The Determination of Water

A Spectrophotometric Titration

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Common Problem & Need

General Chapters: <921> WATER DETERMINATION - first page
www.pharmacopeia.cn/v29240/usp29nf24s0_c921.html
Many Pharmacopeial articles either are hydrates or contain **water** in adsorbed form. As a result, the **determination** of the **water** content is important in ...

Introduction-Methods for Determination of Water - Analytical Chemistry ...
pubs.acs.org/doi/abs/10.1021/ac60056a002
by JW Stillman - 1951 - Cited by 3 - Related articles
Introduction-Methods for **Determination of Water**. J. W. Stillman. Anal. Chem. , 1951, 23 (8), pp 1058–1058. DOI: 10.1021/ac60056a002. Publication Date: ...

Determination of Water Content in Candies | Sigma-Aldrich
www.sigmaaldrich.com/technical-documents/articles/.../**determination-of-water**.html
Water content **determination** by Karl Fischer titration is a fast, accurate, and reliable method. Especially for food samples, the Karl Fischer (KF) titration shows ...

Water Determination in Pharmaceutical Compounds | Sigma-Aldrich
www.sigmaaldrich.com/technical-documents/articles/.../**water-determination**0.html
Sigma-Aldrich.com presents an article concerning **water determination** in pharmaceutical compounds.

Determination of Water Content in Acetone Using Karl Fischer Titration ...
www.sigmaaldrich.com/.../**water-determination**-in-acetone-r676-by-karl-fischer-titrati...
At the same time **water** is formed. Cyclohexanone and acetone react rapidly, long chain ketones react more slowly. Aromatically substituted ketones only react ...

determination of water content
www.uta.edu/ce/geotech/lab/Main/wtrcnt/wtrcnt.htm
Determination of Water Content in Soil. Testing objectives: · Determination of the natural content of the given soil sample. · Testing conforms to ASTM D2216-90.

DETERMINATION OF WATER CONTENT OF SOIL - The Constructor
theconstructor.org › Geotechnical Engineering › GE Lab Tests
A) By oven dry method Theory: The **water** content (w) of a soil sample is equal to the mass of **water** divided by the mass of solids. Where M1=mass of empty co.

DETERMINATION OF MOISTURE AND TOTAL SOLIDS
people.umass.edu/~mcclemen/581Moisture.html
Where mw is the mass of the **water** and msample is the mass of the sample. ... Sometimes food analysts are interested in **determining** the amounts of **water** in ...

ASTM D2216 - 10 Standard Test Methods for Laboratory ...
www.astm.org/Standards/D2216.htm
In fine-grained (cohesive) soils, the consistency of a given soil type depends on its **water** content. ... 1.1 These test methods cover the laboratory **determination** of the **water** (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due ...

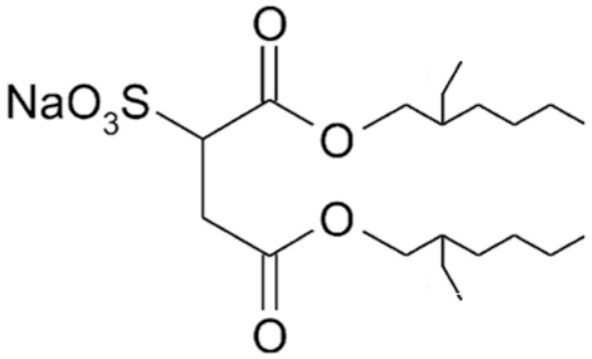
[PDF] The determination of water by coulometric titration - Oak Ridge ...
web.ornl.gov/info/reports/1955/3445603607568.pdf
THE **DETERMINATION OF WATER** BY COULOMETRIC TITRATION. A. S. Meyer, Jr. and CM. Boyd. May 20, 1955. ANALYTICAL CHEMISTRY DIVISION.

Determination of water in forages and animal feeds by Karl Fischer ...
www.ncbi.nlm.nih.gov/pubmed/9477559
by T Van Erem - 1998 - Cited by 7 - Related articles
J AOAC Int. 1998 Jan-Feb;81(1):25-32. **Determination of water** in forages and animal feeds by Karl Fischer titration. Van Erem T(1), Thiex N, Pohmer J, ...

