

Separation and Identification of Surfactants Found in Glyphosate-Based Herbicides

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Abstract

Using High-Performance Liquid Chromatography (HPLC), an automated procedure was developed to separate and characterize surfactant molecules found in commercial glyphosate-based herbicides. Currently, The method that achieved the best separation was determined by gradually increasing the solvent polarity over a short period of time .

Introduction

In recent years, Roundup and other glyphosate-based herbicides have shown correlation to causing non-Hodgkin lymphoma in humans.¹ Researchers have identified a potentially toxic series of chemicals that function as dispersal agents in herbicide products, called surfactants, however little information regarding the toxicity of surfactants is available in the literature.² Some of the most common type of surfactants used in broad-based herbicides are called polyethoxylated tallow amine (POEA) surfactants², and they are most notable for containing for being water soluble and amphiphilic.

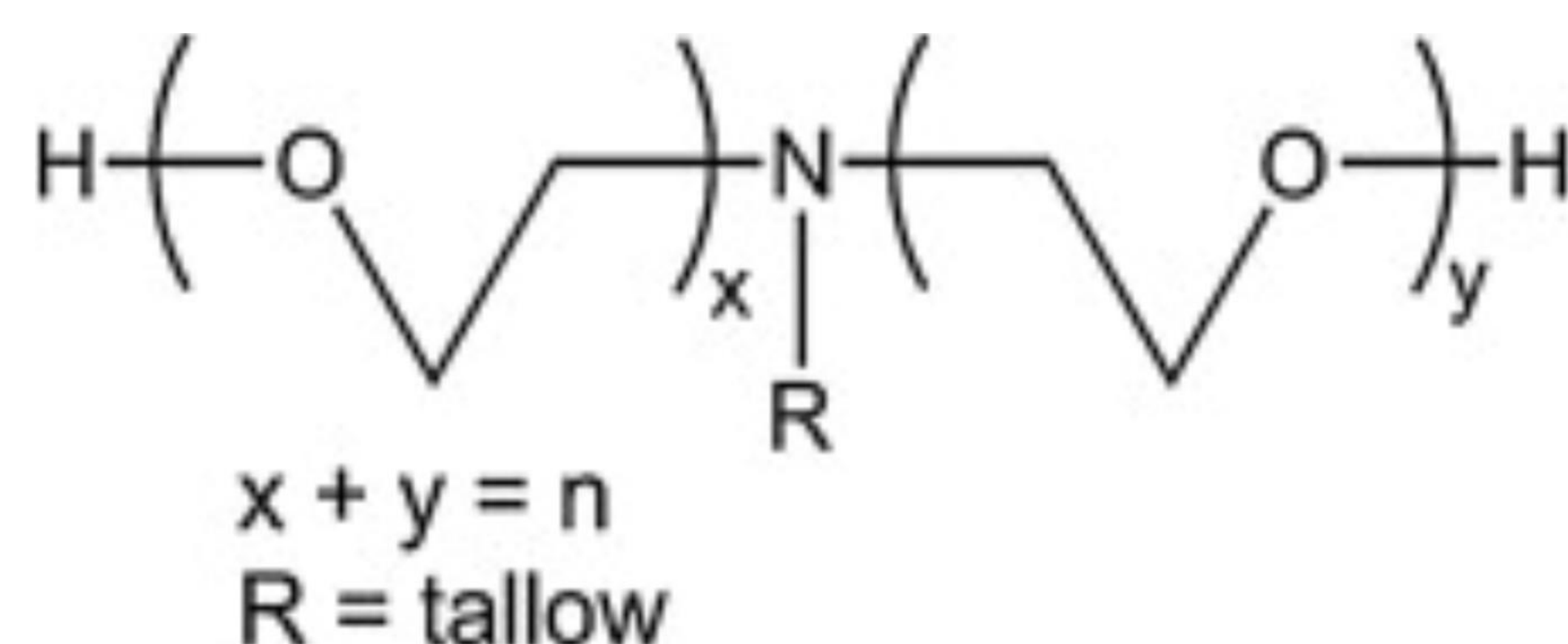


Figure 1: General structure of polyethoxylated tallow amine (POEA).³

In this project, we propose a method to separate, isolate, and characterize compounds found in store-bought herbicides using HPLC and ¹H NMR. Using this method, we seek to develop an automated method which will isolate and separate our target compounds from a sample of commercial pesticides to better characterize these compounds and provide a starting point for and provide a starting point for future synthesis. Ultimately, we hope to use this data to synthesize new and less toxic surfactants made from natural and renewable sources.

Wavelength of absorbed light for UV Spectroscopy	Concentration of Solvent System from 0 to 2.5 minute retention times	Concentration of Solvent System from 2.5 to 5 minute retention times
289 nm (Top)	98:2 CH ₃ N:H ₂ O	90:10 CH ₃ N:H ₂ O
319 nm (Bottom)	98:2 CH ₃ N:H ₂ O	90:10 CH ₃ N:H ₂ O

Table 1: Experimental conditions of the HPLC experiment.

