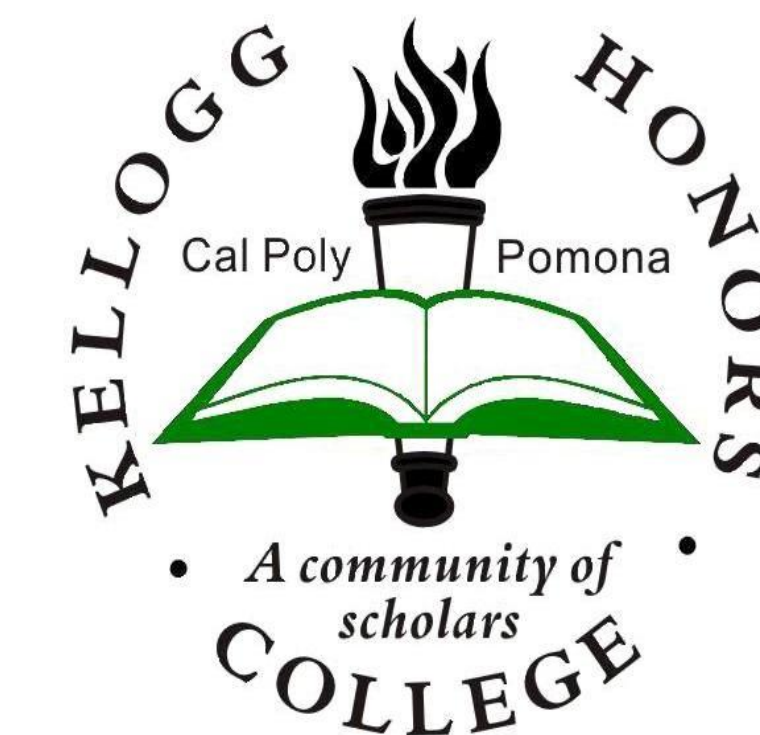




# Study of the Hydrolysis of Trifluoroacetanilide



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## Introduction

Hydrolysis is the reaction of an organic compound with water. A number of organic compounds can undergo hydrolysis including amides, esters, and halogenoalkanes. Currently, all organic textbooks have based the hydrolysis mechanism of amides on a kinetics study performed in 1965 by Research Laboratories of Eastman Kodak Company. However, the approach used for the investigation is poor in comparison to modern techniques practiced for similar studies today. The purpose of this investigation is to repeat the previous work by using an improved UV-Vis spectrophotometer fitted with a circulating temperature bath for better temperature control to obtain more refined data for the kinetic profile of trifluoroacetanilide. Furthermore, another aspect being studied is the possible deprotonation of the hydrogen on the nitrogen of trifluoroacetanilide before it undergoes hydrolysis which may result in an unexpected variation of the spectra observed.

## Methods

Trifluoroacetanilide was synthesized by first cooling a solution of 5.6 mL of aniline, and 10 g of  $Na_2CO_3$ , in 50 mL of diethyl ether to 0°C. Slowly, 11.1 mL of trifluoroacetic acid anhydride was added to the solution. The reaction mixture was then stirred overnight at room temperature producing solid crude product. The work up for the purification of trifluoroacetanilide was accomplished by performing an extraction with diethyl ether to separate the product into the organic layer. The organic layer was then rotovapped and trifluoroacetanilide crystals were isolated. For the study of the kinetic profile of trifluoroacetanilide, the following solutions were prepared for the single UV scans:

- 0.001 M, 0.01 M, 0.1 M KOH in water
- 0.001 M, 0.01 M, 0.1 M KOH in acetonitrile ( $CH_3CN$ )
- 0.001 M, 0.01 M, 0.1 M KOH in 50% water and 50% acetonitrile

3  $\mu$ L of 0.1 M of trifluoroacetanilide in acetonitrile was added directly into the cuvette containing a 3 mL sample of each solution and the spectrum was collected. Following this, time scans were then collected of the 0.001 M and 0.1 M KOH in water, and 0.001 M and 0.1 M KOH in 50% water: 50% acetonitrile solutions.

