

A SIMPLE METHOD FOR THE DETERMINATION OF WATER CONTENT IN TETRAHYDROFURAN

Maria Xiberras¹, Everaldo Attard², Nicolette Sammut Bartolo¹, Anthony Serracino-Inglott¹

¹Department of Pharmacy, University of Malta, Msida, Malta

²Rural Sciences and Food Systems, University of Malta, Msida, Malta

email: maria.xiberras.15@um.edu.mt

INTRODUCTION

Water content in organic solvents used for chemical synthesis frequently influences the reaction outcome and yield.¹ Water content in solvents is usually determined using a Karl Fischer titration and azeotropic distillation. Both methods have limitations, the Karl Fischer apparatus is easily disturbed while the azeotropic distillation is complicated to conduct, time consuming and has a low precision in reading the volume of water collected.^{1,2}

AIM

To develop a simple method to determine the water content in tetrahydrofuran THF using UV/Vis spectroscopy with cobalt chloride (CoCl₂).

METHOD

A method developed by Bai and Yang¹ to determine the water content in alcohol-based solvents using CoCl₂ as an indicator and a UV/Vis spectrometer as an indicator was adapted to THF.

The development of the method for the determination of water content in THF was conducted in two phases:

- In phase 1, the suitability of CoCl₂ as an indicator for water content in THF was investigated. Samples of anhydrous THF and anhydrous THF with cobalt chloride (CoCl₂) were analysed using UV/Vis spectrometry to determine the maximum absorbance of both compounds.
- Phase 2 of the study entailed finding and analysing the difference in absorbance in relation to different water concentration in THF and CoCl₂ with UV/Vis spectroscopy. Vials for each water concentration were prepared in triplicates, by first measuring the HPLC grade water to be added (0.1, 1, 2, 5, 10 µl), with a micropipette, and then were topped up by the mixture of CoCl₂ with THF to reach a total volume of 1 ml.
- The stock solution used for the method development contained 3.51x10⁻³ mol/L CoCl₂ in THF and for the UV/Vis spectroscopy analysis, a 0.5mm cuvette and a wavelength range between 200nm to 800nm were used.

RESULTS

- THF has an absorbance between 200-320nm while absorbance in the region between 548-721nm was attributed to CoCl₂ with a maximum absorbance at 680nm. (Figure 1).
- The average absorbance of each water concentration was calculated and a graph of absorbance at 680nm against water concentration was plotted (Figure 2). It was observed that as the water concentration increased, the absorbance decreased with a polynomial second order trend.
- The equation obtained from the plotted graph (Figure 2), $y = 0.0016x^2 - 0.0241x + 0.3645$ with an R² value of 0.9625 may be used to measure water content in THF if the same concentration of CoCl₂ which was used.

