pHc(init)      12.74      MET pH        1-8
smpl size          2 ml
EP1            5.819 ml               10.77
EP2            6.536 ml                8.25
EP3            7.385 ml                3.94
RS1           147.69 g/l
RS2           119.02 g/l
RS3           116.38 g/l
RS4           113.73 g/l
RS5             5.15 g/l
RS6            37.99 g/l
stop V reached
          ============
```

```
cu
date  91-07-16       time  15:25        10
start V       4.000 ml  MET pH         1-8
1.0 ml/div       dpH=2.0/div
```




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

Remarks:

- Pass a stream of nitrogen through solution during tirtation.

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

- Calculations:
  RS1 = total alcali (NaOH + Na_2S +Na_2CO_3) as g NaOH per liter
  RS2 = active alcali (NaOH + Na_2S) as g NaOH per liter
  RS3 = effective alcali (NaOH + ½ Na_2S) as g NaOH per liter
  RS4 = concentration of NaOH in g/l
  RS5 = concentration of Na_2S in g/l
  RS6 = 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)

References:

SCAN - N2:63 (1963)

Example 2-1

Determination: **Peroxide Number**

(Food industry)

Reagent: $c(Na_2S_2O_3)$ = 0.01 mol/l — Prepared daily from c = 0.1 mol/l

Sample: app. 5 g sunflower oil
50 ml solvent
1 ml KJ sat.
100 ml H_2O

Solvent:
acetic acid : chloroform
3 : 2

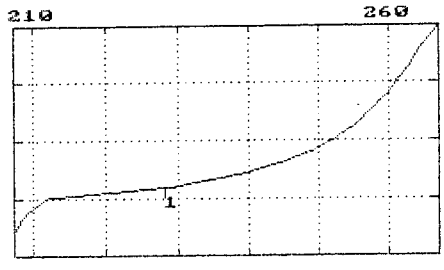
Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U              Peroxide
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              50 mV/min
  equilibr.time             26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                99.99 ml
  stop U                   OFF mV
  stop EP                    1
  filling rate            max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:          all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:            ON
  activate pulse:          OFF
          ------------
```

```
MET  U              Peroxide
>calculations
Perox.No=C01*(EP1-C02)/C00;2;
C00=                  5.034
C01=                     10
C02=                      0
          ------------
```

```
fr
date  91-07-17       time  09:00          2
U(init)          268 mV  MET U     Peroxide
smpl size      5.034 g
EP1            1.406 ml                228 mV
Perox.No        2.79
stop  EP reached
            ============
```

```
cu
date  91-07-17       time  09:00          2
start V       0.000 ml  MET U     Peroxide
0.5 ml/div       dU=10.0 mV/div
```




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

Remarks:

- Calculations:

  Perox.No = Peroxide number (meq.O_2/kg)
  C01 = factor (10)
  C02 = consumption of blank sample (0 ml)

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

References:

Metrohm Application Bulletin No. 141

Example 2-2

Determination: **Bromine Index**

(Petroleum products industry)

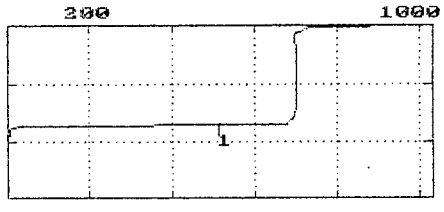
Reagent:	$c(BrO_3^-/Br^-)$ = 0.05 mol/l	Dissolve 5.1 g KBr and 1.4 g $KBrO_4$ each in dist. H_2O and add up to 1 l.
Sample:	50 µl c(cyclohexene) = 10 % in solvent 25 ml solvent	Solvent: 714 ml acetic acid 134 ml CCl_4 134 ml CH_3OH 18 ml H_2SO_4 (20%)
Electrodes:	6.0308.100 double Pt electrode polarized I_{pol} = 1 µA Voltametric indication	

```
MET  Ipol          Br-Index
parameters
>titration parameters
  V step                0.05 ml
  titr.rate             max. ml/min
  signal drift           OFF mV/min
  equilibr.time           20 s
  start V:               OFF
  pause                    0 s
  I(pol)                   1 uA
  electrode test:        OFF
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V               10.00 ml
  stop U                   5 mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:    greatest
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
          ------------
```

```
MET  Ipol          Br-Index
>calculations
Br-Index=(EP1-C01)*C02*C03/C00;1;mg
C00=               0.050
C01=                   0
C02=                0.05
C03=                7990
          ------------
```

```
fr
date  91-07-17      time  11:22        4
U(init)          450 mV  MET IpolBr-Index
smpl size      0.050 g
EP1            1.713 ml              510 mV
Br-Index     13684.5 mg
stop meas.val.reached
             ============
```

```
cu
date  91-07-17      time  11:22        4
start V      0.000 ml  MET Ipol Br-Index
1.0 ml/div       dU=200.0 mV/div
```




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

Remarks:

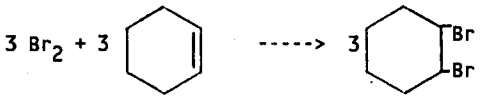
- Bromine index is the number of milligrammes of bromine consumed per 100 g of sample.

- Standardisation of BrO_3^-/Br^- solution:
  Determination reaction:
  $3\ Br_2 + 6\ S_2O_3^{2-} \text{ -----> } 6\ Br^- + 3\ S_4O_6^{2-}$

  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:

$BrO_3^- + 5\ Br^- + 6\ H^+ \text{ -----> } 3\ Br_2 + 3\ H_2O$



- 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)
  C03 = molecular mass of Br multiplied with 100 (7990 g/mol)

References:

Metrohm Application Bulletin No. 177

Example 2-3

Determination: **Total Acid Number (TAN)**

(Petroleum products industry)

Reagent: c(TBAOH) = 0.1 mol/l in isopropanol/methanol — TBAOH = Tetrabutyl ammonium hydroxide

Sample: app. 1.5 g of used motor oil
50 ml solvent

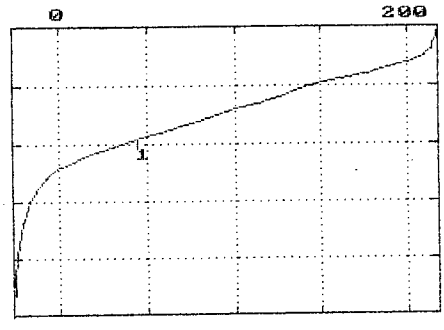
Solvent:
chlorbenzene : isopropanol
2 : 1

Electrodes: 6.0102.102 pH glass electrode
6.0729.100 Ag/AgCl reference electrode (LiCl sat. in ethanol)
6.0331.000 Pt auxiliary electrode
Differential potentiometry

```
MET  U                TAN/TBN
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift           OFF mV/min
  equilibr.time           50 s
  start V:               OFF
  pause                  100 s
  meas.input:           diff.
  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                     20 mV
  EP recognition:       last
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
          ------------
```

```
fr
date  91-07-17       time  14:26          5
U(init)          194 mV  MET U     TAN/TBN
smpl size      1.509 g
EP1            1.916 ml                45 mV
TAN/~~TBN~~          7.12 mg
stop V reached
            ============
```

```
cu
date  91-07-17       time  14:26          5
start V       0.000 ml  MET U      TAN/TBN
1.0 ml/div       dU=50.0 mV/div
```




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

```
MET  U                TAN/TBN
>calculations
TAN/~~TBN~~=(EP1-C01)*C02*C03/C00;2;mg
C00=                  1.509
C01=                      0
C02=                    0.1
C03=                 56.106
          ------------
```

Remarks:

- Calculations:

  TAN/~~TBN~~ = consumption of titrating agent 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 H_2O over night. Before titrating, precondition it in solvent during 10-30 min.

References:

Metrohm Application Bulletin No. 80
ASTM D 2896 - 80
DIN 51596

Example 2-4

Determination: **Total Base Number (TBN)**

(Petroleum products industry)

Reagent: $c(HClO_4)$ = 0.1 mol/l in acetic acid

Sample: app. 1.5 g of used motor oil
50 ml solvent

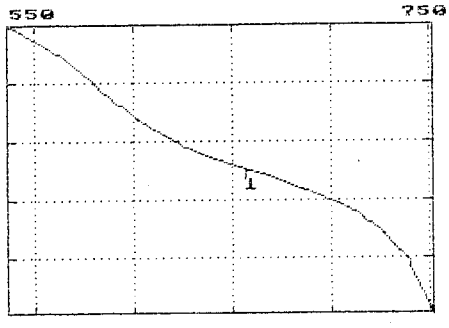
Solvent:
chlorbenzene : acetic acid
2 : 1

Electrodes: 6.0102.102 pH glass electrode
6.0729.100 Ag/AgCl reference electrode (LiCl sat. in ethanol)
6.0331.000 Pt auxiliary electrode
Differential potentiometry

```
MET  U                TAN/TBN
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift           OFF mV/min
  equilibr.time           50 s
  start V:               OFF
  pause                  100 s
  meas.input:           diff.
  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                     20 mV
  EP recognition:       last
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
         ------------
```

```
fr
date  91-07-17       time  15:55          6
U(init)          529 mV  MET U      TAN/TBN
smpl size      1.509 g
EP1            2.479 ml              657 mV
~~TAN~~/TBN          9.22 mg
stop V reached
          ============
```

```
cu
date  91-07-17       time  15:55          6
start V       0.000 ml  MET U       TAN/TBN
1.0 ml/div       dU=50.0 mV/div
```




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

```
MET  U                TAN/TBN
>calculations
~~TAN~~/TBN=(EP1-C01)*C02*C03/C00;2;mg
C00=                  1.509
C01=                      0
C02=                    0.1
C03=                 56.106
         ------------
```

Remarks:

- Calculations:

  ~~TAN~~/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. H_2O over night. Before titrating, precondition it in solvent during 10 - 30 min.

References:

Metrohm Application Bulletin No. 80

ASTM D 2896 - 80

DIN 51596

Example 2-5

Determination: **Oxalic Acid**

Reagent: c(TBAOH) = 0.1 mol/l in isopropanol/methanol

TBAOH = Tetrabutyl ammonium hydroxide

Sample: 2 ml $c(C_2H_2O_4) \cong 0.1$ mol/l
25 ml ethanol

Oxalic acid:
HOOC-COOH

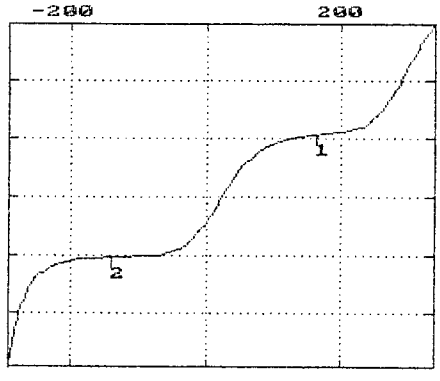
Electrodes: 6.0102.102 pH glass electrode
6.0726.100 Ag/AgCl reference electrode (LiCl sat. in ethanol)

```
MET  U                    2-5
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    6 ml
  stop U                   OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                      30 mV
  EP recognition:        all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
MET  U                    2-5
>calculations
RS1=EP2*C01/C02/C00;3;mol/l
C00=                        2
C01=                      0.1
C02=                        2
          ------------
```

```
fr
date  91-07-18       time  09:26          5
U(init)            340 mV  MET U         2-5
smpl size            2 ml
EP1              1.954 ml              161 mV
EP2              4.053 ml              -140 mV
RS1              0.101 mol/l
stop V reached
            ============
```

```
cu
date  91-07-18       time  09:26          5
start V       0.000 ml  MET U           2-5
1.0 ml/div       dU=200.0 mV/div
```




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

Remarks:

- Calculations:

RS1 = concentration of acid in mol/l
CO2 = concentration of titrating agent (0.1 mol/l)
CO2 = factor for "normality" (2)

- Compare also titration in aqueous medium, page 1-4

References:

Example 2-6

Determination: **Nitrating Acid**

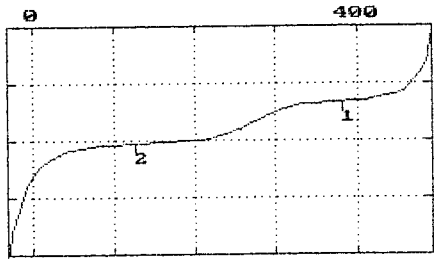
(Explosives and plastics industry)

