

Application Bulletin

Of interest to: General analytical laboratories
Organic chemistry; Plastics

B 1, 3, 6

Polarographic determination of 4-carboxybenzaldehyde in terephthalic acid

Summary

4-Carboxybenzaldehyde, in the following referred to as 4-CBA, can be reduced directly on the dropping mercury electrode (DME) in an ammoniacal solution. After a very simple sample preparation, it is thus possible to determine the concentration of 4-CBA in terephthalic acid quickly and precisely by polarography down to the lower ppm range.

Instruments and accessories

- 746 VA Trace Analyzer with 747 VA Stand or
- 757 VA Computrace

Reagents

All reagents used should have the highest possible degree of purity (p.a. or suprapur). Only ultrapure water should be used.

- Ammonia solution, $w(\text{NH}_3) = 25\%$
- Hydrochloric acid, $w(\text{HCl}) = 32\%$
- 4-Carboxybenzaldehyde 4-CBA, puriss., CAS 619-66-9
- Sodium hydroxide solution, $w(\text{NaOH}) = 32\%$

Ready-to-use solutions

- Ammonium buffer (pH = 9.6): $c(\text{NH}_4\text{Cl}) = 1 \text{ mol/L}$, $c(\text{NH}_3) = 2 \text{ mol/L}$:
Make up 112 mL $w(\text{NH}_3) = 25\%$ and 49 mL $w(\text{HCl}) = 32\%$ to 500 mL with ultrapure water.
- 4-Carboxybenzaldehyde standard solution, $\beta(4\text{-CBA}) = 1 \text{ g/L}$:
Dissolve 100 mg 4-CBA in 10 mL ammonium buffer and make up to 100 mL with ultrapure water.

Sample preparation

Weigh 5 g sample into a beaker and mix to a slurry with 40 mL ultrapure water. While adding 15 mL $w(\text{NH}_3) = 25\%$, heat up the solution. If the sample does not dissolve completely, add $w(\text{NaOH}) = 32\%$ (approx. 1 mL) until a clear solution is obtained.

After cooling down, transfer the solution to a 100 mL volumetric flask, add 3 mL ammonium buffer and fill to the mark with ultrapure water.

Analysis

Pipet 10 mL sample solution into the polarographic vessel and record the polarogram using the following parameters:

working electrode	DME
stirrer speed	2000 rpm
mode	DP
purge time	300 s
equilibration time	10 s
pulse amplitude	50 mV
start potential	-1050 mV
end potential	-1350 mV
voltage step	6 mV
voltage step time	0.4 s
sweep rate	15 mV/s
peak potential 4-CBA	-1200 mV

The concentration is determined by standard addition.

Figures

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===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Method: AB190 .mth OPERATION SEQUENCE
Title : 4-Carboxybenzaldehyde in terephthalic acid
-----
Instructions  t/s  Main parameters  Auxiliary parameters
-----
1  SMPL>M  V.fraction  10.000 mL  V.total  0.1 L
2  PURGE
3  STIR  300.0  Rot.speed  2000 /min
4  (ADD
5  PURGE
6  STIR  30.0  Rot.speed  2000 /min
7  OSTIR
8  OPURGE  10.0
9  (REP
10 SEGMENT  Segm.name  pol
11 REP)1
12 ADD>M  Soln.name  4CBA-std  V.add  0.010 mL
13 ADD)2
14 END

Method: AB190 SEGMENT
                pol
-----
Instructions  t/s  Main parameters  Auxiliary parameters
-----
1  DME
2  DPMODE  U.ampl  -50 mV  t.meas  20.0 ms
           t.step  0.40 s  t.pulse  40.0 ms
3  SWEEP  21.6  U.start  -1050 mV  U.step  6 mV
           U.end  -1350 mV  Sweep rate  15 mV/s
4  OMEAS  U.standby  mV
5  END
    
```

Fig. 1: Method for the determination of 4-carboxybenzaldehyde with the 746 VA Trace Analyzer.