## Compounds & Reactions

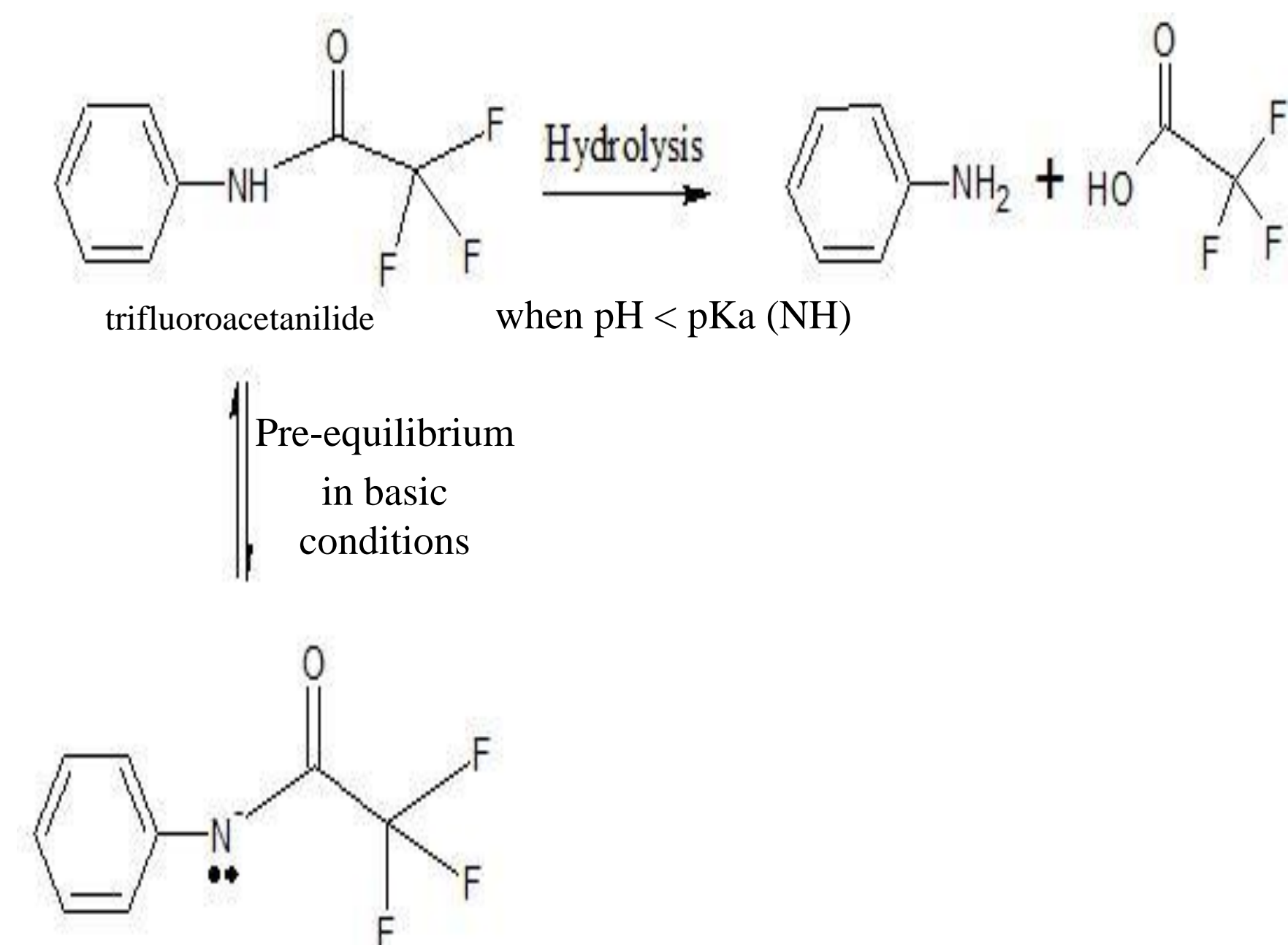


Figure 1. Pre-equilibrium and hydrolysis reaction of trifluoroacetanilide.