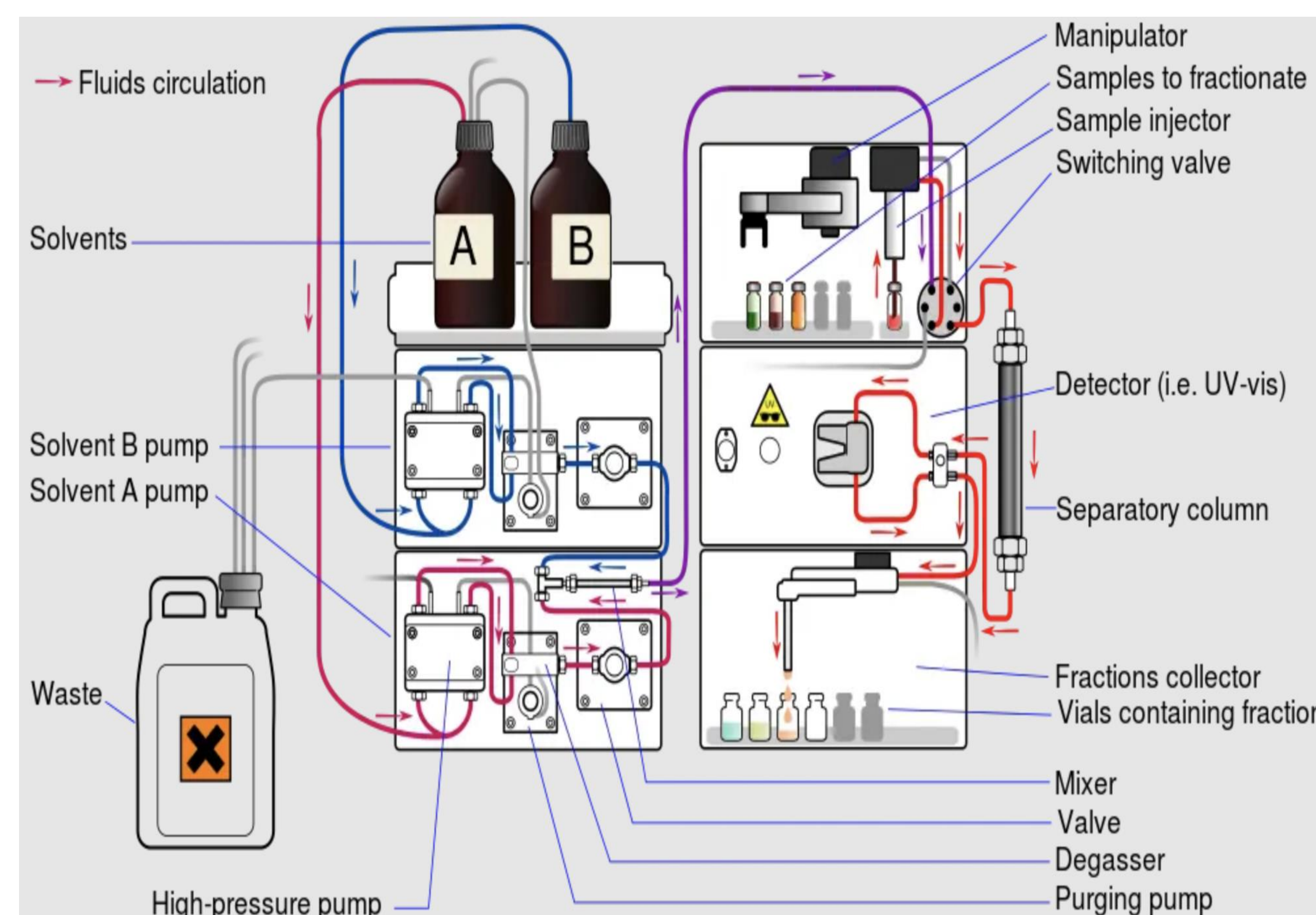


Figure 2: Diagram of Agilent Technologies High-Performance Liquid Chromatography [HPLC] instrument. ⁴

Results

After much experimentation, a method to achieve adequate separation of the components in a prepared sample of Roundup was achieved. However, this separation is not sufficient for collecting samples and taking further spectral analysis.

