HIRANUMA APPLICATION DATA	Automatic Titrator	Date No.	B2	Sep. 12, 2018
Drugs and Medicines	**Determination of sodium acetate with perchloric acid titration**			

1. Abstract

Sodium acetate is widely used in chemical and medical industry (E.g. Buffer, Antifreezing agent, Alkalizing supplements, Dye chemical, Hydragogue). Determination method of sodium acetate is described on Japanese Pharmacopoeia and Japanese Industrial Standard (JIS K8371). Both methods employ perchloric acid titration method, dissolve the sample in glacial acetic acid and titrate with perchloric acid – acetic acid standard solution. Endpoint is detected by potentiometric titration with glass / reference electrodes.

$$CH_3COONa + HClO_4 \rightarrow CH_3COOH + NaClO_4$$

2. Configuration of instruments and Reagents

(1) Instruments

Main unit	: Hiranuma Automatic Titrator	COM Series
Electrode	: Glass electrode	GE-101B
	Reference electrode	RE-201Z
	(Inner solution should be changed; it is described below)	

(2) Reagents

Titrant	: 0.1 mol/L perchloric acid in glacial acetic acid standard solution
Titration solvent	: Glacial acetic acid
Electrolyte	: Saturated sodium perchlorate in glacial acetic acid
	(For inner solution of reference electrode)

3. Measurement procedure

(1) Take 0.2 g of the sample into a 100 mL tall-beaker and accurately weigh it.
(2) Add 50 mL of acetic acid and dissolve the sample.
(3) Immerse the electrodes and start titration with 0.1 mol/L perchloric acid in glacial acetic acid standard solution. Perform blank test without sample.

4. Measurement Conditions and Results

Example of titration condition

Measurement of blank

Cndt No.	1							
Method	Auto		ConstantNo.	1		Mode No.	17	
Buret No.	1		Size	0	g	Pre Int	0	sec
Amp No.	1		Blank	0	mL	Del K	0	
D. Unit	mV		Molarity	0.1	mol/L	Del Sens	0	mV
S-Timer	60	sec	Factor	1.000		Int Time	5	sec
C.P. mL	0	mL	K	0		Int Sens	3	mV
T Timer	0	sec	L	0		Brt Speed	2	
D.P. mL	0	mL				Pulse	16	
End Sens	300		Unit	mL				
Over mL	0.1	mL	Formula	D				
Max.Vol.	1	mL	Digits	3				
			Auto In Pram.		Non			

Measurement of sample

Cndt No.	2							
Method	Auto		ConstantNo.	2		Mode No.	8	
Buret No.	1		Size	0	g	Pre Int	0	sec
Amp No.	1		Blank	0.01	mL	Del K	5	
D. Unit	mV		Molarity	0.1	mol/L	Del Sens	0	mV
S-Timer	60	sec	Factor	1.000		Int Time	5	sec
C.P. mL	0	mL	K	82.03		Int Sens	3	mV
D.P. mL	0	mL	L	0		Brt Speed	2	
End Sens	300					Pulse	40	
Over mL	0.2	mL	Unit	%				
Max.Vol.	40	mL	Formula	(D-B)*K*F*M/(S*10)				
			Digits	3				
			Auto In Pram.		Non			

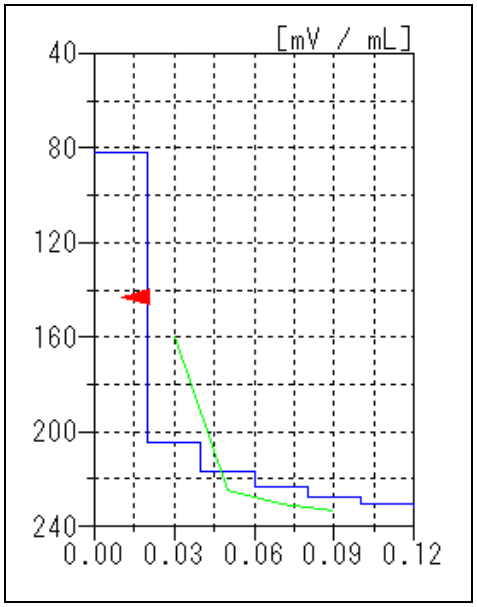
Measurement results

Measurement of blank

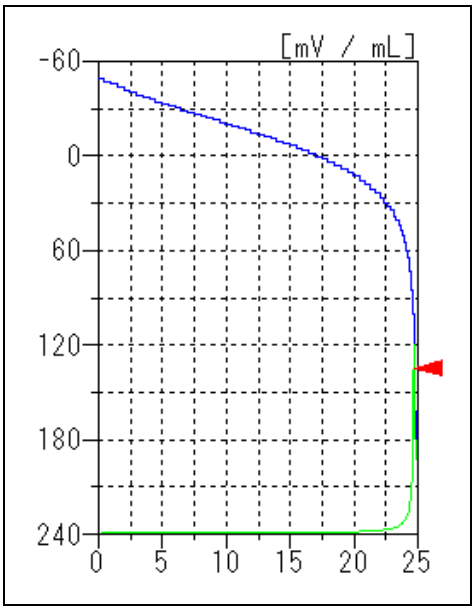
Measurement No.	Size (g)	Titrant volume (mL)
1	—	0.01
2	—	0.01
Statistical result	Avg. (mL)	0.01

Measurement of sample

Measurement No.	Size (g)	Titrant volume (mL)	Conc (%)
1	0.2029	24.690	99.778
2	0.2021	24.553	99.617
3	0.2010	24.440	99.701
Statistical result		Avg. (%)	99.70
		SD (%)	0.0805
		RSD (%)	0.08



Blank measurement



Sample measurement

Examples of titration curves

5. Note

(1) Effect of water on perchloric acid titration

Water mixed in sample solution 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 sample solution. 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 sample solution.

(2) Preparation of inner solution for reference electrode

The inner solution of the reference electrode RE-201Z is filled with saturated KCl aqueous solution normally. For this measurement, it is necessary to replace inner solution to saturated sodium perchlorate in acetic acid solution. Replacement procedure is described below.

i) Prepare the saturated solution of sodium perchlorate in glacial acetic acid with reagent grade of these.

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) Experiment temperature

Acetic acid used as a solvent for the titrant has a relatively large thermal expansion coefficient, and when the temperature changes by 1 °C, the titrant causes a volume change of 0.1 %. For accurate measurement, factor titration and sample measurement should be performed at the same room temperature as much as possible.

Keywords : Sodium acetate, Perchloric acid titration, Nonaqueous titration, Neutralization titration,

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