

Determination of lead and tin by stripping voltammetry

Of interest to:

General analytical laboratories; Environmental protection; Food analysis, Metals

B 1, 2, 7, 10

Summary

In most electrolytes the peak potentials of lead and tin are so close together, that a voltammetric determination is impossible. Difficulties occur especially if one of the metals is present in excess.

Method 1 describes the determination of Pb and Sn. Differential pulse anodic stripping voltammetry (DPASV) is used under addition of cetyltrimethylammonium bromide. This method is used when:

- one is mainly interested in Pb
- Pb is in excess
- the Sn:Pb ratio is not higher than 200:1

According to method 1, Sn and Pb can be determined simultaneously if the difference in the concentrations is not too high and Cd is absent.

Method 2 is applied when traces of Sn and Pb are found or interfering Tl and/or Cd ions are present. This method also uses DPASV in an oxalate buffer with methylene blue addition.

Method 3 in this bulletin describes the determination of Sn(II) in presence of Sn(IV) by DPASV. Using an electrolyte containing fluoride, Sn(IV) gives no signal, so that a speciation is possible.

Apparatus and accessories

- 746 VA Trace Analyzer with 747 VA Stand or
- 757 VA Computrace
- 705 UV Digester

Sample preparation

Organic matter often interferes with voltammetric determinations and therefore sample solutions usually have to be digested.

- Ground water, surface waters, mineral waters and drinking waters can usually be analysed without pretreatment.
- Low polluted waste waters can be digested with the 705 UV-Digester.
Add 50 μL hydrogen peroxide solution $w(\text{H}_2\text{O}_2) = 30\%$ and 10 μL hydrochloric acid $w(\text{HCl}) = 30\%$ to 10 mL acidified sample ($\text{pH} = 2$) and irradiate for 60 minutes at 90°C .
- Samples with organic matter (foods, pharmaceuticals etc.) must be digested.
 - High-pressure asher
 - Microwave digestion

Both techniques oxidise the sample in a closed digestion vessel by means of a mixture of concentrated mineral acids.

- Open wet digestion with H_2SO_4 and H_2O_2 According to Application Bulletin 113.

Method 1: Determination of lead and tin with cetyltrimethylammonium bromide

Reagents

All of the used reagents must be of purest quality possible (analytical grade or suprapur). Only ultrapure water should be used.

- Hydrochloric acid, suprapur, $w(\text{HCl}) = 30\%$
- Trisodium citrate dihydrat puriss.p.a., CAS 6132-04-3
- Oxalic acid monohydrate puriss.p.a. or suprapur, CAS 6153-56-6
- Cetyl trimethylammonium bromide, CTAB, CAS 57-09-0

- Pb standard stock solution $\beta(\text{Pb}^{2+}) = 1 \text{ g/L}$, commercially available
- Sn standard stock solution $\beta(\text{Sn}^{4+}) = 1 \text{ g/L}$, commercially available

Ready-to-use solutions:

Base electrolyte	c(citrate) = 0.1 mol/L c(oxalic acid) = 0.1 mol/L c(HCl) = 0.2 mol/L pH = 2.5
	14.7 g sodium citrate and 6.3 g oxalic acid are dissolved in ultrapure water. 10.5 mL hydrochloric acid are added. The solution is made up to 500 mL with ultrapure water.
CTAB solution	c(CTAB) = 0.005 mol/L
	0.46 g CTAB are dissolved in 250 mL ultrapure water
Pb standard solution	$\beta(\text{Pb}^{2+}) = 1 \text{ mg/L}$
Sn standard solution	$\beta(\text{Sn}^{4+}) = 1 \text{ mg/L}$
	The solution is diluted with c(HCl) = 0.01 mol/L. It is stable for max. 1 week.

Literature

- Hernandez Mendez J., Carabis Martinez R., Gonzales Lopez M.E.
 Simultaneous determination of tin and lead by AC anodic stripping voltammetry at a hanging mercury drop electrode sensitized by cetyltrimethylammonium bromide
 Anal Chim Acta 138 (1982), 47-54
- Ciszewski A., Lukaszewski Z.
 The influence on long-chain amine and ammonium salts on the anodic stripping voltammetry of thallium, lead, tin, cadmium, and indium
 Anal Chim Acta 146(1983), 51-59

Method 1a: Determination of lead in presence of tin

Principle

In presence of cetyltrimethylammonium bromide it is possible to determine Pb as well as Sn, even when a great excess of Sn is present. The maximum Sn:Pb ratio is approx. 200:1.

The detection limit is 1 µg/L of Pb and with high excess of Sn, 5 µg/L of Pb.

Analyse

5 mL (diluted) sample
 + 5 mL base electrolyte
 + 0.05 mL CTAB solution

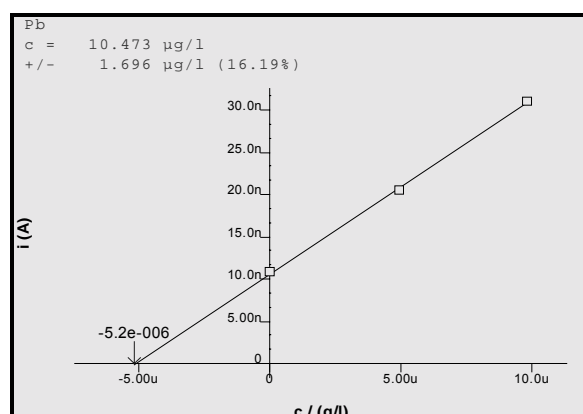
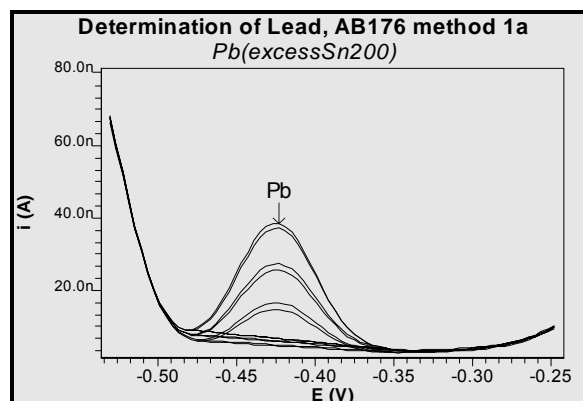
The voltammogram is recorded with the following parameters:

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
drop size	4
Purge time	300 s
Deposition potential	-480 mV
Deposition time	90 s
Equilibration time	20 s
Pulse amplitude	50 mV
Start potential	-530 mV
End potential	-250 mV
Voltage step	4 mV
Voltage step time	0.2 s
Sweep rate	20 mV/s
Peak potential Pb	-420 mV

The concentration is determined by standard addition.

Example:

Determination of Pb in presence of Sn (200 fold excess)



Sample Volume 5 mL
 Results 10.5 µg/L Pb

Method 1b: Determination of tin and lead simultaneously

Principle

In presence of Cetyltrimethylammonium bromide it is possible to determine Pb and Sn simultaneously. The maximum Pb:Sn ratio is appr. 50:1. The simultaneous determination of tin and lead is only possible if Cd is absent.

The detection limit is 1 µg/L of Pb. The detection limit for Sn is 2 µg/L on the 746 and 10 µg/L on the 757.

Analyse

5 mL (diluted) sample
 + 5 mL base electrolyte
 + 0.05 mL CTAB solution

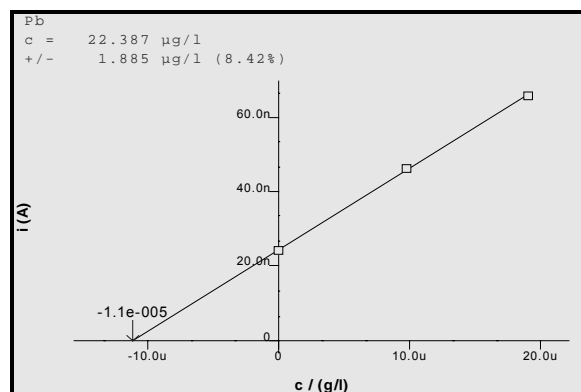
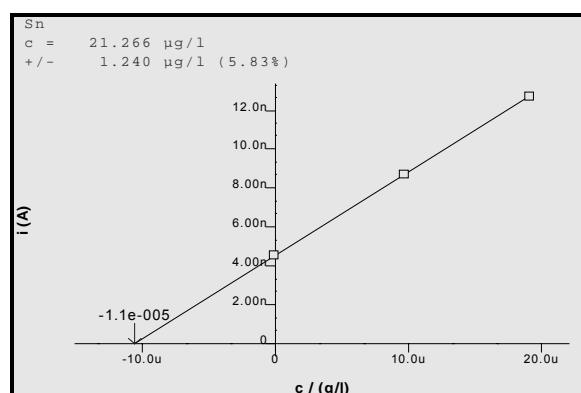
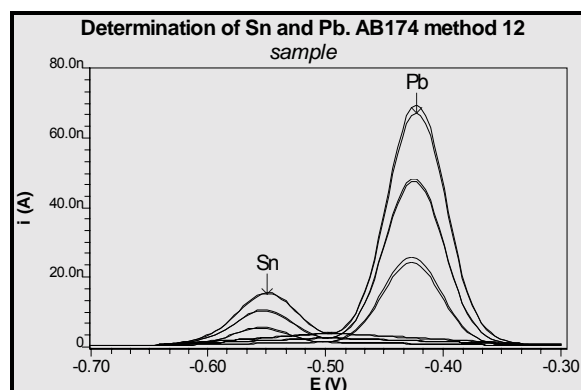
The voltammogram is recorded with the following parameters:

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
drop size	4
Purge time	300 s
Deposition potential	-700 mV
Deposition time	90 s
Equilibration time	20 s
Pulse amplitude	50 mV
Start potential	-800 mV
End potential	-300 mV
Voltage step	4 mV
Voltage step time	0.2 s
Sweep rate	20 mV/s
Peak potential (Sn)	-550 mV
Peak potential (Pb)	-420 mV

The concentration is determined by standard addition.

Example:

Determination of Pb and Sn simultaneously



Sample Volume 5 mL
 Results 21.3 µg/L Sn
 22.4 µg/L Pb

Appendix

Full Report of a determination of Pb in presence of Sn (200 fold excess) acc. to method 1a with the 757 VA Computrace

```

===== METROHM 757 VA COMPUTRACE (5.757.0010) =====
Determ.   : 06161543_Pb(excessSn200).dth
Date      : 1999-06-16      Time: 14:43:09
Modified  : 2000-12-05 11:37:32  User:
Cell volume: 10.050 ml
-----
Ident     : Pb(excessSn200)
Sample volume: 5.000 ml
-----
Method   : AB176_1.mth
Title    : Determination of Pb. AB176 method 1a
Remark1  : 5ml sample + 5ml electrolyte + 50µl cetyltrimethylammoniumbromide
Remark2  : el.: 0.1mol/l trisodiumcitrate + 0.1mol/l oxalic acid + 0.2mol/l HCl
-----
Substance : Pb
Mass conc.: 5.211 ug/l
MC.dev.   : 0.844 ug/l ( 16.19%)
Mass      : 52.366 ng
Add.mass  : 50.000 ng
-----
          VR      V      nA      i.mean  Std.Dev.  i.delta  Comments
-----
          1-1    -0.423  11.57  10.80   1.096
          1-2    -0.423  10.02
          2-1    -0.423  21.18  20.44   1.040   9.64
          2-2    -0.423  19.71
          3-1    -0.423  30.37  31.01   0.907   10.57
          3-2    -0.423  31.66
-----
Substance  Calibr.      Y.reg/offset  Slope      Nonlin.      Mean deviat.
-----
          Pb      std.add.      1.060e-008  2.058e-003  -----      9.879e-010
-----
Final results
-----
          Pb =      10.473 µg/l      1.696      16.190
-----

```

Method parameters of a determination of Pb in presence of Sn (200 fold excess) acc. to method 1a with the 746 VA Trace Analyzer

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB176_1a.mth      OPERATION SEQUENCE
Title : Det.of Pb beneath Sn with CTAB. AB176 part 1a
-----
Instructions  t/s  Main parameters      Auxiliary parameters
-----
1  DOS/M      -----
2  REM        V.added      5.050 mL
3  SMPL/M     5mL sample + 5 mL electrolyte + 50 µL meth. blue
4  PURGE      V.fraction   mL      V.total   L
5  STIR       300.0      Rot.speed      2000 /min
6  (ADD
7  PURGE
8  STIR       30.0      Rot.speed      2000 /min
9  OPURGE
10 (REP
11 SEGMENT   Segm.name   asv
12 REP)1
13 PURGE
14 STIR      Rot.speed   2000 /min
15 ADD>M    Soln.name   Pb-std      V.add      0.100 mL
16 ADD)2
17 END
-----
Method: AB176_1a      SEGMENT
                        asv
-----
Instructions  t/s  Main parameters      Auxiliary parameters
-----
1  STIR       5.0      Rot.speed      2000 /min
2  HMDE      Drop size   4
3  DPMODE     U.ampl     50 mV      Meas.cell   normal
                t.step     0.20 s     t.meas     20.0 ms
                U.meas     -480 mV    t.pulse    40.0 ms
4  MEAS      90.0
5  OSTIR     20.0
6  SWEEP     14.8     U.start      -530 mV     U.step      4 mV
                U.end      -250 mV    Sweep rate  20 mV/s
                U.standby  mV
7  OMEAS
8  END
-----
Method: AB176_1a      CALCULATION
                        max. 15 lines
-----
Quantity      Formula (R##, C##, A##)      Res.unit      Sig.dig.
-----
Pb            R1000=MC:Pb                  #g/L          5
-----

```

Full Report of a determination of Pb and Sn simultaneously acc. to method 1b with the 757 VA Computrace

```

===== METROHM 757 VA COMPUTRACE (5.757.0010) =====
Determ.      : 06151514_Sn_Pb.dth
Date         : 1999-06-15           Time: 14:14:57
Modified    : 2000-12-05 13:48:06   User:
Cell volume  : 10.050 ml

-----
Ident sample                               Sample volume
                                         5.000 ml
-----
Method      : AB176_1.mth
Title       : Determination of Sn and Pb. AB174 method 1a
Remark1     : 5ml sample + 5ml electrolyte + 50µl cetyltrimethylammoniumbromide
Remark2     : el.: 0.1mol/l trisodiumcitrate + 0.1mol/l oxalic acid + 0.2mol/l HCl
-----
Substance   : Sn
Mass conc.  : 10.580 ug/l
MC.dev.     : 0.617 ug/l           ( 5.83%)
Mass        : 106.331 ng
Add.mass    : 100.000 ng
-----
          VR      V      nA      i.mean  Std.Dev.  i.delta  Comments
          -----
1-1      -0.554   4.42   4.52    0.145
1-2      -0.554   4.62   8.71    0.167   4.19
2-1      -0.554   8.59   12.71   0.184   4.00
2-2      -0.554   8.83
3-1      -0.550  12.84
3-2      -0.550  12.58
-----
Substance   : Pb
Mass conc.  : 11.138 ug/l
MC.dev.     : 0.938 ug/l           ( 8.42%)
Mass        : 111.936 ng
Add.mass    : 100.000 ng
-----
          VR      V      nA      i.mean  Std.Dev.  i.delta  Comments
          -----
1-1      -0.427  23.24  24.03   1.126
1-2      -0.427  24.83
2-1      -0.423  45.69  46.13   0.615  22.09
2-2      -0.423  46.56
3-1      -0.423  66.87  65.76   1.564  19.64
3-2      -0.423  64.66
-----
Substance   Calibr.      Y.reg/offset      Slope      Nonlin.      Mean deviat.
-----
Sn          std.add.      4.523e-009      4.282e-004
Pb          std.add.      2.442e-008      2.201e-003
                                         1.360e-010
                                         9.876e-010
-----
Final results                                     +/- Res. dev.  %      Comments
-----
Sn =        21.266 µg/l                          1.240      5.829
Pb =        22.387 µg/l                          1.885      8.419

```

Method parameters of a determination of Pb and Sn simultaneously acc. to method 1b with the 746 VA Trace Analyzer

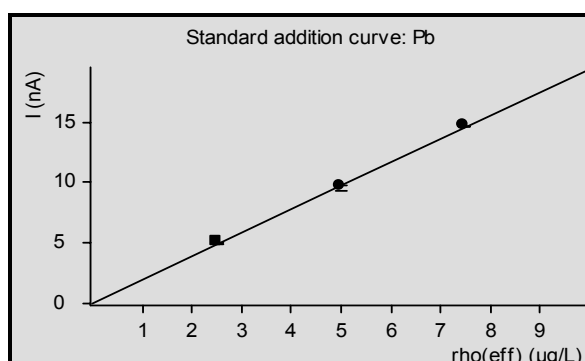
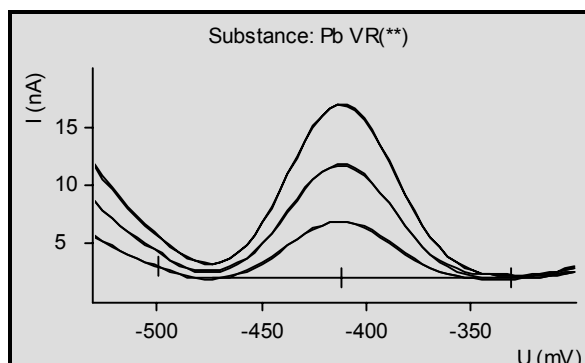
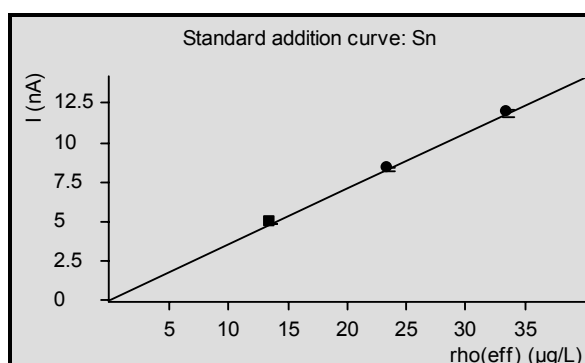
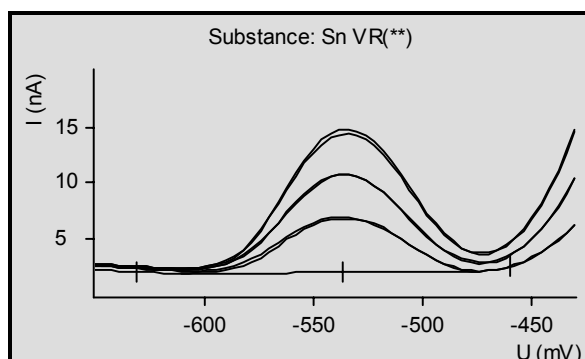
```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB176_lb.mth           OPERATION SEQUENCE
Title : Det.of Sn and Pb with CTAB. AB176 part 1b
-----
Instructions  t/s      Main parameters      Auxiliary parameters
-----
1  DOS/M
2  REM        5mL sample + 5 mL electrolyte + 50 µL meth. blue
3  SMPL/M     V.fraction           mL      V.total
4  PURGE
5  STIR       300.0      Rot.speed           2000 /min
6  (ADD
7  PURGE
8  STIR       30.0      Rot.speed           2000 /min
9  OPURGE
10 (REP
11 SEGMENT    Segm.name      asvSn
12 REP)1
13 PURGE
14 STIR       Rot.speed      2000 /min
15 ADD>M     Soln.name      Pb-std           V.add            0.100 mL
16 ADD>M     Soln.name      Sn-std           V.add            0.100 mL
17 ADD)2
18 END
-----
Method: AB176_lb           SEGMENT
                           asvSn
-----
Instructions  t/s      Main parameters      Auxiliary parameters
-----
1  STIR       5.0      Rot.speed           2000 /min
2  HMDE      Drop size      4
3  DPMODE     U.ampl         50 mV
                           t.step         0.20 s
                           U.meas         -700 mV
4  MEAS      90.0
                           Meas.cell      normal
                           t.meas         20.0 ms
                           t.pulse        40.0 ms

```


Example

Determination of Pb and Sn



Sample Volume 5 mL
 Results 27.4 µg/L Sn
 5.1 µg/L Pb

Remarks

- If the tin excess is great, one must work with two segments, intermediate electrolysis (intermediate electrolysis potential approx. -540 mV) and perhaps two standard addition loops.
- If the sample contains Tl, the Pb peak can be adjusted to more positive values by raising the pH value to 2.4 (addition of ammonia solution w(NH₃) = 25%). One must work fast because at this pH value tin already hydrolyses.

peak potentials:

Pb	-370 mV
Tl	-410 mV
Sn	-540 mV

A good separation under these conditions can still be performed when the ratio Sn:Tl lies at 1:2. Lead cannot be determined.

- If the sample contains Cd, the pH value can be lowered to 1.6 with hydrochloric acid (w(HCl) = 30%). The Sn peak adjusts then to more positive values improving the separation between Cd and Sn. The Pb and the Tl concentration should, however, not be too high.

peak potentials:

Pb	-400 mV
Sn	-500 mV
Cd	-600 mV

For separation, it is better to perform an intermediate electrolysis (intermediate electrolysis potential approx. -580 mV). An excess Cd:Pb of 50:1 does not show any interference.

Appendix

Full Report of a determination of Pb and Sn acc. to method 2 with the 746 VA Trace Analyzer

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Determ.      : 03111419          User:                Date: 1999-03-11
Modified     : 2000-12-05 19:30:39 Run : 0                 Time: 14:19:11
Sample table: -
-----
  Pos.  Ident.1/S1  Ident.2/S2  Ident.3/S3  Method.call  Sample size/S0
        sample
-----
Method   : AB176_2
Title    : Det.of Sn and Pb with methylene blue. AB176 part 2
Remark1  : 5 ml sample + 5 ml electrolyte + 50 µl methylene blue (1g/l)
Remark2  : el.: 0.14mol/l oxalate + 0.17mol/l NH4Cl + 0.15mol/l HCl
-----
Substance : Sn
Mass conc.: 27.41 ug/L          Mass      : 137.1 ng
MC.dev.   : 0.904 ug/L (3.3%)  Add.mass  : 100 ng
Cal.dev.  : -                  V0.sample: 5 mL
-----
      VR  U/mV  I/nA  I.mean  Std.dev.  I.delta  Comments
-----
      00 -537  4.808  4.824  0.0222
      01 -537  4.839
      10 -536  8.227  8.166  0.0871  3.342
      11 -536  8.104
      20 -536  11.41  11.55  0.1883  3.381
      21 -536  11.68
-----
Substance : Pb
Mass conc.: 5.068 ug/L          Mass      : 25.34 ng
MC.dev.   : 0.228 ug/L (4.51%) Add.mass  : 25 ng
Cal.dev.  : -                  V0.sample: 5 mL
-----
      VR  U/mV  I/nA  I.mean  Std.dev.  I.delta  Comments
-----
      00 -412  4.895  4.965  0.0987
      01 -413  5.034
      10 -412  9.579  9.435  0.2038  4.470
      11 -412  9.291
      20 -412  14.24  14.26  0.0323  4.823
      21 -412  14.28
-----
Substance  Techn.  Y.reg/offset  Slope  Nonlin.  Mean deviat.
-----
Sn         std.add.  4.804e-09    3.522e-04
Pb         std.add.  4.888e-09    0.001938
-----
Final results          +/- Res.dev.  %  Comments
-----
Sn = 27.414 ug/L      0.904  3.30
Pb = 5.0681 ug/L     0.228  4.51

```

Method parameters of a determination of Pb and Sn acc. to method 2 with the 746 VA Trace Analyzer

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB176_2 .mth          OPERATION SEQUENCE
Title : Det.of Sn and Pb with methylene blue. AB176 part 2
-----
Instructions  t/s  Main parameters  Auxiliary parameters
-----
1  DOS/M
2  REM          V.added 5.050 mL
3  SMPL/M      5mL sample + 5 mL electrolyte + 50 µL meth. blue
4  PURGE       V.fraction mL  V.total L
5  STIR        300.0  Rot.speed 2000 /min
6  (ADD
7  PURGE
8  STIR        30.0  Rot.speed 2000 /min
9  OPURGE
10 (REP
11 SEGMENT    Segm.name asvSn
12 REP)1
13 PURGE
14 STIR       Rot.speed 2000 /min
15 ADD>M     Soln.name Pb-std  V.add 0.025 mL
16 ADD>M     Soln.name Sn-std  V.add 0.100 mL
17 ADD)2
18 END

```

Method: AB176_2		SEGMENT asvSn		
Instructions	t/s	Main parameters		Auxiliary parameters
1 STIR	2.0	Rot.speed	2000 /min	
2 HMDE		Drop size	4	Meas.cell normal
3 DPMODE		U.ampl	50 mV	t.meas 20.0 ms
		t.step	0.20 s	t.pulse 40.0 ms
4 MEAS	90.0	U.meas	-800 mV	
5 MEAS	20.0	U.meas	-580 mV	
6 OSTIR	10.0			
7 SWEEP	28.2	U.start	-800 mV	U.step 4 mV
		U.end	-250 mV	Sweep rate 20 mV/s
8 OMEAS		U.standby	mV	
9 END				

Method: AB176_2		CALCULATION max. 15 lines	
Quantity	Formula (R##, C##, A##)	Res.unit	Sig.dig.
Sn	R1000=MC:Sn	#g/L	5
Pb	R1001=MC:Pb	#g/L	5

Method 3: Determination of Sn(II) besides Sn(IV) in an electrolyte containing NaF

Principle

In weak alkaline solutions, containing sodium fluoride it is possible to determine Sn(II) besides Sn(IV) by DPASV. The Sn(IV) ions do not give any voltammetric signal under these conditions. The detection limit is 2.5 µg/L.

Accessories

- 6.1450.210 Measuring vessel of PFA

Reagents

All of the used reagents must be of purest quality possible (analytical grade or suprapur). Only ultrapure water should be used.

- Piperazine-1,4-bis(2-ethane sulfonic acid), PIPES, CAS 5625-37-6
- Sodium hydroxide solution, suprapur, w(NaOH) = 30%
- Ammonia solution, suprapur, w(NH₃) = 25%
- Hydrochloric acid, suprapur, w(HCl) = 30%
- NaNO₃, suprapur, CAS 7631-99-4
- NaF suprapur, CAS 7681-49-4
- SnCl₂ dihydrate, p.a., CAS 10025-69-1

Ready-to-use solutions:

PIPES buffer	c(PIPES) = 1 mol/L, pH 8
	7.6 g PIPES are mixed with 1 mL sodium hydroxide solution and 5 mL high purity water. The pH is adjusted to 8 ± 0.1 with ammonia solution. The solution is filled up to 25 mL.

NaNO ₃ solution	c(NaNO ₃) = 1 mol/L
	4.25 g NaNO ₃ are dissolved in 50 mL ultrapure water.
NaF solution	c(NaF) = 1 mol/L
	2.1 g NaF are dissolved in 50 mL ultrapure water.
Sn standard stock solution	c(Sn ²⁺) = 1 g/L
	0.190 g SnCl ₂ are dissolved in 50 mL oxygen free water. 10 mL hydrochloric acid are added and the solution is made up to 100 mL using oxygen free water. The solution is sensitive against oxidation and should be prepared freshly in regular intervals.
Sn(II) standard solution	c(Sn ²⁺) = 1 mg/L
	The diluted working solutions are prepared from standard stock solutions by dilution in c(HCl) = 0.01 mol/L. Oxygen free water has to be used.

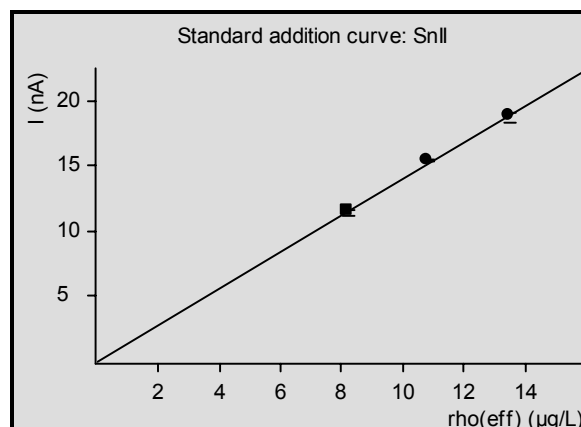
Analysis

- 5 mL sample
- + 0.5 mL NaNO₃ solution
- + 3.5 mL NaF solution
- + 0.5 mL PIPES buffer

The pH is adjusted to 8.0 with ammonia solution.

The voltammogram is recorded with the following parameters:

Working electrode	HMDE
Stirrer speed	2000 rpm
Mode	DP
drop size	4
Purge time	300 s
Deposition potential	-800 mV
Deposition time	90 s
Equilibration time	10 s
Pulse amplitude	50 mV
Start potential	-800 mV
End potential	-500 mV
Voltage step	4 mV
Voltage step time	0.1 s
Sweep rate	40 mV/s
Peak potential (Sn)	-640 mV



Sample Volume 5 mL
 Results 15.6 µg/L Sn(II)

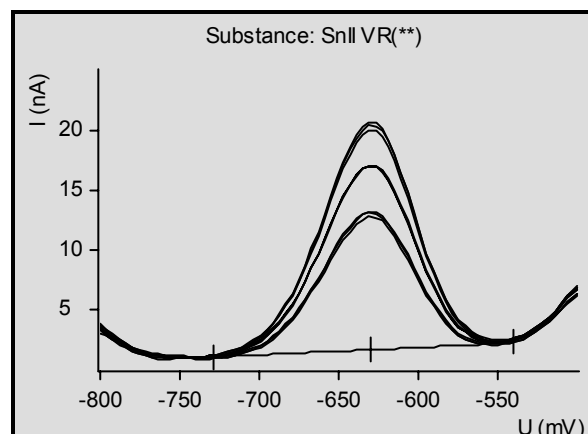
Literature

- Lejeune,R. Thunus,J. Thunus,L.
 Polarographic determination of Sn(II) in samples containing Sn(IV) such as in 99m-technitium radiopharmaceutical kits
 Anal. Chim. Acta 332, (1996) 67-71

The concentration is determined by standard addition.

Example

Determination of Sn(II)



Appendix

Full Report of a determination of Sn(II) acc. to method 3 with the 746 VA Trace Analyzer

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Determ.      : 09111121          User:                Date: 1997-09-11
Modified     : 2000-12-08 16:52:16  Run : 0            Time: 11:21:01
Sample table: -
-----
Pos.  Ident.1/S1  Ident.2/S2  Ident.3/S3  Method.call  Sample size/S0
-----
      sample SnII
-----
Method  : AB176_3
Title   : Det. of Sn(II) besides Sn(IV). AB176 method 3
Remark1 :
Remark2 :
-----
Substance : SnII
Mass conc.: 15.64 ug/L          Mass      : 78.21 ng
MC.dev.   : 0.632 ug/L (4.04%)  Add.mass  : 25 ng
Cal.dev.  : -                  V0.sample: 5 mL
-----
      VR  U/mV  I/nA  I.mean  Std.dev.  I.delta  Comments
      ---  ---  ---  ---  ---  ---  -----
      00  -630  11.50  11.40  0.1664
      01  -629  11.50
      02  -629  11.21
    
```

