

## INTRODUCTION

Water content in organic solvents used for chemical synthesis frequently influences the reaction outcome and yield.<sup>1</sup> The Karl Fischer titration is one of the main methods used to measure water content in solvents.<sup>1</sup> It is established that the UV/VIS spectrometer is readily available in laboratories.

## AIMS

To develop an alternative method to the standard Karl Fischer method for the determination of water content in tetrahydrofuran (THF).

## METHOD

A method developed by Bai and Yang<sup>1</sup> to determine the water content in alcohol-based solvents using cobalt chloride (CoCl<sub>2</sub>) as an indicator and a UV/Vis spectrometer, was adapted for THF.

The development of the method for the determination of water content in THF was conducted in three steps.

Three different solutions of CoCl<sub>2</sub> in anhydrous THF were prepared (1.69x10<sup>-3</sup> mol/L, 3.42x10<sup>-3</sup> mol/L, 6.98x10<sup>-3</sup> mol/L)

Eight dilutions were prepared, in triplicates, for each CoCl<sub>2</sub>/ THF concentration by adding HPLC grade water (0.1, 0.5, 1, 2, 5, 8, 10, 15 μl) to reach a total volume of 1ml.

The prepared samples were analysed using UV/Vis spectrometry at a wavelength range between 200nm to 800nm

## RESULTS

- THF has an absorbance between 200-320nm while CoCl<sub>2</sub> has an absorbance between 480-720nm (Figure 1).
- The average absorbance for each dilution was calculated at 672nm and was plotted against the wavelength. Their respective polynomial equations were derived (Figures 2-4).
- An initial increase in absorbance was unexpectedly observed with the addition of water. This unexpected increase was however followed by a decrease in absorption on further addition of water. This could be due to an incomplete dissolution of CoCl<sub>2</sub> in THF which forms a very fine suspension, escaping detection.
- Addition of more water could result in affecting the complex equilibrium between the various forms of hydrates of CoCl<sub>2</sub> resulting in a decrease in the 672nm peak and introducing lower peaks due to the formation of the hydrates.

## CONCLUSION

The equations obtained from the plotted graphs of the average absorbance at 672nm against water added can be theoretically used to measure water content in THF if the same concentration of CoCl<sub>2</sub> is used. In practice, their respective R<sup>2</sup> values are low.

The method for determination of water investigated in this research requires further investigation and elucidation before it could be considered as a possible substitute for the established Karl Fischer method.