[PDF] Water Determination (Karl Fischer Method)
www.ffcr.or.jp/zaidan/ffcrhome.nsf/.../$file/b43.pdf
The **Water Determination** Test (Karl Fischer Method) is designed to determine **water** content in substances, utilizing the quantitative reaction of **water** with iodine.

Ubiquitous Measurement

- Measured in many systems
- Ours is sodium bis(ethylhexyl)sulfosuccinate



Also known as: AOT
An anionic surfactant useful in forming micelles
Known to be hydroscopic and contains an undetermined amount of water as received from supplier

Standard Methods

- Karl Fisher Titration
  - SO_2, pyridine and iodine (Ugh)
- IR spectroscopy
  - Good method, expensive instrument
- NMR spectroscopy
  - Great method, more expensive instrument
- Spectrophotometry
  - Intriguing method

Spectrophotometric Titration

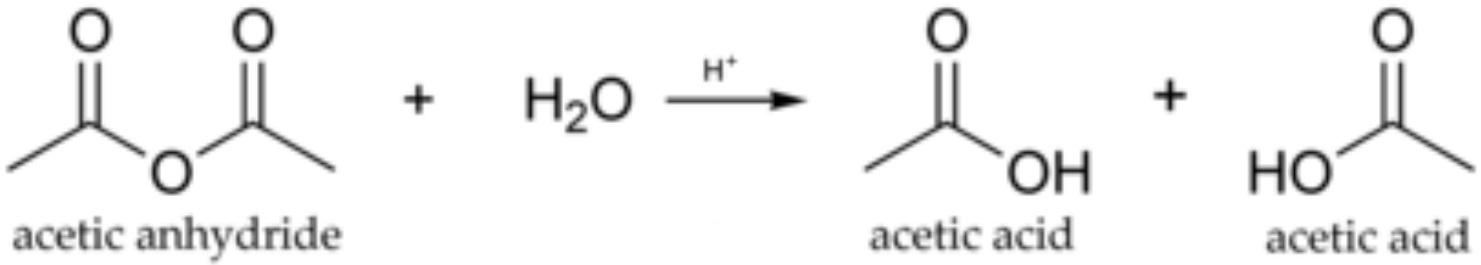
8.1 Water. Bruckenstein[265] has determined water in glacial acetic acid by spectrophotometric titration at 256 mμ with acetic anhydride, in the presence of sulphuric acid as a catalyst. Acetic anhydride absorbs appreciably at 256 mμ while water, acetic acid and sulphuric acid scarcely absorb at that wavelength. Water in the concentration range of 0·009 to 1·7% was satisfactorily determined, the maximum error for amounts in excess of 1% being 0·1%, for amounts between 0·1 and 1%, 0·004%, and for amounts less than 0·1%, 0·0006%.

PHOTOMETRIC TITRATIONS

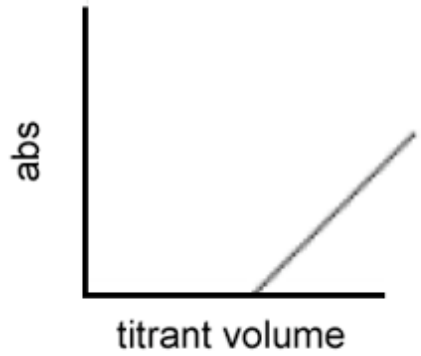
J. B. HEADRIDGE

PERGAMON PRESS 1961

Reaction & Method Details



- Start out with acetic acid$_{(l)}$ containing some water in the titration vessel.
- Add acetic anhydride which consumes water until no water remains.
- Additional acetic anhydride results in absorption at 256 nm.



