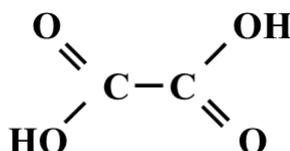


Organic acid

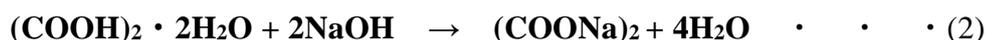
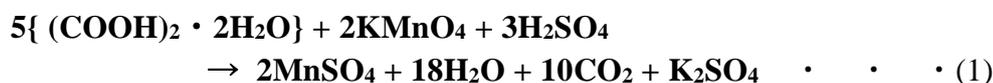
Purity determination of oxalic acid

1. Abstract

Oxalic acid has two carboxy groups (-COOH) in the molecule, it is called dicarboxylic acid. There are two crystallization water molecules in the molecule. Oxalic acid is ortho acid and forms the following structure. It is readily oxidized by the acid stronger than formic acid.



Oxalic acid is used as raw material of organic compound. In addition, sodium salt of oxalic acid is used as standard substance of quantitative analysis because it quantitatively reacts with oxidizing agent like potassium permanganate and is stably conservable. The determination method for oxalic acid is prescribed in JIS K8519, it is determined by potassium permanganate standard solution. This report introduces an example of the purity determination for oxalic acid using redox titration as described below (formula 1) and neutralization titration with sodium hydroxide titrant (formula 2).



2. Configuration of instruments and reagents

(1) Redox titration with potassium permanganate

(i) Configuration of instruments

Main unit	:	Hiranuma Automatic Titrator COM series
Electrodes	:	Platinum electrode PT-301
		Reference electrode RE-201Z

*Instead of the above electrodes, the following electrode is usable.

- Platinum-reference combination electrode PR-701BZ

(ii) Reagents

Titration	:	0.02 mol/L Potassium permanganate standard solution
Additive	:	Diluted sulfuric acid (1:1, [v/v])

(2) Neutralization titration with sodium hydroxide

(i) Configuration of instruments

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

*Instead of the above electrodes, the following electrodes are usable.

- Glass reference combination electrode GR-501BZ···Fixed sleeve type
- Glass reference combination electrode GR-511BZ···Moveable sleeve type

(ii) Reagent

Titrant	:	1 mol/L Sodium hydroxide standard solution
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3. Measurement procedure

(1) Redox titration with potassium permanganate

- Take 0.2 g of sample into a 300 mL beaker and weigh accurately with 0.1 mg digit.
- Add stirring bar and 200 mL of DI water and 20 mL of diluted sulfuric acid.
- Dispense 30 mL of 0.02 mol/L potassium permanganate with stirring solution.
- Heat the beaker at about 60 °C.
- Immerse electrodes and start titration with 0.02 mol/L potassium permanganate standard solution.
Perform the blank test in the same procedure without sample and procedure (iii).

(2) Neutralization titration with sodium hydroxide

- Take 1.0 g of sample into a 100 mL beaker and weigh accurately with 0.1 mg digit.
- Add stirring bar and 50 mL of DI water. Dissolve the sample by stirring.
- Immerse electrodes and start titration with 1 mol/L sodium hydroxide standard solution.

4. Measurement conditions and results

(1) Redox titration with potassium permanganate standard solution

Example of titration condition

(i) Measurement of blank

Cndt No.	1	ConstantNo.	1	Mode No.	18
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	2	Molarity	0.02 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.003	Int Time	5 sec
S-Timer	5 sec	K	0	Int Sens	5 mV
C.P. mL	0 mL	L	0	Brst Speed	2
T Timer	0 sec	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Sens	100	Decimal Places	3		
Over mL	0.3 mL	Auto In Pram.	Non		
Max. Vol.	20 mL				

(ii) Dispense 0.02 mol/L potassium permanganate standard solution.

Cndt No.	2
Method	Disp
Buret No.	1
S-Timer	5 sec
Disp Vol.	30 mL

(iii) Measurement of oxalic acid dihydrate

Cndt No.	3	ConstantNo.	3	Mode No.	22
Method	Auto	Size	0.2 g	Pre Int	0 sec
Buret No.	1	Blank	0.074 mL	Del K	0
Amp No.	2	Molarity	0.02 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.003	Int Time	5 sec
S-Timer	10 sec	K	126.07	Int Sens	5 mV
C.P. mL	0 mL	L	2.5	Brst Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	0.1 mL	Formula	$(D+30-B)*K*F*M*L/(S*10)$		
End Sens	500	Decimal Places	3		
Over mL	1.00 mL	Auto In Pram.	Non		
Max. Vol.	20 mL				

* Enter the blank value to "B", the molecular weight of oxalic acid dihydrate to "K", and the reaction ratio between oxalic acid dihydrate and potassium permanganate to "L".

