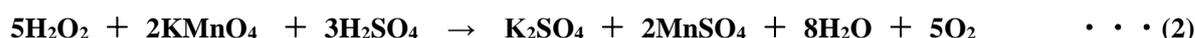


HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	E9	Oct. 7, 2022
PLATING & ETCHING SOLUTION	Determination of hydrogen peroxide and sulfuric acid in etching solution			

1. Abstract

Sulfuric acid and hydrogen peroxide are main components of the etching solution which is used for cleaning the copper surface (soft etching) or half etching. The concentration of sulfuric acid and hydrogen peroxide have to be properly controlled to maintain constant etching speed because hydrogen peroxide readily decomposes by itself. This report introduces a measurement example as below.

- 1) Determination of sulfuric acid by neutralization titration with sodium hydroxide . . . (1)
- 2) Determination of hydrogen peroxide by redox titration with potassium permanganate standard solution. . . (2)



2. Configuration of instruments and reagents

(1) Configuration

Main unit	:	Hiranuma Automatic Titrator COM series
Option	:	Buret, buret head, and simple dispenser (peristaltic type), one by one
Electrodes	:	Glass reference combination electrode GR-501BZ Connect to IE-1. Platinum electrode PT-301 Connect to IE-2.

(2) Reagents

Titrant	:	0.1 mol/L sodium hydroxide standard solution (for sulfuric acid titration) 0.02 mol/L potassium permanganate standard solution (for hydrogen peroxide titration)
Additive solution	:	10 mL of diluted sulfuric acid (1:9, v/v) (for pH adjustment)

3. Measurement procedure

- (1) Dispense 1 mL of sample into a 200 mL beaker with volumetric pipette.
- (2) Add about 100 mL of DI water.
- (3) Immerse electrodes and start titration with 0.1 mol/L sodium hydroxide standard solution.
- (4) After that, 10 mL of diluted sulfuric acid is automatically dispensed. (Option: Simple dispenser)
- (5) Titrate with 0.02 mol/L potassium permanganate standard solution. (Option: Buret)

4. Measurement conditions and results

Examples of titration conditions

(1) Titration of sulfuric acid

Cndt No.	1	Constant No.	1	Mode No.	20
Method	Auto	Size	1.0000 mL	Pre Int	0 sec
Buret No.	1	Blank	0.0000 mL	Del K	9
Amp No.	1	Molarity	0.1000 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.0040	Int Time	2 sec
S-Timer	10 sec	K	49.040	Int Sens	3 mV
C.P. mL	20 mL	L	0.000	Brt Speed	2
T Timer	15 sec	Unit	g/L	Pulse	40
D.P. mL	0.5 mL	Formula			0.05 mL
End Sens	1000				
Over mL	0.2 mL		(D-B)*K*F*M/S		
Max. Vol.	40 mL	Digits	4		
		Auto In Pram.	None		

(2) Dispensing diluted sulfuric acid

Cndt No.	2				
Method	Disp				
Buret No.	2				
S-Timer	0 sec				
Disp Vol.	10 mL				

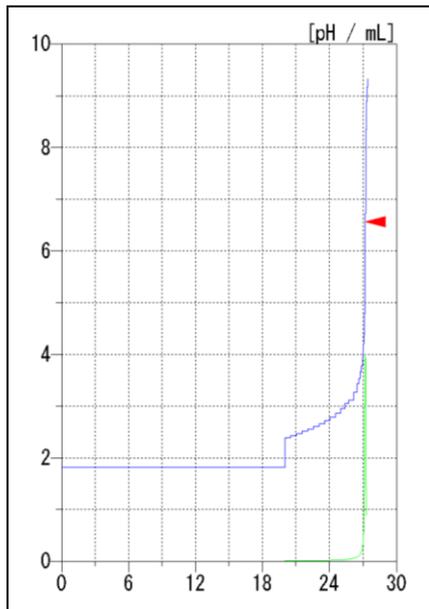
(3) Titration of hydrogen peroxide

Cndt No.	3	Constant No.	3	Mode No.	5
Method	Auto	Size	1.0000 g	Pre Int	0 sec
Buret No.	3	Blank	0.0000 mL	Del K	5
Amp No.	3	Molarity	0.0200 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.0050	Int Time	3 sec
S-Timer	10 sec	K	85.040	Int Sens	3 mV
C.P. mL	0.5 mL	L	0.000	Brt Speed	2
T Timer	60 sec	Unit	g/L	Pulse	40
D.P. mL	0.5 mL	Formula			0.05 mL
End Sens	1000				
Over mL	0.5 mL		(D-B)*K*F*M/S		
Max. Vol.	20 mL	Digits	4		
		Auto In Pram.	None		

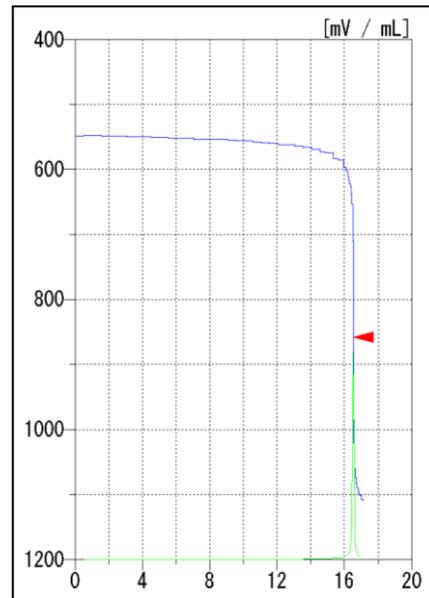
Measurement results

Number of Measurement	Size (mL)	Titrant volume (mL)	Sulfuric acid (g/L)	Titrant volume (mL)	Hydrogen peroxide (g/L)
1	1.0000	27.155	133.701	16.479	28.1676
2	1.0000	27.182	133.834	16.492	28.1898
3	1.0000	27.197	133.908	16.523	28.2428
		Avg.	133.81 g/L	Avg.	28.20 g/L
Statistic calculation		SD	0.10 g/L	SD	0.04 g/L
		RSD	0.08 %	RSD	0.14 %

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



Measurement of sulfuric acid



Measurement of hydrogen peroxide

Examples of titration curves

5. Note

Potassium permanganate standard solution is used for hydrogen peroxide titration in this report because copper ion (Cu^{2+}) coexists in the sample. If the sample does not contain copper ion (Cu^{2+}) or iron ion (Fe^{3+}) which oxidize iodine ion, the determination of hydrogen peroxide with the iodometry method using sodium thiosulfate titrant is also possible.

Keywords: Etching solution, Sulfuric acid, Neutralization titration, Hydrogen peroxide, Potassium permanganate