DETERMINATION OF AN ACID BY TITRATION & POTENTIOMETRY

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CHEM 334, Section L04

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Titration

- Volumetric analysis
  - $\mathrm{V_{titrant}M_{titrant} = N_{titrant} = n_{stoichiometry}N_{analyte}}$
- Titrant
  - 0.1M NaOH
- Two unknowns
  - $KHC_8H_4O_{4(aq)} + NaOH_{(aq)} \rightarrow KNaC_8H_4O_{4(aq)} + H_2O_{(l)}$
  - $H_3PO_4 + 2NaOH \rightarrow Na_2HPO_4 + 2H_2O$
- Titrating weak acids with a strong base

Potentiometry

- Use of electrodes to measure voltages that provide chemical information
- Indicator electrode
  - Transfers electrons to or from analyte
  - Variable potential
- Reference electrode
  - Fixed composition
  - Constant potential
- Difference between electrodes under zero current flow = cell voltage

Instrumentation

- pH meter
  - Glass electrode
  - Ion-selective electrode
- Two reference electrodes measure electric potential difference across glass membrane
- Rinse and blot between measurements
- Store in aqueous solution to prevent dehydration of glass

J. Diverdi, CHEM 334 Quantitative Analysis Laboratory Handout: Determination of an Acid by Titration & Potentiometry, 2012.

Protocol

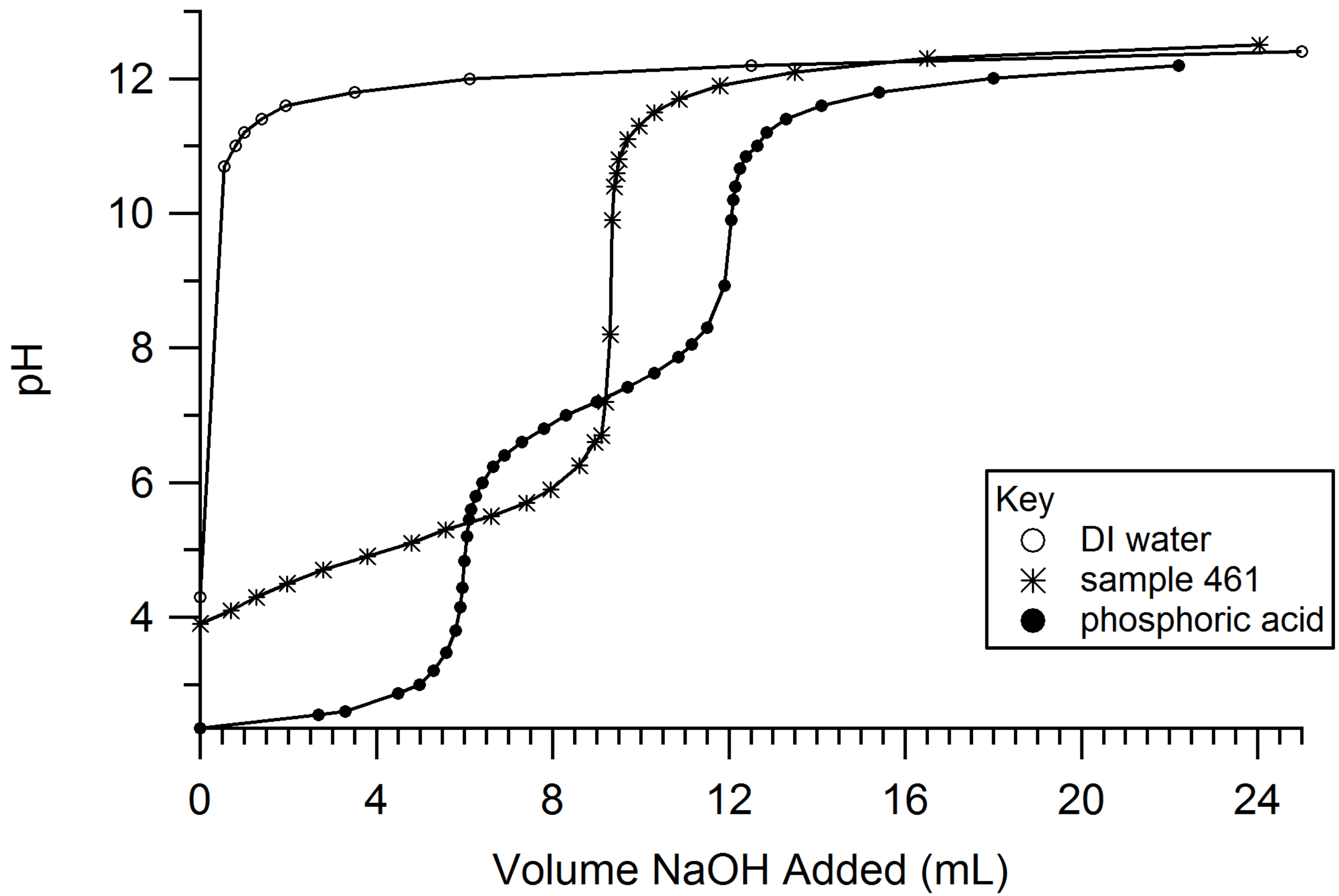
- Prepare NaOH solution
  - 50mL of 1M NaOH stock + 450mL DI water
- Standardize NaOH solution against dried KHP with phenolphthalein (3x)
- Calibrate pH meter by immersing into buffer solutions
- Prepare blank and analyte solutions
  - 50mL DI water
  - 0.35g unknown 461 + 50mL DI water
  - 0.5mL of ~1M phosphoric acid + 50mL DI water
- Titrate with pH meter and stir plate
  - Record data points every 0.2 change in pH