## Data and Results

Single UV Scans of the Hydrolysis of Trifluoroacetanilide in Various Solutions

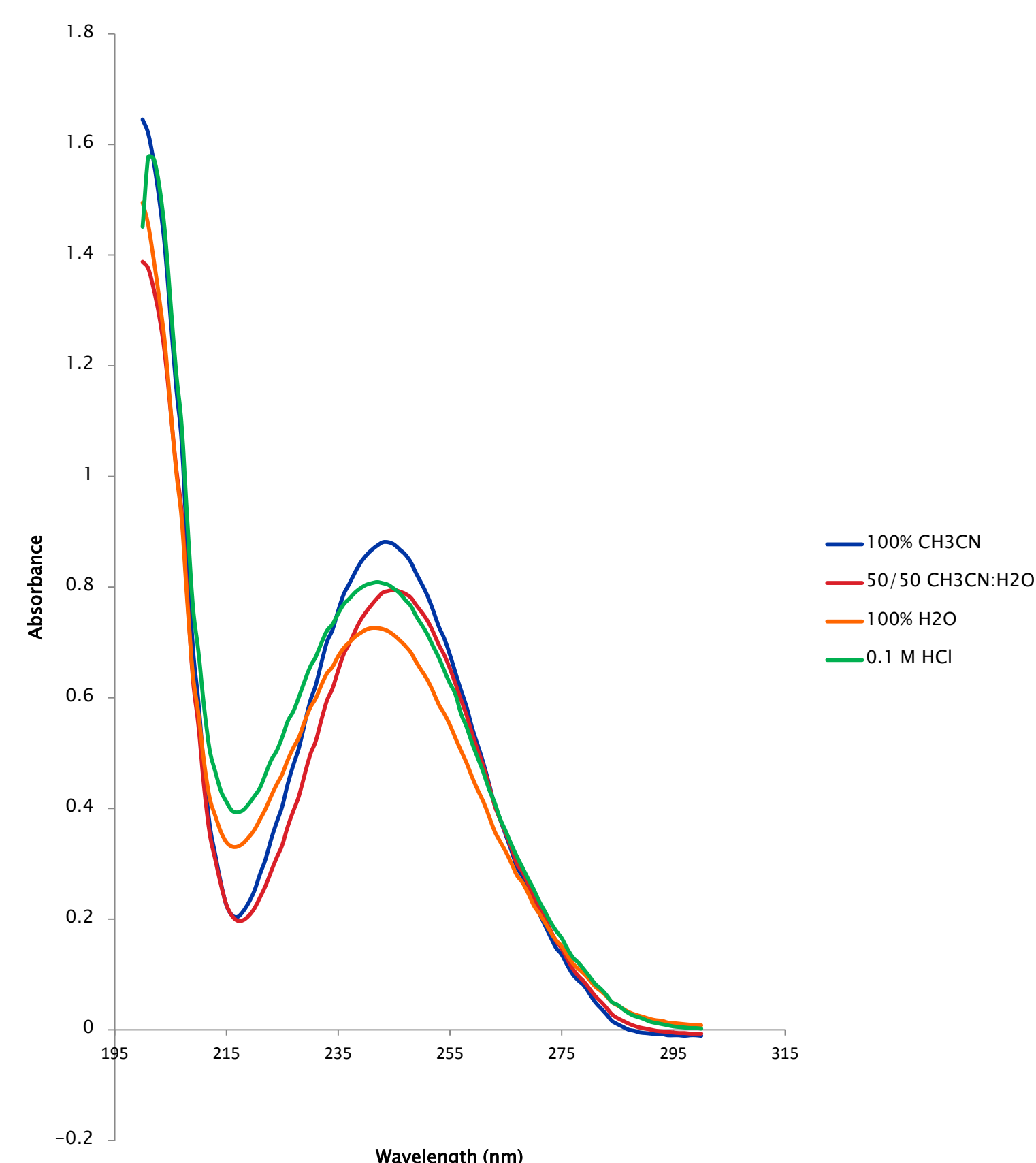


Figure 2. Single UV-Vis Scans of the hydrolysis of trifluoroacetanilide in various solutions.

Single UV-Vis Scans of the Hydrolysis of Trifluoroacetanilide at Various Concentrations of KOH in Water