Analyte + Titrant –> Product

abs(A)	abs(T)	abs(P)
=0	≠0	=0

Specified Procedure

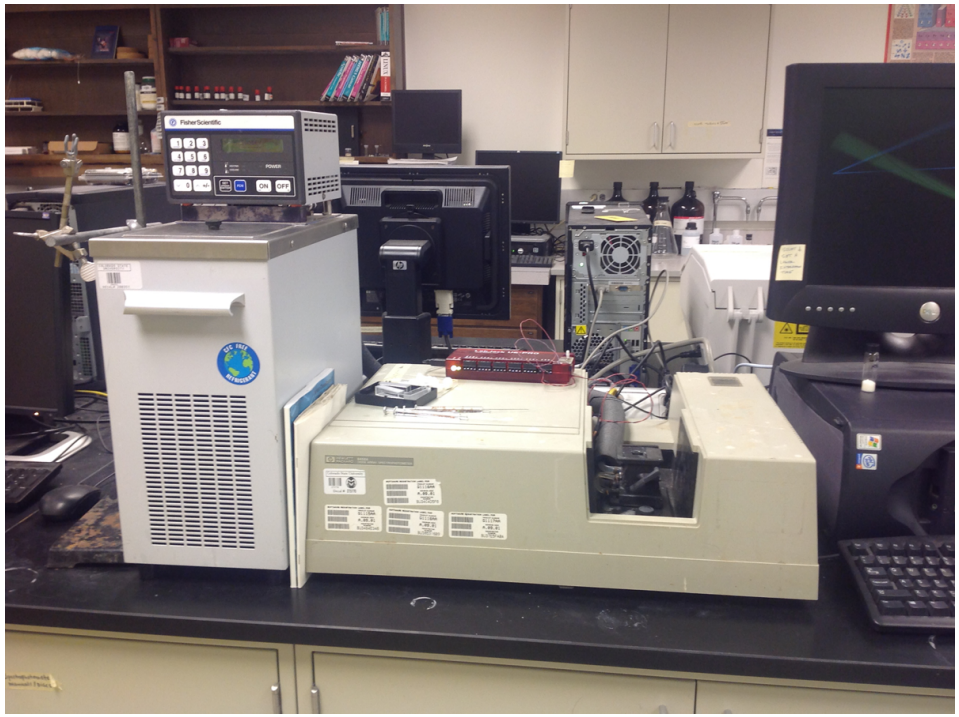
Procedure. Transfer about 3 ml of the acetic acid sample to a weighed 1 cm square silica cell in a dry box. Remove the stoppered cell from the dry box, weigh it and replace it in the dry box. Add 0·1 ml of water-free 2 M sulphuric acid in acetic acid. Place the cell in a spectrophotometer, set the wavelength to 256 mμ and the optical density reading to zero. Return the cell to the dry box and add a suitable volume of acetic anhydride from a 0·1 or 0·01 ml ultramicroburette. Measure the optical density of the solution and carry on in this manner until about six optical density readings after the equivalence point have been obtained. Locate the end-point from a plot of optical density against volume of titrant added.