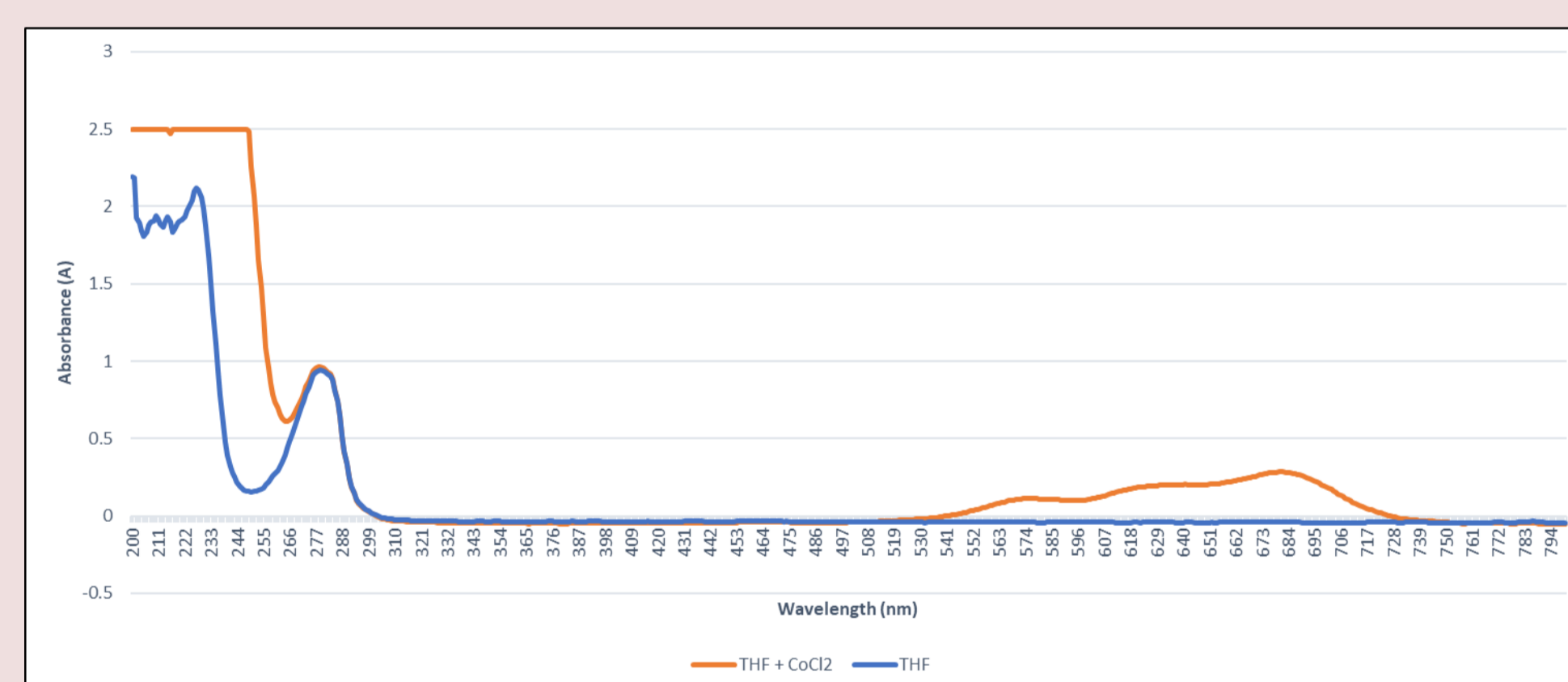


Figure 1: Uv/Vis spectra of THF and THF with CoCl₂

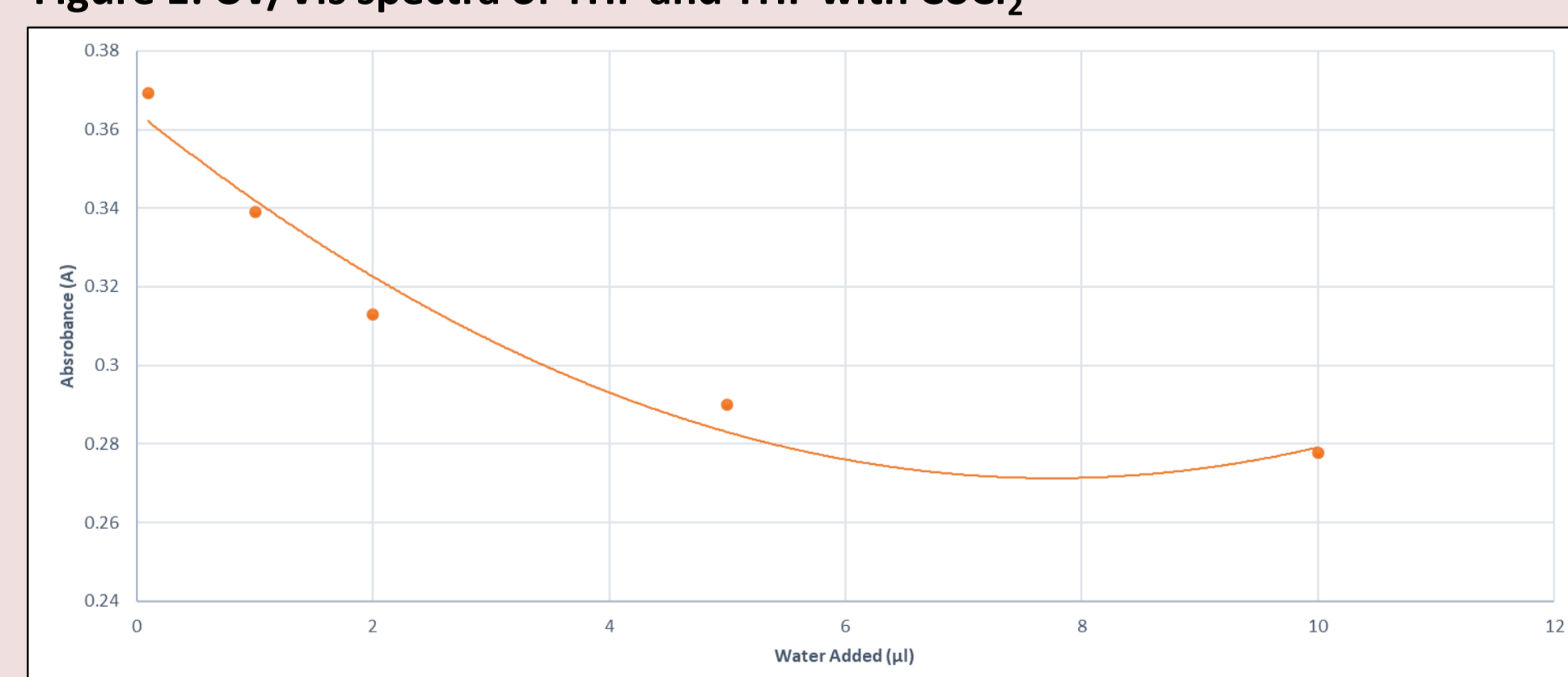


Figure 2: Average absorbance of THF with CoCl₂ at 680nm against water added

CONCLUSION

The developed method is simple, practical and makes use of a UV/Vis spectrometer, an apparatus which is both easy to use and available in most laboratories. The method may be used to calculate the efficacy of dehydrating agents such as magnesium sulphate, molecular sieves and calcium chloride to dry THF.

REFERENCES

¹Bai H, Yang X. Spectrophotometric determination of water content in alcohol organic solvents. Journal of the Chinese Chemical Society. 2007;54(3):619-624

²Fetzer W. Determination of Moisture by Distillation. Analytical Chemistry. 1951;23(8):1062-1069.