SI Analytics
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Determination of Kjeldahl-Nitrogen

Application

Use

The method is suitable for samples which contents nitrate or protein. The sample is digested with sulfuric acid in the presence of a catalyst. This converts organically bound nitrogen to ammonium sulfate.

In a distillation apparatus the digestion solution is treated with NaOH and the released ammonia (NH_3) is distilled into a solution of boric acid. This solution is then titrated with an acid titrant (HCl or H_2SO_4) to a pH endpoint of 4.65.

Appliances

- Titrator: TL 6000/7000 (TL 6000/7000 M2/20) consists of
- Basic device
- Magnetic stirrer TM 235
- 20 mL Exchange unit WA 20, with amber glass bottle for the titrant, complete
- pH combination electrode A 162 DIN ID

Electrodes

- Electrode: A 162 DIN ID
- Calibration: DIN buffer pH= 4.00 and pH= 7.00

Application

Reagents

- Titrant: HCl 0.01 mol/ - 1 mol/l or H_2SO_4 0.005 – 0.5 mol/l
- Titer determination: TRIS or potassium carbonate

Description

Calibration

The pH combination electrode is calibrated in technical buffer pH=4.00 and pH= 7.00 or in DIN buffer pH= 4.01 and pH= 6.87.

Example of the calibration documentation:

Calibration

Buffers used

pH buffer 1:	TEC_4.000	
pH buffer 2:	TEC_7.000	

Measured values

pH buffer 1:	TEC_4.000	165.6 mV / 23.4 °C
pH buffer 2:	TEC_7.000	-11.2 mV / 23.0 °C

Calibration data

Slope:	99.4 % / -58.8 mV/pH
Zero point:	pH 6.81 / -11.2 mV
Temperature:	23.4 °C (a)
Date and time:	07.03.13 / 15:04

Application

Determination of the exact concentration of the standard solution (0.1 mol/l /0.05 mol/l)

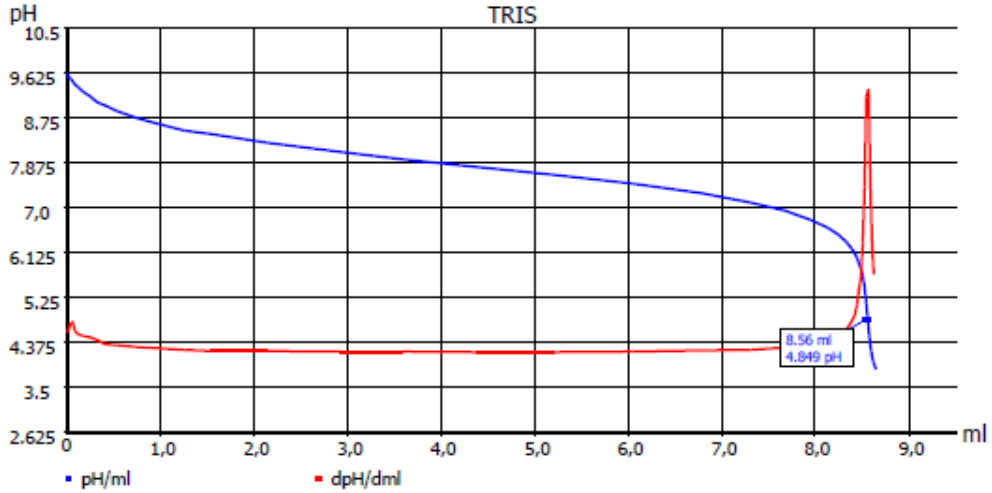
The exact concentration of the acid titrant can be determined using a standard TRIS (recommended) or potassium carbonate. The TRIS is dried for 24 hours in a desiccator at room temperature.

In a 150 mL beaker, 0.15 g TRIS are weighed accurately and dissolved in 80 mL of dist. water with stirring. It is titrated with 0.1 mol/l sodium hydroxide solution.

Pic. left: titer

GLP documentation

Titration graph



Method data

Method name:	Titre HCl	Titration duration:	3 m 8 s
End date:	13.09.12	End time:	14:39:30

Titration data

Sample ID:	TRIS	Weight:	0.1038 g
Start pH:	pH 9.590	End pH:	pH 3.864
Start temperature:	25.0 °C (m)	End temperature:	25.0 °C (m)
Zero point:	pH 6.83 / -10.0 mV	Slope:	100.6 % / -59.5 mV/pH
EQ:	8.560 ml / pH 4.849	Titre:	0.1001 mol/l

Calculation formula

Titre:	(W*F2)/((EQ1-B)*M*F1) -> M103
Mol (M):	121.14000

Weight (W):	man	Factor 2 (F2):	1000.0000
Blank value (B):	0.0000 ml	Factor 1 (F1):	1.0000
Statistics:	Off		

Page 2: Method parameters Titer determination:

Method data overall view

Method name:	Titre HCl	Created at:	09/13/12 14:23:02
Method type:	Automatic titration	Last modification:	09/13/12 14:27:56
Measured value:	pH	Damping settings:	None
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	Steep		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	2.500 pH		
EQ:	On (1)		
Slope value:	Steep	Value:	700

Dosing parameter

Dosing speed:	100 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Unit values