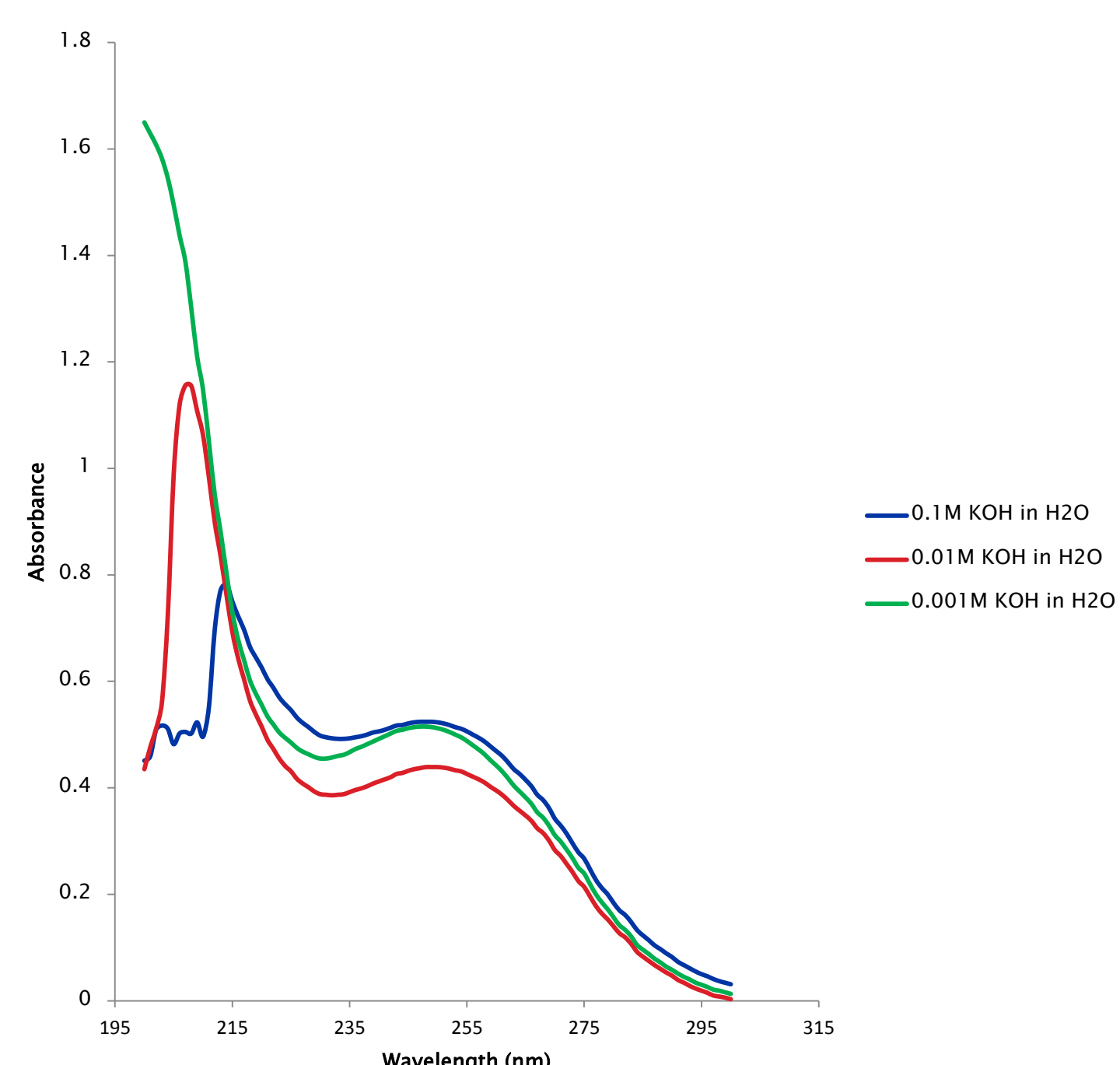


Figure 4. Single UV-Vis scans of the hydrolysis of trifluoroacetanilide at various concentrations of KOH in water.