Reagent:	c(CHA) = 0.5 mol/l	CHA = Cyclohexylamine
Sample:	2 ml nitrating acid 25 ml methanol	Nitrating acid: 1 ml H_2SO_4 (96%, δ = 1.84 g/l) 1 ml HNO_3 (app. 60%) 20 ml H_2O ad. 100 ml methanol
Electrodes:	6.0102.102 pH glass electrode 6.0726.100 Ag/AgCl reference electrode (LiCl sat. in ethanol)	

```
MET  U                    2-6
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             50 mV/min
  equilibr.time            26 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    4 ml
  stop U                   OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:        all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
fr
date  91-07-18       time  12:13        12
U(init)          494 mV  MET U          2-6
smpl size          3 ml
EP1            1.333 ml                382 mV
EP2            2.061 ml                126 mV
RS1            23.80 g/l
RS2            12.70 g/l
stop V reached
            ============
```

```
cu
date  91-07-18       time  12:13        12
start V       0.000 ml  MET U          2-6
1.0 ml/div       dU=100.0 mV/div
```




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

```
MET  U                    2-6
>>calculations
RS1=(EP2-EP1)*C01*C02/C00;2;g/l
RS2=(EP1-(EP2-EP1))*C01*C03/C00;2;g/l
C00=                       3
C01=                       1
C02=                   98.08
C03=                   63.01
          ------------
```

Remarks:

- 1st break: $HNO_3 + H_2SO_4 + 2\ CHA \text{ -----> } NO_3^- + HSO_4^- + 2\ HCHA^+$

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

- Calculations:

  RS1 = sulphuric acid in g/l nitrating acid
  RS2 = nitric acid in g/l nitrating acid
  CO1 = concentration of titrating agent (0.5 mol/l)
  CO2 = molecular mass of H_2SO_4 (98.08 g/mol)
  CO3 = molecular mass of HNO_3 (63.01 g/mol)

References:

Metrohm Application Bulletin No. 39

Example 2-7

Determination: **Epoxy Number**

(Plastics industry)

Reagent: $c(HClO_4)$ = 0.1 mol/l in acetic acid

Sample: app. 0.2 g Araldit resin
25 ml c(TBA) =0.2 mol/l in acetic acid

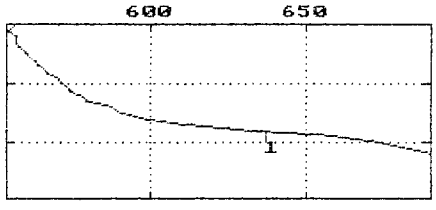
TBA = tetrabutyl ammonium bromide

Electrodes: 6.0102.102 pH glass electrode
6.0729.100 Ag/AgCl reference electrode (LiCl sat. in ethanol)
6.0331.100 Pt auxiliary electrode
Differential potentiometry

```
MET  U                    2-7
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              10 mV/min
  equilibr.time             52 s
  start V:                abs.
  start V                    7 ml
  dos.rate                max. ml/min
  pause                      0 s
  meas.input:             diff.
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                    12 ml
  stop U                   OFF mV
  stop EP                    1
  filling rate            max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:          all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
MET  U                    2-7
>calculations
RS1=EP1*C01*C02*C03/C00;3;
C00=                  0.194
C01=                  0.001
C02=                    0.1
C03=                    100
          ------------
```

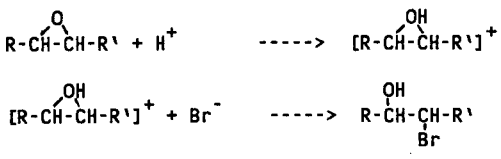
```
fr
date  91-08-05      time  09:52          4
U(init)          509 mV  MET U          2-7
smpl size       0.194 g
EP1             8.824 ml              637 mV
RS1             0.455
stop  EP reached
          ============

cu
date  91-08-05      time  09:52          4
start V      7.000 ml  MET U            2-7
1.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Determination reactions:



- Calculations:

RS1 = epoxy number, number of epoxide groups per 100 g of sample
CO1 = factor for conversion ml -> l (0.001)
CO2 = concentration of titrating agent (0.1 mol/l)
CO3 = factor for 100 g of sample (100)

- Stir sample in solvent during 3 minutes before titration

References:

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

Example 2-8

Determination: **Nicotinamide**

(Pharmaceutical industry)

Reagent: $c(HClO_4)$ = 0.1 mol/l in acetic acid

Sample: app. 0.060 g nicotinamide
50 ml solvent

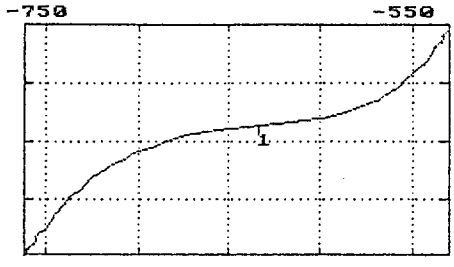
Solvent:
chlorbenzol : acetic acid
2 : 1

Electrodes: 6.0102.102 pH glass electrode
6.0729.100 Ag/AgCl reference elektrode (LiCl sat. in ethanol)
6.0331.000 Pt auxiliary electrode
Differential potentiometry

```
MET  U                    2-8
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              20 mV/min
  equilibr.time             38 s
  start V:                abs.
  start V                    3 ml
  dos.rate                max. ml/min
  pause                      0 s
  meas.input:             diff.
  temperature              25.0  C
>stop conditions
  stop V:                 abs.
  stop V                     7 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 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:            ON
  activate pulse:          OFF
          ------------
```

```
MET  U                    2-8
>calculations
RS1=(EP1-C01)*C02*C03*C04/C00;2;%
C00=              0.05753
C01=                    0
C02=               0.9943
C03=               12.213
C04=                  0.1
          ------------
```

```
fr
date  91-09-06       time  15:35          6
U(init)          -469 mV  MET U          2-8
smpl size    0.05753 g
EP1            4.736 ml              -633 mV
RS1            99.98 %
stop V reached
            ============

cu
date  91-09-06       time  15:35          6
start V       3.000 ml  MET U          2-8
1.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Calculations:

  RS1 = content of nicotinamide in %
  C01 = blank value (0 ml)
  C02 = titer (0.9943)
  C03 = concentration of titrating agent * molecular mass of nicotinamide (12.213)
  C04 = factor for %

- Nicotinamide can also be titrated uesing normal potentiometry instead of diff. The parameter "meas input:" has to be changed to 1. As electrode 6.0430.100 Ag Titrode may be used. If the curve is strongly asymmetric, you may use a 6.0133.100 pH glass electrode with a 6.0726.100 Ag/AgCl reference electrode. (LiCl sat. in Ethanol)

References:

Metrohm Application Bulletin No. 174

Example 3-1

Determination: **Chloride, Bromide, Iodide simultaneous**

Reagent: $c(AgNO_3)$ = 0.01 mol/l

Sample:
1.5 ml of halide mixture
2 ml $c(HNO_3)$ = 2 mol/l
25 ml H_2O

Halide mixture:	pk_L
10 ml c(KBr) = 0.1 mol/l	12.3
10 ml c(KCl) = 0.1 mol/l	9.77
10 ml c(KJ) = 0.1 mol/l	16.1
70 ml H_2O	

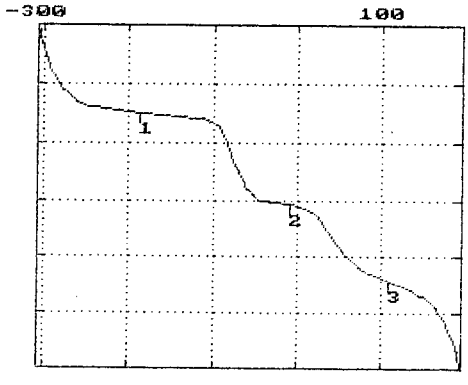
Electrodes: 6.0404.100 combined massive Ag electrode

```
MET  U                    3-1
parameters
>titration parameters
  V step                  0.1 ml
  titr.rate               max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                   0 s
  meas.input:             1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                  6 ml
  stop U                  OFF mV
  stop EP                 9
  filling rate            max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                     30 mV
  EP recognition:         all
  fix EP1 U               OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
MET  U                    3-1
>calculations
RS1=EP1*C01/C00;4;mol/l
RS2=(EP2-EP1)*C01/C00;4;mol/l
RS3=(EP3-EP2)*C01/C00;4;mol/l
C00=                    1.5
C01=                   0.01
          ------------
```

```
fr
date  91-08-12       time  12:04        18
U(init)          -284 mV  MET U         3-1
smpl size           1.5 ml
EP1               1.503 ml             -187 mV
EP2               3.083 ml               -9 mV
EP3               4.442 ml              109 mV
RS1             0.0100 mol/l
RS2             0.0105 mol/l
RS3             0.0091 mol/l
stop V reached
             ============
```

```
cu
date  91-08-12       time  12:04        18
start V       0.000 ml  MET U           3-1
1.0 ml/div       dU=100.0 mV/div
```




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

Remarks:

- Calculations:

RS1 = concentration of I^- in mol/l $pk_L = 16.1$

RS2 = concentration of Br^- in mol/l $pk_L = 12.3$

RS3 = concentration of Cl^- in mol/l $pk_L = 9.77$

C01 = concentration of titrating agent (0.01 mol/l)

References:

Example 3-2

Determination: **Sulphate**

Reagent: $c(Pb(ClO_4)_2)$ = 0.005 mol/l

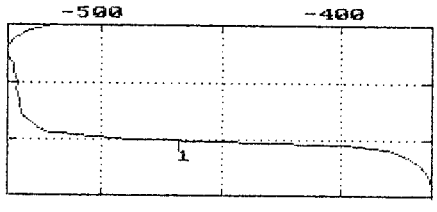
Sample: 0.1 ml $c(H_2SO_4)$ = 0.1 mol/l
25 ml isopropanol

Electrodes: 6.0502.170 Pb^{2+} sensitive electrode
6.0808.000 Glassy carbon electrode

```
MET  U                    3-2
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                   3 ml
  stop U                OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:               OFF
>evaluation
  EPC                    30 mV
  EP recognition:       all
  fix EP1 U             OFF mV
>preselections
  req.ident:            OFF
  req.smpl size:        OFF
  activate pulse:       OFF
          ------------
```

```
fr
date  91-08-12       time  15:25        21
U(init)          -501 mV  MET U         3-2
smpl size         0.1 ml
EP1             2.024 ml              -468 mV
RS1             9.71 g/l
stop V reached
          ============
```

```
cu
date  91-08-12       time  15:25        21
start V       0.000 ml  MET U           3-2
1.0 ml/div       dU=50.0 mV/div
```




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

```
MET  U                    3-2
>calculations
RS1=EP1*C01*C02/C00;2;g/l
C00=                    0.1
C01=                  0.005
C02=                     96
          ------------
```

Remarks:

- Calculations:

RS1 = concentration of SO_4^{2-} in g/l

C01 = concentration of titrating agent (0.005 mol/l)

C02 = molecular mass of SO_4^{2-} (96 g/mol)

References:

Determination: **Silver in Film Emulsions** Example 3-3

(Film industry)

Reagent: c(Thioacetamide) = 0.025 mol/l in buffer pH = 5

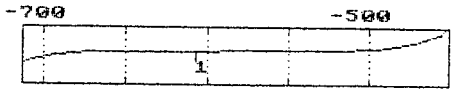
Sample:
10 ml solution
20 ml c(EDTA) = 0.1 mol/l
20 ml c(NaOH) = 2 mol/l
10 ml w(gelatine) = 1.2 %

Solution:
100 cm^2 Ilford black and white film
100 ml fixing agent (300 g/l $Na_2S_2O_3$)

Electrodes: 6.0404.100 combined Ag electrode with Ag_2S coating

```
MET  U                    3-3
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                   5 ml
  stop U                OFF mV
  stop EP                  1
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
          ------------
```

```
fr
date  91-08-13       time  15:59          8
U(init)          -453 mV  MET U          3-3
smpl size          10 ml
EP1              0.857 ml              -608 mV
RS1              3.641
stop  EP reached
          ============

cu
date  91-08-13       time  15:59          8
start V        0.000 ml  MET U          3-3
2.0 ml/div       dU=50.0 mV/div
```

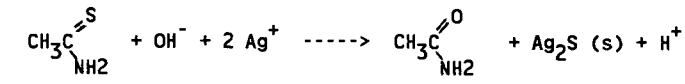



```
          ============
```

```
MET  U                    3-3
>>calculations
RS1=EP1*C01*C02/C00*C03/C04/C05;3;
C00=                       10
C01=                    0.025
C02=                   169.87
C03=                      100
C04=                     0.01
C05=                     1000
          ------------
```

Remarks:

- Determination reaction:



- Calculations:

RS1 = 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 electrode in alcalnie 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.