Unit size:	20ml
Unit ID:	10039005
Reagent:	HCl 0.1 mol/L
Batch ID:	no Charge
Concentration [mol/l]:	0.10070
Determined at:	12/05/11 19:18:45
Expire date:	08/18/12
Opened/compounded:	09/10/11
Test according ISO 8655:	05/10/11
Last modification:	09/13/12 14:35:18

Device information

Device:	TitroLine 7000
Serial number:	00012
Software version:	1230

Titre_HCl_13_09_12-14_36_21.pdf 2/2

Application

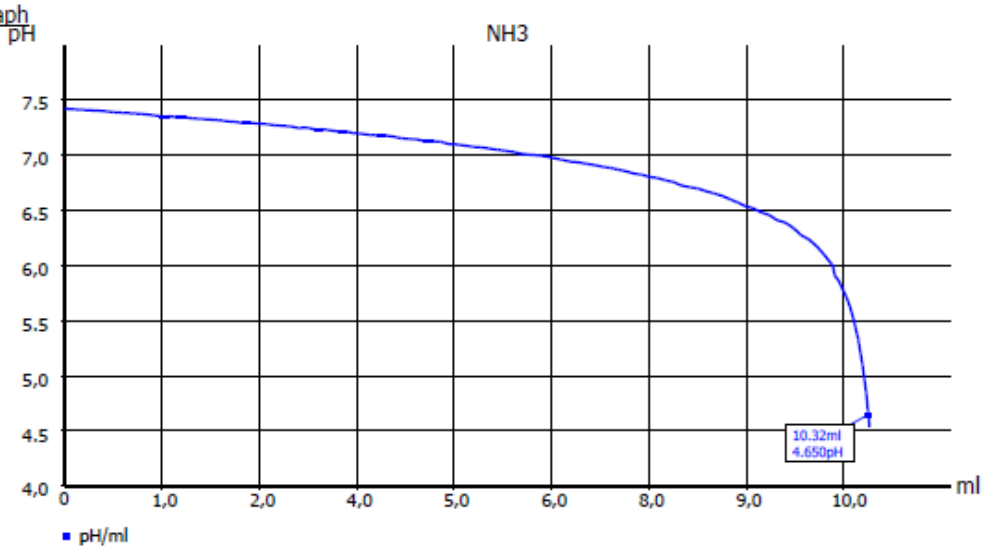
Titration of the sample

When the distillation is finish the beaker (250 ml or bigger) is placed on the magnetic stirrer. The electrode and burette tip are immersed into the solution, the Kjeldahl method is selected and started. The titrator titrate until an endpoint of pH 4.65 is reached. A blank titration with more carefully titration parameetrs should be also carried out before to find a blank value for the Kjeldahl instrument.

Result example:

Standard documentation

Titration graph



Method data

Method name:	Kjehldal		
End date:	30.11.11		
End time:	16:37:56	Titration duration:	2 m 31 s

Titration data

Sample ID:	NH3	Pattern:	1.5500ml
Start pH/temp:	pH7.420	End pH/temp:	pH4.529
start temperature:	25.0 °C (m)	end temperature:	25.0 °C (m)
Zero point:	pH 6.82 / 0.1 mV	Slope:	98.4% / -58.2 mV/pH
EP1:	10.317 ml/pH4.650		%s
Nitrogen:	9.325 g/l		

Calculation formula

Formulas	(EP1-B)*T*M*F1/(V*F2)	Factor (F1):	2.0000
		Factor (F2):	1.0000
		Factor (F3):	1.0000
Blank value (B):	0.0000ml		
Titre (T):	0.05000000(f)		
Mol (M):	14.01000		

Method

Method data

Method name:	Kjeldahl	Created at:	04/23/13 15:24:04
Method type:	Automatic titration	Last modification:	05/16/13 12:54:59
Measured value:	pH		
Titration mode:	End pt.	Documentation:	GLP
Linear steps:	0.040 ml		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
Endpoint 1:	pH 4.650	delta endpoint 1:	pH 1.200
		Endpoint delay 1:	3 s
Endpoint 2:	Off		

Dosing parameter

Dosing speed:	60.00 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Calculation formula

Nitrogen:	(EP1-B)*T*M*F1/(W*F2)	Mol (M):	14.01000
Unit:	%	Decimal places:	2
Blank value (B):	M02	Titre (T):	auto
Factor 1 (F1):	0.1000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

Application

Blank method:

Method data

Method name:	Kjeldahl blank	Created at:	05/16/13 12:56:24
Method type:	Automatic titration	Last modification:	05/16/13 12:57:33
Measured value:	pH		
Titration mode:	End pt.	Documentation:	GLP
Linear steps:	0.010 ml		
Measuring speed / drift:	Normal:	minimum holding time:	02 s
		maximum holding time:	15 s
		Measuring time:	02 s
		Drift:	20 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
Endpoint 1:	pH 4.650	delta endpoint 1:	pH 1.200
		Endpoint delay 1:	3 s
Endpoint 2:	Off		

Dosing parameter

Dosing speed:	30.00 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		

Calculation formula

blank value:	EP1 -> M02		
Unit:	ml	Decimal places:	3
Statistics:	Off		

Application

Notes

If you have any questions on the application, you can feel free to contact us..

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