AQUACOUNTER Application Sheet	COM series	DATA No. J2	1st edition

Inorganic Acid — Measurement of phosphoric acid purity

1. Measurement outline

Phosphoric acid is an important chemical as a raw material for industrial chemicals and as a raw material for fertilizers, and it has a wide range of field for use as well as a large production volume. The method for measuring the purity of phosphoric acid is stipulated in JIS K 9005 as a titration method by indicator with end point at pH8.8. The main impurity in phosphoric acid is moisture.

This section introduces an example in which the sample was weighed and collected precisely for measurement by potentiometric titration with sodium hydroxide titrant.

Though phosphoric acid should give a titration curve with 3 inflection points since it is a tribasic acid, its titration curve shows only 2 inflection points in aqueous solution. This is because the dissociation constant for the third step (12.36) shows a pH value that cannot be titrated with strong base.

$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O$ ---------- (1)

$NaH_2PO_4 + NaOH \rightarrow Na_2HPO_4 + H_2O$ ---------- (2)

$Na_2HPO_4 + NaOH \rightarrow Na_3PO_4 + H_2O$ ---------- (3)

2. Reagents and Electrodes

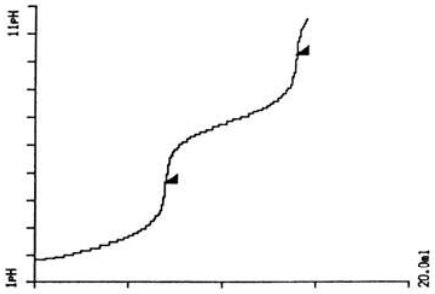
(1) Reagents	Titrant	1mol/L sodium hydroxide titrant
(2) Electrodes *standard accessories	Indicator electrode	*Glass electrode GE-101B to IE jack
	Reference electrode	*Reference electrode RE-201 to RE jack

3. Measurement conditions example (for COM-1600S)

Master File No.1 Condition file: 1+ 2	
Parameters for Condition file 1, 2	
Method	AUTO
Amp No.	1
Buret No.	1
Meas Unit	pH
S-Timer	10 sec / 0 sec (for file 2)
CP	0 mL
DP	0 mL
Direction	N/A
End Sens	250
Over mL	0 mL
Max Vol	20 mL
Mode No.	4
Unit	ML
Formula	(D-B)×K×F×M/(S×10)
Blank	0
Molarity	1
Factor	Titer of titrant
K	98.00001

Mode No.4	
Pre Int	0 sec
Del K	9
Del Sens	0 mV
Int Time	3 sec
Int Sens	3 mV
Brt Speed	2
Pulse	40

4. Measurement example



Measurement results on phosphoric acid purity

Sample No.	Sample volume (g)	Titration value (mL)	Concentration (%)
1	0.8257	14.527	85.706
2	0.8006	13.833	85.764
3	0.7913	13.669	85.743
	Avg.		85.737 %
	Std. Dev.		0.029 %
	C.V.		0.034 %

5. Outline

(1) About collection of the sample

This section adopted the sample collection method to collect approximately 0.6g directly into a 100mL beaker and weigh. Caution is required that the sample weighing precision affects the measurement precision greatly in this measurement.

(2) About control of the titrant

A high-concentration sodium hydroxide titrant is used as the titrant for this measurement. Since sodium hydroxide tends to absorb carbon dioxide gas in air (Formula 2), it is important that the carbon dioxide gas absorbent (soda lime) in the reagent bottle be replaced regularly. Titrant that has absorbed carbon dioxide gas contains sodium carbonate and delivers a titration curve that shows inflection points at around pH 4 and pH9.5 (Formulae 3 and 4).

$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$ ———— (2)

$2Na_2CO_3 + H_2SO_4 \rightarrow 2NaHCO_3 + Na_2SO_4$ ———— (3)

$2NaHCO_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2CO_2 + 2H_2O$ ———— (4)

(3) About titration end point detection and concentration calculation

As shown in the previous titration example, the titration curve for phosphoric acid by strong base (sodium hydroxide) has 2 inflection points. Though the titration value for each of these 2 inflection points should be equal theoretically, the titration value for the first step shows a slightly smaller value in actual measurement. For the results of this measurement, concentration was calculated using the titration value to the second step according to JIS.

Key words

Measurement of phosphoric acid purity, neutralization titration

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