
Application Bulletin

Of interest to: Pasta production, food

AF 7

Titrimetric methods for the chemical analysis of pasta

Summary

The quality of egg-based pasta is above all determined by its egg content. Also of importance, however, is the water content, which influences the storage life of the product, as well as the degree of acidity, which, in the case of high values, indicates undesirable acidification during processing or drying. A check of the chloride content shows whether salt has been added to the pasta.

Sample preparation

The sample is ground finely in a laboratory mill (if possible under cooling). Cooling is particularly important for the determination of the water content, because if the sample heats up during the grinding process it may lose water.

1. Determination of the water content

Instruments and accessories

- 701 KF Titrino, 720 KFS Titrino, 758 KFD Titrino or 784 KFP Titrino
- 703 Titration Stand
- 6.3014.223 Exchange Unit
- 6.1418.220 titration vessel with thermostatic jacket
- Possibly printer with printer cable
- Laboratory thermostat

Reagents

- Titrant: e.g. Hydranal Composite 5 (Riedel-de Haën) or another suitable Karl Fischer reagent
- Solvent mixture: methanol : formamide = 2 : 1 (volume ratio)

Analysis

Pour 30 mL solvent mixture into the titration vessel, heat up to 50 °C and titrate to dryness (conditioning). Using the weighing spoon, add approx. 0.5 g of the finely ground sample and titrate the water content also at 50 °C. Exchange the solvent mixture after each determination.

Remarks

- Extraction of the water from the powdery sample is accelerated by the addition of formamide and by working at 50 °C. Hence, the time required for the analysis is shortened considerably.
 - The determination of the water content using a drying cabinet at 130 °C yields results that are too high as organic constituents of the sample are destroyed during the drying process (strong browning of the sample).
 - The water content should not be much more than 12%, otherwise the storage life of the pasta is shortened (becomes musty and moldy).
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2. Determination of the degree of acidity**Instruments and accessories**

- 702 SET/MET Titrino, 716 DMS Titrino, 736 GP Titrino, 751 GPD Titrino or 785 DMP Titrino
 - 2.728.0040 Magnetic Stirrer
 - 6.3014.223 Exchange Unit
 - 6.0222.100 combined LL pH glass electrode with 6.2104.020 electrode cable
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Reagents

- Titrant: $c(\text{NaOH}) = 0.1 \text{ mol/L}$
 - Calcium chloride solution:
Dissolve 149 g $\text{CaCl}_2 \cdot 6 \text{ H}_2\text{O}$ in 100 mL dist. water and adjust the pH value to 8.3 with NaOH.
-

Analysis

Grind 10 g of the finely ground sample in a porcelain bowl for 3 min with 20 mL dist. water, then rinse the mixture into a glass beaker with 80 mL hot dist. water and cool down immediately to room temperature. After addition of 1 mL CaCl_2 solution, titrate with $c(\text{NaOH}) = 0.1 \text{ mol/L}$ to $\text{pH} = 8.3$ using the SET mode.

Calculation

1 mL $c(\text{NaOH}) = 0.1 \text{ mol/L}$ corresponds to 1 degree of acidity for a sample weight of exactly 10 g. The result is given with one decimal place.

degree of acidity = EP1 (in mL)

Remarks

- CaCl_2 is added in order to bind any phosphates present in the sample.
 - For egg-based pasta degrees of acidity up to 8 are to be considered normal. Higher degrees of acidity indicate that the product has either been stored too long or undesirable acidification has taken place during processing or drying.
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3. Determination of the sodium chloride content**Instruments and accessories**

- 702 SET/MET Titrino, 716 DMS Titrino, 736 GP Titrino, 751 GPD Titrino or 785 DMP Titrino
 - 2.728.0040 Magnetic Stirrer
 - 6.3014.223 Exchange Unit
 - 6.0430.100 Ag Titrode with Ag_2S coating
-

Reagents

- Titrant: $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$
 - Nitric acid, $c(\text{HNO}_3) = 2 \text{ mol/L}$
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Analysis

Shake 10 g of the finely ground sample with 400 mL dist. water for 30 min, then make up to 500 mL with dist. water. Filter through a chloride-free paper filter. Add 5 mL $c(\text{HNO}_3) = 2 \text{ mol/L}$ to 100 mL of the filtrate (corresponding to 2 g of the original sample) and titrate with $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$.

Calculation

1 mL $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$ corresponds to 5.8443 mg NaCl

$$\% \text{ NaCl} = \text{EP1} * \text{C01} * \text{C02} / \text{C00}$$

EP1 = titrant consumption in mL

C00 = 2 (sample mass used in g original sample)

C01 = 5.8443

C02 = 0.1 (conversion factor for %)

Remarks

- Unsalted pasta yields values of approx. 0.1% NaCl.
- Values higher than 0.3% NaCl indicate that salt has been added during processing.

Figures

```
'pa
758 KFD Titrino      01108  758.0020
user                Appl Lab
date 2000-01-12    time 17:18      9
KFT Ipol          AB69 1
parameters
>control parameters
  EP at U           250 mV
  dynamics          100 mV
  max.rate          max. ml/min
  min.volume incr.  min. ul
  stop crit:       drift
  stop drift       20 ul/min
>titration parameters
  pause 1           0 s
  start V:         OFF
  pause 2           0 s
  extr.time         0 s
  temperature       25.0 °C
  time interval    2 s
>stop conditions
  stop V:          abs.
  stop V           99.99 ml
  filling rate     max. ml/min
>statistics
  status:          OFF
>preselections
  display drift:   ON
  drift corr:      OFF
  req.ident:       OFF
  req.smpl size:   value
  limit smpl size: OFF
  oven:            no
  activate pulse:  OFF
=====
```

```
'fm
758 KFD Titrino      01108  758.0020
user                Appl Lab
date 2000-01-12    time 17:24      9
KFT Ipol          AB69 1
>calculations
w(H2O)=EP1*C01*C02/C03/C00;2;%
C00=                0.2696
C01=                 5
C02=                100
C03=                1000
=====
```

Fig. 1: Parameter settings and calculation formula for the determination of the water content.

```
'pa
785 DMP Titrino     01102  785.0011
user                th
date 2000-01-12    time 09:34      7
SET pH             AB69 2
parameters
>SET1
  EP at pH         8.30
  dynamics         2
  max.rate         10.0 ml/min
  min.rate         25.0 µl/min
  stop crit:       drift
  stop drift       20 µl/min
>SET2
  EP at pH         OFF
>titration parameters
  titr.direction:  auto
  pause 1         0 s
  start V:        OFF
  pause 2         0 s
  extr.time       0 s
  meas.input:     1
  temperature     19.8 °C
  time interval   2 s
>stop conditions
  stop V:         abs.
  stop V         99.99 ml
  filling rate    max. ml/min
>statistics
  status:         OFF
>preselections
  conditioning:   OFF
  req.ident:      OFF
  req.smpl size:  value
  limit smpl size: OFF
  activate pulse:  OFF
=====
```

```
'fm
785 DMP Titrino     01102  785.0011
user                th
date 2000-01-12    time 09:34      7
SET pH             AB69 2
>calculations
acidity=EP1*C01*C02/C00;1;
C00=                10.0192
C01=                 0.1
C02=                100
=====
```

Fig. 2: Parameter settings and calculation formula for the determination of the degree of acidity.

```

'pa
785 DMP Titrino          01102  785.0011
user                    th
date 2000-01-10        time 16:12      3
DET U                   AB69 3
parameters
>titration parameters
  meas.pt.density        4
  min.incr.              10.0 µl
  dos.rate               max. ml/min
  signal drift           50 mV/min
  equilibr.time          26 s
  start V:               OFF
  pause                  0 s
  meas.input:            1
  temperature            25.0 °C
>stop conditions
  stop V:                abs.
  stop V                 6.5 ml
  stop U                 OFF mV
  stop EP                 9
  filling rate           max. ml/min
>statistics
  status:                OFF
>evaluation
  EPC                    5
  EP recognition:        greatest
  fix EP1 at U           OFF mV
  pK/HNP:                OFF
>preselections
  req.ident:             OFF
  req.smpl size:         OFF
  limit smpl size:      OFF
  activate pulse:       OFF
  =====

'fm
785 DMP Titrino          01102  785.0011
user                    th
date 2000-01-10        time 16:12      3
DET U                   AB69 3
>calculations
w(NaCl)=EP1*C01*C02*C03/C04/C00;2;%
C00=                     2.0753
C01=                     0.1
C02=                     58.45
C03=                     100
C04=                     1000
  =====
    
```

Fig. 3: Parameter settings and calculation formula for the determination of the sodium chloride content.

```
'fr
785 DMP Titrino      01102  785.0011
user                th
date 2000-01-10    time 16:12      3
card label:785 Lab1
U(init)            145 mV DET U      AB69 3
smp1 size          2.0753 g
EP1                2.363 ml          36 mV
w(NaCl)            0.67 %
stop V reached
=====
```

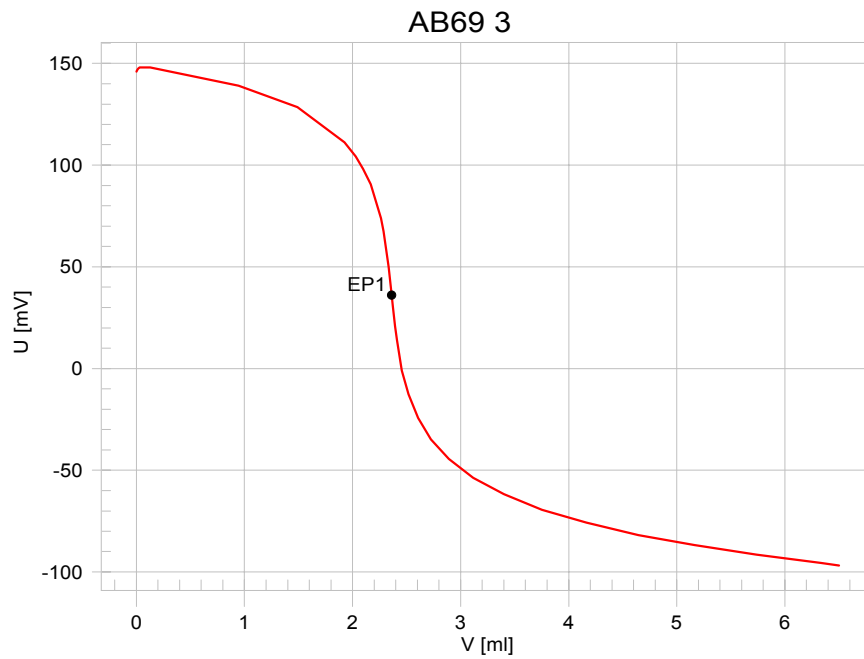


Fig. 4: Result block and titration curve for the determination of sodium chloride in pasta.