

PREREQUISITES FOR THE ANALYSIS OF METAL-EDTA COMPLEX USING LIQUID CHROMATOGRAPHY

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INTRODUCTION

Ethylenediaminetetraacetic acid (EDTA) can preferentially bind certain metal ions depending on the pH of the solution, which greatly facilitates the selectivity of the complexation itself. The complex thus prepared can be analyzed by liquid chromatography, however, the instrument itself is made of metal parts, whereby EDTA from the prepared drug sample can additionally bind free metal ions from the instrument, which can lead to inaccurate and unreliable results. Prior the analysis, thorough priming of the instrument with EDTA solution is mandatory.



METHODOLOGY AND RESULTS

Isocratic elution on C18 column is used with mobile phase consisting of TBAOH buffer and methanol mixture set to desired pH. Below are examples of chromatograms of Diluent and Blank solution obtained during the analyses of metal-EDTA complex, showing the possible interferences with the peak of interest.

DILUENT: OCCURANCE OF UNKNOWN PEAKS AS A MOBILE PHASE FLOWS THROUGH THE SYSTEM

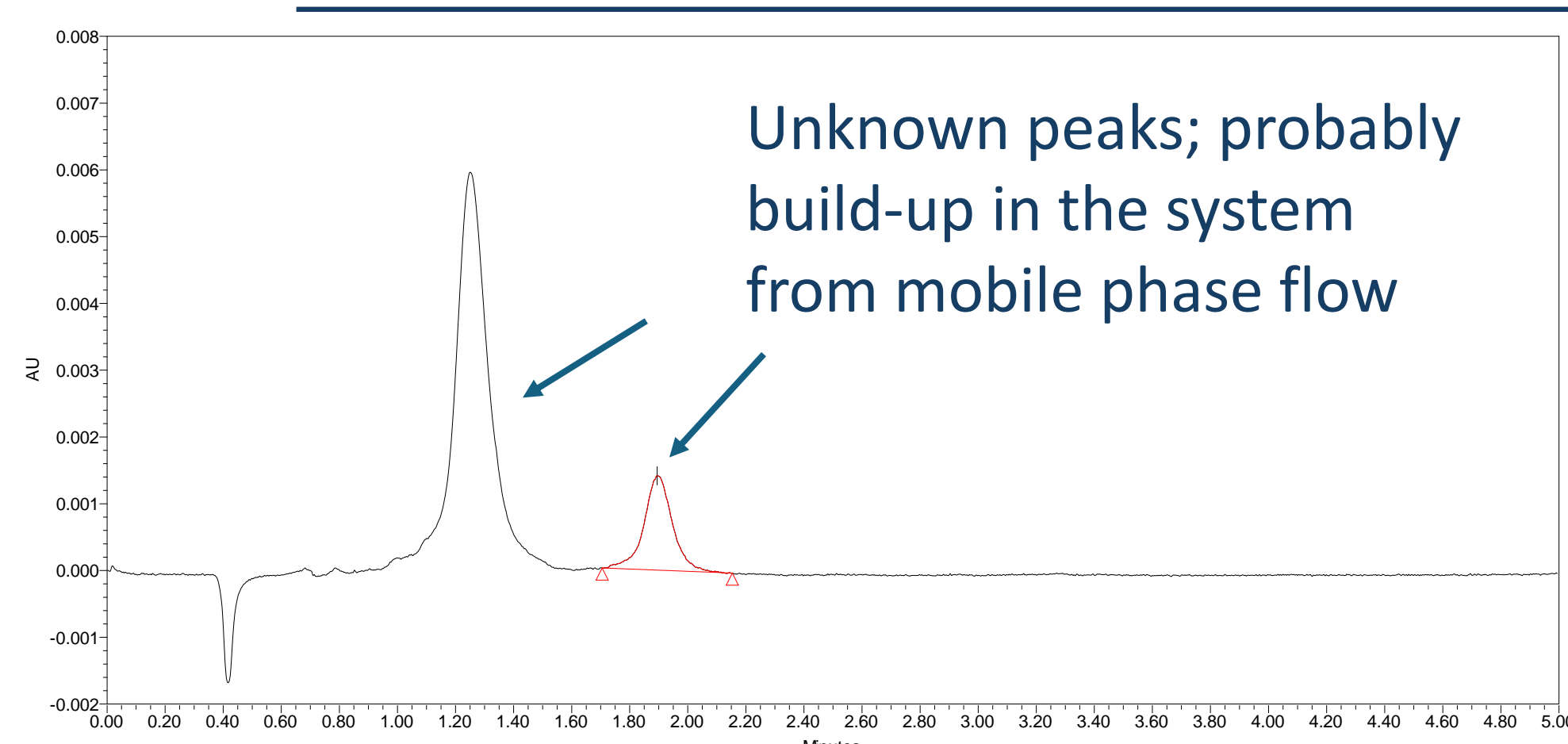


Figure 1. First injection of Diluent (contains no EDTA)