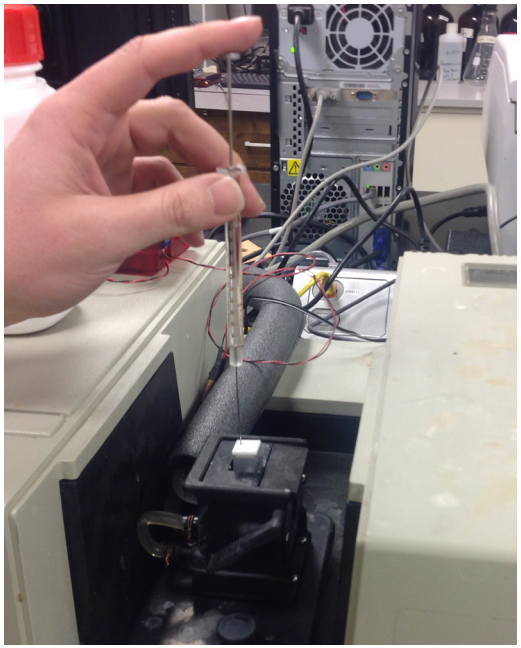
PHOTOMETRIC TITRATIONS

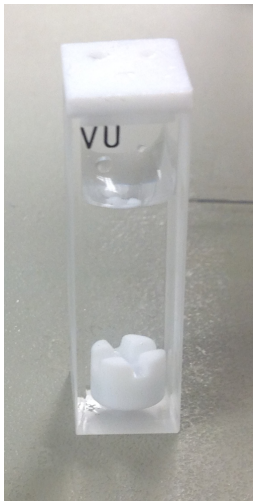
J. B. HEADRIDGE

PERGAMON PRESS 1961

Actual Method Used

