

Fat and Oil

Formol Nitrogen in Hydrolyzed Protein

Acid-base titration by
Automatic Potentiometric Titrator

StandardTest method prescribed by
National Tax Administration

1. Abstract

Formol nitrogen in hydrolyzed protein (HAP) is measured by titrating with 0.1mol/L sodium hydroxide up to pH 8.5 by adding neutral formaldehyde after the first titration with 0.1mol/L sodium hydroxide to pH 8.5.

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

7. Measurement procedure

—Pretreatment—

- 1) To make neutral formaldehyde, add 0.1mol/L sodium hydroxide to pH=8.5.
- 2) Add pure water to 5.0mL sample making it 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	<Calculation>の続き
Method No. : 50(Combine)	Conc.2 : Set
Number of Method : 2	CO2=(EP2-EP1)*TF*C1*K1
No.1 Method No. : 20	/(
No.2 Method No. : 21	SIZE*C2)
	Unit :
	EP No. : 2
Method No. : 20	Temp.Comp. : Off
<Auto Titration>	
<Titration>	<Constant>
Form : Level Stop	C1(mg/mL) : 0.0014
APB No. : 1	C2(mg/mL) : 0.1
Unit No. : 1	K1 : 100
Detector No. : 1	
Unit : pH	Method No. : 21
Max. Volume : 20.0mL	<Auto Titration>
Wait Time : 0s	<Titration>
Direction : Auto	Form : Level Stop
<Control>	APB No. : 1
End Point No. : 1	Unit No. : 1
1st Level : 8.5pH	Detector No. : 1
Over Titr.Vol. : 0mL	Unit : pH
Gain : 1	Max. Volume : 20.0mL
Data samp.Pot. : 4.0mV	Wait Time : 60s
Data samp.Vol. : 0.5mL	Direction : Auto
Control Speed : Medium	<Control>
<Calculation>	End Point No. : 1
Calc.Type : Sample	1st Level : 8.5pH
Conc.1 : Set	Over Titr.Vol. : 0mL
CO1=EP1	Gain : 1
	Data samp.Pot. : 4.0mV
Unit : ml	Data samp.Vol. : 0.5mL
EP No. : 1	Control Speed : Medium

-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
[ 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%)	Formol nitrogen batch processed	
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 combination 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 a family member of amino acids.

The sample measurement shows a good repeatability with 0.4% relative standard deviation. Precise and reliable measurement is assured by the automated potentiometry.

The analysis of hydrolyzed protein can be perfectly made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric・volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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