Application Note

Determination of ammonium ions using a multiple sample changer

Industry	Chemicals
Instrument	Multiple sample changer, Automatic potentiometric titrator
Measurement method	Potentiometric titration / Neutralization titration
Standards	

1. Scope

CAUTION
When following this application note, wear masks, gloves, and any other necessary PPE; and conduct operations inside a fume hood.

The multiple sample changer can be connected to an automatic potentiometric titrator system to create an automatic measurement system. This system contributes to efficiency and labor savings in analytical operations by automatically titrating many samples with high accuracy. This Application Note introduces an example of measuring ammonium ions contained in ammonium nitrate (NH_4NO_3) solution (8. Notes).

2. Post-measurement procedure

Seal the refill port for internal liquid of the reference electrode with a rubber plug so that the internal liquid is prevented from leaking out or concentrating. Once done, store the electrode in accordance with the following:

- For short-term storage (less than one month) store immersed in pure water.
- For long-term storage (longer than one month) store immersed in a solution of pH 4 standard solution and 3.3 mol/L KCl solution mixed in an equal volume ratio.

3. Apparatus

Equipment	Automatic potentiometric titrator (preamplifier STD)
Electrode	Combined glass electrode (Inner liquid 3.3 mol/L potassium chloride solution)
Options	Multiple sample changer

4. Reagents

Titrant	0.05 mol/L Sodium hydroxide solution
Reagents	Ion exchanged water Aqueous formaldehyde solution (37 %)

5. Procedure

-Preparation-

To remove dissolved gas present in the titrant, degas the titrant for 30 minutes under reduced pressure in an ultrasonic cleaner.

-Measurement-

1) Dispense 10 mL of the sample accurately into a beaker.
2) Set the beaker in the multiple sample changer.
3) Add 10mL of aqueous formaldehyde solution (37 %) and 30 mL of ion exchanged water.
4) Titrate with 0.05 mol/L Sodium hydroxide solution.

6. Calculation

Acidity (mass%)* = EP1×TF×K1×C1/S

EP1	Titration amount of sample (mL)
TF	Factor of titrant (1.000)
C1	Concentration conversion coefficient (0.902 mg)※
K1	Unit conversion factor (1000)
S	Quantity of sample (mL)

*Ammonium ion equivalent of 1mL of 0.05 mol/L sodium hydroxide solution (mg)

7. Example of measurement

-Titration parameter-

<Titr. Mode>	Auto Int.
<Titr. Form>	Level Stop

<Titr. Para.>

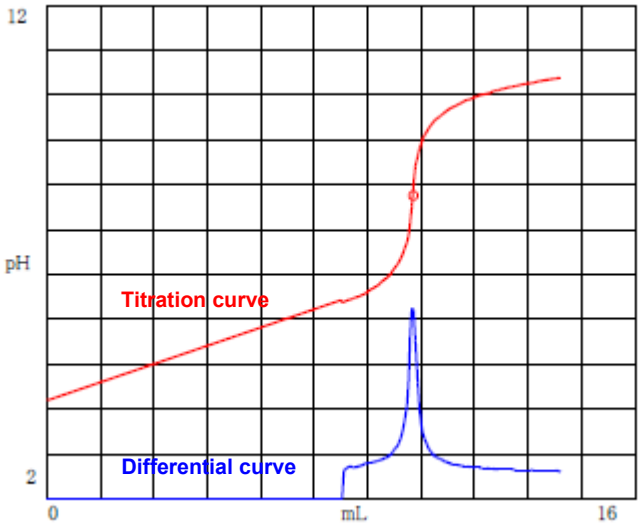
Burette No.	1
Max. Volume	14 (mL)
Channel/Unit(Ctrl.)	Ch1, pH
Channel/Unit(ref.)	off
pH Polarity	Standard
Type of Titration	Not check
EP Direction	Auto
Wait Time	30 (s)
Dose Mode	Volume Stop
Stop volume	10 (mL)
Dispense Speed	10 (s/mL)