References:

Metrohm Application Bulletin No. 72

Example 3-4

Determination: **Pure Silver Content**

(Noble metal analytics)

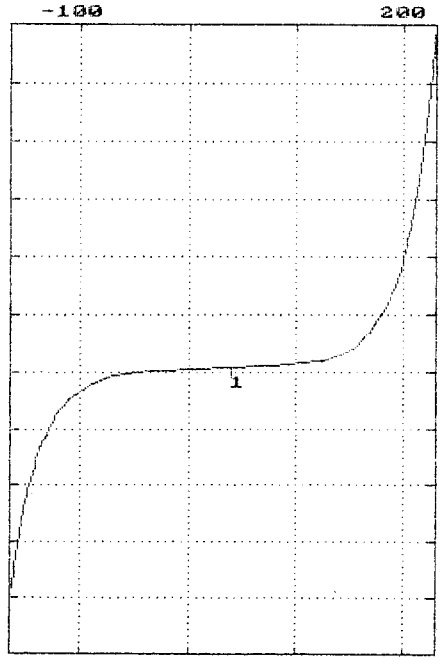
Reagent: c(KBr) = 0.1 mol/l

Sample: app. 220 mg Ag with
10 ml HNO_3 65%
100 ml H_2O

Electrodes: 6.0404.100 combined massive Ag electrode with AgBr coating

```
MET  U                    3-4
parameters
>titration parameters
  V step                   0.1 ml
  titr.rate               max. ml/min
  signal drift              10 mV/min
  equilibr.time             52 s
  start V:                 abs.
  start V                   15 ml
  dos.rate                 max. ml/min
  pause                      0 s
  meas.input:                1
  temperature              25.0  C
>stop conditions
  stop V:                  abs.
  stop V                    25 ml
  stop U                   OFF mV
  stop EP                  OFF
  filling rate             max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:          all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------

MET  U                     3-4
>calculations
RS1=EP1*C01*C02*C03*C04/C00;3;%
C00=                  225.72
C01=                     0.1
C02=                  0.9998
C03=                  107.87
C04=                     100
          ------------
```

```
fr
date  91-08-14       time  16:34        10
U(init)          264 mV  MET U          3-4
smpl size     225.72 mg
EP1            20.933 ml               39 mV
RS1           100.018 %
stop V reached
          ============

cu
date  91-08-14       time  16:28        10
start V      15.000 ml  MET U          3-4
1.0 ml/div       dU=100.0 mV/div
```




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

Remarks:

- Calculations:

RS1 = content of silver in % (purity)
C01 = concentration of titratng agent (0.1 mol/l)
C02 = titer (0.9998)
C03 = molecular mass of Ag (107.87 g/mol)
C04 = factor for % (100)

- Sample preparation:

Heat silver in HNO_3. Allow nitrous gases to evaporate.

- AgBr coating of electrode:

Clean Ag electrode with emery cloth and electrolyze in w(HBr) = 10 % during 2 h with 5 mA using a 6.0305.000 Pt electrode as cathode. Stir during electrolysis.

References:

Metrohm Application Bulletin No. 61

Example 3-5

Determination: **Chloride in Tap Water**

(Water analytics)

Reagent: $c(AgNO_3)$ = 0.01 mol/l

Sample: 100 ml tap water
5 ml $c(HNO_3)$ = 2 mol/l

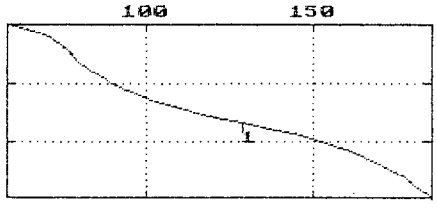
Electrodes: 6.0404.100 combined massive Ag electrode

```
MET  U                    3-5
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              20 mV/min
  equilibr.time             38 s
  start V:                 OFF
  pause                      0 s
  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 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
         ------------
```

```
MET  U                    3-5
>calculations
RS1=EP1*C01*C02*C03/C00;2;ppm
C00=                    100
C01=                   0.01
C02=                  35.45
C03=                   1000
         ------------
```

```
fr
date  91-08-14       time  17:21        12
U(init)            59 mV  MET U         3-5
smpl size         100 ml
EP1             1.679 ml               129 mV
RS1              5.95 ppm
stop V reached
          ============
```

```
cu
date  91-08-14       time  17:21        12
start V       0.000 ml  MET U          3-5
1.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Calculations:

RS1 = 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)

References:

Example 3-6

Determination: **Silver in Fixing Baths**

(Photo industry)

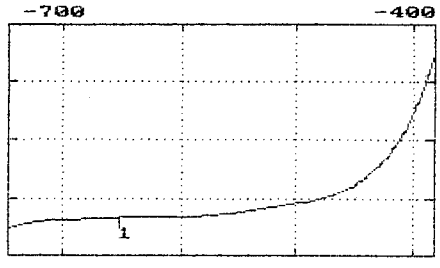
Reagent: c(Thioacetamide) = 0.025 mol/l in buffer pH = 5

Sample:
5 ml of sample
20 ml c(NaOH) = 2 mol/l
20 ml c(EDTA) = 0.1 mol/l
10 ml w(gelatine) = 1.2 %

Electrodes: 6.0404.100 combined massive Ag electrode with Ag_2S coating

```
MET  U                    3-6
parameters
>titration parameters
  V step                   0.1 ml
  titr.rate               max. ml/min
  signal drift              10 mV/min
  equilibr.time             52 s
  start V:                 abs.
  start V                    5 ml
  dos.rate                 max. ml/min
  pause                      0 s
  meas.input:                1
  temperature              25.0  C
>stop conditions
  stop V:                  abs.
  stop V                    20 ml
  stop U                   OFF mV
  stop EP                    1
  filling rate             max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:          all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------

MET  U                     3-6
>calculations
 RS1=EP1*C01*C02/C00;3;g/l
C00=                     1.0
C01=                   5.394
C02=                   1.000
          ------------
```

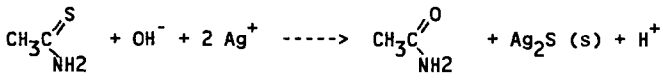
```
fr
date  91-08-14       time  11:45          6
U(init)          -358 mV  MET U          3-6
smpl size           5 ml
EP1            10.695 ml                -651 mV
RS1            11.538 g/l
stop  EP reached
            ============

cu
date  91-08-14       time  11:45          6
start V       5.000 ml  MET U          3-6
2.0 ml/div       dU=100.0 mV/div
```




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

Remarks:

- Determination reaction:



- Calculations:

RS1 = Ag coating in g/l
C01 = Ag aquivalent (5.394 mg/ml titrating agent)
C02 = titer of tirating agent (1.000)

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

- Ag_2S coating of electrode:
Keep Ag electrode in alcaline 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.

References:

Metrohm Application Bulletin No. 72

Example 3-7

Determination: **Detergents in Liquid Soap**

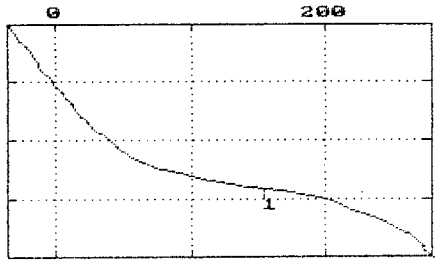
(Detergents industry)

Reagent:	c(HDPCl) = 0.01 mol/l	HDPCl: Hexadecyl pyridinium chloride
Sample:	4 ml sample solution 25 ml H_2O	Sample solution: 2.527 g liquid soap in 100 ml H_2O
Electrodes:	6.0504.130 BF_4^- sensitive indicator electrode 6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/l)	

```
MET  U                    3-7
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             10 mV/min
  equilibr.time            52 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    4 ml
  stop U                   OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                      30 mV
  EP recognition:     greatest
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
         ------------
```

```
fr
date  91-08-19       time  09:41          8
U(init)          -31 mV  MET U           3-7
smpl size     103.08 mg
EP1             2.845 ml                155 mV
RS1              7.96 %
stop V reached
           ============
```

```
cu
date  91-08-19       time  09:41          8
start V       0.000 ml  MET U           3-7
1.0 ml/div       dU=100.0 mV/div
```

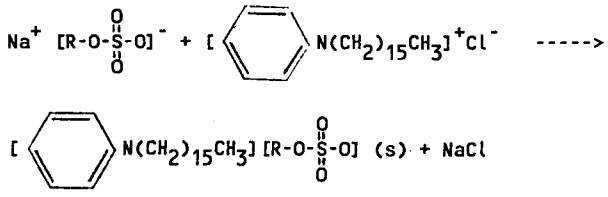



```
           ============
```

```
MET  U                    3-7
>calculations
RS1=EP1*C01*C02*C03/C00;2;%
C00=                103.08
C01=                  0.01
C02=                288.38
C03=                   100
         ------------
```

Remarks:

- Determination reactions:



- Calculations:

RS1 = content of detergent calculated as sodium dodecyl sulphate in %
CO1 = concentration of titrating agent (0.01 mol/l)
CO2 = molecular mass of sodium dodecyl sulphate $NaC_{12}H_{25}O_4S$ (288.38 g/mol)
CO3 = factor for % (100)

- Clean BF_4^- electrode after every titration with a moist cloth.

References:

W.Seilig, Z. anal. Chem. 300, 183 (1980)

Determination: **Cetrimide in Antiseptic Disinfectant** Example 3-8

(Pharmaceutical industry)

Reagent: c(Na laurylsulphate) = 0.01 mol/l

Sample: 1 ml sample, diluted 1:10
40 ml H_2O

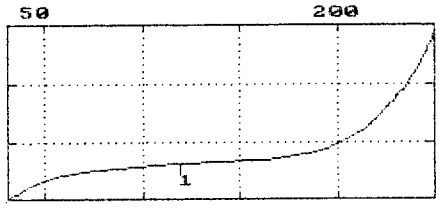
Sample:
Desogen

Electrodes: 6.0504.130 BF_4^- sensitive indicator electrode
6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/l)

```
MET  U                    3-8
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                   3 ml
  stop U                OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:               OFF
>evaluation
  EPC                     30 mV
  EP recognition:    greatest
  fix EP1 U             OFF mV
>preselections
  req.ident:            OFF
  req.smpl size:        OFF
  activate pulse:       OFF
        ------------
```

```
MET  U                    3-8
>calculations
RS1=EP1*C01*C02*C03/C00;2;%
C00=                    0.1
C01=                  3.364
C02=                  1.000
C03=                    0.1
        ------------
```

```
fr
date  91-08-19       time  13:19        15
U(init)          247 mV  MET U          3-8
smpl size          0.1 ml
EP1              2.382 ml              120 mV
RS1               8.01 %
stop V reached
          ============

cu
date  91-08-19       time  13:19        15
start V       0.000 ml  MET U          3-8
1.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Determination reaction:

- Calculations:

RS1 = % Cetrimid in g per liter
C01 = molecular mass of Cetrimid * concentration of titrating agent (3.364)
C02 = titer of titrating agent (1.000)
C03 = factor for % (0.1)

- Clean BF_4^- electrode after every titration with a moist cloth.