Hydrolysis of Trifluoroacetanilide in 0.100 M KOH in H2O

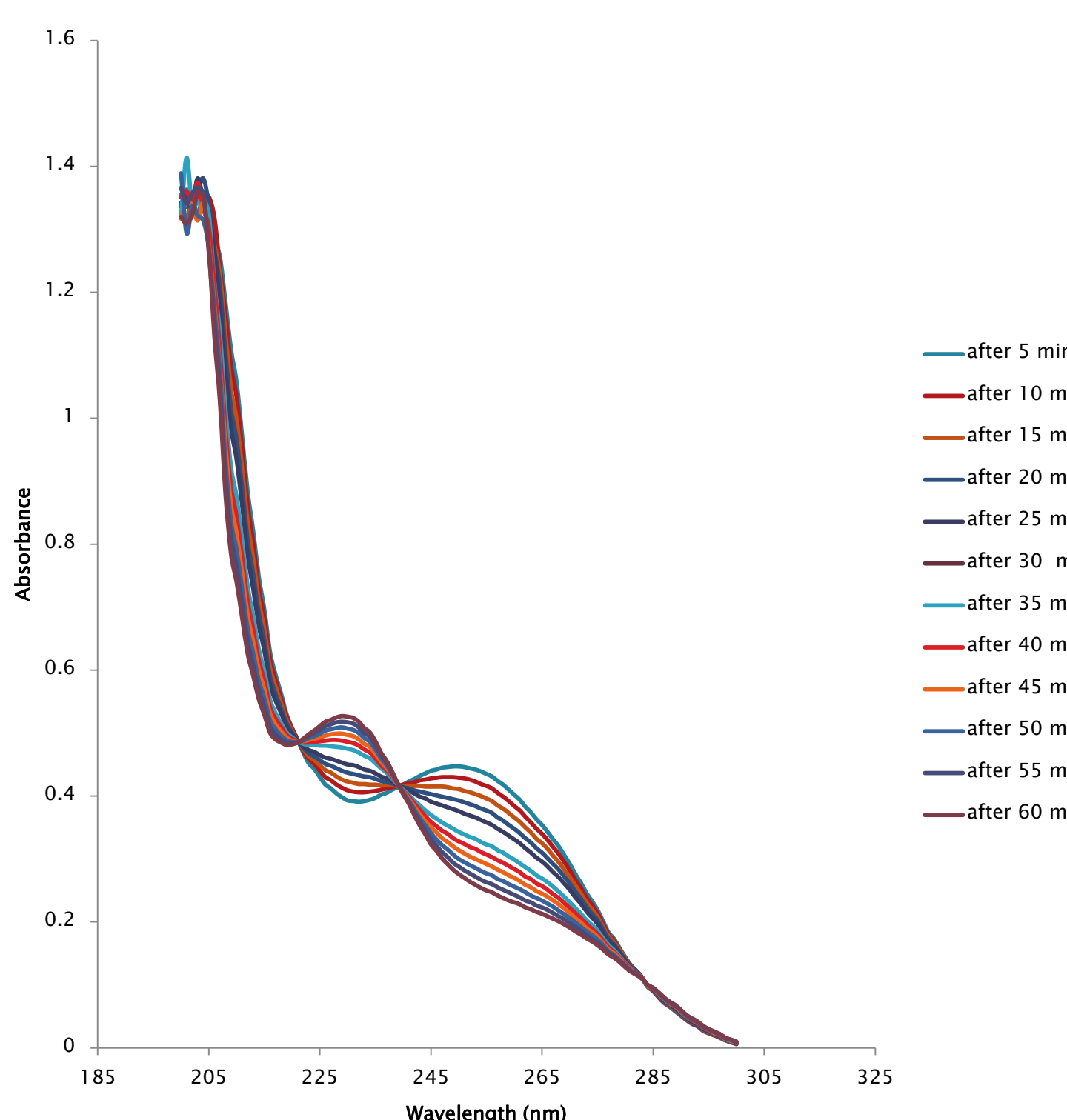


Figure 6. Time scan of the hydrolysis of trifluoroacetanilide in 0.100 M KOH in water.

Single UV Scans of the Hydrolysis of Trifluoroacetanilide at Various Concentrations of KOH in 50% acetonitrile: 50% water

