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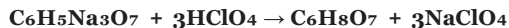
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HIRANUMA APPLICATION DATA		Automatic Titrator	Data No.	B21	Nov. 14,2018
Drugs and Medicines	Quantitative determination of sodium citrate				

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

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

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

Measurement of sodium citrate

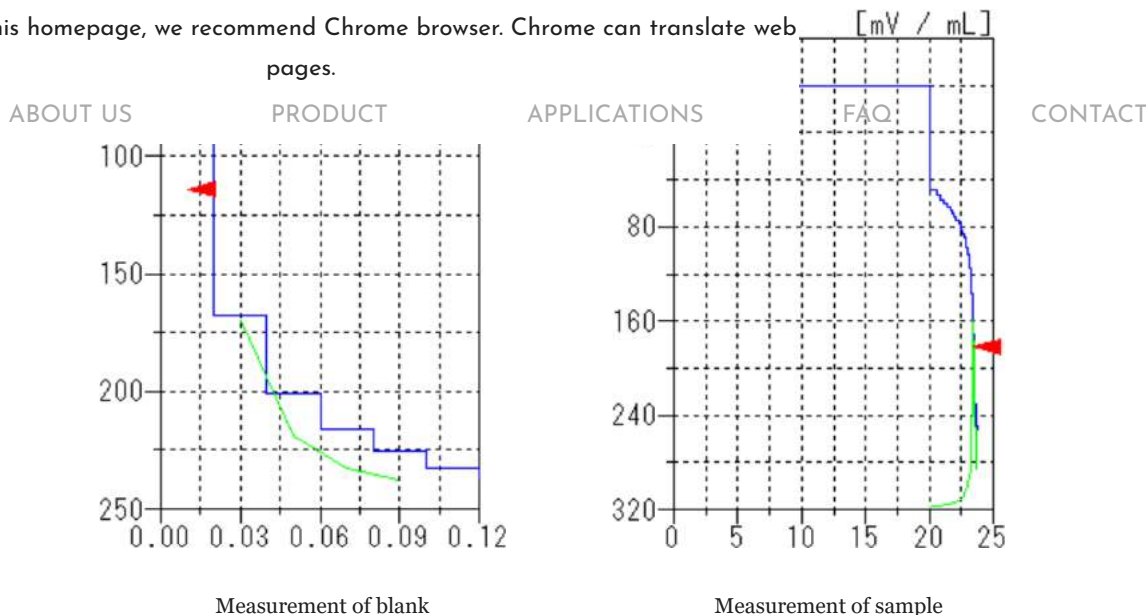
Cndt No.	2		ConstantNo.	2		Mode No.	8	
Method	Auto		Size	0	g	Pre Int	0	sec
Buret No.	1		Blank	0.0100	mL	Del K	5	
Amp No.	1		Molarity	0.1000	mol/L	Del Sens	0	mV
D. Unit	mV		Factor	1.004		Int Time	5	sec
S-Timer	10	sec	K	86.02		Int Sens	3	mV
C.P. mL	20	mL	L	0		BrT Speed	2	
T Timer	5	sec				Pulse	40	
D.P.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

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 %

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Examples of measurement 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

*Some measurement would not be possible depending on optional configuration of system.

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