Determination of Salicylic Acid Concentration in Clear Pore® Solution via Spectroscopy

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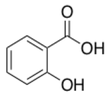
Abstract



salicylic acid

- UV-vis and fluorescence spectroscopy were used to determine the concentration of salicylic acid in Neutrogena Clear Pore® using two methods
1. Two calibration curves were generated using solvents water and 1:1 water:ethanol mixture to mimic the Neutrogena environment. The concentration was determined using the best fit line.
2. A standard addition curve was generated to back calculate the salicylic acid concentration.

Ultraviolet – Visible (UV-vis) Spectroscopy

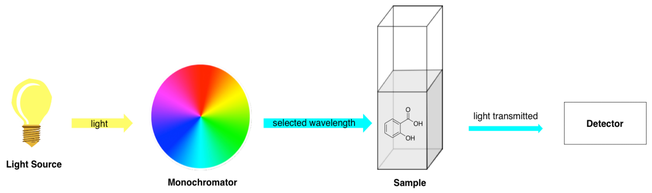


Figure 1. Ultraviolet-visible spectroscopy (aka UV-Vis) is an instrumentation method that determines absorbance of light by detecting transmitted light through a sample.

Fluorescence Spectroscopy

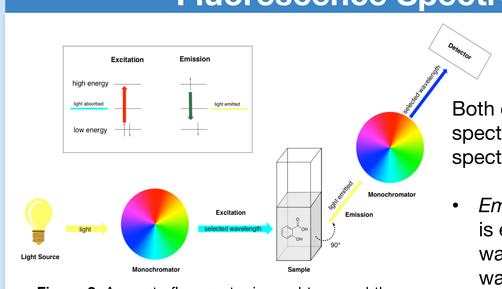


Figure 2. A spectrofluorometer is used to record the fluorescence of samples by exciting electrons into excited stated and detecting the light emitted as they return to the ground state.

Both excitation and emission spectra can be measured in a spectrofluorometer.

- *Emission Spectrum*: the sample is excited with a specific wavelength of light and the wavelengths of emission are monitored. When fluorescence is present, this yields a detectable signal.
- *Excitation Spectrum*: the sample is excited with variable wavelengths and the emission is monitored at a specific wavelength.
- When the excitation wavelength causes emission to occur, it yields a detectable response.
- This often mimics the absorption spectrum because it is probing the same electronic transitions.
- The excitation wavelength (265 nm) was chosen in order to ensure the intensity did not exceed 1000 a.u. which would damage the detector.

Excitation and Emission Spectra

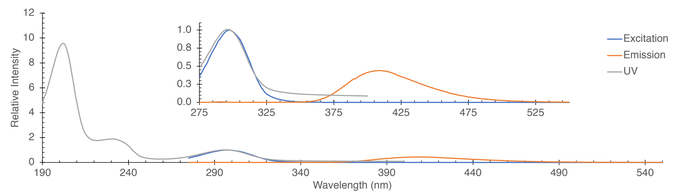


Figure 3. Using fluorescence spectroscopy, excitation (blue) and emission (orange) spectra of a Clear Pore ® solution diluted with distilled water were taken, and an absorption scan (grey) was taken using UV-vis spectroscopy. The relative intensities of all spectra were scaled for comparison.

Building a Calibration Curve

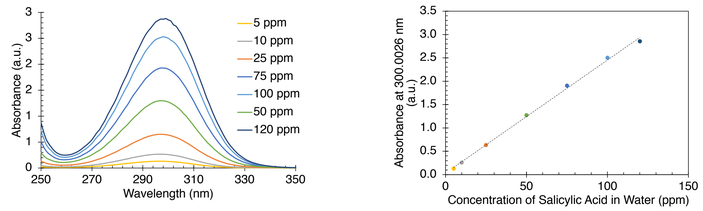


Figure 4: Calibration curves are developed from varying concentrations of salicylic acid and extracting the maximum absorbance or emission value from each spectrum (ex: 300 nm from the absorbance spectrum. Then, this maximum intensity value is plotted against the respective concentrations.

Determining the Limit of Detection in UV-vis

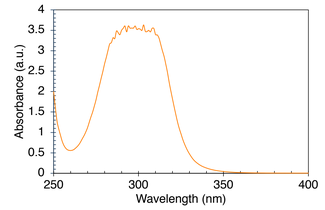


Figure 5: Oversaturated UV-vis scan of 240 ppm solution.

- Beer's Law applies only for dilute solutions (where 0.1 < A < 1 a.u.) which is where absorbance is proportional to concentration.
- A higher absorbance, correlating to a greater concentration, means a lower transmittance, or much less light is hitting the detector causing more error in the results.
- The line of fit for the calibration curve becomes less accurate due to the higher concentration data points not being linearly proportional to concentration. (Compare the orange R^2 values in **Figure 6** and **Figure 7**.)

