# Application Bulletin

| Of interest to: | Tobacco industry; Pharmaceutical industry | B 1, 3, 4, 7 |
|---|---|---|

## Polarographic determination of nicotine

### *Summary*

The quantitative determination of the alkaloid nicotine, which is an essential constituent of the tobacco plant, can be carried out by polarography. The limit of quantitation is less than 0.1 mg/L in the polarographic vessel.

### *Instruments and accessories*

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

### *Reagents*

All reagents used should be of the highest purity (p.a. or «suprapur»). Only ultrapure water should be used.

- Acetic acid, w($CH_3COOH$) = 96%
- Phosphoric acid, w($H_3PO_4$) = 85%, puriss. p.a.
- Boric acid, puris.s p.a.
- Sodium hydroxide solution, c(NaOH) = 0.2 mol/L
- Sodium hydroxide solution, c(NaOH) = 2 mol/L
- (±)-Nicotine, CAS 22083-74-5

***Ready-to-use solutions***

- Supporting electrolyte: ***Britton-Robinson buffer pH = 6.0:***
  c($CH_3COOH$) = 0.4 mol/L
  c($H_3PO_4$) = 0.4 mol/L
  c($H_3BO_3$) = 0.4 mol/L
  c(NaOH) = 1 mol/L
  Dissolve 2.5 g boric acid in 50 mL c(NaOH) = 2 mol/L. After addition of 4.6 g (2.7 mL) w($H_3PO_4$) = 85% and 2.5 g (2.35 mL) w($CH_3COOH$) = 96% make up to 100 mL with water.
- Nicotine standard solution: ρ(nicotine) = 1 g/L in water

## Sample preparation

### Determination of nicotine in tobacco

Place 1 g tobacco in a 100 mL volumetric flask, add 50 mL dist. water and 1 mL c(NaOH) = 2 mol/L, mix now and again and allow to stand for at least 12 h. After this time, fill to the mark with dist. water and mix well, then filter the mixture.

### Determination of nicotine in tobacco smoke

The tobacco smoke is passed through a glass frit (G1) and absorbed in 50 mL supporting electrolyte.

## Analysis

10 mL supporting electrolyte + 100 µL … 1 mL filtrate (dilute the sample solution if necessary)
or
10 mL absorption solution

The polarogram is recorded using the following parameters:

| | |
|---|---|
| working electrode | DME |
| stirrer speed | 2000 rpm |
| mode | DP |
| purge time | 300 s |
| equilibration time | 30 s |
| pulse amplitude | 50 mV |
| start potential | –1000 mV |
| end potential | –1500 mV |
| voltage step | 6 mV |
| pulse time | 40 ms |
| voltage step time | 0.6 s |
| sweep rate | 10 mV/s |
| peak potential | –1280 mV |

The concentration is determined by standard addition.

## Remarks

- Since nicotine is sensitive to light, it is recommended to store the standard solution in an opaque vessel and renew them frequently.
- Tests have been made to determine nicotine with supporting electrolytes of other pH values. However, with lower pH values the proportional relationship between concentration and peak height is lost and in the alkaline region the sensitivity of the determination is greatly reduced.
- The linear working range lies between 0.1 mg/L and 10 mg/L nicotine in the measuring vessel. Higher concentrations must be diluted.

## Literature

- Krjukowa, Sijakowa, Arefjewa
  Polarographische Analyse
  VEB Deutscher Verlag für Grundstoffindustrie, Leipzig, 1964.

## *Figures*

```
================== METROHM 746 VA TRACE ANALYZER (5.746.0101) ==================
 Method: AB057   .mth         OPERATION SEQUENCE
 Title : Determination of nicotine with DME. AB 57
--------------------------------------------------------------------------------

      Instructions   t/s   Main parameters            Auxiliary parameters
      ------------ -----  ------------------------  ------------------------
 1    DOS/M                V.added      10.000 mL
 2    REM                  10 mL buffer
 3    PURGE
 4    STIR          300.0  Rot.speed      2000 /min
 5    0PURGE
 6    SMPL>M               V.fraction    1.000 mL     V.total          0.1 L
 7    (ADD
 8      PURGE
 9      STIR         60.0   Rot.speed      2000 /min
10      SEGMENT             Segm.name   pol
11      ADD>M               Soln.name   nic-Std        V.add         0.020 mL
12    ADD)2
13    END

 Method: AB057                SEGMENT
                                   pol
--------------------------------------------------------------------------------

      Instructions   t/s   Main parameters            Auxiliary parameters
      ------------ -----  ------------------------  ------------------------
 1    (REP
 2      0STIR
 3      0PURGE        5.0
 4      DME
 5      DPMODE              U.ampl          -50 mV     t.meas         20.0 ms
                            t.step         0.60 s      t.pulse        40.0 ms
 6      SWEEP        52.2   U.start       -1000 mV     U.step            6 mV
                            U.end         -1500 mV     Sweep rate       10 mV/s
 7      STIR          2.0   Rot.speed      2000 /min
 8      0MEAS               U.standby           mV
 9    REP)1
10    END
```

***Fig. 1:*** *Method for the determination of nicotine with the 746 VA Trace Analyzer.*

```
================== METROHM 746 VA TRACE ANALYZER (5.746.0101) ==================
 Determ.     : 06100915             User:                      Date: 1999-06-10
 Modified    : no                   Run :  0                   Time: 09:15:24
 Sample table: -
--------------------------------------------------------------------------------
  Pos.  Ident.1/S1    Ident.2/S2    Ident.3/S3    Method.call   Sample size/S0
        nicotine            1.0                                          1 g
--------------------------------------------------------------------------------
 Method  :  AB057
 Title   :  Determination of nicotine with DME. AB 57
 Remark1 :  Determination of nicotine
 Remark2 :
--------------------------------------------------------------------------------

  Substance : nicotine                                          Comments
  Mass conc.:    3.769 mg/L            Mass     : 3.769 ug     ----------
  MC.dev.   :    0.120 mg/L (3.18%)    Add.mass :     2 ug
  Cal.dev.  :    -                     V0.sample:     1 mL

              VR   U/mV    I/nA   I.mean  Std.dev. I.delta   Comments
              ---  -----  ------  ------  -------  -------   ----------
              00   -1276  -32.12  -32.54   0.5921
              01   -1276  -32.96
              10   -1272  -50.87  -50.79   0.1133   -18.25
              11   -1273  -50.71
              20   -1270  -67.08  -67.15   0.0893   -16.36
              21   -1270  -67.21

 Substance    Techn.     Y.reg/offset   Slope        Nonlin.      Mean deviat.
 ---------    --------   ------------   ----------   ----------   ------------
 nicotine     std.add.     -3.284e-08   -9.585e-05                 6.101e-10

 Final results                        +/- Res.dev.   %      Comments
 ---------------------------------    -----------------     ----------
   nicotine =   376.93 ug/g               12.0    3.18
```

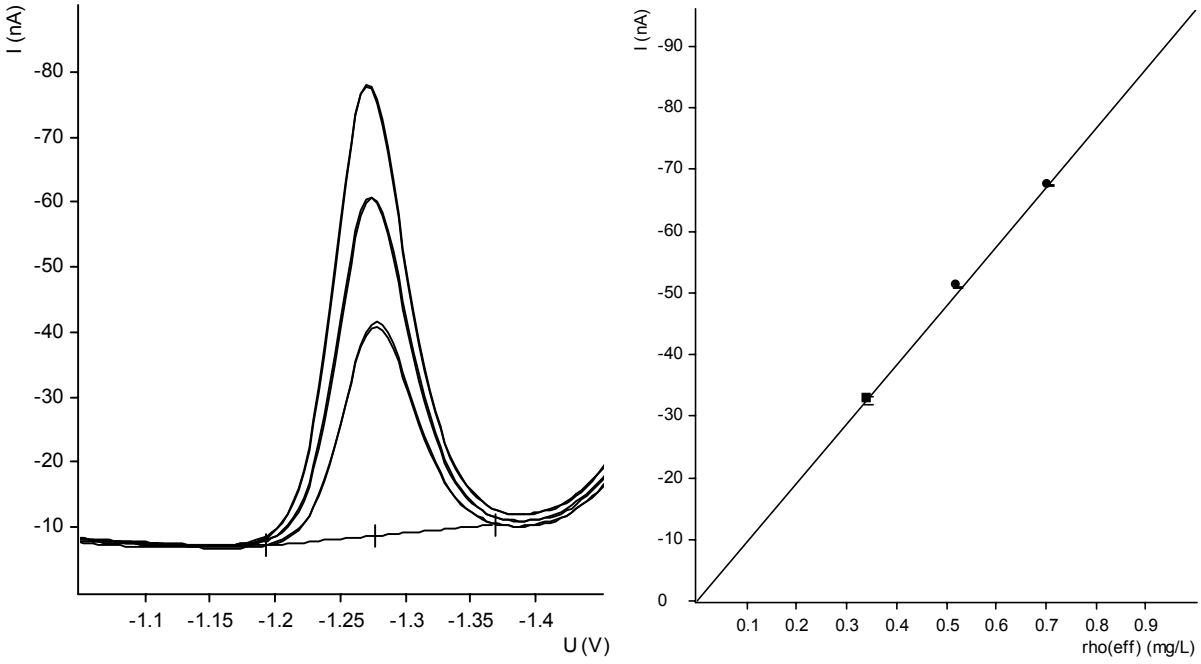


***Fig. 2:*** *Example of a determination of nicotine with the 746 VA Trace Analyzer.*