References:

Example 4-1

Determination: **Calcium/Magnesium in Tap Water**

(Water analytics)

Reagent: c(EDTA) = 0.05 mol/l in c(KOH) = 0.1 mol/l

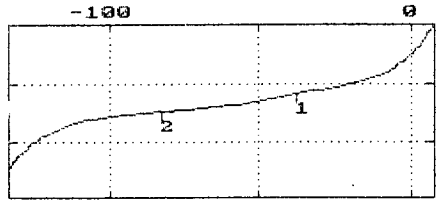
Sample: 50 ml tap water
15 ml auxiliary complexing agent (pH app. 8.5)

Electrodes: 6.0504.100 Ca^{2+} sensitive indicator electrode
6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/l)

```
MET  U                    4-1
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:                OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                   5 ml
  stop U                OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     10 mV
  EP recognition:       all
  fix EP1 U             OFF mV
>preselections
  req.ident:            OFF
  req.smpl size:        OFF
  activate pulse:       OFF
          ------------
```

```
fr
date  91-08-15      time  09:44        8
U(init)           8 mV  MET U         4-1
smpl size        50 ml
EP1           2.350 ml              -38 mV
EP2           2.974 ml              -83 mV
RS1            2.35
RS2            0.62
RS3            2.97
stop V reached
          ============
```

```
cu
date  91-08-15      time  09:44        8
start V      0.000 ml  MET U          4-1
2.0 ml/div      dU=50.0 mV/div
```




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

```
MET  U                    4-1
>calculations
RS1=EP1*C01*C02/C00;2;
RS2=(EP2-EP1)*C01*C02/C00;2;
RS3=EP2*C01*C02/C00;2;
C00=                    50
C01=                  0.05
C02=                  1000
          ------------
```

Remarks:

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

- Calculations:

  RS1 = calcium hardness in mmol/l
  RS2 = magnesium hardness in mmol/l
  RS3 = total hardness in mmol/l
  C01 = concentration of titrating agent (0.05 mol/l)
  C02 = factor for conversion mol -> mmol (1000)

- Sample preparation:

  Auxiliary complexing agent: c(Acetylacetone) = 0.1 mol/l
  c(Trishydroximethylamino methane) = 0.2 mol/l

- Electrode preparation:

  Soak electrode in $c(CaCl_2)$ = 0.1 mol/l solution during app. 15 min.

References:

Metrohm Application Bulletin No. 125

Example 4-2

Determination: **EDTA/NTA in Detergents**

(Detergent industry)

Reagent: $c(Cu^{2+})$ = 0.01 mol/l

Sample:
10 ml sample solution
10 ml buffer pH = 9.6 [$c(NH_3/NH_4NO_3)$ = 1 mol/l]
2 ml c(EDTA) = 0.01 mol/l
30 ml H_2O

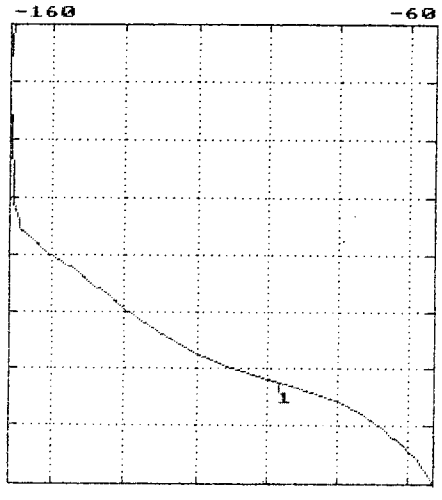
Electrodes:
6.0502.140 Cu^{2+} sensitive indicator electrode
6.0726.100 Ag/AgCl reference electrode (c(KCl) = 3 mol/l)

```
MET  U                    4-2
parameters
>titration parameters
  V step                  0.1 ml
  titr.rate               max. ml/min
  signal drift             10 mV/min
  equilibr.time            52 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                    4 ml
  stop U                   OFF mV
  stop EP                   9
  filling rate            max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
MET  U                    4-2
>calculations
RS1=(EP1-C01)*C02*C03/C00;2;%
C00=                   88.8
C01=                    2.0
C02=                    100
C03=                 1.9114
          ------------
```

```
fr
date  91-08-16       time  08:46          6
U(init)         -162 mV  MET U          4-2
smpl size        88.8 mg
EP1             3.129 ml              -97 mV
RS1              2.43 %
stop V reached
          ============
```

```
cu
date  91-08-16       time  08:46          6
start V       0.000 ml  MET U          4-2
0.5 ml/div       dU=20.0 mV/div
```




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

Remarks:

- Calculations:
  RS1 = content of EDTA/NTA in %
  CO1 = amount of EDTA added (2 ml)
  CO2 = factor for % (100)
  CO3 = 1ml $c(Cu^{2+})$ = 0.01 mol/l = 1.9114 mg NTA (resp. 2.9225 mg EDTA)

- Sample preparation:
  Dissolve 0.5 ... 1 g detergent in 50 ml dist. H_2O at 40°C.
  Allow solution to cool and add up to 100 ml.

- Enter sample size CO0 in mg, 1/10 aliquot.

References:

Metrohm Application Bulletin No. 143

Example 4-3

Determination: **Nickel (back titration)**

(Galvanic industry)

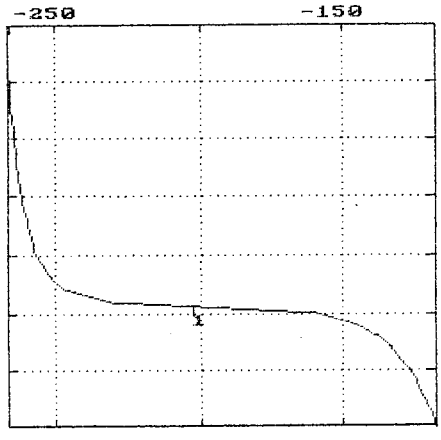
Reagent: $c(Cu^{2+})$ = 0.01 mol/l

Sample: 1.5 ml $c(Ni^{2+}) \cong$ 0.05 mol/l
10 ml H_2O
10 ml buffer pH = 9.6 [$c(NH_3/NH_4NO_3)$ = 1 mol/l]
1.00 ml c(EDTA) =0.1 mol/l

Electrodes: 6.0502.140 Cu^{2+} sensitive indicator electrode
6.0726.100 Ag/AgCl reference electrode (c(KCl) =3 mol/l)

```
MET  U                    4-3
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             10 mV/min
  equilibr.time            52 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                  3.5 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 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
          ------------

MET  U                    4-3
>calculations
RS1=(C01-EP1*C02*C03)*C04/C00;3;g/l
C00=                    1.5
C01=                    0.1
C02=                   0.01
C03=                  1.002
C04=                  58.71
          ------------
```

```
fr
date  91-08-15       time  11:20        14
U(init)          -250 mV  MET U         4-3
smpl size          1.5 ml
EP1              2.438 ml             -202 mV
RS1              2.958 g/l
stop V reached
          ============

cu
date  91-08-15       time  11:20        14
start V       0.000 ml  MET U          4-3
0.5 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Calculations:

RS1 = concentration of Ni^{2+} in g/l
C01 = amount of EDTA added (0.1 mmol)
C02 = concentration of titrating agent (0.01 mol/l)
C03 = titer (1.002)
C04 = molecular mass of Ni (58.71 g/mol)

References:

Determination: **Ca^{2+} with Amalgamated Ag Electrode**

Reagent: c(EDTA) = 0.1 mol/l

Sample: 2 ml $c(CaCl_2) \cong 0.1$ mol/l
10 ml buffer pH = 10
40 ml H_2O

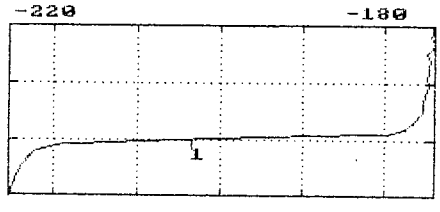
Electrodes: 6.0404.100 combined amalgamated Ag electrode

```
MET  U                    4-4
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift            OFF mV/min
  equilibr.time            10 s
  start V:                OFF
  pause                     0 s
  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 U               OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
MET  U                    4-4
>calculations
RS1=EP1*C01/C00;3;mol/l
C00=                        2
C01=                      0.1
          ------------
```

```
fr
date  91-08-19       time  14:53        20
U(init)         -173 mV  MET U          4-4
smpl size          2 ml
EP1             2.000 ml              -203 mV
RS1             0.100 mol/l
stop V reached
            ============
```

```
cu
date  91-08-19       time  14:53        20
start V       0.000 ml  MET U          4-4
1.0 ml/div       dU=10.0 mV/div
```




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

Remarks:

- Calculations:

  RS1 = concentration of Ca^{2+} solution in mol/l
  CO1 = concentration of titrating agent (0.1 mol/l)

- Coating of Ag electrode:

  Clean electrode first by immersing it in conc. HNO_3, then immerse it shortly in Hg.

References:

Determination: **Diazotation of 1-Naphthylamine-5-sulfonic acid**

Reagent: $c(NaNO_2)$ = 0.1 mol/l

Sample: 0.05 ... 0.1 g sample
20 ml w(HCl) = 36 %
300 ml H_2O

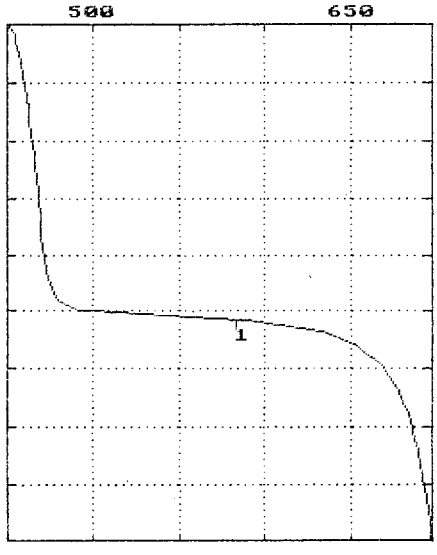
Electrodes: 6.0413.100 combined Au electrode

```
MET  U                 Diazo
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift           OFF mV/min
  equilibr.time           20 s
  start V:              abs.
  start V                0.5 ml
  dos.rate              max. ml/min
  pause                   80 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                   5 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:    greatest
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
         ------------
```

```
MET  U                 Diazo
>calculations
Content=EP1*C01*C02*C03/C00;2;%
C00=              0.0691
C01=              22.325
C02=               1.000
C03=                 0.1
         ------------
```

```
fr
date  91-08-19      time  17:06       22
U(init)         448 mV  MET U      Diazo
smpl size    0.0691 g
EP1           3.089 ml            583 mV
Content       99.80 %
stop V reached
         ============
```

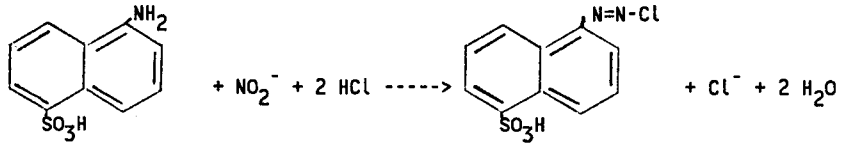
```
cu
date  91-08-19      time  17:06       22
start V      0.500 ml  MET U       Diazo
0.5 ml/div      dU=50.0 mV/div
```




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

Remarks:

- Determination reaction:



- Calculations:

  Content = fraction of "acid" in %
  C01 = molecular mass of "acid"/10 (22.3)
  C02 = titer (1.000)
  C03 = factor for conversion ml -> l and for % (0.001 * 100 = 0.1)

- Clean electrode from time to time as follows:
  First with scouring powder, then immerse it in a solution of 0.5 g quinhydrone in 50 ml buffer pH = 4. Leave it in solution for 5 minutes.

References:

Example 5-2

Determination: **Diazotation of Cyclamate**

(Food industry)

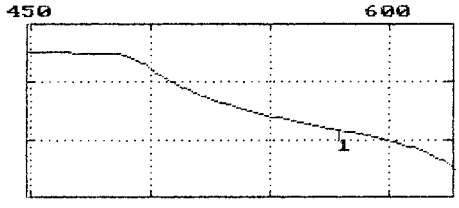
Reagent: $c(NaNO_2)$ = 0.1 mol/l

Sample: app. 100 mg sweetener
40 ml H_2O
10 ml w(HBr) = 20 %

Electrodes: 6.0413.100 combined Au electrode

```
MET  U                 Diazo
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift           OFF mV/min
  equilibr.time           20 s
  start V:               abs.
  start V                  1 ml
  dos.rate              max. ml/min
  pause                   80 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:                abs.
  stop V                   5 ml
  stop U                 OFF mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     10 mV
  EP recognition:    greatest
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:          ON
  activate pulse:        OFF
          ------------

MET  U                 Diazo
>calculations
Content=EP1*C01*C02*C03/C00;2;%
C00=               0.0993
C01=              20.106
C02=               1.000
C03=                 0.1
          ------------
```

```
fr
date  91-08-21       time  16:04        15
U(init)          450 mV  MET U       Diazo
smpl size     0.0993 g
EP1            3.664 ml             579 mV
Content         74.18 %
stop V reached
          ============

cu
date  91-08-21       time  16:04        15
start V       1.000 ml  MET U        Diazo
2.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Calculations:

  Content = content of cyclamate in %
  CO1 = molecular mass of cyclamate * concentration of
  titrating agent (20.106)
  CO2 = titer (1.000)
  CO3 = factor for % (0.1)

- Clean electrode from time to time as follows:
  First with scouring powder, then immerse it in a solution of 0.5 g quinhydrone in 50 ml buffer pH = 4. Leave it in this solution for 5 minutes.