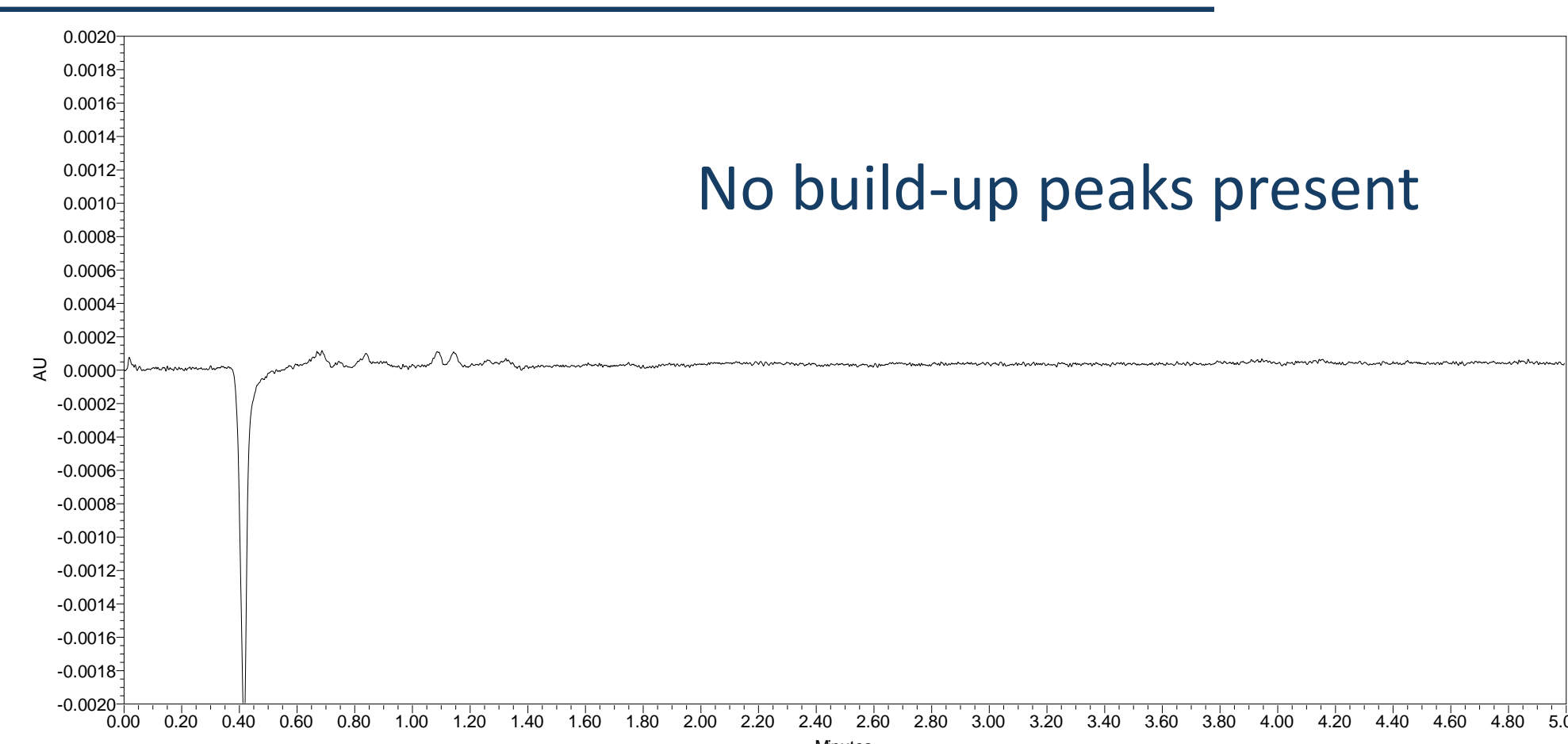


Figure 2. Following injection of Diluent

BLANK: OCCURANCE OF UNKNOWN PEAKS AS A MOBILE PHASE FLOWS THROUGH THE SYSTEM

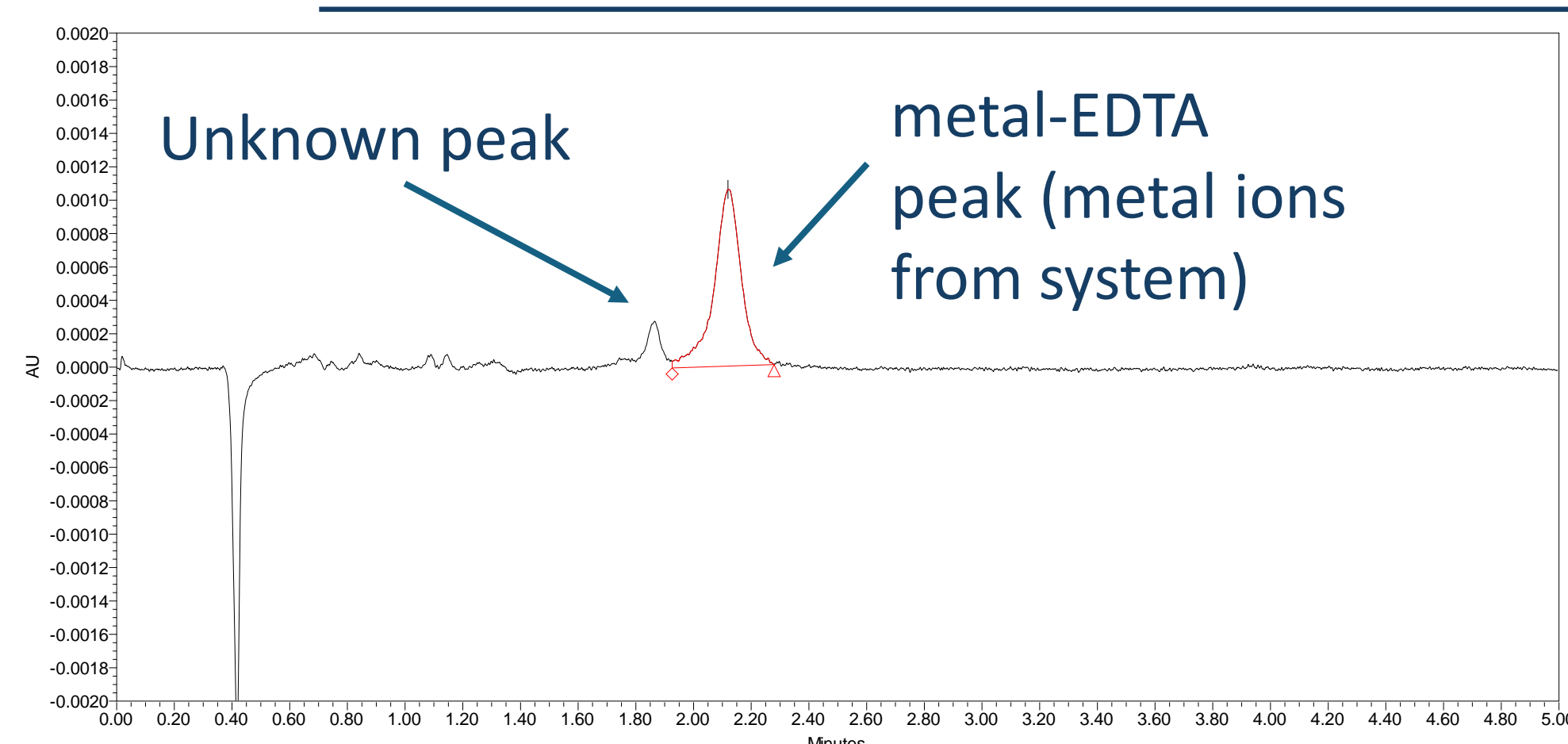


