

# Titration of a Known Acid Using a pH Meter to Determine the Endpoint of Titration

## Introduction

Up to this point in your chemistry career, you have utilized visual endpoints to know when to stop titrating. In this experiment, you will use a simple pH meter to determine the end point of titrating a monoprotic acid.

The technique to accomplish this is similar to how you completed titrating an antacid and determining the equivalent weight of an unknown acid. The difference is that a pH electrode instead of an acid-base indicator is used to collect data so that the endpoint may be calculated at a later date.

## Applied Theory

How does one determine the endpoint of a titration with a pH meter? Using the data in the following table, this technique becomes fairly simple.

Volume base added	pH	Volume base added	pH
0.00	3.04	39.50	9.60
4.00	3.50	39.60	10.00
8.00	3.70	39.70	10.25
10.00	3.92	39.90	10.45
14.00	4.10	40.50	10.80
18.00	4.25	41.00	10.95
22.00	4.40	43.00	11.25
26.00	4.60	45.00	11.40
30.00	4.80	47.00	11.49
34.50	5.10	48.50	11.58
37.00	5.50	50.00	11.62
38.45	6.00		
39.00	6.50		
39.30	8.40		
39.40	9.15		

The first step is to calculate the change in volume ( $\Delta V$ ) between each pair of data values (in order from top to bottom, then left to right in columns 1 and 3, below):

$\Delta V$		$\Delta V$	
4.00-0.00 = 4.00 mL		39.60-39.50 = 0.10 mL	
8.00-4.00 = 4.00 mL		39.70-39.60 = 0.10 mL	
10.00-8.00 = 2.00 mL		39.90-39.70 = 0.20 mL	
14.00-10.00 = 4.00 mL		40.50-39.90 = 0.60 mL	
18.00-14.00 = 4.00 mL		41.00-40.50 = 0.50 mL	
22.00-18.00 = 4.00 mL		43.00-41.00 = 2.00 mL	
26.00-22.00 = 4.00 mL		45.00-43.00 = 2.00 mL	
30.00-26.00 = 4.00 mL		47.00-45.00 = 2.00 mL	
34.50-30.00 = 4.50 mL		48.50-47.00 = 1.50 mL	
37.00-34.50 = 2.50 mL		50.00-48.50 = 1.50 mL	
38.45-37.00 = 1.45 mL			
39.00-38.45 = 1.45 mL			
39.30-39.00 = 0.30 mL*			
39.40-39.30 = 0.10 mL			
39.50-39.40 = 0.10 mL			

Step 2 consists of calculating the  $\Delta$  pH between each pair of data values. These operations/values are placed in columns 2 and 4 of the following table:

$\Delta V$	$\Delta$ pH	$\Delta V$	$\Delta$ pH
4.00-0.00 = 4.00 mL	3.50-3.04 = 0.46	39.60-39.50 = 0.10 mL	10.00-9.60 = 0.40
8.00-4.00 = 4.00 mL	3.70-3.50 = 0.20	39.70-39.60 = 0.10 mL	10.25-10.00 = 0.25
10.00-8.00 = 2.00 mL	3.92-3.70 = 0.22	39.90-39.70 = 0.20 mL	10.45-10.25 = 0.20
14.00-10.00 = 4.00 mL	4.10-3.92 = 0.18	40.50-39.90 = 0.60 mL	10.80-10.45 = 0.35
18.00-14.00 = 4.00 mL	4.25-4.10 = 0.15	41.00-40.50 = 0.50 mL	10.95-10.80 = 0.15
22.00-18.00 = 4.00 mL	4.40-4.25 = 0.15	43.00-41.00 = 2.00 mL	11.25-10.95 = 0.30
26.00-22.00 = 4.00 mL	4.60-4.40 = 0.20	45.00-43.00 = 2.00 mL	11.40-11.25 = 0.15
30.00-26.00 = 4.00 mL	4.80-4.60 = 0.20	47.00-45.00 = 2.00 mL	11.49-11.40 = 0.09
34.50-30.00 = 4.50 mL	5.10-4.80 = 0.30	48.50-47.00 = 1.50 mL	11.58-11.49 = 0.09
37.00-34.50 = 2.50 mL	5.50-5.10 = 0.40	50.00-48.50 = 1.50 mL	11.62-11.58 = 0.04
38.45-37.00 = 1.45 mL	6.00-5.50 = 0.50		
39.00-38.45 = 1.45 mL	6.50-6.00 = 0.50		
39.30-39.00 = 0.30 mL*	8.40-6.50 = 1.90		
39.40-39.30 = 0.10 mL	9.15-8.40 = 0.75		
39.50-39.40 = 0.10 mL	9.60-9.15 = 0.45		