References:

Example 5-3

Determination: **Hydrogen Peroxide**

Reagent: $c(KMnO_4)$ = 0.02 mol/l

Sample:

2 ml sample
2 ml $c(H_2SO_4)$ = 0.5 mol/l
0.1 g $MnSO_4 \cdot H_2O$ (catalyst)
100 ml H_2O

Sample:
app. 1.2 g $w(H_2O_2)$ = 30 % in
100 ml $c(H_2SO_4)$ = 0.5 mol/l

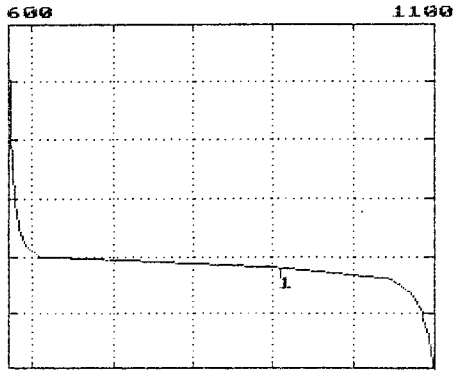
Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-3
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            10 mV/min
  equilibr.time           52 s
  start V:              abs.
  start V                  1 ml
  dos.rate              max. ml/min
  pause                    0 s
  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:                 ON
  mean                n=   3
  res.tab:            original
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 U               OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:           ON
  activate pulse:         OFF
          ------------
```

```
MET  U                    5-3
>calculations
RS1=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                  1.190
C01=                0.02791
C02=                   2.5
C03=                 34.02
C04=                   0.1
C05=                    50
          ------------
```

```
fr
date  91-08-21       time  13:40          6
U(init)           572 mV  MET U          5-3
smpl size       1.190 g
EP1             3.099 ml              910 mV
RS1             30.91 %
                mean( 3)    +/-s          s/%
RS1               30.89    0.020 %       0.06
stop V reached
          =============
```

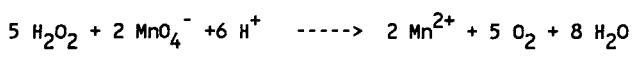
```
cu
date  91-08-21       time  13:40          6
start V       1.000 ml  MET U          5-3
0.5 ml/div       dU=100.0 mV/div
```




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

Remarks:

- Determination reaction:



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

- Calculations:

RS1 = fraction of H_2O_2 in %
C01 = concentration of tirating agent * titer (0.02781)
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 * factor for % (0.001 * 100 = 0.1)
C05 = dilution factor (50)

References:

Example 5-4

Determination: **Perborates in Detergents**

(Detergent industry)

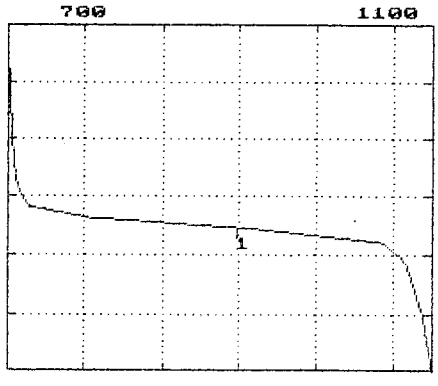
Reagent: $c(KMnO_4)$ = 0.02 mol/l

Sample: 5 ml detergent solution
20 ml $w(H_2SO_4)$ = 30%
20 ml H_2O

Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-4
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             10 mV/min
  equilibr.time            52 s
  start V:                 OFF
  pause                     0 s
  meas.input:               1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                    3 ml
  stop U                   OFF mV
  stop EP                   9
  filling rate           max. ml/min
>statistics
  status:                  OFF
>evaluation
  EPC                       30 mV
  EP recognition:        all
  fix EP1 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:            ON
  activate pulse:          OFF
          ------------
```

```
fr
date  91-08-22       time  16:00          6
U(init)          604 mV  MET U          5-4
smpl size       2500 mg
EP1            1.786 ml                897 mV
RS1            15.34 %
stop V reached
          ============

cu
date  91-08-22       time  16:00          6
start V       0.000 ml  MET U          5-4
0.5 ml/div       dU=100.0 mV/div
```

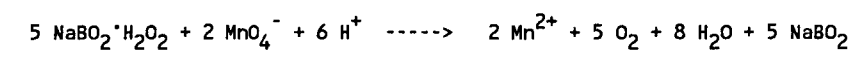



```
          ============
```

```
MET  U                    5-4
>calculations
RS1=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                    2500
C01=                 0.02791
C02=                     2.5
C03=                  153.86
C04=                     100
C05=                      20
          ------------
```

Remarks:

- Determination reaction:



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

- Calculations:

RS1 = fraction of perborate in %
C01 = concentration of tirating agent (0.02 mol/l)
C02 = factor for "normality" (5/2 = 2.5)
C03 = molecular mass of $NaBO_2 \cdot H_2O_2 \cdot 3H_2O$ (153.86 g/mol)
C04 = factor for % (100)
C05 = factor for dilution (20)

- Sample preparation:
Weigh 2.5 g sample 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 $w(H_2SO_4)$ = 30 % and 20 ml H2O and titrate.

References:

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

Determination: **Iron (II)**

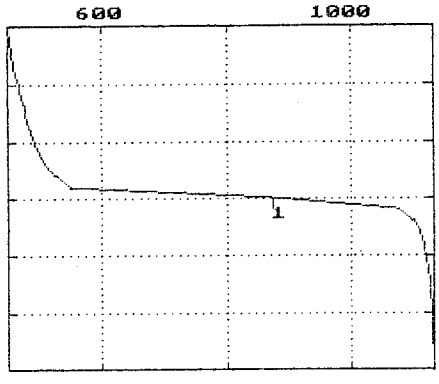
Reagent: $c(KMnO_4)$ = 0.02 mol/l

Sample: 2 ml $c[(NH_4)_2Fe(SO_4)_2] \tilde{=} 0.1$ mol/l
10 ml $c(H_2SO_4)$ = 0.5 mol/l
40 ml H_2O

Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-5
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              10 mV/min
  equilibr.time             52 s
  start V:                  OFF
  pause                      0 s
  meas.input:                1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                     3 ml
  stop U                    OFF mV
  stop EP                    9
  filling rate            max. ml/min
>statistics
  status:                   OFF
>evaluation
  EPC                        30 mV
  EP recognition:           all
  fix EP1 U                 OFF mV
>preselections
  req.ident:                OFF
  req.smpl size:            OFF
  activate pulse:           OFF
          ------------

MET  U                    5-5
>calculations
RS1=EP1*C01*C02/C00;2;g/l
C00=                         2
C01=                   0.13955
C02=                     55.85
          ------------
```

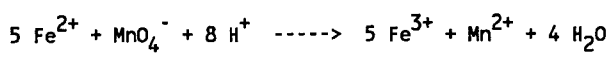
```
fr
date  91-08-22       time  17:15        11
U(init)           489 mV  MET U         5-5
smpl size           2 ml
EP1             1.500 ml               874 mV
RS1             5.84 g/l
stop V reached
          ============

cu
date  91-08-22       time  17:15        11
start V       0.000 ml  MET U          5-5
0.5 ml/div       dU=200.0 mV/div
```




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

Remarks:

- Determination reaction:



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

- Calculations:

RS1 = concentration of Fe in g/l

C01 = concentration of titrating agent * titer * "normality"
(0.02 * 1.3955 * 5 = 0.13955)

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

References:

Example 5-6

Determination: **COD Determination**

(Water analytics)

Reagent: $c[(NH_4)_2Fe(SO_4)_2] = 0.12$ mol/l

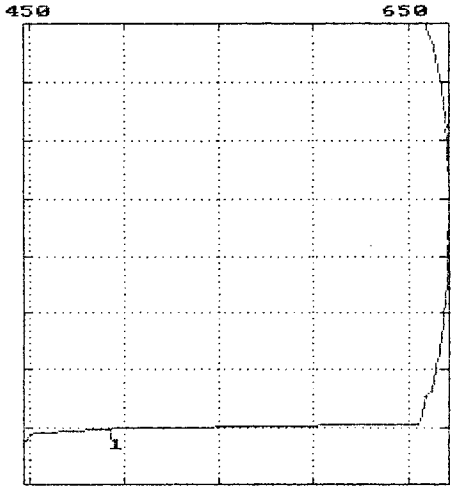
Sample:
20 ml waste water
2 ml $c(K_2Cr_2O_7) = 0.02$ mol/l containing 80 g/l $HgSO_4$
30 ml $c(Ag_2SO_4) = 0.01$ mol/l in $w(H_2SO_4) = 30$ %
100 ml H_2O

Electrodes:
6.0331.030 massive Au electrode
6.0726.100 Ag/AgCl reference electrode (KNO_3 sat.)

```
MET  U                      1
parameters
>titration parameters
  V step                0.08 ml
  titr.rate             max. ml/min
  signal drift            70 mV/min
  equilibr.time           22 s
  start V:               OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                  25 ml
  stop U                OFF mV
  stop EP                  1
  filling rate          max. ml/min
>statistics
  status:               OFF
>evaluation
  EPC                     40 mV
  EP recognition:       all
  fix EP1 U             OFF mV
>preselections
  req.ident:            OFF
  req.smpl size:        OFF
  activate pulse:       OFF
          ------------
```

```
MET  U                      1
>calculations
RS1=(C30-EP1)*C01*C02/C00;0;ppm
C00=                      20
C01=                    0.12
C02=                    8000
C30=                   8.325
          ------------
```

```
fr
date  91-08-16       time  10:55          4
U(init)           659 mV  MET U           1
smpl size          20 ml
EP1              7.040 ml                 493 mV
RS1                62 ppm
stop  EP reached
             ============

cu
date  91-08-16       time  10:55          4
start V        0.000 ml  MET U           1
1.0 ml/div        dU=50.0 mV/div
```




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

Remarks:

- COD = Chemical Oxygen Demand

  which means the amount of O_2 that would be consumed to oxidize all the substances which are oxidized by $K_2Cr_2O_7$.
  (1 mol $K_2Cr_2O_7$ = 1.5 mol O_2)

- Ag_2SO_4 is used as a catalyst.

- Calculations:

  RS1 = COD value in ppm
  C01 = concentration of tirating agent (0.12 mol/l)
  C02 = equivalent factor (8000)
  C30 = consumption of blank sample (8.325), common variable!

- The consumption of blank sample has been determined out of 2 titrations as a mean value by a previous method. The determined mean value was stored as the common variable C30.

- Sample preparation:

  Add auxiliary solutions to sample. Heat to boiling during 10 min and keep refluxing during 110 min at a temperature of 148±3°C. Allow to cool below 60°C and dilute to 100 ml. If sample is at room temperature, titrate.

References:

DIN 38 409 Teil 41
Metrohm Application Bulletin No. 212

Example 5-7

Determination: **Iodine**

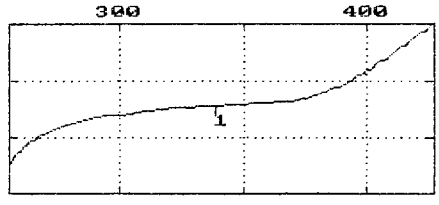
Reagent: $c(Na_2S_2O_3)$ = 0.1 mol/l

Sample: 3 ml $c(KI_3) \tilde{=}$ 0.05 mol/l
5 ml $c(H_2SO_4)$ = 0.5 mol/l
20 ml H_2O

Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-7
parameters
>titration parameters
  V step                 0.10 ml
  titr.rate              max. ml/min
  signal drift             20 mV/min
  equilibr.time            38 s
  start V:                 OFF
  pause                     0 s
  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 U                OFF mV
>preselections
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------

MET  U                     5-7
>calculations
RS1=EP1*C01/(C02*C00);4;mol/l
C00=                       3
C01=                     0.1
C02=                       2
          ------------
```

```
fr
date  91-08-23       time  08:31          7
U(init)          427 mV  MET U          5-7
smpl size          3 ml
EP1            2.911 ml                339 mV
RS1           0.0485 mol/l
stop V reached
          ============

cu
date  91-08-23       time  08:31          7
start V       0.000 ml  MET U          5-7
2.0 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Determination reaction:

$$I_2 + 2\ S_2O_3^{2-} \quad \text{-----} > \quad 2\ I^- + S_4O_6^{2-}$$

- Calculations:

RS1 = concentration of KI_3 solution in mol/l
C01 = concentration of tirating agent (0.1 mol/l)
C02 = factor for "normality" (2)

References:

Determination: **Ascorbic Acid**

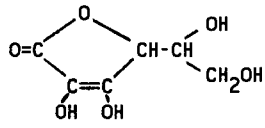
Example 5-8

(Pharmaceutical and food industry)

Reagent: $c(KI_3)$ = 0.05 mol/l

Sample: 2 ml $c(C_6H_8O_6)$ = 0.1 mol/l
5 ml $c(H_2SO_4)$ = 2 mol/l
20 ml H_2O

Ascorbic acid:



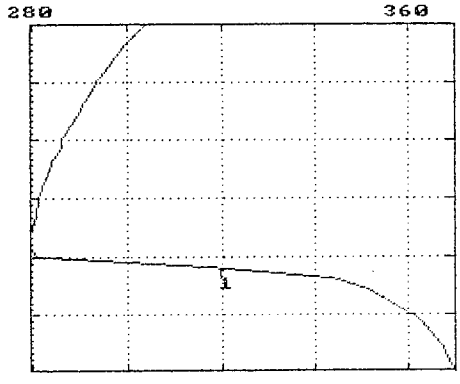
Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-8
parameters
>titration parameters
  V step                  0.10 ml
  titr.rate               max. ml/min
  signal drift              50 mV/min
  equilibr.time             26 s
  start V:                abs.
  start V                    2 ml
  dos.rate                max. ml/min
  pause                      0 s
  meas.input:                1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                     5 ml
  stop U                  OFF mV
  stop EP                    9
  filling rate            max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                       30 mV
  EP recognition:         all
  fix EP1 U               OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------
```

```
MET  U                    5-8
>calculations
RS1=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                    1770
C01=                    0.05
C02=                  0.9802
C03=                   176.1
C04=                     100
C05=                      50
          ------------
```

```
fr
date  91-08-23       time  15:10        12
U(init)           389 mV  MET U         5-8
smpl size        1770 mg
EP1             4.099 ml              319 mV
RS1             99.95 %
stop V reached
          ============
```