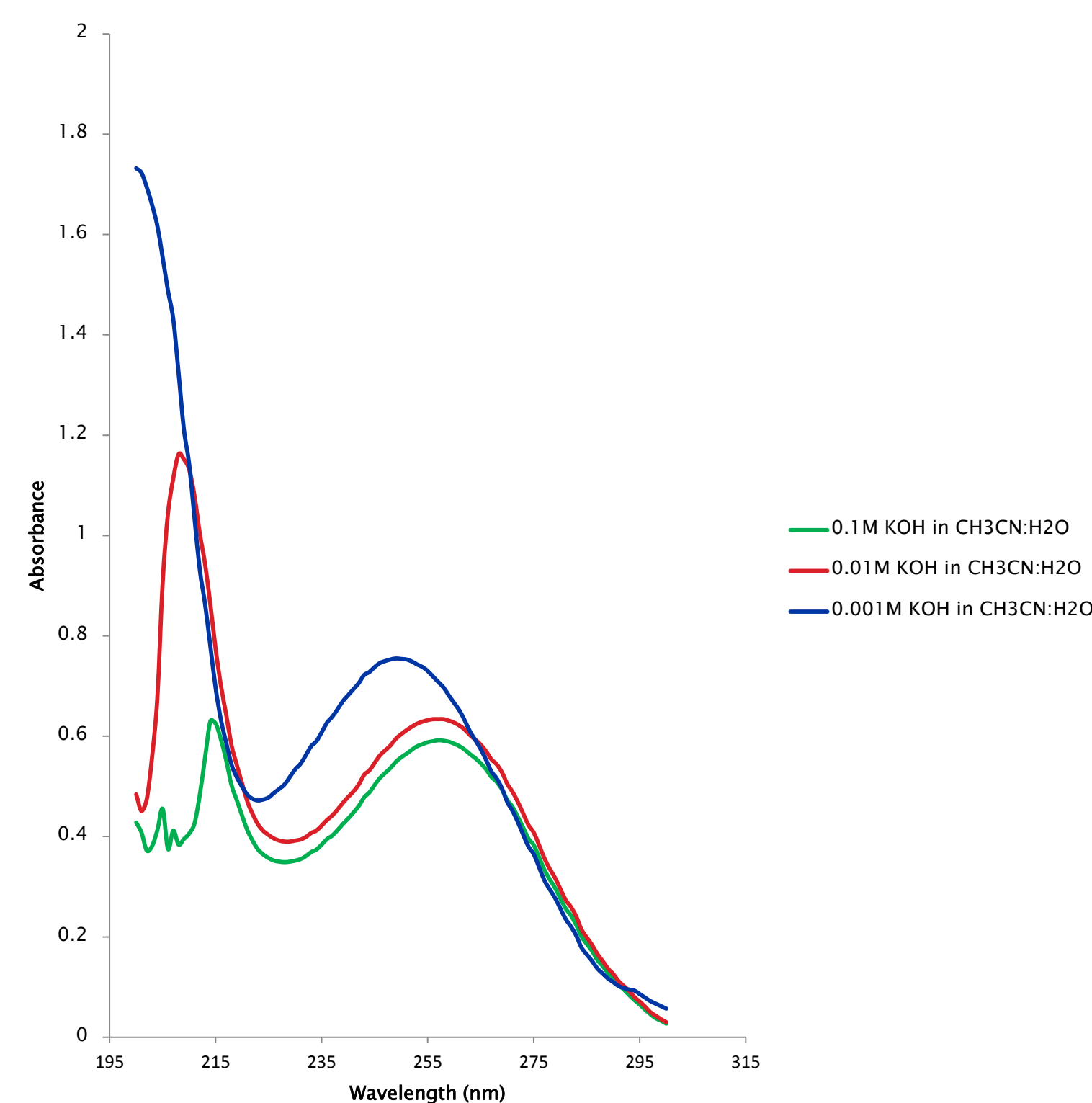


Figure 3. Single UV-Vis scans of the hydrolysis of trifluoroacetanilide at various concentrations of KOH in 50% water: 50% acetonitrile solutions.