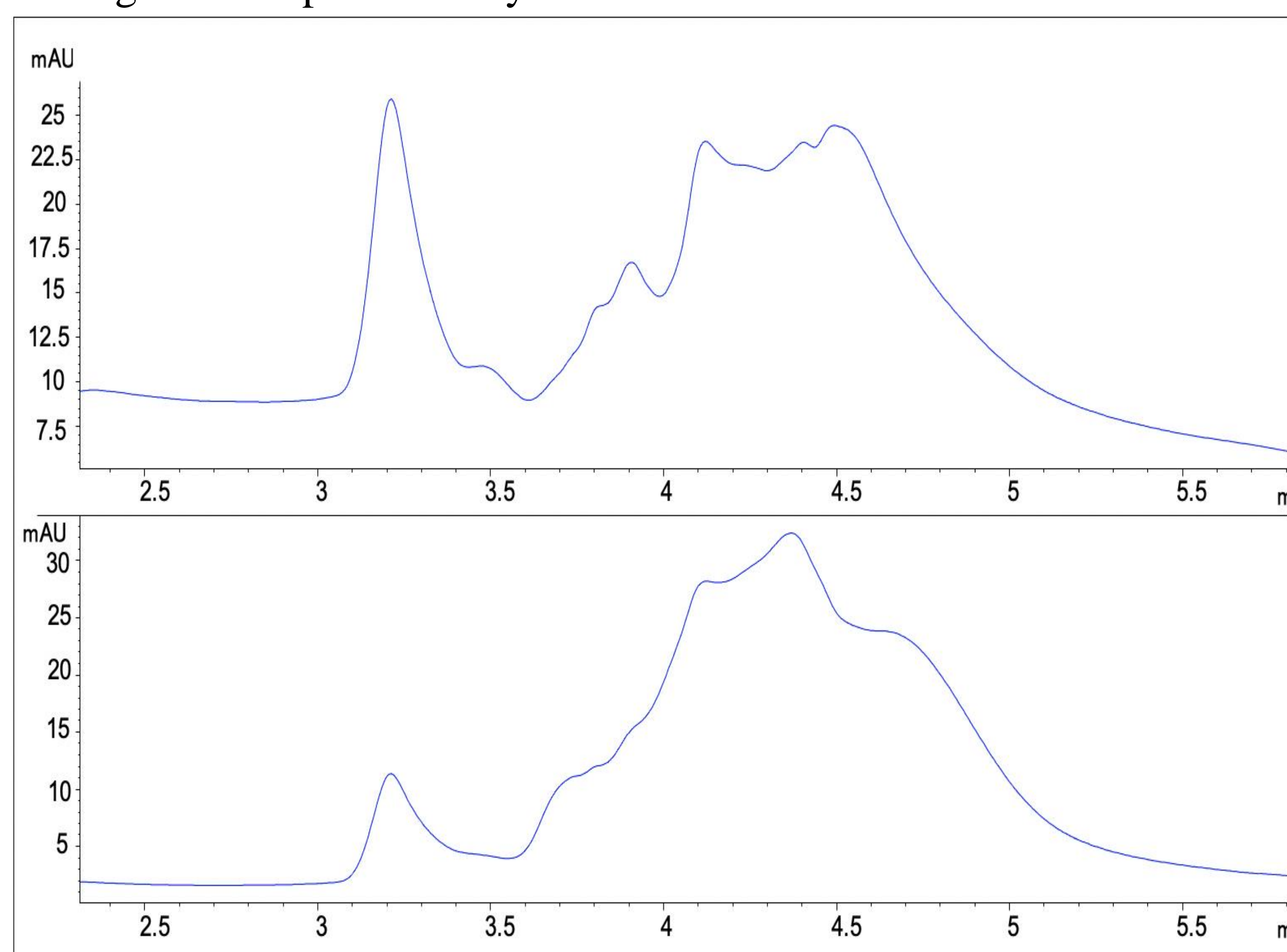


Figure 3: UV chromatogram of prepared Roundup sample under the conditions listed in Table 1.

Conclusion

Both chromatograms illustrate that one of the compounds in the mixture is being sufficiently separated with a retention time of 3 minutes. However, both spectra illustrate that several peaks are overlapping to produce a large, lumpy signal starting from a retention time of 3.5 minutes and ending at a retention time of 5 minutes. Therefore, the target separation has not been achieved.

However, the data does demonstrate that several unique peaks are starting to resolve around the four-minute mark on the 289 nm (top) graph. Namely, three separate peaks seem to be resolving at retention times of 3.75, 4.25, and 4.5 minutes, respectively. Adaptations to the experimental procedure are will be necessary to acquire better resolution for these peaks.

Future Direction

For future experiments, the components of these herbicides tend to run faster down the column in more non-polar solvents. Therefore, better separation will likely be achieved by gradual manipulating the polarity of mobile phase during the HPLC experiment. Better separation is also achieved through longer experiment run times with a more gradual increase in mobile phase polarity, therefore a longer method run time will be explored as well.

Furthermore, the quality of the data that was collected in the HPLC compared to the type of data that has been advertised seems to suggest that the column currently equipped to the machine may need to be replaced. If this is accomplished, sharper peaks with a much a higher resolution should be expected.

References

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