Figure 3. First injection of Blank

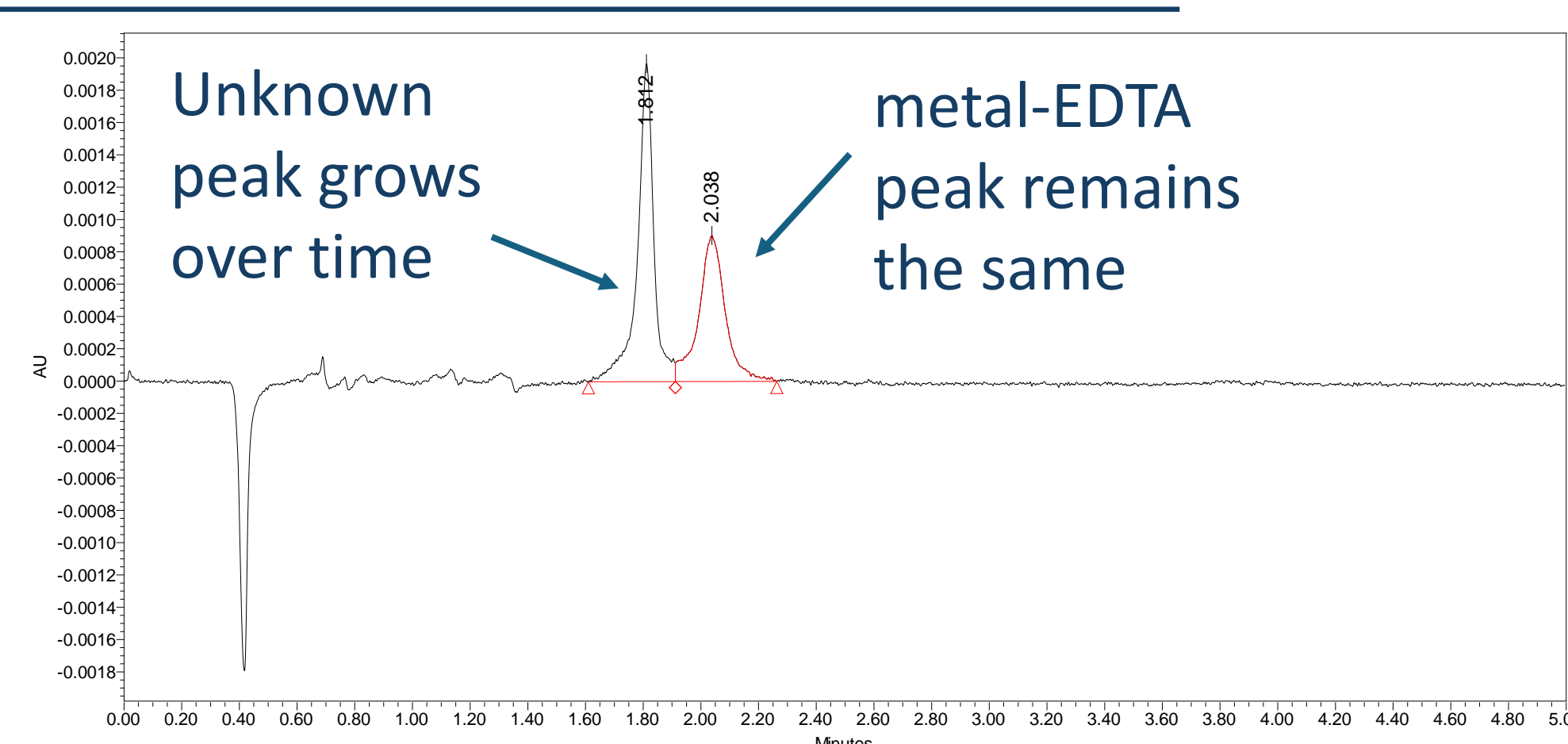


Figure 4. Blank after 24h hours of mobile phase flow

BLANK: AS IT SHOULD LOOK LIKE, AFTER THOROUGH PRIMING OF INSTRUMENT AND COLUMN WITH EDTA SOLUTION

Solution for instrument priming contains EDTA dissolved in mobile phase. Running EDTA solution through the system and chromatographic column removes majority of free metal ions from the system.