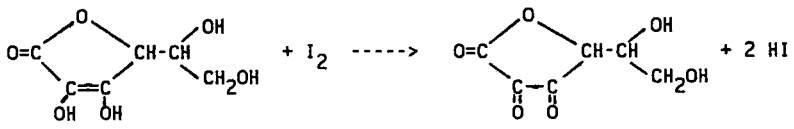
```
cu
date  91-08-23       time  15:10        12
start V       2.000 ml  MET U          5-8
0.5 ml/div       dU=20.0 mV/div
```




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

Remarks:

- Determination reaction:



- Calculations:

RS1 = fraction of ascorbic acid in % (purity)

C01 = concentration of titrating agent (0.05 mol/l)

C02 = titer (0.9802)

C03 = molecular mass of ascorbic acid (176.1 g/mol)

C04 = factor for % (100)

C05 = sample size, aliquot (1:50)

References:

Example 5-9

Determination: **Tin (II) in Plating Bath**

(Galvanic industry)

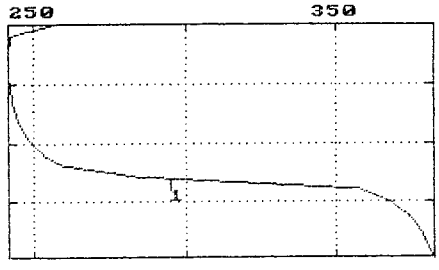
Reagent: $c(KI_3)$ = 0.05 mol/l

Sample: 2 ml $c(Sn^{2+})$ = 0.1 mol/l in $c(H_2SO_4)$ = 2 mol/l
10 ml $c(H_2SO_4)$ = 2 mol/l
40 ml H_2O

Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                    5-9
parameters
>titration parameters
  V step                  0.1 ml
  titr.rate               max. ml/min
  signal drift             10 mV/min
  equilibr.time            52 s
  start V:                abs.
  start V                   2 ml
  dos.rate                max. ml/min
  pause                     0 s
  meas.input:               1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                    4 ml
  stop U                  OFF mV
  stop EP                   9
  filling rate            max. ml/min
>statistics
  status:                 OFF
>evaluation
  EPC                      30 mV
  EP recognition:         all
  fix EP1 U               OFF mV
>preselections
  req.ident:              OFF
  req.smpl size:          OFF
  activate pulse:         OFF
          ------------

MET  U                    5-9
>calculations
RS1=EP1*C01*C02/C00;3;g/l
>C00=                        2
C01=                     0.05
C02=                   118.69
          ------------
```

```
fr
date  91-08-26      time  09:23        15
U(init)          338 mV  MET U         5-9
smpl size          2 ml
EP1            3.314 ml              295 mV
RS1            9.835 g/l
stop V reached
          ============

cu
date  91-08-26      time  09:23        15
start V      2.000 ml  MET U          5-9
0.5 ml/div       dU=50.0 mV/div
```




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

Remarks:

- Determination reaction:

$$Sn^{2+} + I_2 \text{ -----> } Sn^{4+} + 2\ I^-$$

- Calculations:

RS1 = concentration of Sn^{2+} in g/l
C01 = concentration of titrating agent (0.05 mol/l)
C02 = molecular mass of Sn (118.69 g/mol)

References:

Example 5-10

Determination: **Oxidizability of Waste Water**

(Water analytics)

Reagent: $c(KMnO_4)$ = 0.01 mol/l

Sample:
25 ml waste water
5 ml $w(H_2SO_4)$ = 35 %
75 ml H_2O
15 ml $c(KMnO_4)$ = 0.01 mol/l
15 ml c(Oxalic acid) = 0.01 mol/l

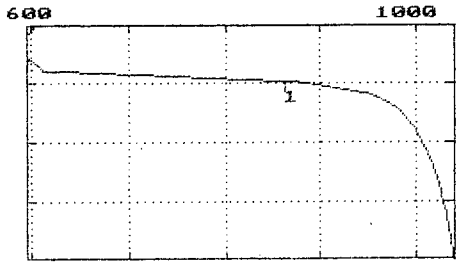
Electrodes: 6.0415.100 combined massive Pt electrode

```
MET  U                  5-10
parameters
>titration parameters
  V step                0.10 ml
  titr.rate             max. ml/min
  signal drift            20 mV/min
  equilibr.time           38 s
  start V:              abs.
  start V                  5 ml
  dos.rate              max. ml/min
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                  15 ml
  stop U                1040 mV
  stop EP                  9
  filling rate          max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                     30 mV
  EP recognition:        all
  fix EP1 U              OFF mV
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
         ------------
```

```
MET  U                  5-10
>calculations
RS1=EP1*C01*C02/C00;2;mg
C00=                     25
C01=                  0.316
C02=                   1000
         ------------
```

```
fr
date  91-08-27      time  09:51        3
U(init)          664 mV  MET U        5-10
smpl size         25 ml
EP1             5.493 ml             864 mV
RS1             69.43 mg
stop meas.val.reached
          ============
```

```
cu
date  91-08-27      time  09:51        3
start V       5.000 ml  MET U         5-10
0.5 ml/div       dU=100.0 mV/div
```




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

Remarks:

- Prepare sample as follows:
  Add 75 ml H_2O dist. and 5 ml $w(H_2SO_4)$ = 35 % to 25 ml of sample and heat up to a boiling mixture. Add 15.00 ml of $c(KMnO_4)$ = 0.01 mol/l and keep boiling for 10 minutes. Then add 15 ml c(Oxalic acid) = 0.01 mol/l.
  Titrate with $c(KMnO_4)$ = 0.01 mol/l according to parameters.

- Calculations:

  RS1 = 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)

References:

Deutsche Einheitsverfahren zur Wasseruntersuchung
Kapitel H4, Abschnitt 1

Determination: **Chloride**

Reagent: $c(AgNO_3)$ = 0.01 mol/l

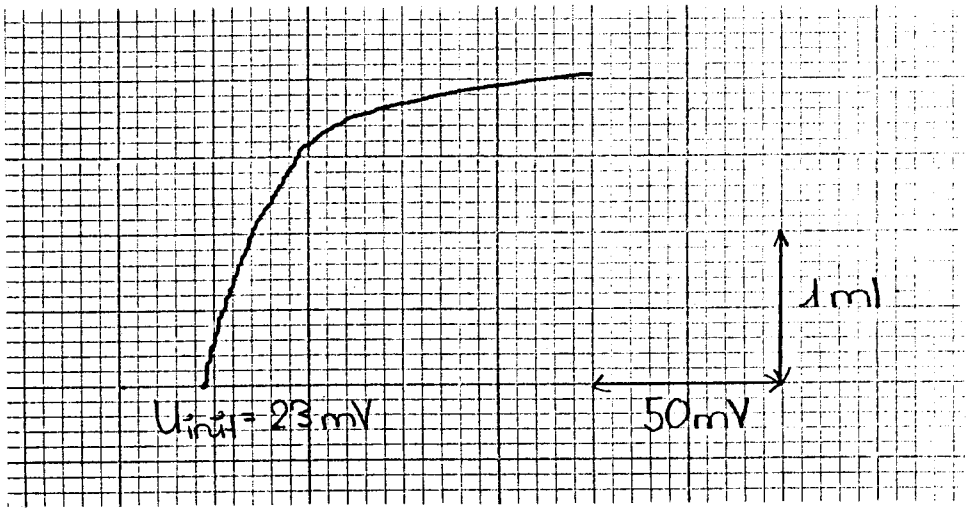
Sample: 0.2 ml $c(KCl) \cong$ 0.1 mol/l
2 ml $c(HNO_3)$ = 2 mol/l
25 ml H_2O

Electrodes: 6.0404.100 combined massive Ag electrode

```
pa
date  91-05-14      time 10:32        14
SET  U                  6-1
parameters
>SET1
  EP at U                118 mV
  dynamics                70 mV
  max.rate              10.0 ml/min
  min.rate                25 ul/min
  stop.crit:             drift
  stop drift              20 ul/min
>SET2
  EP at U                OFF mV
>titration parameters
  titr.direction:        auto
  start V:               OFF
  pause                    0 s
  meas.input:              1
  temperature            25.0  C
>stop conditions
  stop V:                abs.
  stop V                99.99 ml
  filling rate           max. ml/min
>statistics
  status:                OFF
>preselections
  conditioning:          OFF
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
         ============
```

```
fm
date  91-05-14      time 10:32        14
SET  U                  6-1
>calculations
RS1=EP1*C01/C00;3;mol/l
C00=                     0.2
C01=                    0.01
         ============
fr
date  91-05-14      time 10:32        14
U(init)          23 mV  SET U        6-1
smpl size       0.2 ml
EP1           2.013 ml             119 mV
RS1           0.101 mol/l
         ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph



Remarks:

- Calculations:

  RS1 = concentration of Cl^- in mol/l
  CO1 = concentration of titrating agent (0.01 mol/l)

- Determine EP with MET U. The EP may change depending on electrode.