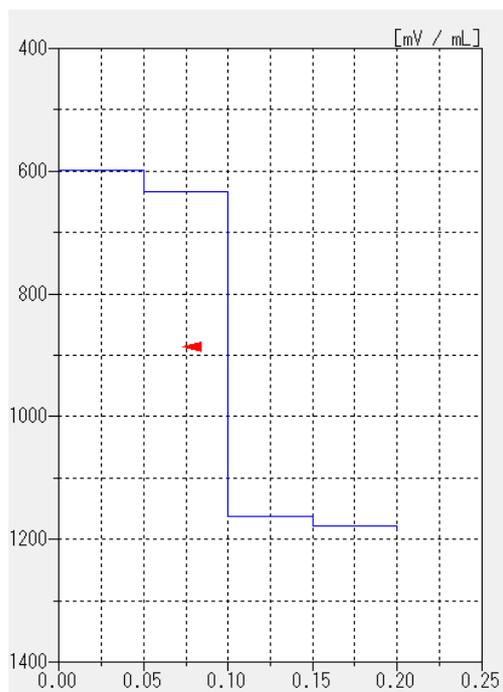
Measurement results

Measurement results of blank

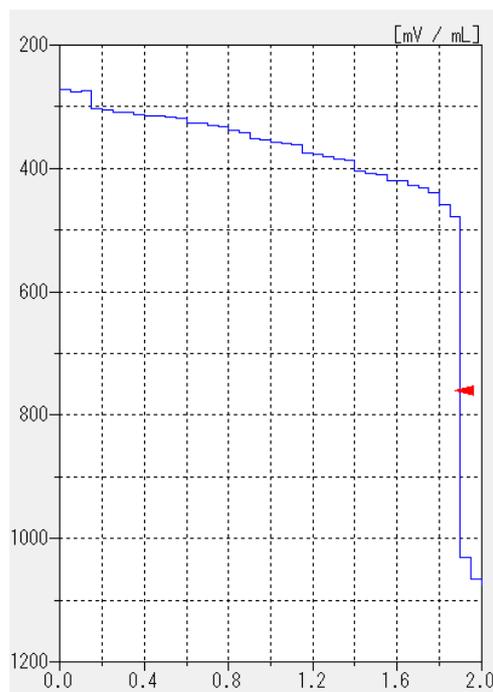
Number of Measurement	Size (g)	Titrant Volume (mL)
1	-	0.073
2	-	0.074
Avg. (Blank)		0.074 mL

Measurement results of sample

Number of Measurement	Size (g)	Titrant Volume (mL)	Purity (%)
1	0.2007	1.779	99.876
2	0.2017	1.976	99.999
3	0.2012	1.876	99.933
Statistic calculation		Avg.	99.94 %
		SD	0.0616 %
		RSD	0.0616 %



Measurement of blank



Measurement of sample

Examples of titration curves

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

(2) Neutralization titration with 1 mol/L sodium hydroxide standard solution

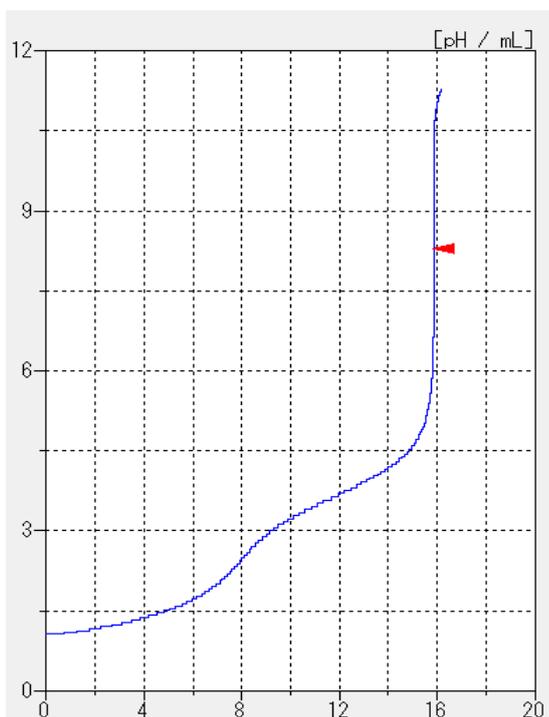
Example of titration condition

(i) Measurement of oxalic acid dihydrate

Cndt No.	4	ConstantNo.	4	Mode No.	8
Method	Auto	Size	1.000 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	1	Molarity	1.000 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	5 sec
S-Timer	5 sec	K	126.07	Int Sens	3 mV
C.P. mL	0 mL	L	2.0	BrT Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula	(D-B)*K*F*M/(S*10*L)		
End Sens	1000	Decimal Places	3		
Over mL	0.3 mL	Auto In Pram.	Non		
Max.Vol.	20 mL				

* Enter the molecular weight of oxalic acid dihydrate to “K”, and the valence of oxalic acid dihydrate to “L”.

Measurement results



Measurement Number	Size (g)	Titrant Volume (mL)	Purity (%)
1	1.0067	15.874	99.893
2	1.0024	15.822	99.993
3	1.0001	15.775	99.925
Statistic calculation	Avg.	99.94	%
	SD	0.0511	%
	RSD	0.0511	%

Example of titration curve

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

5. Note

(1) Measurement result

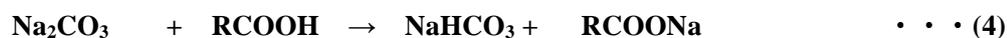
The purity of oxalic acid was determined by the following two methods: redox titration with potassium permanganate and neutralization titration with sodium hydroxide. There is no difference between the results on these methods, the titration was possible without problem in each method. The neutralization titration is particularly easy because of no pretreatment and the less waste solution.

(2) Collection of sample

The sample is collected directly to the beaker and weighed accurately. The accuracy of sample collection influences the measurement accuracy. Please note that the sample should be carefully taken and accurately weighed.

(3) Control of titrant

The concentrated sodium hydroxide standard solution is used as titrant in this report. The carbon dioxide gas absorber (soda lime) on reagent bottle has to be regularly exchanged because sodium hydroxide readily absorbs carbon dioxide gas in the air (formula (3)). The standard solution of sodium hydroxide that has absorbed carbon dioxide contains sodium carbonate, and the inflection point on titration curve may be unclear due to buffer capacity of sodium hydrogen carbonate generated in the reaction with an acidic sample (formula (4)).



Keywords: Oxalic acid, Potassium permanganate, Sodium hydroxide, Redox, Neutralization, JIS K 8519