

Fat and Oil

Formol Nitrogen in Hydrolyzed Protein

Acid-base titration by
Automatic Potentiometric Titrator

Standard

1. Abstract

Formol nitrogen in hydrolyzed protein (HAP) is measured by titrating with 0.1mol/L sodium hydroxide up to pH8.5 by adding neutral formaldehyde after the first titration with 0.1mol/L sodium hydroxide to pH8.5.

The amount of formol nitrogen is calculated from the difference in titration volume of 0.1mol/L sodium hydroxide between the first and second endpoint.

2. Reference

- 1) Test method prescribed by National Tax Administration Agency

3. Cautions in measurement

- 1) Neutralize formaldehyde before use in measurement.
- 2) Take due care when you handle chemicals.

4. Post-measurement care

Clean the electrode with pure water, and keep it dipped in water for use in next measurement.

5. Test equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier: STD-)

Electrode : standard Combination glass electrode

standard Temperature compensation electrode

6. Reagent

Titrant : 0.1mol/L sodium hydroxide solution (f=1.004)

Reagent : Pure water,

Neutral formaldehyde solution (Neutralize 50mL of formaldehyde solution (JIS K 8872) up to pH8.5 with 0.1mol/L sodium hydroxide solution, making it 100mL in total by adding water)

7. Measurement procedure

—Pretreatment—

1) Add pure water to 5.0mL sample making it correctly 250mL in total.

—Measurement—

1) Transfer 25.0mL sample liquid to a 50mL tall beaker.

2) Titrate with 0.1mol/L sodium hydroxide up to pH=8.5.

3) Add 20.0mL of neutral formaldehyde solution.

4) Titrate with 0.1mol/L sodium hydroxide up to pH=8.5 to obtain formol nitrogen.

8. Formula

Formol nitrogen (W/V%) = (EP2 - EP1) × TF × C1 × K1 / (SIZE × C2)

EP1 : First endpoint titration volume (mL)

EP2 : Second endpoint titration volume (mL)

TF : Factor of titrant (1.004)

C1 : Concentration conversion coefficient (0.0014mg/mL)

K1 : Unit conversion coefficient (100)

SIZE : Sample size (mL)

C2 : Dilution ratio (0.1)

9.Example of measurement

— Ambient condition —

Room temperature : 22.0 °C	Humidity : 24 %	Weather : Fair
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-Titration parameter-

Model : AT-510	Continuation of <Calculation>
Method No. : 50 (Combine)	Temp. Comp. : Off
Number of Method : 2	<Constant>
No. 1 Method No. : 20	C1 (mg/mL) : 0.0014
No. 2 Method No. : 21	C2 (mg/mL) : 0.1
Method No. : 20	K1 : 100
<Auto Titration>	
<Titration>	Method No. : 21
Form : Level Stop	Method Type : Dose& Titr.
APB No. : 1	<Dose>
Unit No. : 1	Mode : Volume Stop
Detector No. : 1	APB No. : 2
Unit : pH	Unit No. : 2
Max. Volume : 20.0mL	Volume : 20.0mL
Wait Time : 0s	Cut-off Time : 0s
Direction : Auto	Dispense Speed : 1s/mL
<Control>	Wait Time : 0s
End Point No. : 1	<Titration>
1st Level : 8.5pH	Form : Level Stop
Over Titr. Vol. : 0mL	APB No. : 1
Gain : 1	Unit No. : 1
Data samp. Pot. : 4.0mV	Detector No. : 1
Data samp. Vol. : 0.5mL	Unit : pH
Control Speed : Medium	Max. Volume : 20.0mL
<Calculation>	Wait Time : 60s
Calc. Type : Sample	Direction : Auto
Conc. 1 : Set	<Control>
C01=EP1	End Point No. : 1
Unit : ml	1st Level : 8.5pH
EP No. : 1	Over Titr. Vol. : 0mL
Conc. 2 : Set	Gain : 1
C02=(EP2-EP1)*TF*C1*K1/(Data samp. Pot. : 4.0mV
SIZE*C2)	Data samp. Vol. : 0.5mL
Unit :	Control Speed : Medium
EP No. : 2	

-Titration curve-

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*** R e s u l t ***
Sample No. : 00-01
Date : 2000/02/28 10:52

<Combine Data>
Method No.   Detect No.
  20             1
  21             2

Method No. : 50
Method Name :
             Combine 50

[2nd Combine]
Method No. : 21
             <Auto Titration>
Method Name :
             Auto Titration

<Constant>
C1(mg/mL)   : 0.0014
C2(mg/mL)   : 0.1
K1           : 100

Titr. Time  : 00:03:20
Size        : 5.0mL

           2 End Point Detect

Conc-1      1.1181ml
Conc-2      1.0162

End Point-1
Volume      : 1.1181mL
Potential   : 8.50pH
End Point-2
Volume      : 4.7330mL
Potential   : 8.50pH

5.00 [pH] 9.00
0.000

8.000
[mL]

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(The above printout data were obtained from titration by AT-510)

«Combine: combined titration»

Number of Method: number of connected method / No.x Method No.: Connected method number

«Titration: titration parameter»

Form: titration form / APB No.: the burette used / Unit No. APB Unit File number used in titration

Detector No. detector used in titration / Max Volume: of titration/Wait Time: before titration starts

Direction.: of titration

«Control parameter»

End Point No.: number of EPs / 1st Level: potential at EP1 / Over Titr.Vol. over-titrated volume / Gain: sensitivity of detection signal /Data samp.Pot.: potential change of sampling signal / Data samp.Vol. : titration volume of sampling signal/Control Speed: control mode of titration speed

«Result: calculation parameter»

Calc.Type: of formula / Conc.x: formula x / Unit: of result /EP No.: EP number for calculation

Temp.Comp.: temperature compensation / C1(mg/mL): concentration conversion

C2: dilute ratio / K1: unit conversion coefficient

–Measurement results–

n	Sample (mL)	Titration (mL) (EP2-EP1)	Formol nitrogen (W/V%)	Result of statistical calculation	
1	<u>5.0</u>	<u>3.6149</u>	<u>1.0162</u>	Mean	1.0138 W/V%
2	5.0	3.5888	1.0089	SD	0.0043 W/V%
3	5.0	3.6155	1.0164	RSD	0.4215 %

* The above results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10.Summary

Hydrolyzed protein is the amino acid mixture used for material of “tasty quality” additive in cooking. This is not food additive pertinent to food hygiene law but ought to indicate the name required by JAS.

Formol nitrogen is the amount of nitrogen contained in amino acids titrated by this method and is an index of the amount of amino acids in the sample.

In this measurement, the relative standard deviation is 0.4% and good repeatability is obtained . Stable measurement is possible by using automatic potentiometric titrator.