Application Note

Determination of Arsenic (III) using a multiple sample changer

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

1. Scope

CAUTION

Arsenic compounds are harmful to the human body. When following this application note, wear masks, gloves, protective equipment, etc., and conduct the operations inside a fume hood.

The multiple sample changer can be connected to an automatic potentiometric titrator system to create an automatic measurement system. The multiple sample changer enables high accuracy automated titration of multiple samples, which results in more efficient, labor-saving analysis work.
This Application Note introduces an example of the measurement of arsenic (III) in an aqueous sodium arsenite solution using sequence control (Note 1). Measurement results for arsenic (III) concentration is expressed in (mg/L) (Note 2).

2. Post-measurement procedure

To prevent the internal solution from flowing out and concentrating, seal the internal solution filling port on the reference electrode with a rubber stopper.

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	0.0068 mol/L Iodine solution
Reagents	Pure water 50 g/L Sodium hydrogen carbonate solution

5. Procedure

1) Dispense 10mL of the sample accurately into a beaker.
2) Set the beaker in the multiple sample changer.
3) Add approximately 60 mL of 50 g/L sodium bicarbonate solution.
4) Add 30mL of pure water and titrate with 0.0068 mol/L iodine solution.

6. Calculation

Arsenic (III) (mg/L) = (EP1 – BL1) × TF × C1 × K1/S

EP1	Titration amount of sample (mL)
BL1	Titration amount of Blank test (0 mL)
TF	Factor of titrant (0.9289)
C1	Concentration conversion coefficient (0.5095)※
K1	Unit conversion factor (1000)
S	Quantity of sample (mL)

※Arsenic(III) equivalent in 1mL of 0.0068 mol/L iodine solution (mg)

7. Example of measurement

-Titration parameter-

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

<Titr. Para.>

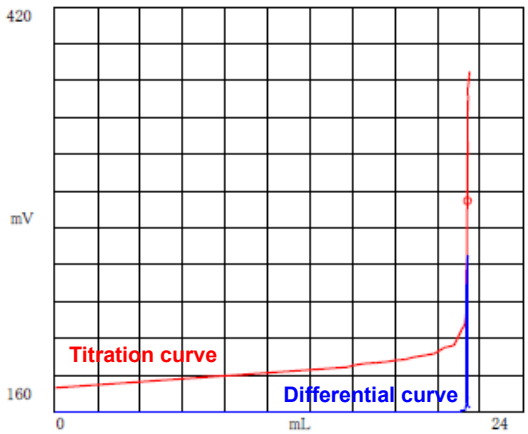
Burette No.	1
Max. Volume	40 (mL)
Channel/Unit(Ctrl.)	Ch1, mV
pH Polarity	Standard
Type of Titration	Not check
EP Direction	Auto
Wait Time	30 (s)
Dose Mode	Volume Stop
Stop volume	15 (mL)
Dispense Speed	10 (s/mL)

<Ctrl. Para.>

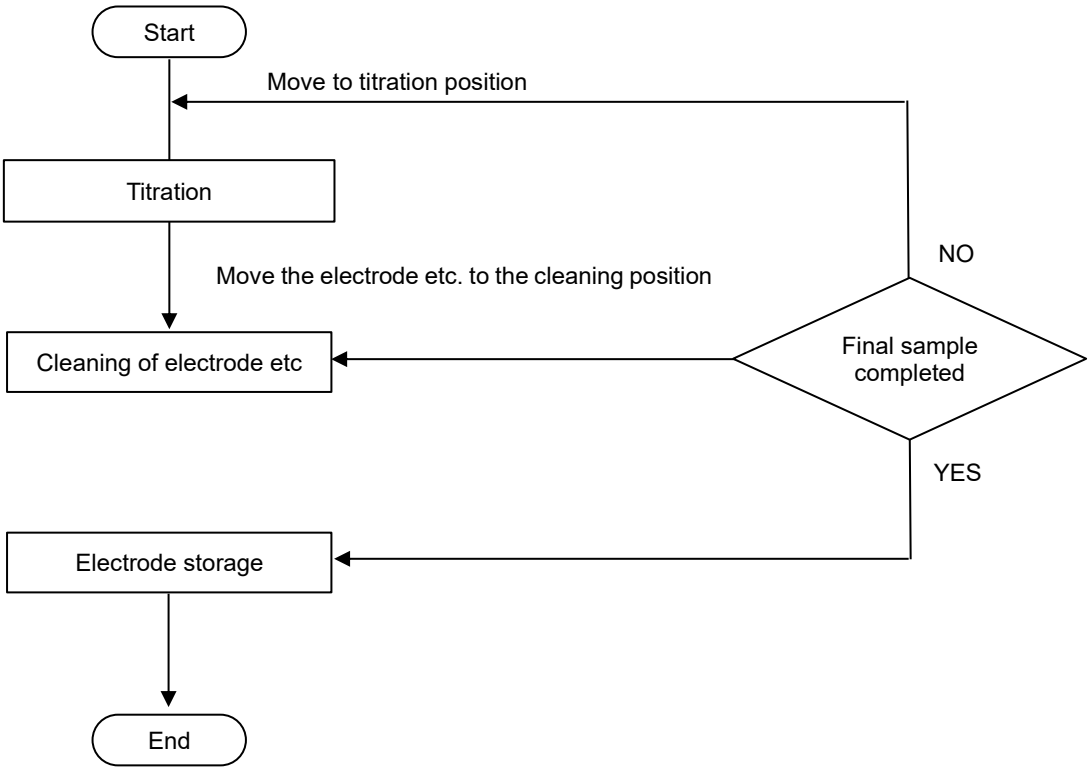
Number of EP	1
End Sense	Auto
Gain	1
Data Sampling	Standard
Ctrl. Speed Mode	Set
Ctrl. Speed	0.5
Other Control	Standard
Stirrer Speed	4

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

—Titration curve—



—Sequence—



— Measurement results —

Table Measurement result

Sequence	Sample (mL)	Titration amount (mL)	result (mg/L)
1	10	21.1977	1003.23
2	10	21.2038	1003.52
3	10	21.2142	1004.01
4	10	21.2199	1004.28
5	10	21.2034	1003.50
6	10	21.2114	1003.88
7	10	21.2232	1004.44
8	10	21.2149	1004.05
9	10	21.2214	1004.35
10	10	21.2283	1004.68
Mean			1003.99
SD			0.465
RSD(%)			0.046

8. Summary

In this test, a standard solution with an indicated arsenic (III) concentration of 1000 mg/L (± 6 mg/L) was measured. After 10 measurements, the average concentration value was 1003.99 mg/L, which is consistent with the indicated value of the standard solution.

9. Notes

Note 1) The additive solutions and indicator solutions can also be automated with the addition of an automatic burette.

Note 2) Arsenic (III) exists in an aqueous solution as the arsenite ion AsO_3^{3-}. In this test, arsenite ions were titrated directly with iodine. The titration reaction is shown below.

$$AsO_3^{3-} + I_2 + H_2O \rightleftharpoons AsO_4^{3-} + 2HI$$

The above reaction is reversible under acidic conditions, owing to the generation of the strong reducing agent, Hydroiodic acid (HI). Consequently, the reaction product AsO_4^{3-} is reduced to AsO_3^{3-} by HI. In the titration, the reaction must proceed in a completely rightward direction, which requires neutralization of HI. In this test, neutralization of HI was achieved by adding sodium bicarbonate.