<Ctrl. Para.>

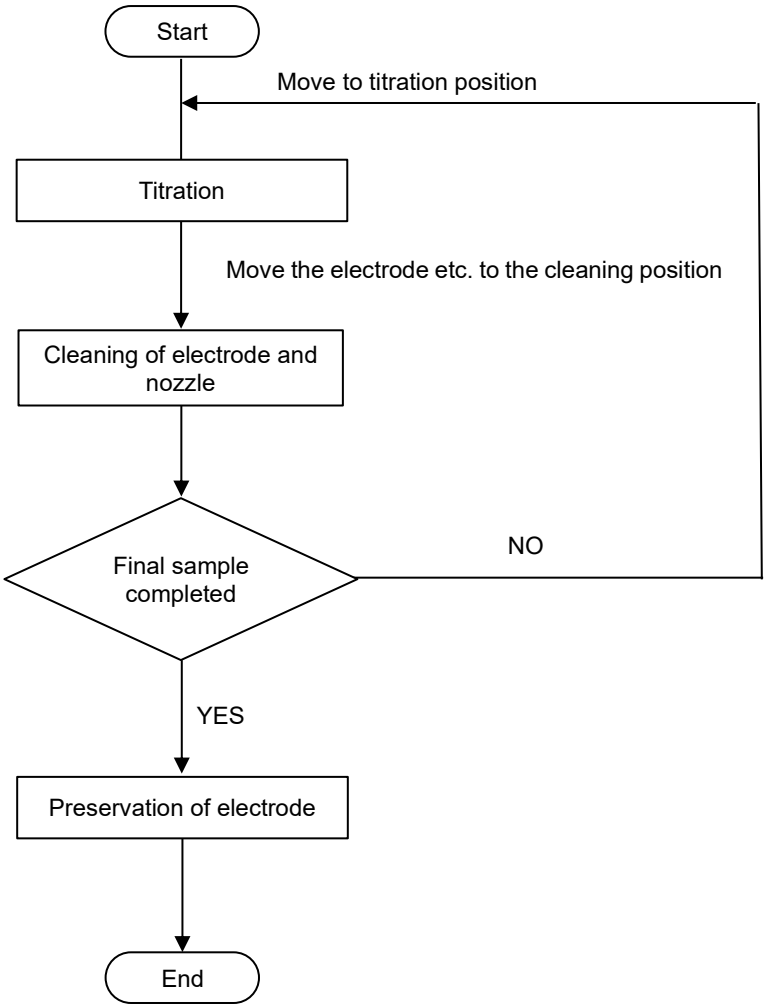
Number of EP	1
End Sense	Set
EP Sense (Potential)	160 (dE)
EP Sense (Differential)	700 (dE/dmL)
Gain	3
Data Sampling	Set
Data Sampling Pot.	4.0 (mV)
Data Sampling Vol.	0.5 (mL)
Ctrl. Speed Mode	slow
Ctrl. Speed	0.5
Other Control	Standard
Other Control	Standard
Stirrer Speed	4

（Listed above are example settings. Availability of settings may vary by instrument model.）

—Titration curve—



—Sequence—



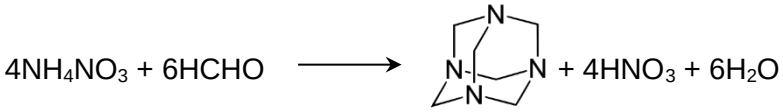
— Measurement results —

Table Measurement result

n	Sample (mL)	Titration amount (mL)	Result (mg/L)
1	10.0	11.1451	1005.29
2	10.0	11.1438	1005.17
3	10.0	11.1440	1005.19
4	10.0	11.1431	1005.11
5	10.0	11.1465	1005.41
6	10.0	11.1476	1005.51
7	10.0	11.1488	1005.62
8	10.0	11.1507	1005.79
9	10.0	11.1515	1005.87
10	10.0	11.1501	1005.74
Mean			1005.47
SD			0.278
RSD(%)			0.028

8. Notes

The addition of formaldehyde to ammonium nitrate produces hexamethylenetetramine, water and an amount of nitric acid that is equal/directly proportional to that of the ammonium ions contained in ammonium nitrate.



This nitric acid is titrated with sodium hydroxide (neutralization titration) to quantify the ammonium ion. While ammonium nitrate, a weak acid, can be titrated directly with a base, it is highly preferential to use the method described here, as the release of nitric acid produces a clear-cut pH jump at the equivalence point, making it highly accurate. This technique is also applicable to other ammonium salts.