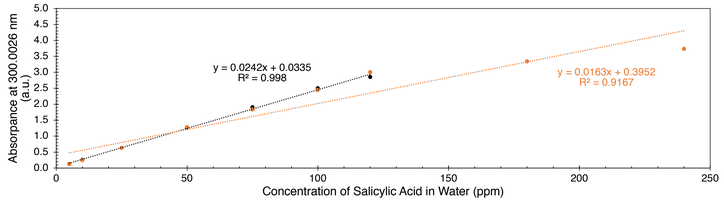


Figure 6: Using UV-vis spectroscopy, two calibration curves of salicylic acid in H_2O (black) and a H_2O/EtOH mixture (orange) were generated with a greater range of concentration.

UV-vis Spectroscopy Calibration Curves

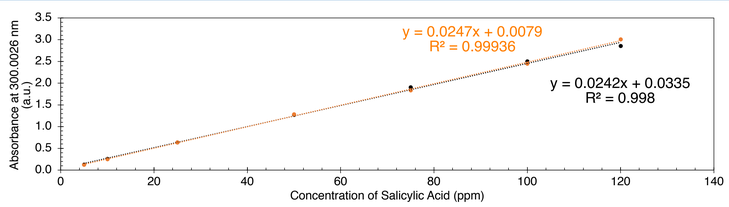


Figure 7: Using UV-vis spectroscopy, two calibration curves of salicylic acid in H_2O (black) and a H_2O/EtOH mixture (orange) were generated.

Fluorescence Calibration Curves

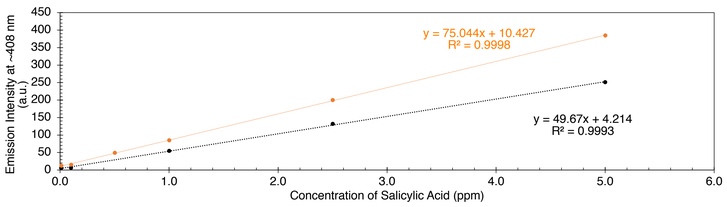


Figure 8: Using fluorescence spectroscopy, two calibration curves of salicylic acid in H_2O (black) and a H_2O/EtOH mixture (orange) were generated.

Standard Addition Curve

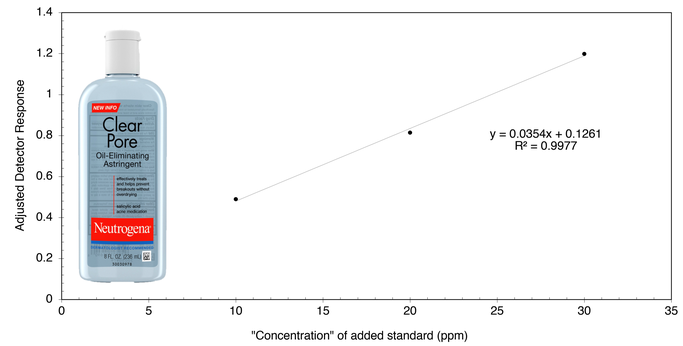


Figure 9: Using UV-vis spectroscopy, a standard addition curve using a diluted CP solution and a 100 ppm salicylic acid in H_2O/EtOH standard was constructed. This was generated using the standard addition equation:

$$\frac{I_x}{I_{x+s}} = \frac{[X_i]}{[S_f] + [X_i]\frac{V_0}{V}}$$

where I_x is the detector response of the unknown, I_{x+s} is the detector response of the unknown plus the addition of the standard, $[X_i]$ is the concentration of the unknown, $[S_f]$ is the final concentration of the standard added, V_o is the initial concentration of the unknown, and V is the total volume of solution.

Conclusions

Table 1: Concentration of salicylic acid in the Clear Pore® solution determined by experimental methods.

Determined [Salicylic Acid] (ppm)	Method
1.44×10^5	Standard addition
1.55×10^5	UV – vis (EtOH:H_2O mixture)
1.52×10^5	UV – vis (H_2O)
4.64×10^3	Fluorescence (EtOH:H_2O mixture)
1.44×10^5	Fluorescence (H_2O)

- The results from both UV-vis best-fit lines were consistent ($\pm 0.03 \times 10^5$ ppm).
- Modifying the solvent resulted in significantly different fluorescent behavior as illustrated by the much lower concentration in the EtOH:H_2O solvent.
- Standard addition and fluorescence with the H_2O solvent best-fit line were consistent ($\pm 0.00 \times 10^5$ ppm).
- Standard addition and both UV-vis spectra best-fit lines were on a similar magnitude.
- Standard addition and fluorescence with EtOH:H_2O solvent best-fit line were not consistent.

Future Directions

- Develop methods for salicylic acid detection in other solvents used in topical products (fatty acids, coconut oil, etc.) since results appear to be solvent dependent.
- Generalize these methods to other compounds found in topical products such as hydroquinone, ascorbic acid, hyaluronic acid, etc.

Acknowledgements

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Utilized Literature for Method Development

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