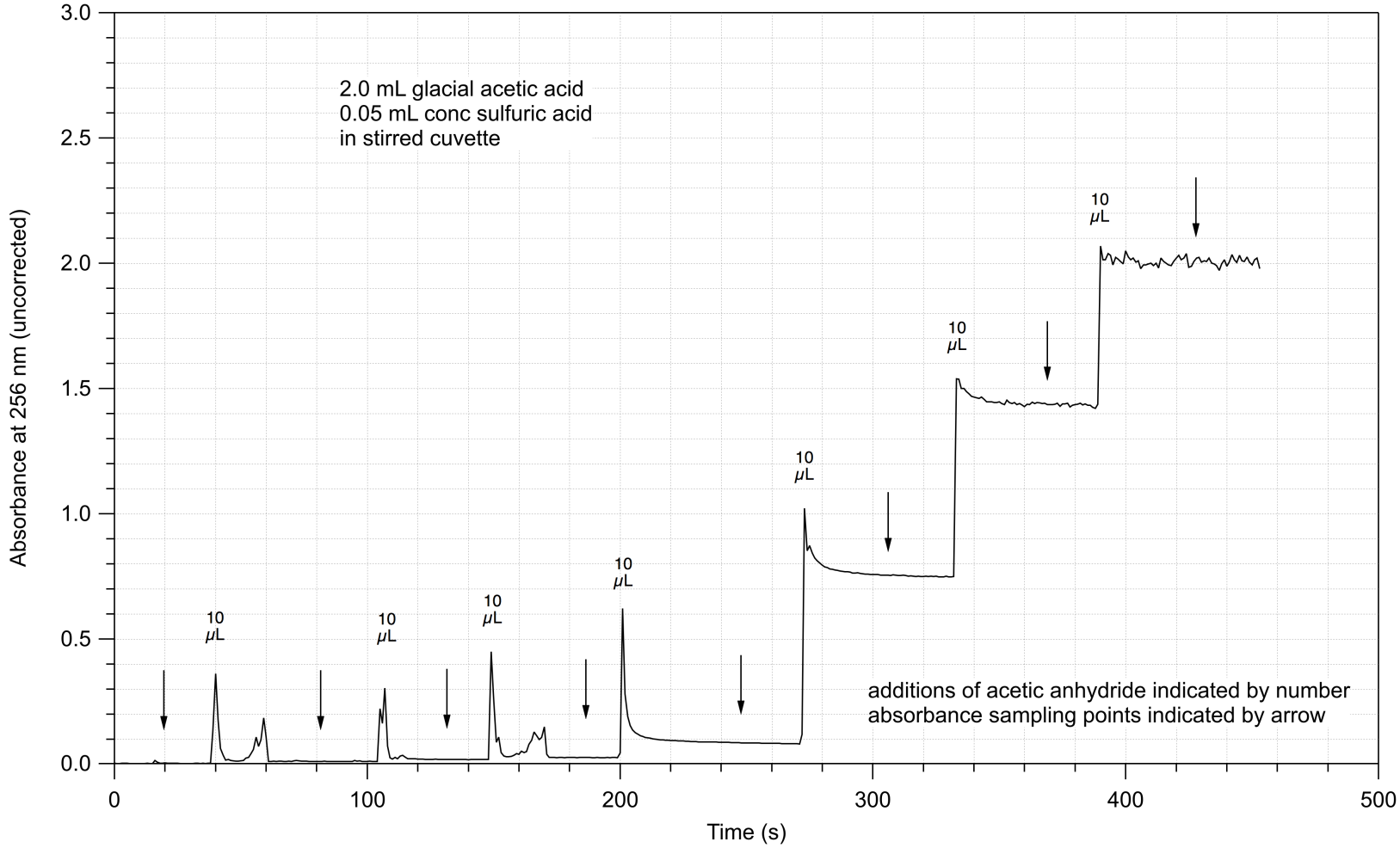




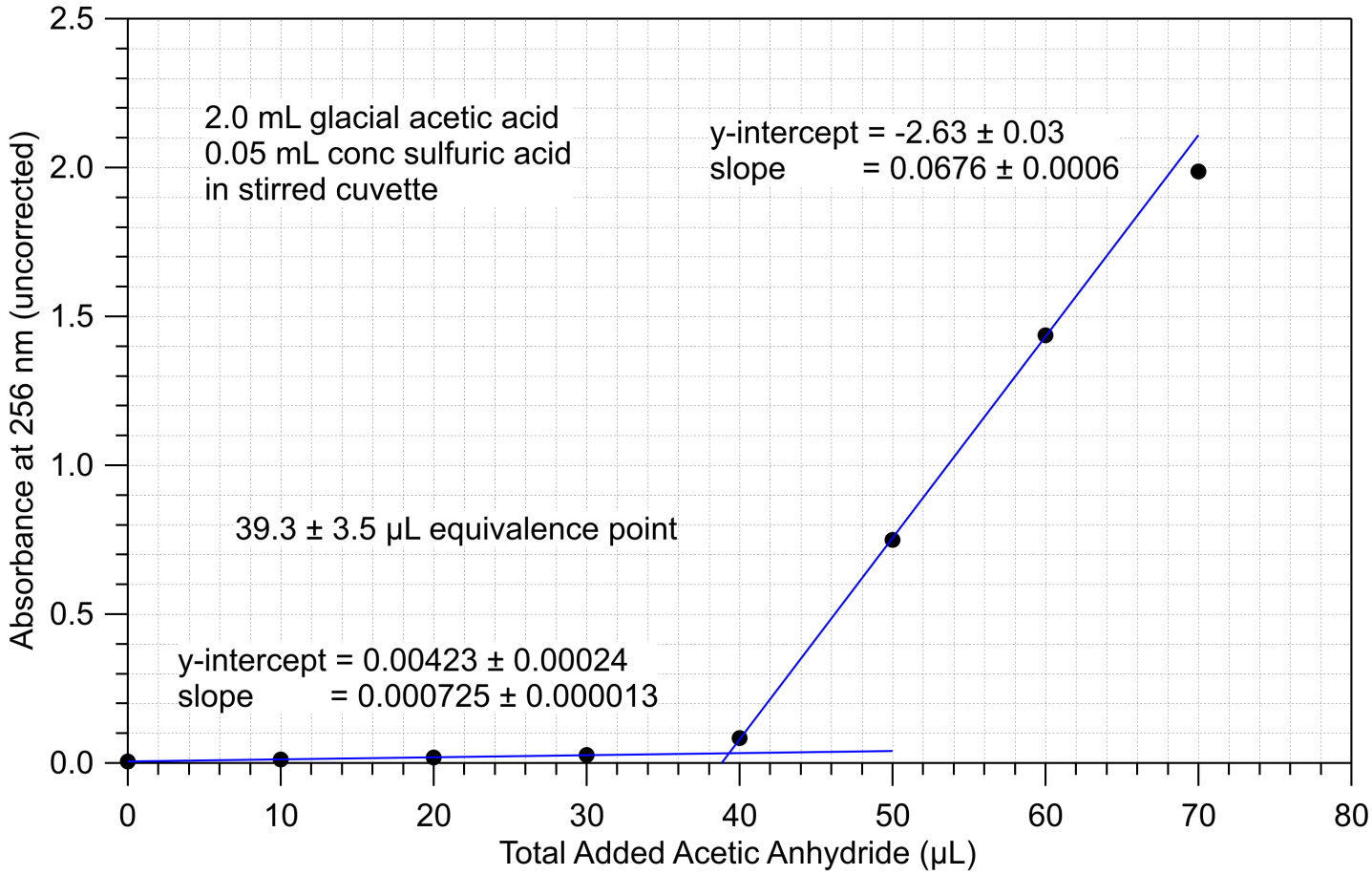


- Recording UV-Vis spectrometer
- Cuvette with stirring disk
- Microsyringe injection (repeating)