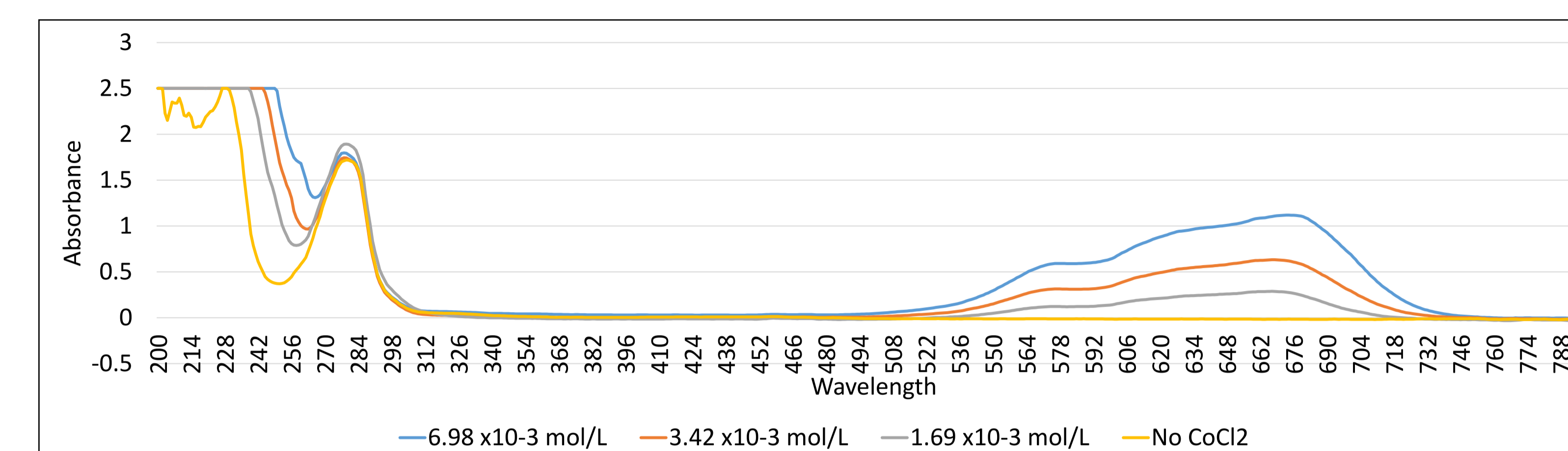


Figure 1: Absorbance against wavelength in different concentrations of CoCl<sub>2</sub>

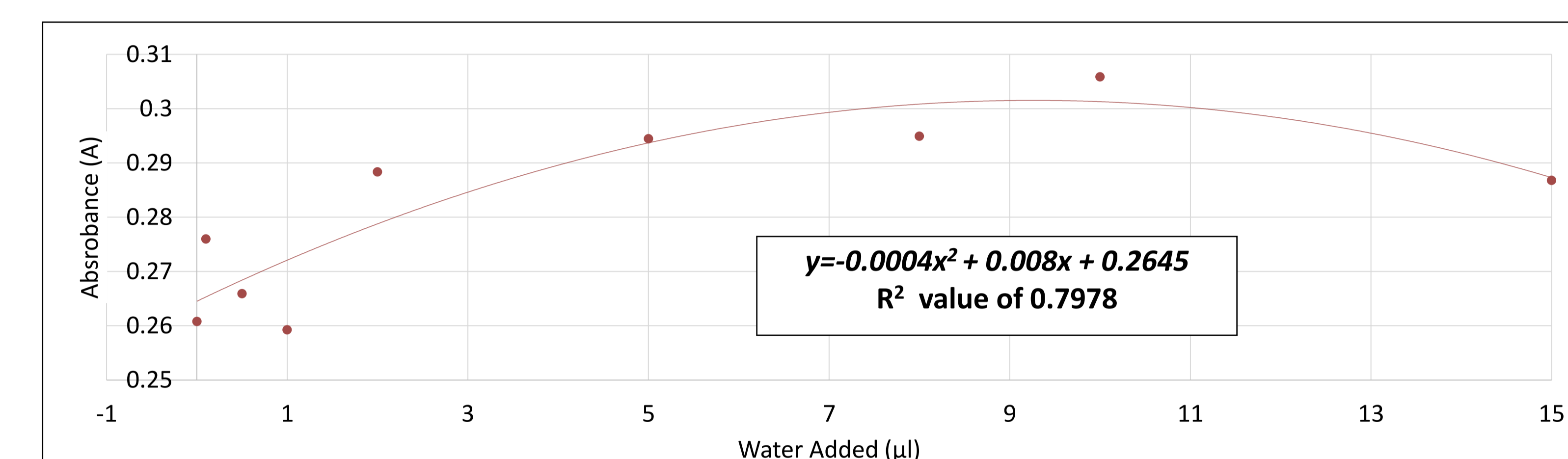


Figure 2: Average absorbance at 672nm against water added in 1.69x10<sup>-3</sup> mol/L CoCl<sub>2</sub> in THF

