

Quantification of Veloutone® in trace amount from aqueous media

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Bachelor thesis, Analytical chemistry

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ABSTRACT

A LC-MS/MS method for the quantification of Veloutone® was developed, using ESI and APCI sources. Different SPE cartridges were screened to enhance the concentration of the analyte before analysis. Derivatization with three different positively charged reagents was tested to improve the LOQ.

INTRODUCTION

Veloutone® (+/-2,2,5-trimethyl-5-pentylcyclopentan-1-one) is a fragrance that slightly smells of flowers and fruits and is currently used in perfume applications¹. LC-MS/MS method is needed to quantify Veloutone® for REACH purposes. A new ESI-LC-MS/MS method with a LOQ of 300 ppb was developed on a Acquity/TSQ Vantage system. A three-time improvement of sensitivity was achieved by using APCI instead of ESI. Concentration of the analyte before analysis was needed to increase the sensitivity. SPEs of different polarities were tried, ranging from C18 to C8, using different desorption solvents and different methods for solvent recovery. One of the challenge to face was the adsorption of Veloutone® on the surfaces, that directly impaired the analytical results.

Derivatization of Veloutone® was attempted to overcome these problems by the formation of a Schiff base with positively charged hydrazides. Different solvents, reaction temperatures and reaction times were screened, but an in depth study is necessary to elucidate this point.

RESULTS

LC-MS/MS Method

UPLC: Waters Acquity

MS: TSQ Vantage

Column: Acquity BEH C18. 2.1 x 100 mm, 1.7µm

Solvents: A: H₂O + 0.1% FA, B: Methanol + 0.1% FA

Internal standard: Tonalide

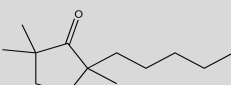


Figure 1: Veloutone®¹

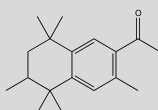


Figure 2: Tonalide²



Figure 3: Waters Acquity³ TSQ Vantage⁴



Figure 4:

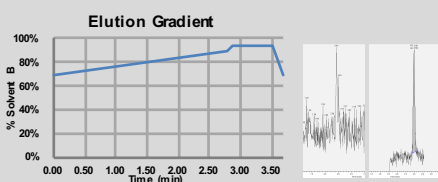


Figure 5: Elution gradient

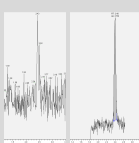


Figure 6:
300 ppb ESI
100 ppb APCI

SPE-Tests

Comparison of six SPE cartridges with different polarities.

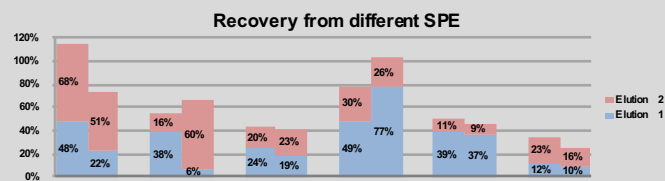


Figure 7: Recovery of different SPE eluted with ACN

Best recoveries were obtained with ENVI-8 and LC-8. For further tests, ENVI-8 was selected due to higher recovery with smaller elution volume.

Solvent screening for desorption

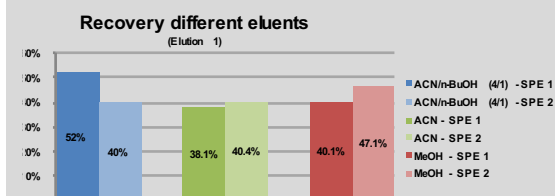


Figure 8: Recovery of different desorption solvents ENVI-8

ACN has been chosen as desorption solvent for further tests.

Recovery evaluation of different concentration levels.

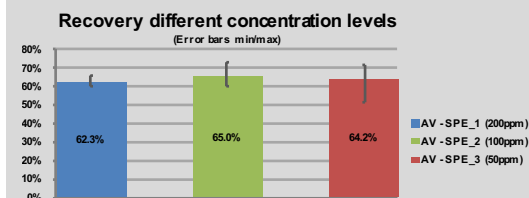


Figure 9: Recovery different concentration levels, ENVI-8, ACN

Average recovery 65%: loss of Veloutone® due to adsorption on surfaces. (glassware, polymeric SPE cartridges)

Derivatization

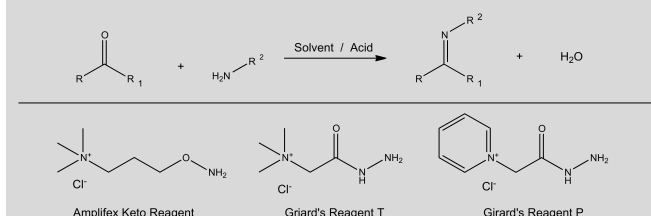


Figure 10: Derivatization⁵⁻⁷

CONCLUSIONS

1. APCI source three-times more sensitive than ESI
2. Best recovery with SPE C8but on average still 35% loss
3. Veloutone® recovery independent from desorption solvent
4. Adsorption on surfaces impact total recovery
5. Preliminary studies on Veloutone® derivatization conducted, to be continued

REFERENCES

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