Raw Experimental Data



Reduced Experimental Data



Blank Computations

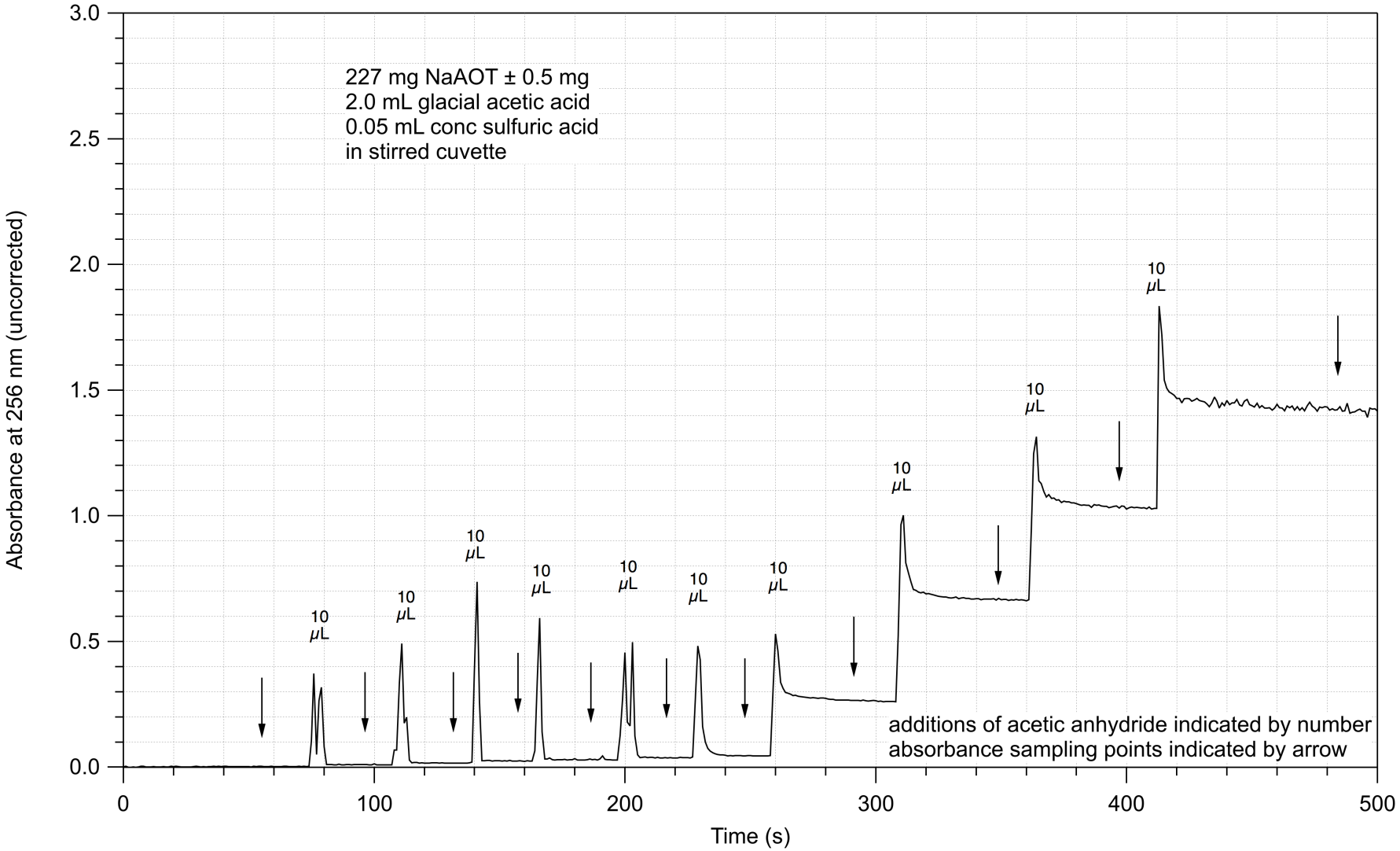
2.0 mL glacial acetic acid
0.05 mL conc sulfuric acid

