

# Quantitative determination of NPEs by LC/MS

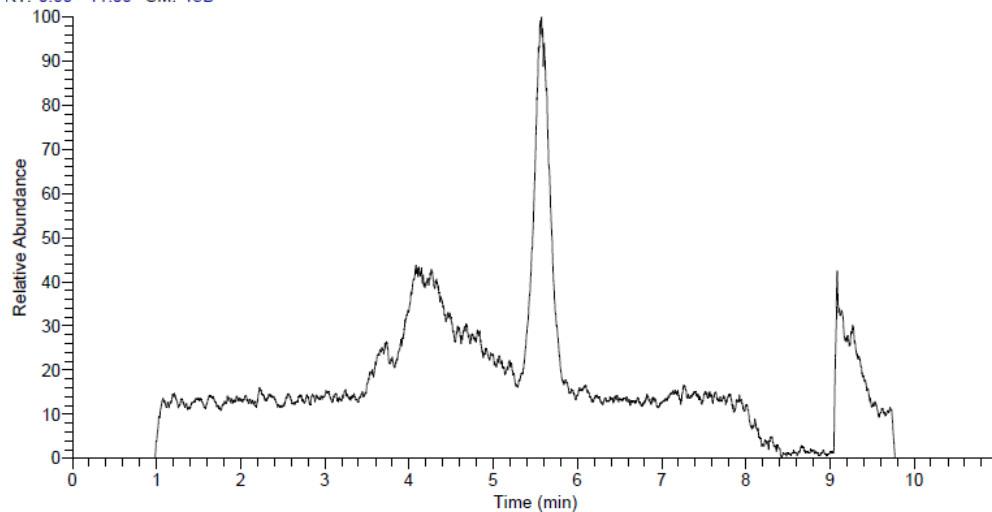
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## INTRODUCTION

NP/NPEs, also known as nonylphenols and nonylphenol ethoxylates, are nonionic surfactants, or detergent-like substances, with uses that lead to widespread release into aquatic environments. NPEs are used as detergents, emulsifiers, wetting agents, industrial processes, personal hygiene, automotive, latex paints, and lawn care products. Their presence in the environment is solely due to human activities, and mainly from WWTPs (waste water treatment plant) and direct discharge. Over 80% of

alkylphenol produced are nonylphenol based compounds. The rest are mostly octylphenol based compounds. NPEs are degraded to shorter chain nonylphenol ethoxylates as well as shorter nonylphenol ethoxycarboxylates under aerobic condition, before being biodegraded to nonylphenol. Shorter ethoxylate chain NPEs have greater toxicity than longer ones. These shorter chain NPEs have been reported to cause a number of estrogenic responses on aquatic organisms and thus they have been classified as endocrine disruptors (EDCs) by several organizations<sup>2</sup>. We have developed a method using liquid chromatography coupled with mass spectrometry for identification and quantitation of NPEs.

RT: 0.00 - 11.00 SM: 15B



NL: 1.63E4  
m/z=  
501.80-502.80+535.40-536.40+545.80-546.80+  
589.70-590.70+633.80-634.80+677.80-678.80+  
721.80-722.80+765.80-766.80+809.90-810.90+  
853.80-854.80+897.80-898.80 F: ITMS + c ESI Full ms  
[300.00-1500.00] MS 2174-9-L5-lcmsJ-05-1

2174-9-L5-lcmsJ-05-1 #1948-2025 RT: 5.46-5.68 AV: 78 NL: 1.98E3  
F: ITMS + c ESI Full ms [300.00-1500.00]

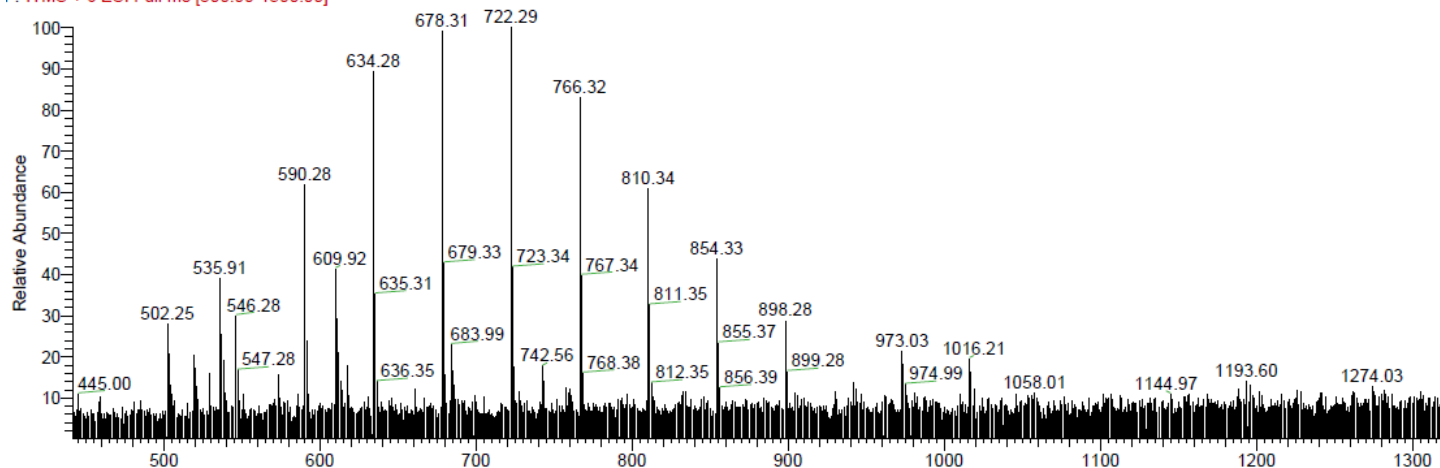


Figure 1. LC/MS chromatograms (above) and mass spectra (below) of NPE

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## LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY (LC/MS)

LC/MS is on the STL 17025 Scopes of Accreditation. LC/MS combines the techniques of HPLC and MS to characterize the structures of components in a complex matrix. HPLC is used for the separation of the compounds in the sample. A mixture of solvents or solutions, called the mobile phase, is forced at high pressure through a packed column, usually of coated silica particles, called the stationary phase. Components in the mixture are separated based on the difference in their affinities for the stationary phase and the mobile phase and can be detected and measured as they elute from the column. Typically, HPLC detection is performed using a UV detector monitoring absorption at a target wavelength or wavelengths. The time a chemical component spends in the column from injection until detection is known as retention time and is an indicator of component identity when compared with the retention time of known standards under the same conditions. The measured peak area or height is concentration dependent and may be used to quantify the component. Figure 1 shows the chromatographic and mass spectra profile of the standard.

## SUMMARY

NPEs are produced in large volumes for use in industrial processes

and in consumer detergents, hygiene products, automotive, paints, and landscaping products. The production and use of NPE's are prohibited in the European Union (EU) due to health and environmental effects. In the United States, the Environmental Protection Agency (EPA), has set recommended limits on NPE concentrations in fresh and salt water. The EPA is pushing for a voluntary phase-out of NPEs and future usage regulations similar to the EU.

EAG provides the quality NPEs testing data to help clients address these concerns and more:

- Qualify a product or manufacturer
- Perform an external quality control check for their industrial process
- Test potential contamination from the use of a product that contains NPEs
- Resolve a dispute which centers around the use of a product that contains NPEs

## REFERENCES

- [2] J.W. Birkett, J.N. Lester, Endocrine Disruptors in Wastewater and Sludge Treatment Processes, CRC Press, Boca Raton, FL, 2003.