References:

Example 6-2

Determination: **Analysis of Rayon Spinning Bath**

(Synthetic fiber industry)

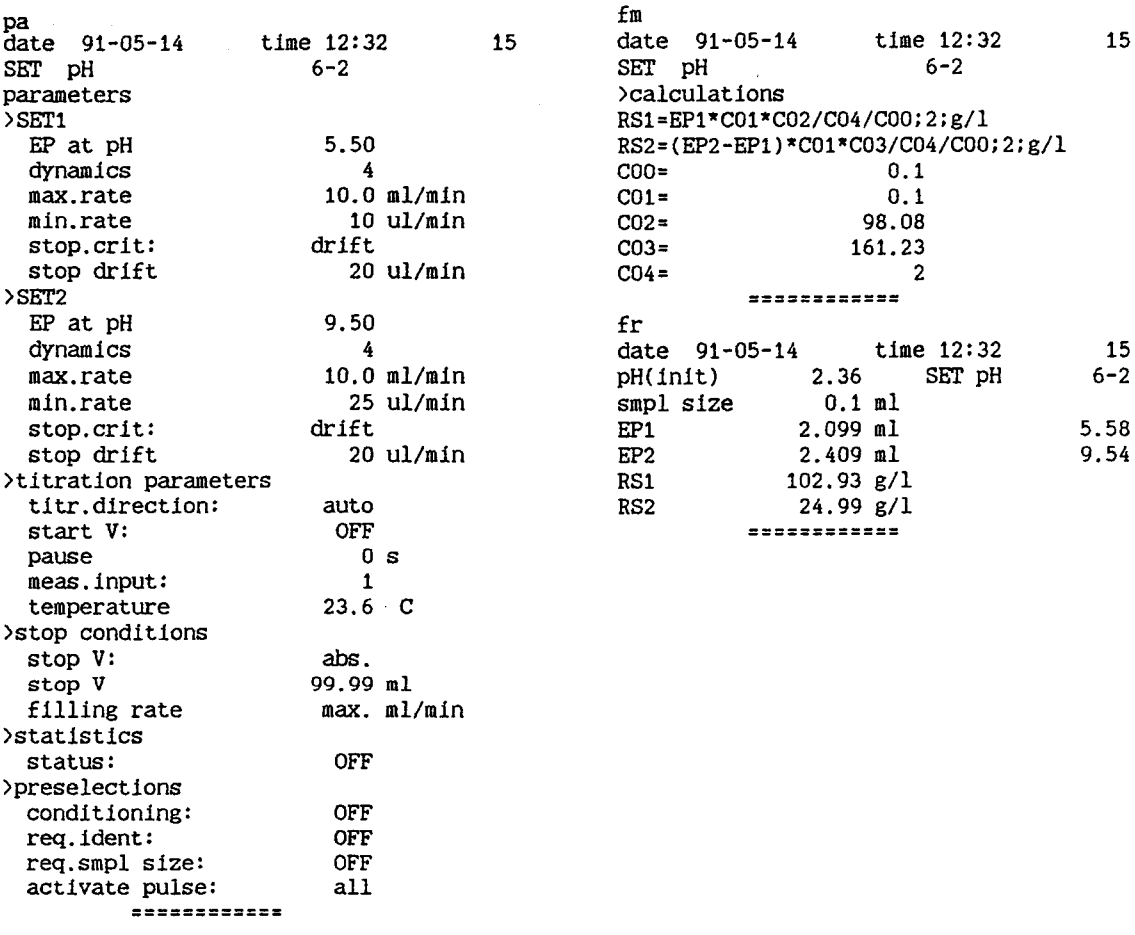
Reagent: c(NaOH) = 0.1 mol/l

Sample: 0.1 ml $c(ZnSO_4)$ = 0.1 mol/l in $c(H_2SO_4)$ = 1 mol/l
25 ml H_2O

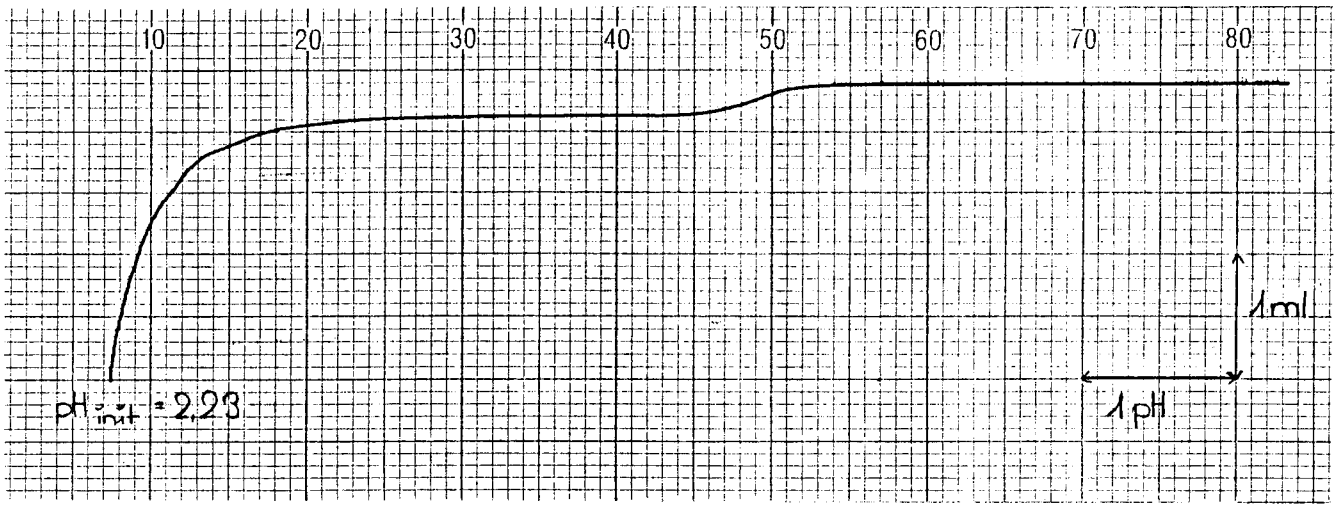
Electrodes: 6.0203.100 combined pH glass electrode

```
pa
date  91-05-14      time 12:32        15
SET  pH                 6-2
parameters
>SET1
  EP at pH              5.50
  dynamics                 4
  max.rate              10.0 ml/min
  min.rate                10 ul/min
  stop.crit:           drift
  stop drift              20 ul/min
>SET2
  EP at pH              9.50
  dynamics                 4
  max.rate              10.0 ml/min
  min.rate                25 ul/min
  stop.crit:           drift
  stop drift              20 ul/min
>titration parameters
  titr.direction:       auto
  start V:               OFF
  pause                    0 s
  meas.input:              1
  temperature           23.6  C
>stop conditions
  stop V:               abs.
  stop V               99.99 ml
  filling rate          max. ml/min
>statistics
  status:                OFF
>preselections
  conditioning:          OFF
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        all
         ============
```

```
fm
date  91-05-14      time 12:32        15
SET  pH                 6-2
>calculations
RS1=EP1*C01*C02/C04/C00;2;g/l
RS2=(EP2-EP1)*C01*C03/C04/C00;2;g/l
C00=                     0.1
C01=                     0.1
C02=                   98.08
C03=                  161.23
C04=                       2
         ============
fr
date  91-05-14      time 12:32        15
pH(init)       2.36     SET pH       6-2
smpl size       0.1 ml
EP1           2.099 ml               5.58
EP2           2.409 ml               9.54
RS1          102.93 g/l
RS2           24.99 g/l
         ============
```



Analog titration curve, recorded on-line with 586 Metrohm Labograph



Remarks:

- EP1: $H_2SO_4 + 2\ OH^- \text{ -----> } SO_4^{2-} + 2\ H_2O$

  EP2: $Zn^{2+} + 2\ OH^- \text{ -----> } Zn(OH)_2\ (s)$

- Calculations:

  RS1 = concentration of H_2SO_4 in g/l
  RS2 = concentration of $ZnSO_4$ in g/l
  CO1 = concentration of titrating agent (0.1 mol/l)
  CO2 = molecular mass of H_2SO_4 (98.08 g/mol)
  CO3 = molecular mass of $ZnSO_4$ (161.23 g/mol)
  CO4 = factor for "normality" (2)

References:

Example 6-3

Determination: **p and m Value**

(Water analytics)

Reagent: c(HCl) = 0.1 mol/l

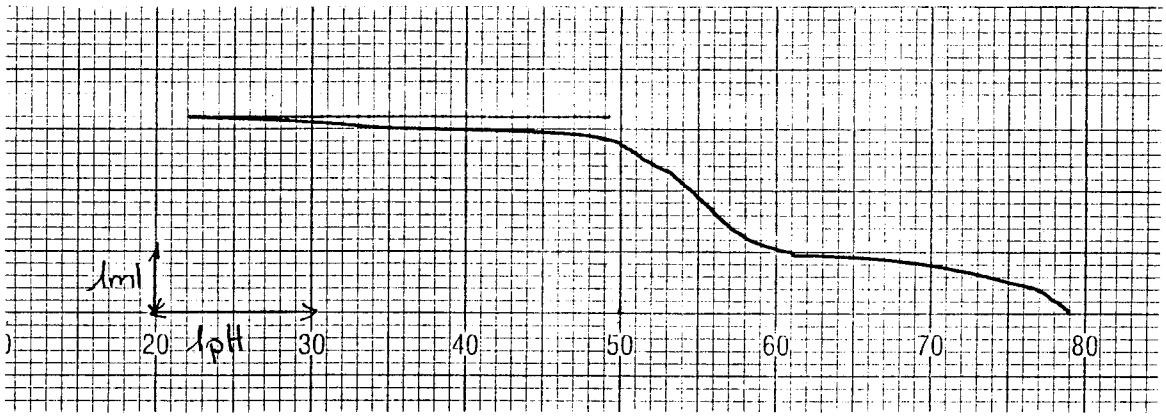
Sample: 25 ml tap water
0.1 ml $c(Na_2CO_3)$ = 1 mol/l

Electrodes: 6.0203.100 combined pH glass electrode

```
pa
date  91-05-15      time  16:50        30
SET  pH                  6-3
parameters
>SET1
  EP at pH               8.2
  dynamics                 2
  max.rate              10.0 ml/min
  min.rate              25.0 ul/min
  stop.crit:           drift
  stop drift               20 ul/min
>SET2
  EP at pH               4.3
  dynamics                 3
  max.rate              10.0 ml/min
  min.rate              25.0 ul/min
  stop.crit:           drift
  stop drift               20 ul/min
>titration parameters
  titr.direction:       auto
  start V:               OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V               99.99 ml
  filling rate          max. ml/min
>statistics
  status:                OFF
>preselections
  conditioning:          OFF
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
          ============
```

```
fm
date  91-05-15      time 16:50        30
SET  pH                  6-3
>calculations
RS1=(EP1*C01-C02)*C03;2;
RS2=(EP2*C00-C02)*C03;2;
C00=                   1.0
C01=                 1.000
C02=                  1.00
C03=                     4
          ============
fr
date  91-05-15      time 16:50        30
pHc(init)      9.82     SET pH       6-3
EP1           0.935 ml               8.08
EP2           3.209 ml               4.29
RS1           -0.26
RS2            8.84
          ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph



Remarks:

- Values for the acid capacity of water.
  p value = value of Phenolphthalein
  m value = value of Methyl orange
  If the titration is executed with NaOH the values give the base capacity of water.

- Calculations:

  RS1 = p value (if negative, the water has an initial pH below 8.2)
  RS2 = m value
  both calculated in mmol/l $[n(H_3O^+)/V(H_2O)]$
  CO1 = concentration of titrating agent x 10 (1.000 mol/l)
  CO2 = amount of CO_3^{2-} added x 10 (1.00 mmol)
  CO3 = factor for sample size (4)

References:

DIN 38 409, Teil 7 (1979)

Example 6-4

Determination: **Bromine Number**

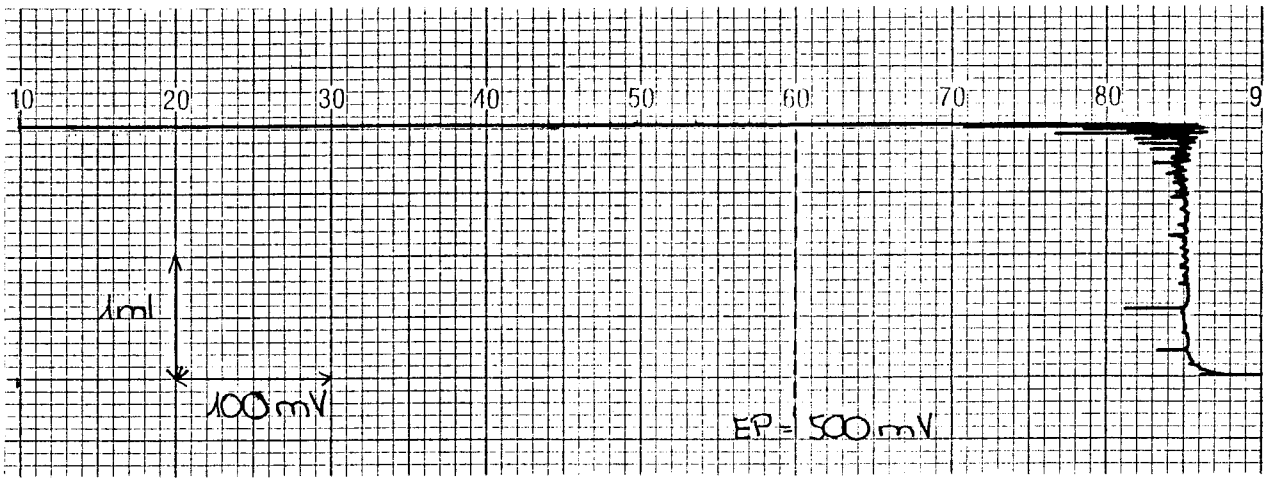
(Petroleum products industry)

Reagent:	c(1/6$KBrO_3$) = 0.5 mol/l	Dissolve 51 g KBr and 13.92 g $KBrO_3$ each in dist H_2O and add up to 1 l.
Sample:	1ml c(cyclohexene) = 6% in solvent 25 ml solvent	Solvent: 714 ml acetic acid 134 ml CCl_4 134 ml CH_3OH 18 ml H_2SO_4 (20%)
Electrodes:	6.0309.100 double Pt electrode, polarized I_{pol} = 10 µA Voltametric indication	