	10	-629	15.37	15.32	0.0630	3.922
	11	-629	15.34			
	12	-629	15.25			
	20	-629	18.72	18.60	0.3264	3.278
	21	-629	18.85			
	22	-629	18.23			
Substance	Techn.	Y.reg/offset	Slope	Nonlin.	Mean deviat.	
SnII	std.add.	1.149e-08	0.001396		2.492e-10	
C#	Workg.com.var	Remark				
Final results			+/- Res.dev.	%	Comments	
	SnII =	15.641 ug/L	0.632	4.04		

Method parameters of a determination of Sn(II) acc. to method 3 with the 746 VA Trace Analyzer

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB176_3 .mth OPERATION SEQUENCE
Title : Det. of Sn(II) besides Sn(IV). AB176 method 3
  
```

Instructions	t/s	Main parameters	Auxiliary parameters
1 DOS>M		Soln.name NaNO3	V.add 0.500 mL
2 DOS>M		Soln.name NaF	V.add 3.500 mL
3 DOS>M		Soln.name PIPES	V.add 0.500 mL
4 SMPL/M		V.fraction mL	V.total L
5 PURGE			
6 STIR	300.0	Rot.speed 2000 /min	
7 (ADD			
8 PURGE			
9 STIR	30.0	Rot.speed 2000 /min	
10 (PURGE			
11 (REP			
12 SEGMENT		Segm.name Tin	
13 REP)2			
14 ADD>M		Soln.name SnIIstd	V.add 0.025 mL
15 ADD)2			
16 END			

```

Method: AB176_3 SEGMENT
Tin
  
```

Instructions	t/s	Main parameters	Auxiliary parameters
1 STIR	5.0	Rot.speed 2000 /min	
2 HMDE		Drop size 7	Meas.cell normal
3 DPMODE		U.ampl 50 mV	t.meas 20.0 ms
		t.step 0.10 s	t.pulse 40.0 ms
4 MEAS	90.0	U.meas -800 mV	
5 OSTIR	10.0		
6 SWEEP	7.8	U.start -800 mV	U.step 4 mV
		U.end -500 mV	Sweep rate 40 mV/s
7 OMEAS		U.standby mV	
8 END			

```

Method: AB176_3 CALCULATION
max. 15 lines
  
```

Quantity	Formula (R##, C##, A##)	Res.unit	Sig.dig.
SnII	R1000=MC:SnII	#g/L	5