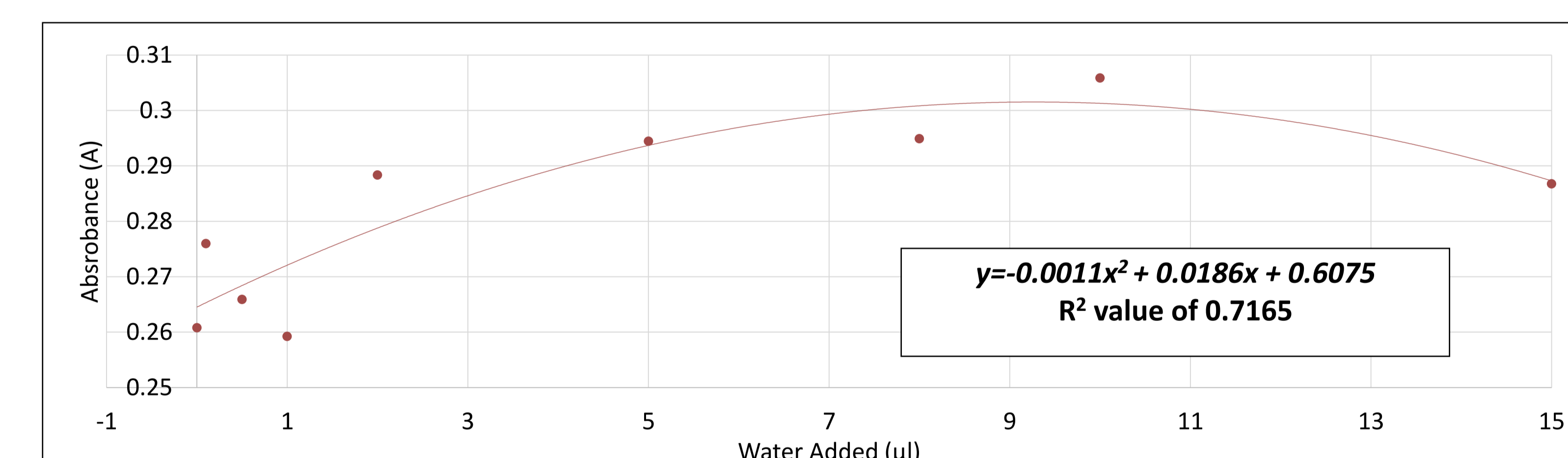


Figure 3: Average absorbance at 672nm against water added in 3.42x10<sup>-3</sup> mol/L CoCl<sub>2</sub> in THF

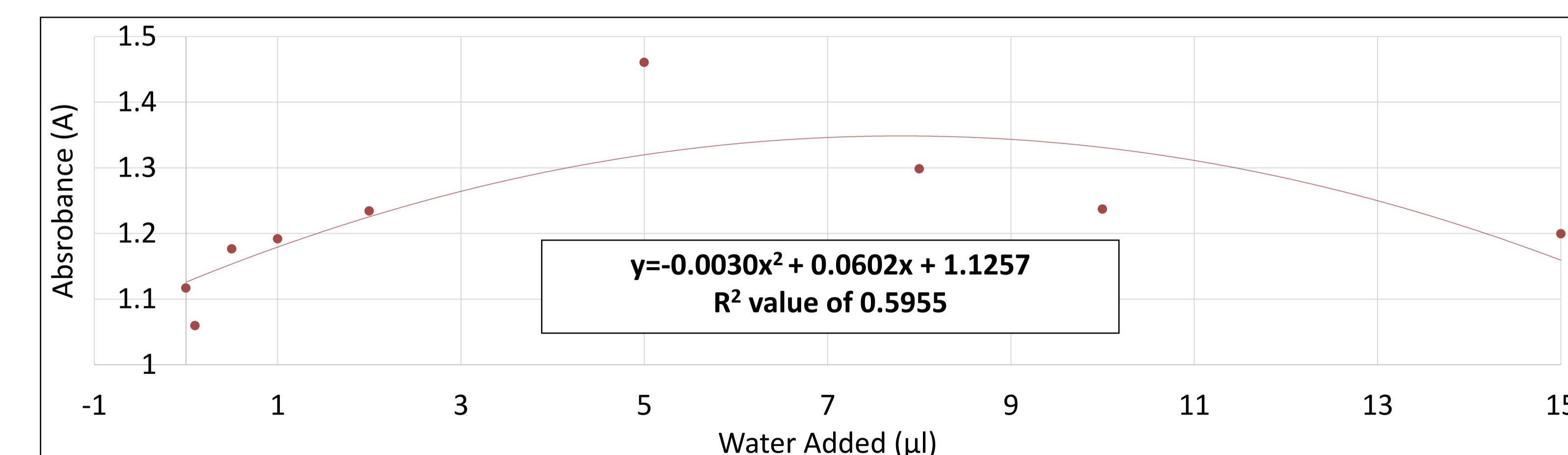


Figure 4: Average absorbance at 672nm against water added in 6.98x10<sup>-3</sup> mol/L CoCl<sub>2</sub> in THF

## REFERENCES

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