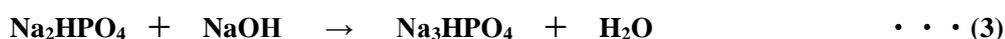
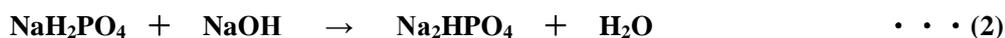


HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	J2	Apr. 5, 2019
Inorganic acids & Mixed acids	Purity determination of phosphoric acid			

1. Abstract

Phosphoric acid is important reagent as a raw material of chemical industrial reagents and fertilizers. Its production amount is large and it is used in a broad range of fields. The determination method of phosphoric acid purity is described in “JIS (Japanese Industrial Standards) K9005:2006 Phosphoric acid (reagent)”; the purity of phosphoric acid is determined by potentiometric titration with sodium hydroxide standard solution. This report introduces an example of the purity determination of phosphoric acid by this method.

Phosphoric acid reacts with sodium hydroxide as described in the reaction formula (1) ~ (3) because phosphoric acid is triacid. The titration curve of neutralization titration for phosphoric acid theoretically has three inflection points, but practically it shows two inflection points. This is because third acid dissociation constant (pK_a) of phosphoric acid is 12.35, the pH at the third inflection point is undetectable value by the titration with strongly basic titrant. The purity of phosphoric acid is determined by the titration to the second inflection point in this measurement.



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

Titant	:	1 mol/L Sodium hydroxide standard solution
--------	---	--

3. Measurement procedure

- (1) Take about 1.5 g of sample into a 100 mL beaker and weigh accurately.
- (2) Add 40 mL of DI water without carbon dioxide.
- (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

Examples of titration conditions

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	5 sec
S-Timer	10 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	250	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.	4
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	3 sec
S-Timer	10 sec	K	49.000 *	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	20 mL	Formula	(D-B)*K*F*M/(S*10)		
End Sens	250	Decimal Places	3		
Over mL	0.2 mL	Auto In Pram.	Non		
Max.Vol.	40 mL				

*"49 (Parameter K)" comes from "98 (H₃PO₄)/2" because titrant volume will be twice according to reaction (1) and (2).

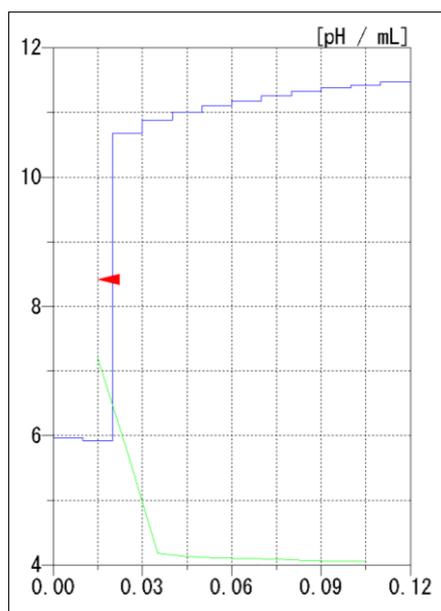
Measurement results

Measurement results of blank

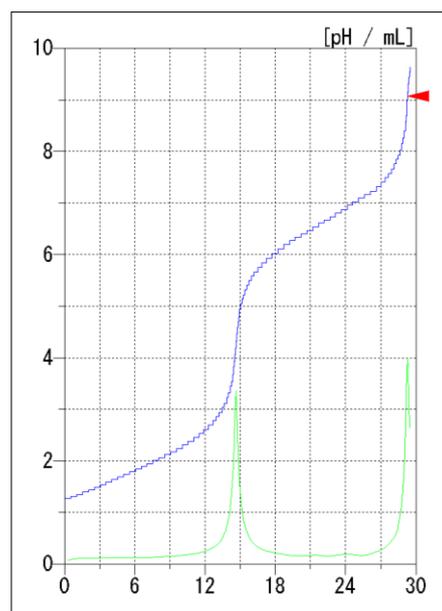
Number of Measurement	Size (g)	Titrant Volume (mL)
1	-	0.015
2	-	0.015
Avg. (Blank)		0.015 mL

Measurement results of sample

Number of Measurement	Size (g)	Titrant Volume (mL)	Concentration (%)
1	1.6693	29.094	85.784
2	1.6743	29.142	85.669
3	1.6797	29.277	85.790
Statistic calculation		Avg.	85.75 %
		SD	0.07 %
		RSD	0.08 %



Measurement of blank



Measurement of sample

Examples of titration curves

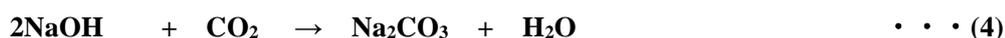
5. Note

(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 (4)).



(3) Endpoint detection and calculation of concentration

The titration curve with strong base (sodium hydroxide) shows two inflection points as described in the abstract. The first inflection point at around pH 4 is not detected (“D.P. mL” is set to “20 mL”) and the second inflection point is detected as endpoint. The titrant volume at second inflection point is used for the calculation of concentration.

(4) Reduction of measurement time

The titrant volume was 25 mL or more, the measurement time was about 8.5 min on this measurement. The sodium hydroxide standard solution can be added continuously up to 20 mL by the function “C.P. mL”, it reduces the measurement time. The measurement time was about 4.5 min when using the function “C.P. mL”.

Keywords: Phosphoric acid, Neutralization titration, Purity