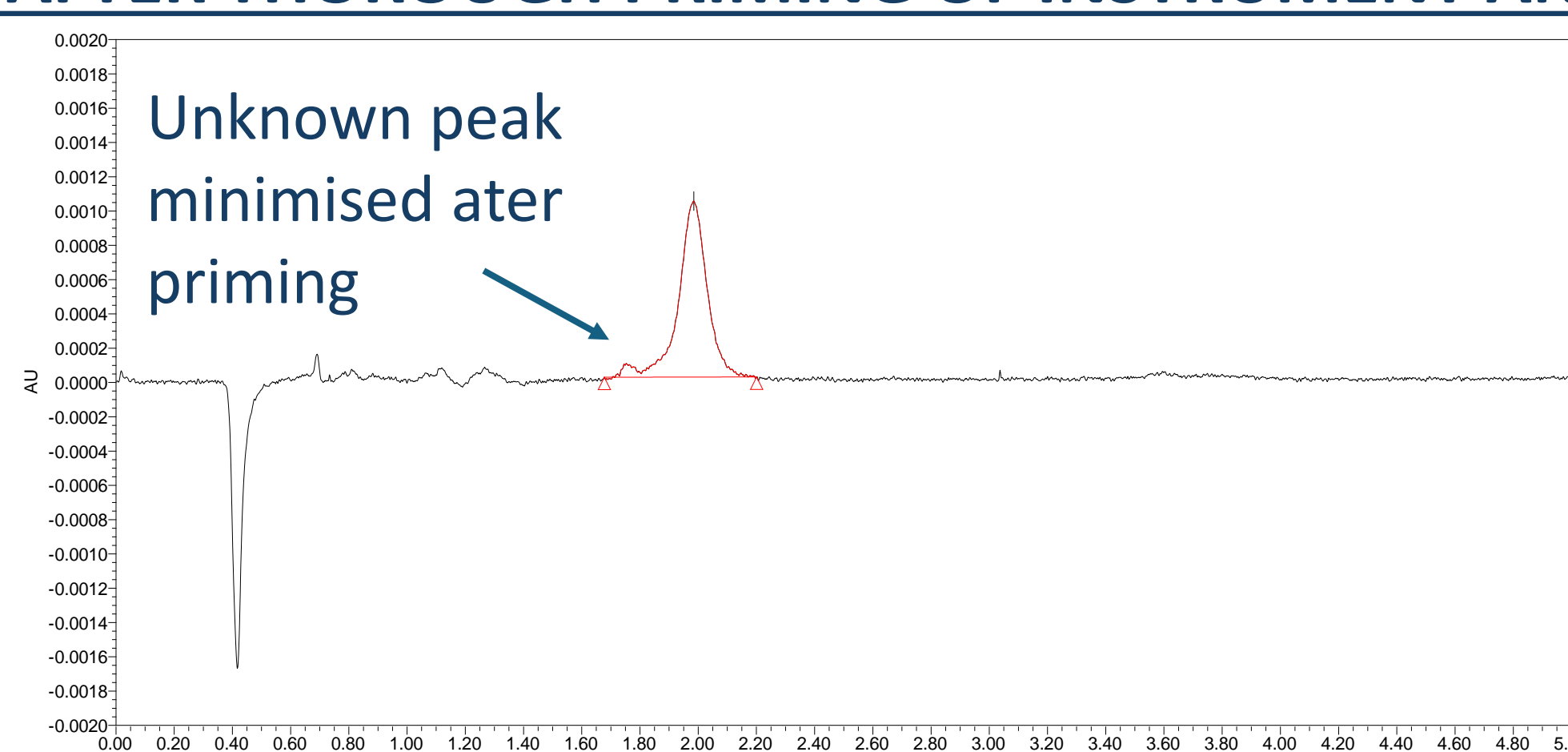


Figure 5. Blank solution after instrument priming

Although, we have metal-EDTA complex peak present in the Blank, it remains constant through whole analysis and therefore does not affect accuracy nor precision of the method.



CONCLUSION

In this poster presentation multiple examples of possible chromatographic interferences are shown. After thorough priming of instrument and column with EDTA solution, unknown peak in Blank is minimised, due to rinsing of majority of free metal ions from the system. Based on the results obtained above, it is recommended to perform instrument and column priming each day prior the analysis of metal-EDTA complex samples to prevent any build up of free metal ions in the system.