

<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	O5	Feb. 03, 2022
<b>Factor standardization</b>	<b>Standardization of ammonium thiocyanate titrant</b>			

## 1. Abstract

The measurement method of chloride ions or salt content by precipitation titration using a silver nitrate standard solution is widely used in the titration method. The back-titration method of silver nitrate titration is used for some substances listed in the *Japanese Pharmacopoeia*. The back-titration method is generally applied when the titration reaction is slow, and the reaction progresses faster than a forward titration by adding an excessive amount of titrant to the sample. An excessive amount of silver nitrate standard solution is added to the sample to react with the target component in the sample, and the remaining silver nitrate is titrated with ammonium thiocyanate standard solution to indirectly quantify the target component in the sample.

This report introduces the factor determination method for ammonium thiocyanate standard solution. Firstly, 0.1 mol/L silver nitrate standard solution, which is a standard material, was dispensed with a buret. Secondly, potentiometric titration was performed with ammonium thiocyanate standard solution to determine the factor of ammonium thiocyanate. 1 mol of silver nitrate and 1 mol of ammonium thiocyanate react quantitatively according to formula 1, and the titration curve shows an inflection point at the end point.



*Japanese Industrial Standard (JIS K8001)* and the *Japanese Pharmacopoeia* describe that standardized silver nitrate solution should be used for factor determination of ammonium thiocyanate standard solution. See Application Data No. O4 for the method of standardizing silver nitrate standard solution.

- 1) Japanese Pharmacopoeia Eighteenth Edition
- 2) Japanese Industrial Standard JIS K8001 General rules for test methods of reagents

## 2. Configuration of instruments and reagents

### (1) Configuration of instruments

Main unit	: Automatic Titrator	COM Series
	Optional buret	1 unit
Electrodes	: Silver-reference combination electrode	AGR-811Z*

\* It can also be applied to combinations of other silver electrodes such as AG-311 and silver comparison electrodes such as RE-241Z.

### (2) Reagents

Titrant	: 0.1 mol/L (0.1 N) ammonium thiocyanate standard solution (Buret No. 2)
Standard sample	: 0.1 mol/L (0.1 N) silver nitrate standard solution (f = 0.995, Buret No.1)

### 3. Measurement procedure

- (1) Add 50 mL of DI water and a stirrer bar to a 100 mL beaker.
- (2) Immerse the electrodes and start the measurement. 10 mL of silver nitrate standard solution is added to the beaker by the buret dispensing.
- (3) Titration is subsequently performed with a 0.1 mol/L ammonium thiocyanate standard solution. The inflection point on the titration curve is detected as the end point.

### 4. Measurement conditions and results

#### Examples of titration conditions

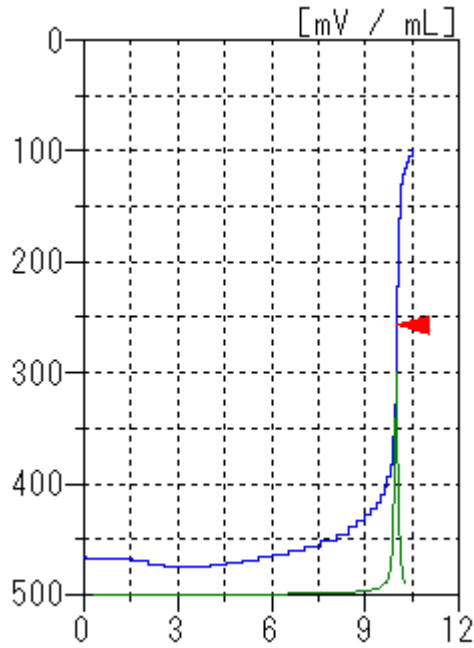
Factor standardization measurement of 0.1 mol/L ammonium thiocyanate standard solution

M. File	1+2		
Cndt No.	1		
Method	Disp		
Buret No.	1		
S-Timer	5 sec		
Disp Vol.	10 mL		
Cndt No.	2		
Method	Auto	ConstantNo.	2
Buret No.	2	Size	10 mL
Amp No.	2	Blank	0 mL
D. Unit	mV	Molarity	0.1 mol/L
S-Timer	5 sec	Factor	0.995 *1
C.P. mL	0 mL	K	0
T Timer	0 sec	L	0
D.P. mL	1 mL		
End Sens	500	Unit	Fact1
Over mL	0.5 mL	Formula	S/(D-B)*F
Max.Vol.	20 mL	Digits	4
		Mode No.	4
		Pre Int	0 sec
		Del K	9
		Del Sens	0 mV
		Int Time	3 sec
		Int Sens	3 mV
		Brt Speed	2
		Pulse	40

\*1: Factor of 0.1 mol/L silver nitrate standard solution

#### Measurement results

Measurement No.	Sample size (mL)	Titrant volume (mL)	Factor	Statistic calculation	
1	10	9.973	0.9977	Avg.	0.997
2	10	9.980	0.9970	SD	0.001
3	10	9.984	0.9966	RSD	0.06 %



Measurement of factor of ammonium thiocyanate standard solution

### Examples of titration curves

#### 5. Note

(1) About the buret for dispensing silver nitrate

In this report, silver nitrate standard solution was dispensed and then immediately titrated with ammonium thiocyanate standard solution. For some substance described in the *Japanese Pharmacopoeia*, back-titration with silver nitrate and ammonium thiocyanate is applied for a quantitative method of its purity. These methods are described in which an excessive amount of silver nitrate is dispensed, allowed to stand the solution overnight, filtered it, and then titrated with an ammonium thiocyanate standard solution. In that case, use dispensing function of the buret or use a volumetric pipette to add a certain amount of silver nitrate to the sample precisely.

Keywords : Factor standardization, Precipitation titration, Back-titration, Silver nitrate, Ammonium thiocyanate