NaOH Standardization

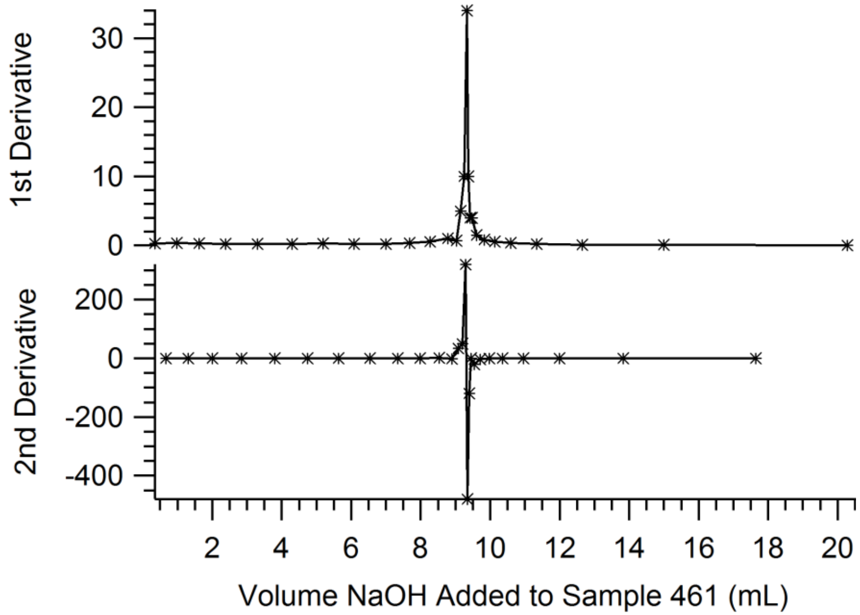
- NaOH is not pure
  - $OH^- + CO_2 \rightarrow HCO_3^-$
  - Absorbed water
- Necessitates use of primary standard KHP
  - High purity
  - Low reactivity
  - Low hygroscopicity

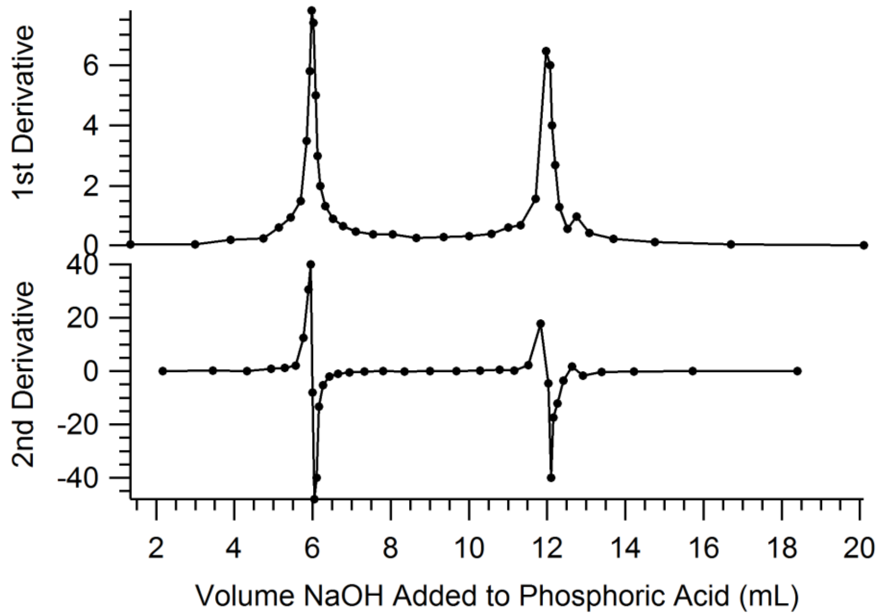
NaOH Solution Concentration			
Average Concentration (M)	Propagated Error (M)	95% C.I.	SD (M)
0.1029	0.0003	±0.0004	0.0002

Titration Curves



1st and 2nd Derivatives





	Added NaOH at End Points		
	Sample 461	H_3PO_4 1	H_3PO_4 2
Graphically derived (mL)	9.33	6.00	12.06
Numerically derived (mL)	9.31	6.00	11.99

Concentration of Sample 461	
Weight %	Error (%)
51.53	0.18

Concentration of H_3PO_4	
Concentration (M)	Error (M)
1.236	0.025

Discussion

- Advantages to potentiometric titration
  - Elimination of indicators and associated human error
  - Easily automated
- Disadvantages
  - Potentially less accessible than colorimetric titration
  - Time consuming
  - Susceptible to pH meter dysfunction