

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	K8	Feb. 10, 2021
Organic acid	Fractional determination of ascorbic acid and sodium ascorbate			

1. Abstract

Ascorbic acid has properties as an acid and a strong reducing agent. On the other hand, sodium ascorbate doesn't have a function as an acid but works as a reducing agent as well as ascorbic acid. The quantitative determination method for ascorbic acid is prescribed in JIS K 9502 and Japanese pharmacopeia. There are two determination methods for ascorbic acid; neutralization titration and iodine titration.

The fractional determination method for ascorbic acid and sodium ascorbate is introduced in this report. First, ascorbic acid is determined by the neutralization titration with sodium hydroxide standard solution (formula (1)). After that, the total amount of ascorbic acid (ascorbic acid and sodium ascorbate) is measured by the redox titration with iodine standard solution (formula (2) and (3)). The sodium ascorbate is determined by the subtraction of the ascorbic acid from the total amount of ascorbic acid.

(I) Reaction formula for neutralization titration



(II) Reaction formulae for redox titration



2. Configuration of instruments and reagents

(1) Configuration

Main unit	:	Automatic Titrator COM series		
Option	:	One buret and one buret head		
Electrode	:	Glass reference electrode	GR-501BZ	Connect to IE-1.
	:	Platinum electrode	PT-301	Connect to IE-2.

(2) Reagents

Titration	:	0.1 mol/L Sodium hydroxide standard solution
		0.05 mol/L Iodine standard solution
Additive	:	2 % Metaphosphoric acid solution

3. Measurement procedure

(1) Measurement for ascorbic acid

- (i) Take approx. 0.4 g of sample into a 100 mL beaker and weigh it accurately.
- (ii) Add approx. 40 mL of DI water.
- (iii) Immerse the electrodes to start titration with 0.1 mol/L sodium hydroxide standard solution.

(2) Measurement for sodium ascorbate

- (i) Take approx. 0.4 g of sample into a 100 mL beaker and weigh it accurately.
- (ii) Add 50 mL of 2 % metaphosphoric acid solution.
- (iii) Immerse the electrodes to start titration with 0.05 mol/L iodine standard solution with using optional buret and buret head.

4. Measurement conditions and results

Examples of titration conditions

(1) Measurement for ascorbic acid

Cndt No.	1	Constant No.	1	Mode No.	4
Method	Auto	Size	0.4036 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	9
Amp No.	1	Molarity	0.1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.004	Int Time	3 sec
S-Timer	5 sec	K	176.13	Int Sens	3 mV
C.P. mL	0 mL	L		BrT Speed	2
T Timer	0 sec	Unit	mg/g	Pulse	40
D.P. mL	0 mL	Formula			
End Sens	200		$(D-B)*K*F*M/S$		
Over mL	0.2 mL	Decimal places	4		
Max Vol.	20 mL	Auto input parameter	None		

(2) Measurement for sodium ascorbate

Cndt No.	2	Constant No.	2	Mode No.	20
Method	Auto	Size	0.4042 g	Pre Int	0 sec
Buret No.	2	Blank	0 mL	Del K	2
Amp No.	2	Molarity	0.05 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.001	Int Time	3 sec
S-Timer	30 sec	K	176.13	Int Sens	3 mV
C.P. mL	7 mL	L	0	Brst Speed	2
T Timer	30 sec	Unit	mg/g	Pulse	40
D.P. mL	0.5 mL	Formula	(D-B)*K*F*M/S		
End Sens	500	Decimal places	4		
Over mL	0.2 mL	Auto input parameter	None		
Max Vol.	20 mL				
Cndt No.	3	Constant No.	3		
Method	Calc	Size	0.4042 g		
		Blank	0 mL		
		Molarity	0 mol/L		
		Factor	0		
		K	116.92 *1		
		L	1.125 *2		
		Unit	mg/g		
		Formula	(CA-K)*L		
		Decimal places	4		
		Auto input parameter	None		

*1 K (coefficient 1): Result of ascorbic acid by neutralization titration

*2 L (coefficient 2): Coefficient to convert ascorbic acid to sodium ascorbate ($C_6H_7NaO_6$ (198.11) / $C_6H_8O_6$ (176.13))

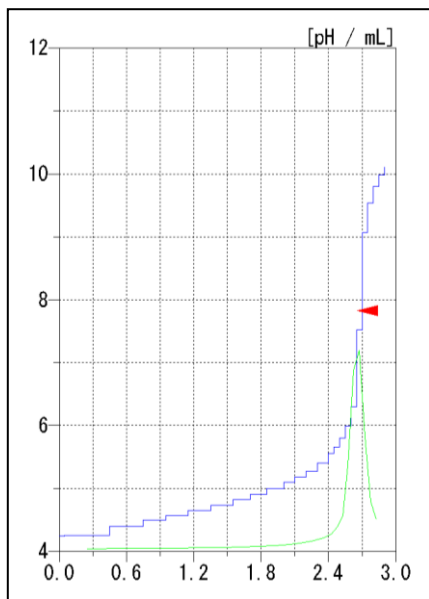
Measurement results

Measurement results of ascorbic acid

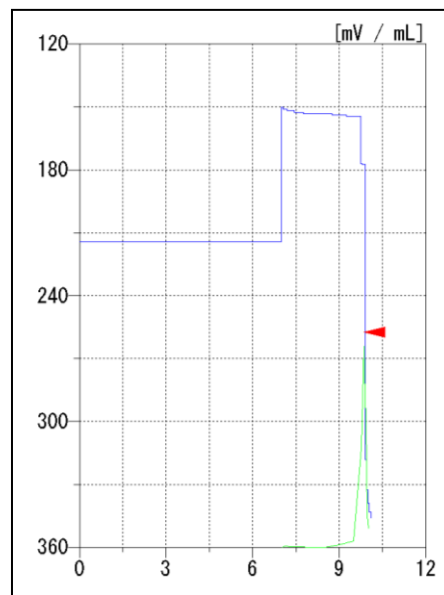
Meas. No.	Size (g)	Titrant volume (mL)	Ascorbic acid (mg/g)	Statistic calculation	
1	0.4036	2.671	117.028	Average	116.92 mg/g
2	0.4021	2.659	116.931	Standard deviation	0.12 mg/g
3	0.4055	2.678	116.796	Coefficient of variation	0.10 %

Measurement results of sodium ascorbate

Meas. No.	Size (g)	Titrant volume (mL)	Total ascorbic acid (mg/g)	Sodium ascorbate (mg/g)	Statistic calculation	
1	0.4042	9.858	215.001	110.341	Average	110.70 mg/g
2	0.4056	9.922	215.671	111.095	Standard deviation	0.38 mg/g
3	0.4064	9.925	215.275	110.649	Coefficient of variation	0.34 %



Measurement for ascorbic acid



Measurement for sodium ascorbate

Examples of titration curves

5. Note

Another determination method of ascorbic acid

The iodine titration method is used for the determination of ascorbic acid in this report, but there is an indophenol method as another determination method for ascorbic acid; the color change of indophenol from blue to red under metaphosphoric acid is detected as the end point of titration in this method. The relatively-high selectivity on the determination of ascorbic acid in fruit juice etc. compared with the iodine titration method is the character of the indophenol method.

Keywords: Fractional determination of ascorbic acid and sodium ascorbate, Neutralization titration, Redox titration, Iodine titration

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