acetic acid solvent
1.049 g mL^{-1}
60.06 g mol^{-1}
17.47 mmol mL^{-1}

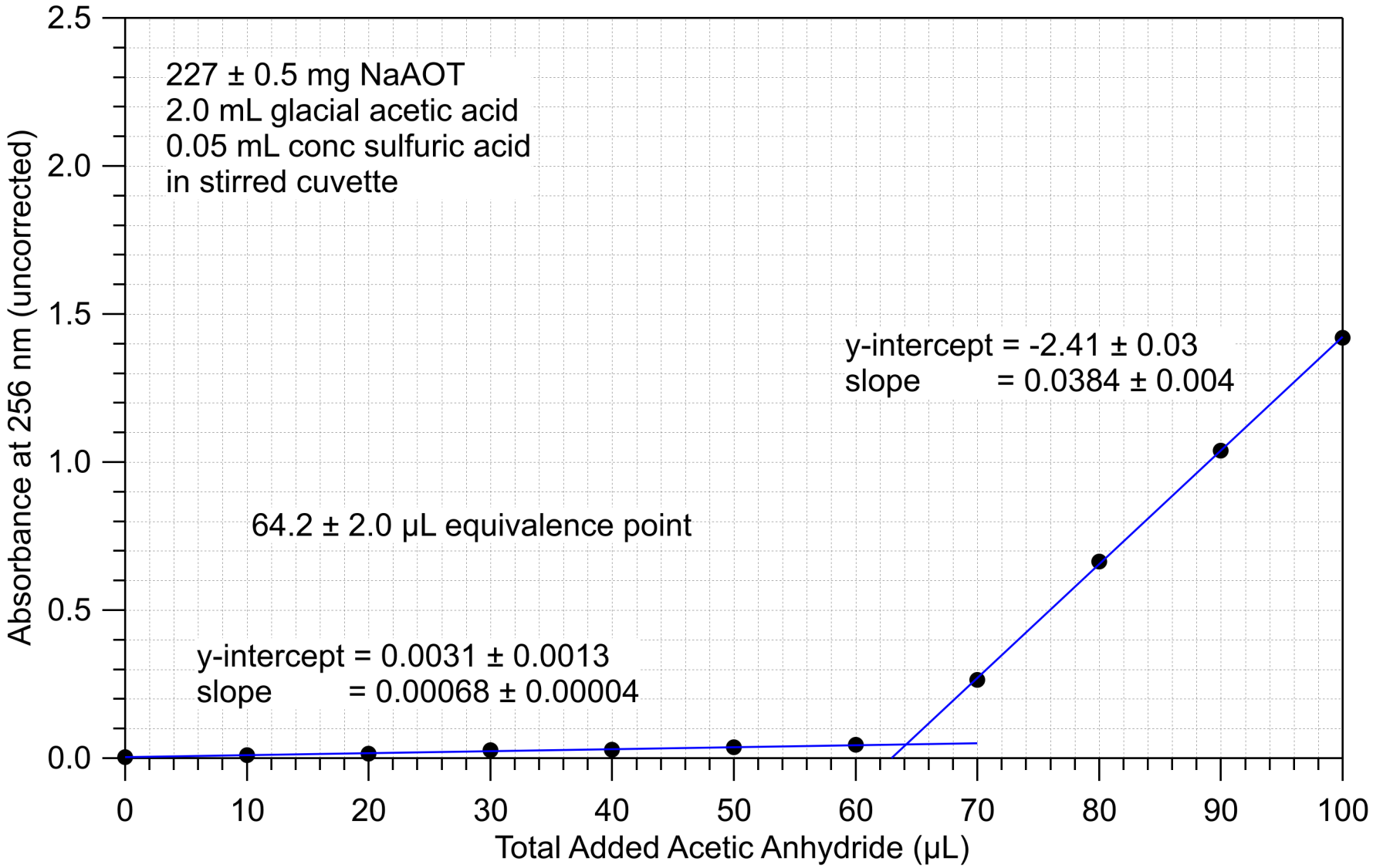
acetic anhydride titrant
1.082 g mL^{-1}
102.09 g mol^{-1}
10.06 µmol $µL^{-1}$

39.3 µL to reach equivalence point
395 µmol H_2O in 34.9 mmol acetic acid
1.13 mol % H_2O in acetic acid

AOT Raw Data


227 mg NaAOT ± 0.5 mg
2.0 mL glacial acetic acid
0.05 mL conc sulfuric acid
in stirred cuvette
10 µL
10 µL
10 µL
10 µL
10 µL
10 µL
10 µL
10 µL
10 µL
10 µL
additions of acetic anhydride indicated by number
absorbance sampling points indicated by arrow
Absorbance at 256 nm (uncorrected)
Time (s)
0.0
0.5
1.0
1.5
2.0
2.5
3.0
0
100
200
300
400
500

