

<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	J1	Apr. 5, 2019
<b>Inorganic acids &amp; Mixed acids</b>	<b>Purity determination of sulfuric acid</b>			

## 1. Abstract

Sulfuric acid is one of the most elemental reagent in chemical industrial reagents. Its production amount is large and it is used in a broad range of fields. Since sulfuric acid absorbs moisture in atmosphere because of its properties, the purity of sulfuric acid is gradually reduced. “JIS (*Japanese Industrial Standards*) K8951: 2006 Sulfuric acid (Reagent)” describes the determination method for purity of sulfuric acid which it is titrated with sodium hydroxide standard solution until the color of bromothymol blue indicator changes from yellow to blue green at the endpoint. However, this report introduces an example of the potentiometric titration with sodium hydroxide for the measurement of the accurately-weighed sample.

The neutralization titration for sulfuric acid with sodium hydroxide proceeds as two step reaction because sulfuric acid is diacid. However, the first and second dissociation of sulfuric acid cannot be discriminated due to the leveling effect of water. Therefore the single inflection point is observed on the titration curve and the reaction formula on this measurement can be described as below.



## 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

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

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

### (2) Reagents

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

- (1) Take about 1 g of sample into a 100 mL beaker and weigh accurately.
- (2) Gently add 40 mL of DI water with the 100 mL beaker iced.
- (3) Immerse electrodes and start titration with 1 mol/L sodium hydroxide standard solution. Perform the blank test with the same procedure of sample measurement.

## 4. Measurement conditions and results

### Example of titration condition

#### Measurement of blank

Cndt No.	1	ConstantNo.	1	Mode No.	14
Method	Auto	Size	0 mL	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	0
Amp No.	1	Molarity	1.000 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	3 sec
S-Timer	20 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brt Speed	2
T Timer	0 sec	Unit	mL	Pulse	8
D.P. mL	0 mL	Formula	D		
End Sens	1000	Decimal Places	3		
Over mL	0.1 mL	Auto In Pram.	Non		
Max.Vol.	1 mL				

#### Measurement of sample

Cndt No.	2	ConstantNo.	2	Mode No.	1
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0.0150 mL	Del K	9
Amp No.	1	Molarity	1.000 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	1 sec
S-Timer	20 sec	K	49.04	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brt Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula			
End Sens	1000				
Over mL	0.2 mL		$(D-B)*K*F*M/(S*10)$		
Max.Vol.	40 mL	Decimal Places	3		
		Auto In Pram.	Non		

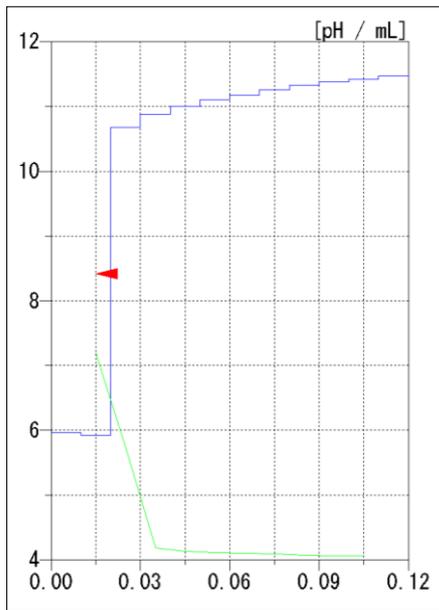
### Measurement results

#### Measurement results of blank

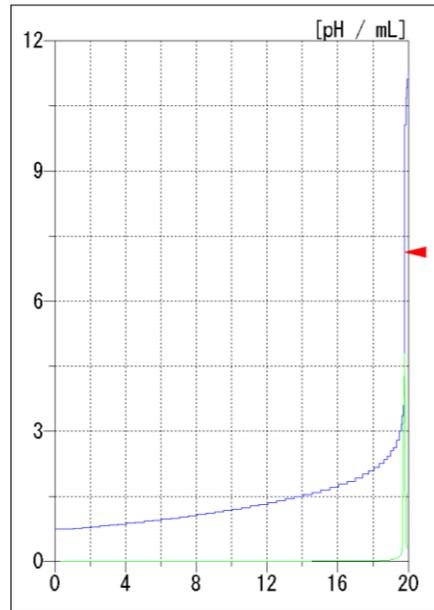
Number of Measurement	Size (g)	Titrant Volume (mL)
1	-	0.015
2	-	0.015
Avg. (Blank)		0.015 mL

#### Measurement results of sample

Number of Measurement	Size (g)	Titrant Volume (mL)	Concentration (%)
1	0.9868	19.325	96.443
2	1.0198	19.975	96.463
3	1.0098	19.775	96.442
		Avg.	96.45 %
Statistic calculation		SD	0.01 %
		RSD	0.01 %



Measurement of blank



Measurement of sample

Examples of titration curves

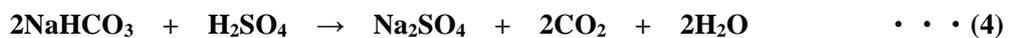
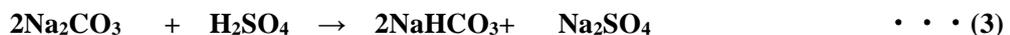
## 5. Note

### (1) Collection of sample

The sample should be collected directly to 100 mL 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.

### (2) 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 (2)). The titrant contains sodium carbonate if it absorbed carbon dioxide gas. When using this titrant, the titration curve would show the inflection points at pH around 4 and 9 (formula (3) and (4)).



Keywords: Sulfuric acid, Neutralization titration, Purity, JIS K8951