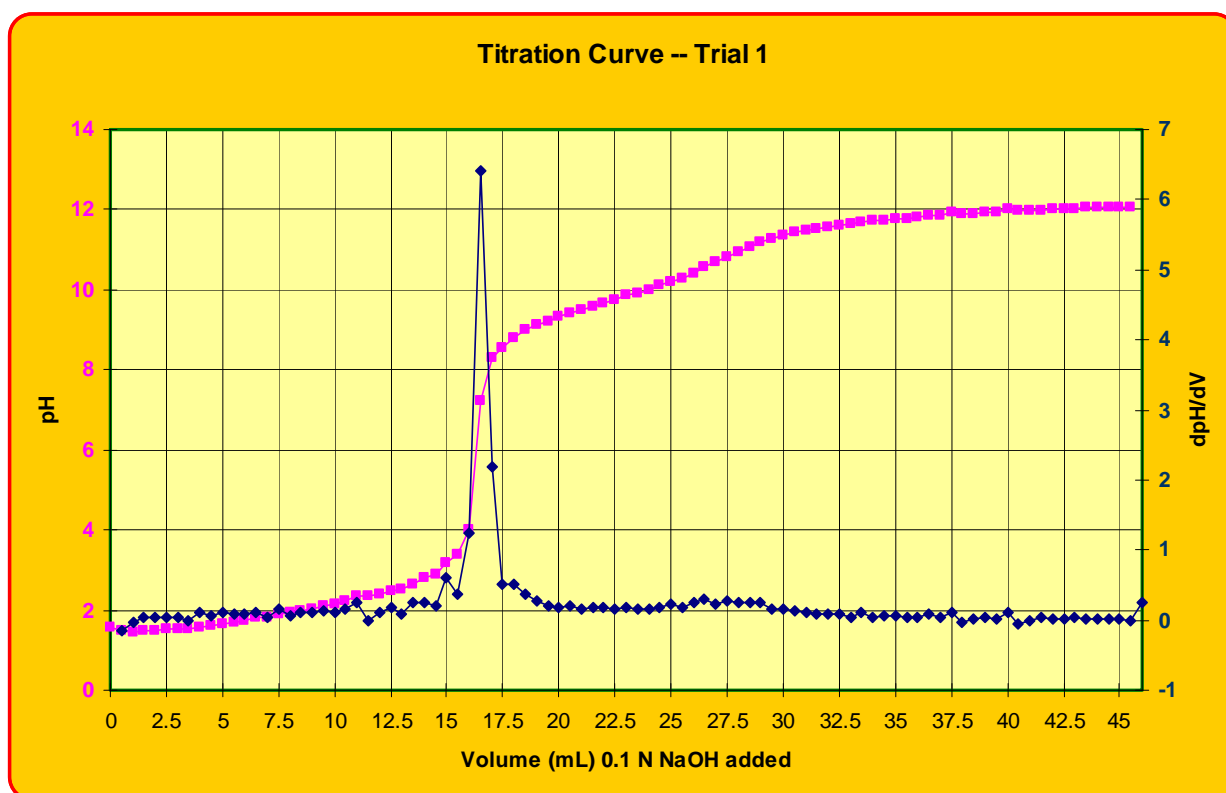
Step three: divide the  $\Delta \text{pH}$  by the  $\Delta V$  for each data pair (in sequential order):