```
pa
date  91-05-14      time 15:55        28
SET  Ipol              6-4
parameters
>SET1
  EP at U              500 mV
  dynamics             500 mV
  max.rate            10.0 ml/min
  min.rate              25 ul/min
  stop.crit:          drift
  stop drift            20 ul/min
>SET2
  EP at U              OFF mV
>titration parameters
  titr.direction:        -
  start V:             OFF
  pause                  0 s
  I(pol)                10 uA
  electrode test:      OFF
  temperature         25.0  C
>stop conditions
  stop V:             abs.
  stop V             99.99 ml
  filling rate        max. ml/min
>statistics
  status:              OFF
>preselections
  conditioning:        OFF
  req.ident:           OFF
  req.smpl size:       OFF
  activate pulse:      OFF
          ============
```

```
fm
date  91-05-14      time 15:55        28
SET  Ipol              6-4
>calculations
RS1=(EP1-C01)*C02*C03*C04/C00;0;
C00=                 6.00
C01=                    0
C02=                  0.5
C03=                 7.99
C04=                  100
          ============
fr
date  91-05-14      time 15:55        28
U(init)          942 mV  SET Ipol     6-4
smpl size       6.00 g
EP1            2.062 ml             35 mV
RS1              137
          ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph

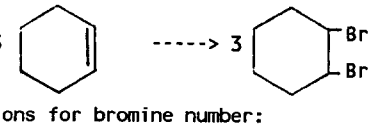


Remarks:

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

- Standardisation of BrO_3^-/Br^- solution:
  Determination reaction:
  $3\ Br_2 + 6\ S_2O_3^{2-}$ -----> $6\ Br^- + 3\ S_4O_6^{2-}$
  Calculation:
  RS1=EP1*C01/C00 normality of titrating reagent (BrO_3^-/Br^-)
  C01 = concentration of titrating agent ($S_2O_3^{2-}$)
  C00 = ml of BrO_3^-/Br^- solution

- Determination reactions for bromine number:
  $BrO_3^- + 5\ Br^- + 6\ H^+$ -----> $3\ Br_2 + 3\ H_2O$



- Calculations for bromine number:
  RS1 = bromine number
  C01 = consumption of blank sample (0 ml)
  C02 = normality of BrO_3^-/Br^- solution as calculated above (0.5)
  C03 = molecular mass of Br_2 multiplied by 0.05 (7.99 g/mol)
  C04 = dilution factor (100)

References:

ASTM D1159-84

Example 6-5

Determination: **pH Stat Titration**

Reagent: c(HCl) = 1 mol/l

Sample: 200 mg "Andursil"
100 ml H_2O pH = 3

1 tablet "Andursil" containing:
750 mg $Al(OH)_3/MgCO_3$
250 mg activ.Polymethyl siloxane

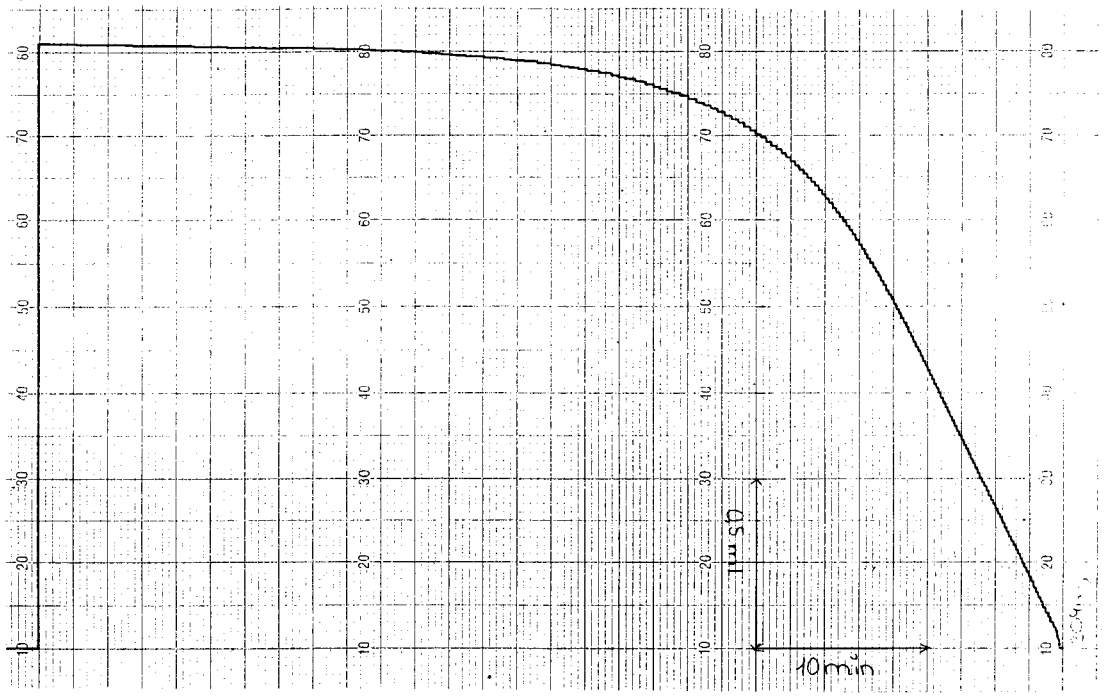
Electrodes: 6.0203.100 combined pH glass electrode

```
pa
date  91-05-17      time 11:01          6
SET  pH                 6-5
parameters
>SET1
  EP at pH              3.00
  dynamics                 1
  max.rate              10.0 ml/min
  min.rate                25 ul/min
  stop.crit:            time
  t(delay)               INF s
  stop time             3600 s
>SET2
  EP at pH               OFF
>titration parameters
  titr.direction:          -
  start V:               OFF
  pause                    0 s
  meas.input:              1
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V               99.99 ml
  filling rate          max. ml/min
>statistics
  status:                OFF
>preselections
  conditioning:          OFF
  req.ident:             OFF
  req.smpl size:         OFF
  activate pulse:        OFF
         ============
```

```
date  91-05-16      time 13:14          6
meas.input:              1     CAL
cal.date        91-05-16
                        pH     U/mV
buffer  1             7.00      -13
buffer  2             4.00      160
cal.temp              22.0  C
slope(rel)           0.984       pH(as)  6.78
          ------------
```

```
fr
date  91-05-17      time 11:01          6
pHc(init)        3.18     SET pH       6-5
EP1           1.728 ml                 3.00
          ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph



Remarks:

- Select curve "V" to be outputted at the analog output in <configuration> key.

```
configuration
>peripheral units
  send to:              Seiko
  balance:           Sartorius
  record:                   V
>auxiliaries
  dialog:             english
  date                91-05-17
  time                   12:26
  run number                 6
  auto start               OFF
  program               02.05.91
>RS232 settings
  baud rate:              9600
  data bit:                  8
  stop bit:                  1
  parity:                 none
  handshake:               HWs
  RS control:              OFF
```

References:

Journal of Pharmaceutical Sciences 1977, 66 , p. 1528-1533

Determination: **Hydrogen Peroxide**

Reagent: $c(KMnO_4)$ = 0.02 mol/l

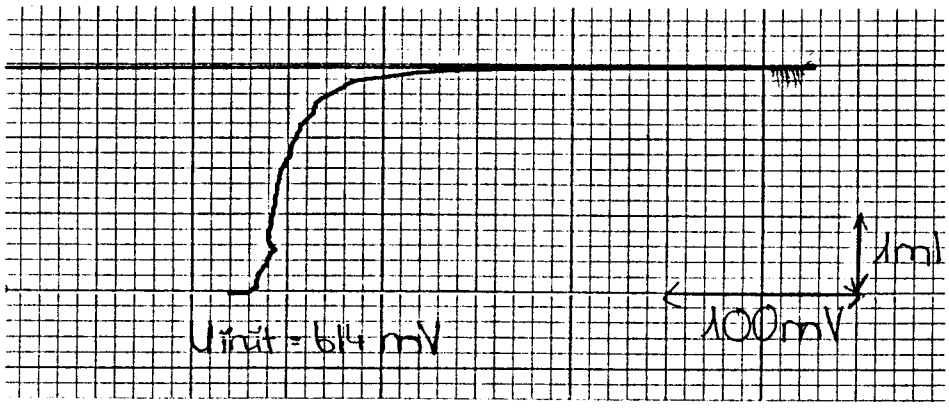
Sample:
2 ml sample
2 ml $c(H_2SO_4)$ = 0.5 mol/l
0.1 g $MnSO_4 \cdot H_2O$ (catalyst)
100 ml H_2O

Electrodes: 6.0415.100 combined massive Pt electrode

```
SET  U                    6-6
parameters
>SET1
  EP at U                  900 mV
  dynamics                 350 mV
  max.rate                   5 ml/min
  min.rate                  10 ul/min
  stop.crit:              time
  t(delay)                  10 s
>SET2
  EP at U                  OFF mV
>titration parameters
  titr.direction:            +
  start V:                 OFF
  pause                      0 s
  meas.input:                1
  temperature             25.0  C
>stop conditions
  stop V:                 abs.
  stop V                     6 ml
  filling rate            max. ml/min
>statistics
  status:                  OFF
>preselections
  conditioning:            OFF
  req.ident:               OFF
  req.smpl size:           OFF
  activate pulse:          OFF
          ------------
```

```
SET  U                         6-6
>calculations
RS1=EP1*C01*C02*C03*C04*C05/C00;2;%
C00=                       1.190
C01=                     0.02791
C02=                         2.5
C03=                       34.02
C04=                         0.1
C05=                          50
              ------------


fr
date  91-08-26       time  11:38        27
U(init)          614 mV  SET U         6-6
smpl size      1.190 g
EP1            3.010 ml              910 mV
RS1            30.02 %
              ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph



Remarks:

- Determination reaction:

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

- Calculations:

RS1 = fraction of H_2O_2 in %
C01 = concentration of tirating agent * titer (0.02781)
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 * factor for % (0.001 * 100 = 0.1)
C05 = dilution factor (50)

References:

Example 6-7

Determination: **KF Water Determination**

Reagent: HYDRANAL Composite 5

Sample: 30 ml Methanol
ca. 0,6 g sample solution

Sample:
ca. 0,5 % water in methanol

Electrodes: 6.0338.000 double Pt electrode, polarized I_{pol} = 25 µA
Voltametric indication

```
 'pa
date  93-02-03      time  15:19       34
SET  Ipol               6-7
parameters
>SET1
  EP at U                250 mV
  dynamics               200 mV
  max.rate                 1 ml/min
  min.rate               0.1 ul/min
  stop.crit:           drift
  stop drift              20 ul/min
>SET2
  EP at U                OFF mV
>titration parameters
  titr.direction:          -
  start V:               OFF
  pause                    0 s
  I(pol)                  25 uA
  electrode test:        OFF
  temperature           25.0  C
>stop conditions
  stop V:               abs.
  stop V                  10 ml
  filling rate            10 ml/min
>statistics
  status:                 ON
  mean              n=     5
  res.tab:          original
>preselections
  conditioning:           ON
  display drift:          ON
  req.ident:             OFF
  req.smpl size:       value
  activate pulse:        OFF
         ------------
```

```
 'fm
date  93-02-03      time  15:12       34
SET  Ipol               6-7
>calculations
RS1=EP1*C30*C01/C00;2;%
C00=                 0.673
C01=                   0.1
C30=                 4.821
         ============
```

```
 'fr
date  93-02-03      time  15:12       34
U(init)          575 mV  SET Ipol     6-7
smpl size      0.673 g
EP1            0.709 ml             215 mV
RS1             0.51 %
              mean( 5)    +/-s         s/%
RS1             0.51    0.000 %      0.00
         ============
```

Analog titration curve, recorded on-line with 586 Metrohm Labograph

Remarks:

- Calculations:

RS1 = content of % H_2O
C01 = factor for % (0,1)
C30 = Titer of KF Reagent (4,821)
C00 = sample size in g

- The parameters "max.rate" and "min.Rate" have to be optimized for a given sample. In the given example, they are set for relatively slow titrations.

References: