

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	J11	Feb. 10, 2021
Inorganic acids & Mixed acids	Fractional determination of nitric acid and phosphoric acid			

1. Abstract

The mixed solution of nitric acid and phosphoric acid works as strong acid, and also has the strong oxidizability and solvency. It is used as the surface treatment solution for metals, glass products, and semiconductors.

This report introduces an example of the fractional and successive determination for nitric acid and phosphoric acid by neutralization titration with sodium hydroxide standard solution. When nitric acid and phosphoric acid are titrated by neutralization titration using a glass electrode for pH measurement, two inflection points appear on the titration curve. The first inflection point appears as the total amount of nitric acid and phosphoric acid (reaction formula (1) and (2)). Sodium dihydrogen phosphate, which is the product of reaction equation (2), subsequently reacts with sodium hydroxide and shows an inflection point in the second stage (reaction equation (3)). Therefore, it is possible to obtain the phosphoric acid concentration from the second inflection point, and nitric acid concentration could be obtained with subtracting titration volume at the second inflection point from the first inflection point.



2. Configuration of instruments and reagents

(1) Configuration of instruments

Main unit	:	Automatic Titrator COM series
Electrodes	:	Glass electrode GE-101B (Connect to IE-1)
	:	Reference electrode RE-201Z (Connect to RE-1)

(2) Reagent

Titrant	:	1 mol/L Sodium hydroxide standard solution (For volumetric analysis)
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3. Measurement procedure

- (1) Take 1 mL of sample into a 100 mL beaker with a micropipette and accurately weigh it.
- (2) Add 50 mL of DI water and a stirrer bar.
- (3) Immerse electrodes and titrate with 1 mol/L sodium hydroxide standard solution until second inflection point is detected.

4. Measurement conditions and results

Examples of titration conditions

(1) Titration condition for total acid

Cndt No.	6	ConstantNo.	6	Mode No.	4
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	9
Amp No.	1	Molarity	1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	3 sec
S-Timer	0 sec	K	63	Int Sens	3 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	500	Digits	3		
Over mL	0 mL				
Max.Vol.	40 mL				

(2) Titration condition for phosphoric acid

Cndt No.	7	ConstantNo.	7	Mode No.	8
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	1	Molarity	1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.005	Int Time	5 sec
S-Timer	0 sec	K	98	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brst Speed	2
T Timer	0 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula	(VB-B)*K*F*M/(S*10)		
End Sens	500	Digits	3		
Over mL	0.3 mL				
Max.Vol.	20 mL				

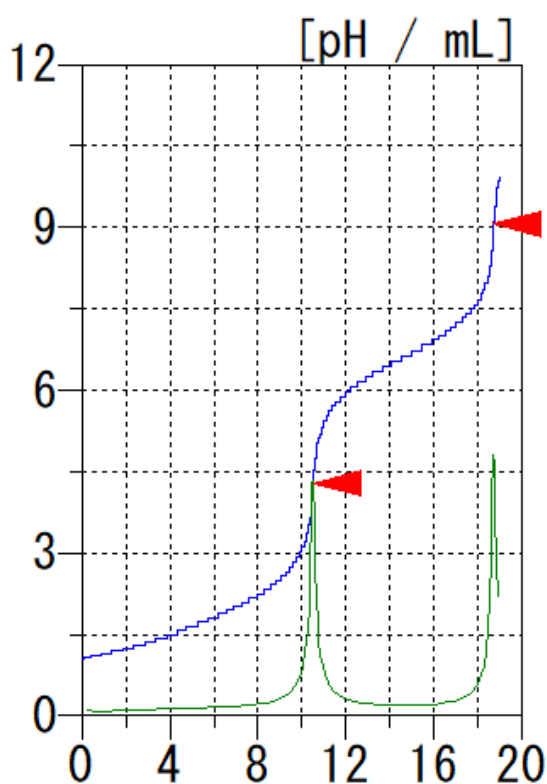
(3) Calculation for nitric acid

Cndt No.	8	ConstantNo.	8
Method	Calc	Size	0 g
		Blank	0 mL
		Molarity	1 mol/L
		Factor	1.005
		K	63.01
		L	0
		Unit	%
		Formula	(VA-VB)*K*F*M/(S*10) ※
		Digits	3

※ If the titrator is a model manufactured before 2009 (COM-1600, etc.), set (x, y) instead of the coefficient (VA, VB) in the formula. The calculation function is the same.

Measurement results

Number of measurement	Sample size (g)	Total acid	Phosphoric acid		Nitric acid	
		Titrant volume (mL)	Titrant volume (mL)	Conc. (%)	Conc. (%)	
1	1.4508	10.501	8.243	55.959	9.9018	
2	1.4511	10.554	8.294	56.294	9.7836	
3	1.4609	10.562	8.298	55.943	9.7890	
				Avg.	56.07 %	9.82 %
				SD	0.20 %	0.07 %
				RSD	0.4 %	0.7 %



Example of titration curve

5. Note

If the sample contains metal ions, the formation of these hydroxide salts may consume the titrant.

Keywords : Nitric acid, Phosphoric acid, Fractional titration, Potentiometric titration, Neutralization titration