Hydrolysis of Trifluoroacetanilide in 0.100 M KOH in 50% H2O: 50% CH3CN

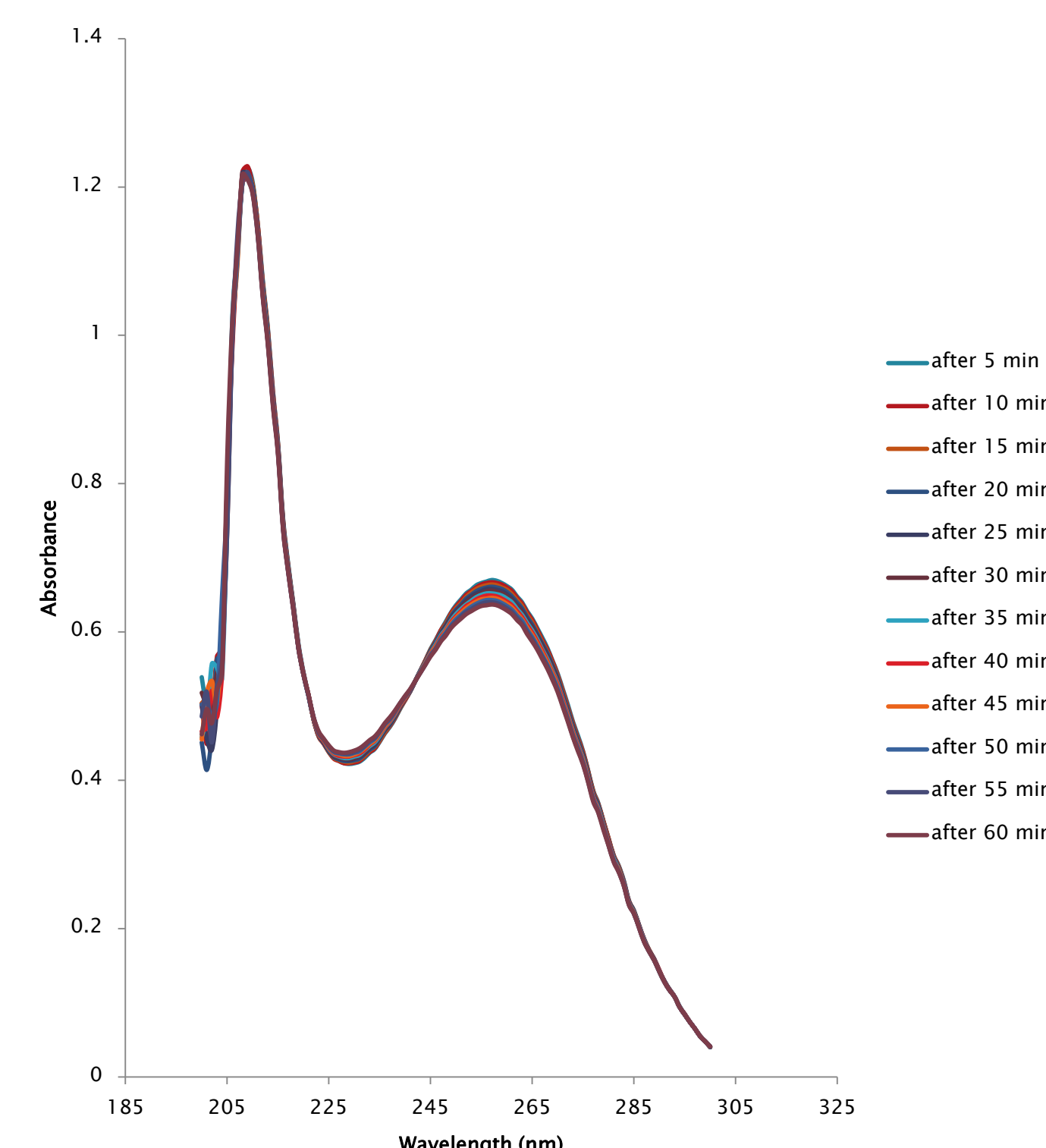


Figure 5. Time scan of the hydrolysis of trifluoroacetanilide in 0.100 M KOH in 50% water: 50% acetonitrile solution.

