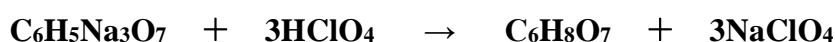


<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	B21	Nov. 14, 2018
<b>Drugs and Medicines</b>	<b>Quantitative determination of sodium citrate</b>			

## 1. Abstract

Sodium citrate is used as anticaking agent for blood in pharmaceutical field. Quantitative determination method for sodium citrate is stipulated in *Japanese Pharmacopoeia*.

This report introduces an example of potentiometric titration for sodium citrate that well-dried sample is dissolved in acetic acid, then it is titrated with perchloric acid - acetic acid standard solution.



## 2. Configuration of instruments and reagents

### (1) Configuration of instruments

Main unit	: Hiranuma Automatic Titrator	COM Series
Electrodes	: Glass electrode	GE-101B**
	: Reference electrode	RE-201Z*

\*Inner solution has to be changed to saturated sodium perchlorate in acetic acid.

\*\*Glass-reference combination electrode GR-511BZ is also usable.

### (2) Reagents

Titration solvent	: 50 mL of acetic acid (reagent grade)
Titration solvent	: 0.1 mol/L perchloric acid - acetic acid standard solution

## 3. Measurement procedure

- (1) Take about 0.1 g of sodium citrate hydrate ( $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) and dry it at 180 °C for 2 hours.  
(This procedure removes the hydrates.)
- (2) Take about 0.2 g of the above dried sample into 100 mL tall beaker and weigh accurately.
- (3) Add 30 mL of acetic acid and dissolve sample by stirring.
- (4) Immerse the electrodes and titrate with 0.1 mol/L perchloric acid - acetic acid standard solution.  
Additionally, perform the blank test with the same procedure of sample measurement.

## 4. Measurement conditions and results

### Examples of titration conditions

#### Measurement of blank

Cndt No.	1	ConstantNo.	1	Mode No.	17
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	0.1000 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.004	Int Time	5 sec
S-Timer	10 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	mL	Pulse	16
D.P. mL	0 mL	Formula	D		
End Sens	1000	Digits	3		
Over mL	0.1 mL	Auto In Pram.	Non		
Max.Vol.	1.0 mL				

#### Measurement of sodium citrate

Cndt No.	2	ConstantNo.	2	Mode No.	8
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0.0100 mL	Del K	5
Amp No.	1	Molarity	0.1000 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.004	Int Time	5 sec
S-Timer	10 sec	K	86.02	Int Sens	3 mV
C.P. mL	20 mL	L	0	BrT Speed	2
T Timer	5 sec	Unit	%	Pulse	40
D.P. mL	0.0 mL	Formula	$(D-B)*K*F*M/(S*10)$		
End Sens	300	Digits	3		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	40 mL				

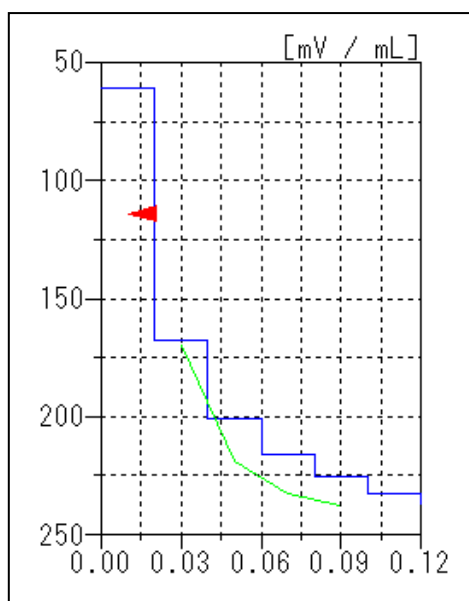
### Measurement results

#### Measurement of blank

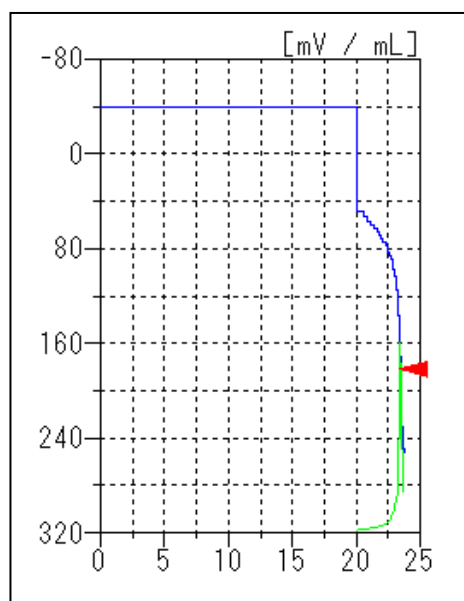
Number of Measurement	Size (g)	Titrant Volume (mL)
1	-	0.010
2	-	0.010
Avg. (Blank)		0.010 mL

#### Measurement of sample

Number of Measurement	Size (g)	Titrant Volume (mL)	Concentration (%)
1	0.2029	23.419	99.640
2	0.2052	23.675	99.601
3	0.2052	23.670	99.580
		Avg.	99.61 %
Statistic calculation		SD	0.0304 %
		RSD	0.03 %



Measurement of blank



Measurement of sample

### Examples of titration curves

## 5. Note

### (1) Effect of water on perchloric acid titration

Water mixed in a measurement system affects to the reaction system of perchloric acid titration because of the leveling effect, which results in a negative effect such as lowered quantitative performance or getting less sensitivity around the end point. Therefore please take care not to mix water in the measurement system. Reference electrode for non-aqueous titration should be prepared as described in the following item (2), because water of KCl solution commonly used as inner solution for reference electrode could be mixed in the measurement system.

### (2) Preparation of inner solution for reference electrode

The inner solution of the reference electrode RE-201Z is filled with 4 M KCl aqueous solution when purchased. This inner solution have to be replaced to saturated sodium perchlorate in acetic acid solution for this measurement. Replacement procedure is described below.

- i) Prepare the saturated solution of sodium perchlorate in acetic acid with reagent grade.
- ii) Discharge inner solution from reference electrode RE-201Z and wash inside it with water and then acetic acid.
- iii) Fill the prepared inner solution into reference electrode from the supply port.
- iv) Cure the electrode for one day before use.

### (3) Influence of experiment temperature on perchloric acid titration

Acetic acid used as a solvent for the titrant has a relatively large thermal expansion coefficient. When the temperature changes by 1 °C, the titrant causes a volume change of 0.1 %. Factor titration and sample measurement should be performed at the same room temperature as much as possible for accurate measurement. (Please refer to application data B19 in regards to factor correction.)

Keywords: Sodium citrate, Perchloric acid titration, Non-aqueous titration