$\Delta \text{pH}/\Delta V$	$\Delta \text{pH}/\Delta V$	$\Delta \text{pH}/\Delta V$	$\Delta \text{pH}/\Delta V$
$0.46/4.00 = 0.115$	$0.30/4.50 = 0.067$	$0.40/0.10 = 4$	$0.09/2 = 0.045$
$0.20/4.00 = 0.05$	$0.40/2.5 = 0.16$	$0.25/0.10 = 2.5$	$0.09/1.50 = 0.06$
$0.22/2.00 = 0.11$	$0.50/1.45 = 0.34$	$0.20/0.20 = 1$	$0.04/1.50 = 0.027$
$0.18/4.00 = 0.045$	$0.50/0.55 = 0.91$	$0.35/0.60 = 0.58$	
$0.15/4.00 = 0.375$	$1.90/0.30 = 6.33$	$0.15/0.5 = 0.3$	
$0.20/4.00 = 0.05$	$0.75/0.1 = 7.5$	$0.30/2 = 0.15$	
$0.20/4.00 = 0.05$	$0.45/0.10 = 4.5$	$0.15/2 = 0.075$	

Step four (4) is to align the data so that  $\Delta \text{pH}/\Delta V$  is on the "Y" axis and the volume of base added is on the "X" axis:

Volume base added	$\Delta \text{pH}/\Delta V$	Volume base added	$\Delta \text{pH}/\Delta V$
4.00	0.115	39.40	7.5
8.00	0.05	39.50	4.5
10.00	0.11	39.60	4
14.00	0.045	39.70	2.5
18.00	0.375	39.90	1
22.00	0.375	40.50	0.58
26.00	0.05	41.00	0.3
30.00	0.05	43.00	0.15
34.50	0.067	45.00	0.075
37.00	0.16	47.00	0.045
38.45	0.34	48.50	0.06
39.00	0.91	50.00	0.027
39.30*	6.33		
<b>Equivalent point</b>			

Step five (5): Plot the data using Lotus 123, QuattroPro or Excel or whatever spreadsheet program you have that you know how to use:



Step 6: Analyze your data. To obtain the actual pH at the potentiometric endpoint/equivalence point, read the **first derivative curve** from the volume of base added (16.50 mL in the graphed example, above) up to where that point intersects the **"S" shaped curve** (called a SIGMOID curve), then read to the left on the pH axis (7.21 in the graphed example, above). Where that line intersects is the actual equivalence point. Take the volume of base added to reach the equivalence point and divide it by 2. Go to that new volume (one half the equivalent point volume; 8.25 mL in the graphed example) on the sigmoid-shaped curve, read that new volume UP to the **"S" shaped curve** curve, then read to the left on the pH axis. This value is the  $pK_a$  of the acid under titration (remember Henderson-Hasselbach) and is roughly 1.95, or so, in the graphed example, above.

#### Materials and Methods

For this experiment, obtain the following supplies:

1 - buret	1 - ring stand	1 - pH checker	0.1 N NaOH
1 - buret clamp	1 - 3 125 mL Erlenmeyer flasks	3 - 10 mL aliquots of 0.1 N HOAc	Phenolphthalein

Assemble the ring stand with the buret clamp. Wash and rinse the buret with soap and water. Rinse the buret with a small amount of 0.1 N NaOH as you've done in the other two titration experiments. Fill your buret with 0.1 N NaOH as you've done in the past. Place 2-3 drops phenolphthalein into the 0.1 N HOAc aliquots.