Single UV-Vis Scan of Compound in 0.1 M KOH Neutralized with HCl

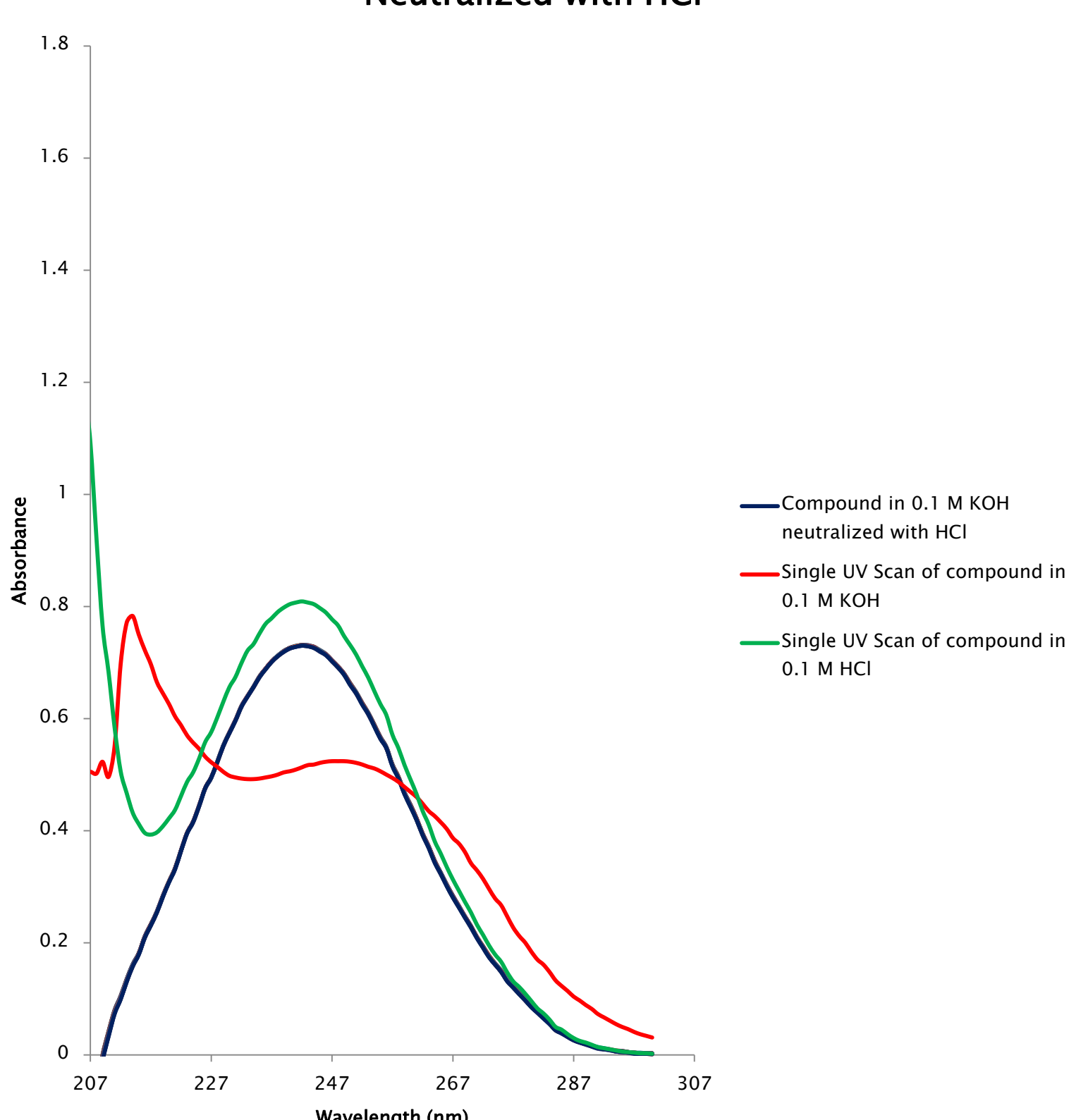


Figure 7. Single UV-Vis scans of the hydrolysis of trifluoroacetanilide at both high and low pH, and neutralized with HCl when at high pH.

Table 1. Single UV-Vis Scans of various solutions with 3  $\mu$ L of 0.1 M trifluoroacetanilide.

Solution	Wavelength Max. (nm)	Absorbance
100% CH <sub>3</sub> CN	244	0.881
50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	245	0.795
100% H <sub>2</sub> O	242	0.726
0.1 M HCl in water	242	0.809
0.1 M KOH in 50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	257	0.592
0.01 M KOH in 50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	258	0.634
0.001 M KOH in 50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	249	0.755
0.1 M KOH in H <sub>2</sub> O	249	0.524
0.01 M KOH in H <sub>2</sub> O	250	0.439
0.001 M KOH in H <sub>2</sub> O	248	0.515

Table 2. UV-Vis Time Scans of various solutions with 3  $\mu$ L of 0.1 M trifluoroacetanilide.

Solution	After 5 min		After 60 min	
	Wavelength Max. (nm)	Absorbance	Wavelength Max. (nm)	Absorbance
0.1 M HCl in water	242	0.784	242	0.809
0.1 M KOH in H <sub>2</sub> O	250	0.447	229	0.527
0.001 M KOH in H <sub>2</sub> O	242	0.736	242	0.752
0.1 M KOH in 50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	257	0.670	257	0.637
0.001 M KOH in 50% CH <sub>3</sub> CN: 50% H <sub>2</sub> O	244	1.010	244	1.007
0.1 M KOH in H <sub>2</sub> O neutralized with HCl	243	0.731	242	0.730

## Discussion

The a UV-Vis study of the hydrolysis of trifluoroacetanilide was obtained by UV-Vis spectroscopy. Using the maximum wavelength from the spectra collected it was observed that under high pH conditions, the wavelength changed the most. Time scans on the UV-Vis were then collected of the hydrolysis of trifluoroacetanilide under both high and low pH, and neutralized when at the high pH. It was observed that under high pH the wavelength was 229 nm and at low pH it was 242 nm, and when it was neutralized the wavelength was 242 nm, meaning that a reversible reaction is occurring because most of the compound was recovered. We predict that a pre-equilibrium reaction occurs at high pH, in which the nitrogen is deprotonated. Whether the reaction occurs at high or low pH determines if there is a pre-equilibrium reaction occurring simultaneously with the hydrolysis reaction affecting the overall kinetics of the hydrolysis reaction of trifluoroacetanilide.

## References

1. Trifluoroacetanilide.  $pK_a$  and Alkaline Hydrolysis Kinetics. P. M. Mader. *Journal of the American Chemical Society* 1965 87 (14), 3191-3195