```

===== METROHM 746 VA TRACE ANALYZER (5.746.0101) =====
Determ.      : 10210847      User: zu      Date: 1999-10-21
Modified     : no           Run : 4             Time: 08:47:18
Sample table: -
    
```

```

-----
Pos.  Ident.1/S1  Ident.2/S2  Ident.3/S3  Method.call  Sample size/S0
-----
      Terephthals      1.0
-----
    
```

```

Method : AB190
Title  : 4-Carboxybenzaldehyde in terephthalic acid
Remark1 : 10ml dissolved sample
Remark2 : sample preparation according to AB190
-----
    
```

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Substance : 4CBA
Mass conc.: 486.8 ug/L      Mass      : 4.868 ug
MC.dev.   : 18.4 ug/L (3.78%)  Add.mass : 10 ug
Cal.dev.  : -              V0.sample: 10 mL
-----
    
```

VR	U/mV	I/nA	I.mean	Std.dev.	I.delta	Comments
00	-1185	-17.39	-17.40	0.0196		
01	-1184	-17.41				
10	-1184	-53.72	-54.40	0.9621	-37.00	
11	-1184	-55.08				
20	-1183	-89.49	-89.77	0.3900	-35.36	
21	-1184	-90.04				

```

Substance  Techn.  Y.reg/offset  Slope  Nonlin.  Mean deviat.
-----
4CBA      std.add.  -1.766e-08  -3.628e-05  -----  6.894e-10
    
```

```

C#  Workg.com.var  Remark
-----
    
```

```

Final results      +/-  Res.dev.  %  Comments
-----
4CBA = 9.7367 ug/g      0.368  3.78
    
```

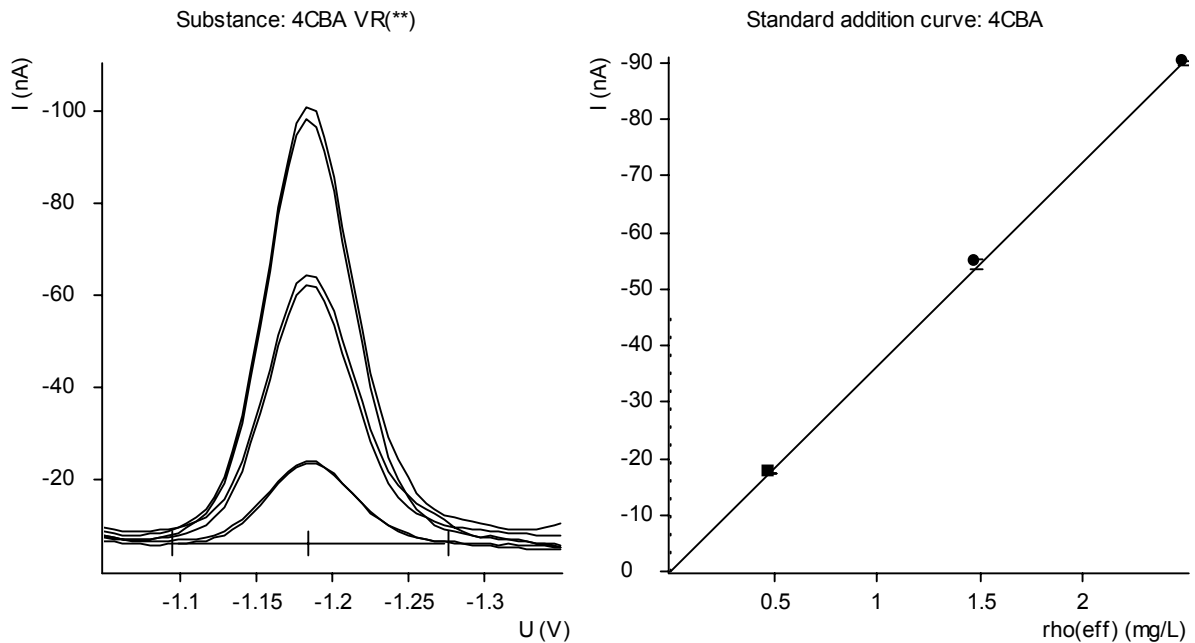


Fig. 2: Determination of 4-carboxybenzaldehyde in terephthalic acid with the 746 VA Trace Analyzer.