ASIDE: The phenolphthalein is IMPORTANT! As you get closer to the visual endpoint, you will use smaller increments of base so that you may accurately determine the equivalence point of HOAc. The phenolphthalein will alert you to this by changing pink for longer periods of time before you reach the endpoint, allowing you to slow your titrations down CONSIDERABLY.

END OF ASIDE

Insert the pH checker into the 0.1 N HOAc solution in the Erlenmeyer flask as your instructor demonstrates. Record the initial volume of 0.1 N NaOH and the pH of the 0.1 N HOAc. Swirl the acid-base mixture as your instructor shows you. Record the pH (out to EVERY number on the LED) at the volumes of base added, below, in triplicate in the data tables provided.

Trial 1			
Volume Base Added (mL)	pH of Solution	Volume Base Added (mL)	pH of Solution
0.00		-----	
0.50		8.00	
1.00		8.50	
1.50		9.00	
2.00		9.50	
2.50		10.00	
3.00		10.50	
3.50		11.00	
4.00		11.50	
4.50		12.00	
5.00		12.50	
5.50		13.00	
6.00		13.50	
6.50		14.00	
7.00		14.50	
7.50		15.00	
Trial 2			
Volume Base Added (mL)	pH of Solution	Volume Base Added (mL)	pH of Solution
0.00		-----	
0.50		8.00	
1.00		8.50	
1.50		9.00	
2.00		9.50	
2.50		10.00	
3.00		10.50	
3.50		11.00	
4.00		11.50	
4.50		12.00	
5.00		12.50	
5.50		13.00	
6.00		13.50	
6.50		14.00	
7.00		14.50	
7.50		15.00	

Trial 3			
Volume Base Added (mL)	pH of Solution	Volume Base Added (mL)	pH of Solution
0.00		-----	
0.50		8.00	
1.00		8.50	
1.50		9.00	
2.00		9.50	
2.50		10.00	
3.00		10.50	
3.50		11.00	
4.00		11.50	
4.50		12.00	
5.00		12.50	
5.50		13.00	
6.00		13.50	
6.50		14.00	
7.00		14.50	
7.50		15.00	

When you've recorded all your data, discard your 0.1 N NaOH as instructed, your HOAc-NaOH mix as instructed, rinse your buret with water and place in the buret cleaner, tip up and valve open. Rinse your glass ware and place it in the dishwasher at the front of the lab.

#### Questions/Problem Set

1. Explain why phenolphthalein is a good indicator for the titration of HCl with NaOH, but not for NaOH with HCl.
2. The pK for formic acid is 3.75. In what molar ratio must formic acid and sodium formate be mixed to give a buffer of pH = 4.35?
3. The pK of HOAc is 4.75. How many grams of sodium acetate must be added to 200 mL of 0.100 N HOAc to give a buffer of pH = 4.40? Assume that volume changes are negligible.
4. If arterial blood has a pH of 7.41 and changes to 7.37, calculate the change that takes place in the bicarbonate/carbonic acid ratio. The pK of carbonic acid is 6.35.
5. If arterial blood has a pH of 7.40 and is 0.0240 M in bicarbonate ion, calculate the molar concentration of carbonic acid.
6. A plasma sample had a pCO<sub>2</sub> of 28 mm Hg and a bicarbonate concentration of 15mM. Calculate the pH of the plasma sample. HINT: look at the answer key, first.
7. A patient swallowed 10 grams of ammonium chloride and after 1 hour, the blood had a pH of 7.35. Calculate the mole ratio of bicarbonate/carbonic acid in his blood.
8. Aspirin can be determined by hydrolyzing the aspirin with a known amount of excess base (boiling for 10 minutes) and then titrating the remaining base with acid (kind of like the antacid titration experiment) If the sample weighed 0.2745 g, 50 mL of 0.1 N NaOH is used and 11.03 mL of 0.21 N HCl is required for the excess base (phenol red is usually used), calculate the percent purity of this sample.

#### Answer Keys for Problems

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## References

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