AOT Reduced Data



AOT Computations

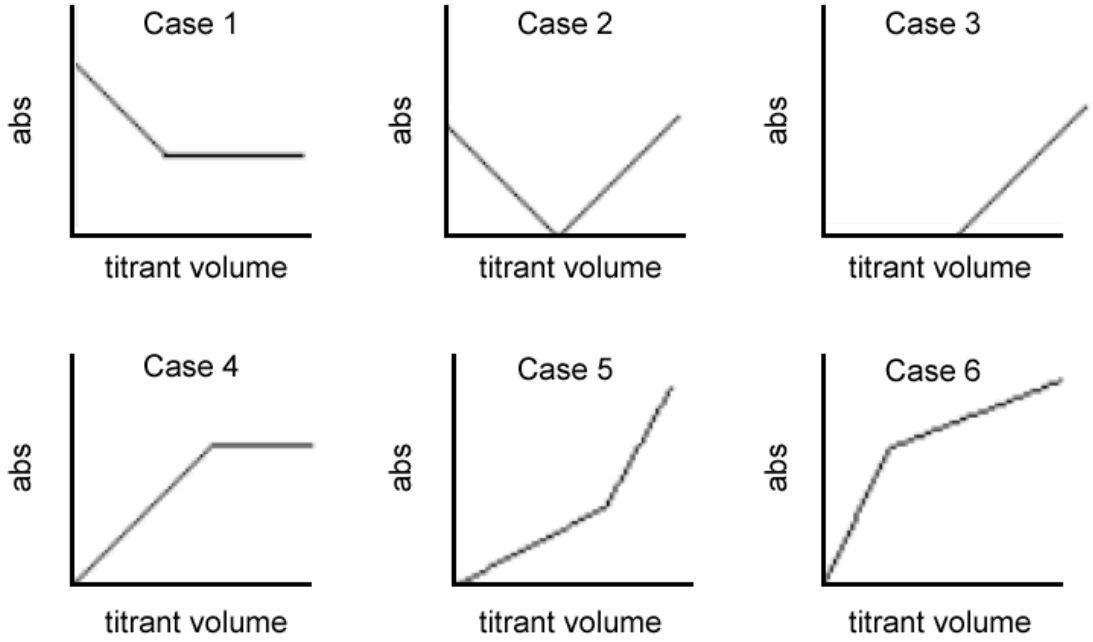
227 ± 0.5 mg NaAOT
2.0 mL glacial acetic acid
0.05 mL conc sulfuric acid

NaAOT analyte
444.56 g mol^{-1}

acetic anhydride titrant
1.082 g mL^{-1}
102.09 g mol^{-1}
10.06 µmol $µL^{-1}$

24.9 ± 4.0 µL to reach equivalence point
250.0 ± 40 µmol H_2O in 511 ± 2.0 µmol NaAOT
48.9 ± 11.1 mol % H_2O in NaAOT

Other Titrations Possible



Analyte + Titrant –> Product

A + T –> P

	abs(A)	abs(T)	abs(P)	
1	≠0	=0	≠0	abs(A) > abs(P)
2	≠0	≠0	=0	abs(A) ~ abs(P)
3	=0	≠0	=0	
4	=0	=0	≠0	
5	=0	≠0	≠0	abs(T) > abs(P)
6	=0	≠0	≠0	abs(T) < abs(P)

Additional Considerations

Effect of pH

In developing the spectrophotometric method for iron using ethylenediamine-di(*o*-hydroxyphenylacetic acid), it was shown[3] that color development was maximal throughout the pH range of 2.5 to 9.5. However, excess reagent was used, whereas

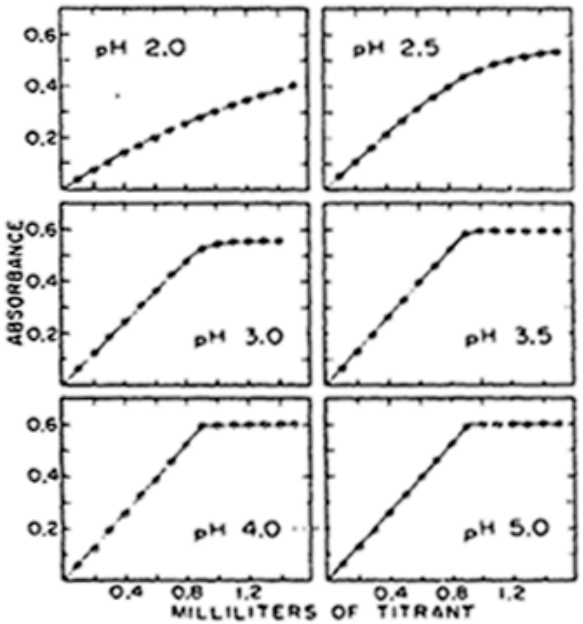


Fig. 1. Effect of pH.

a sharp photometric end-point requires that the chelation reaction go well toward completion with quantities of reagent more nearly stoichiometric. The effect of pH on the titration is shown in Fig. 1, where it may be seen that the pH should be at least 3 in order to obtain a reasonably good titration curve.

Acknowledgements

- Kai Fuller, undergraduate research student

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