Phenols derivatization method in GC-MS/MS

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Introduction

Phenols belong to the pesticide family, which can be found in water, air and ground. Like every pesticide, they are environmentally threatening and very toxic for the human body. Analyses to detect phenol traces are carried out either on drinking water samples or residual waters. These kinds of pesticides are non-volatile compounds; however, in order to be studied by CPG, it is necessary to have volatile or semi-volatile compounds. The goal of this method is to get phenol family compounds volatile with a chemical reaction called the derivatization.
As well, the laboratory would like to apply this operating method routinely. That means manipulations must be short and simple.

Experimental Conditions

The best adapted-operating method found was: to make a reaction of silylation with a silylant agent BSTFA (Bistriméthylsilyltrifluoroacetamide) and 1 % Triméthychlorosilane. All manipulations were carried out on two different mixtures containing several compounds of the phenols' family, as well as all of the mono molecules contained within reference mixtures. Once silylated, vials were injected into the GC-MS/MS (triple quadrupole). The program of chromatography was never modified, because it is the same for all manipulations, whereas the mass spectrometry method was modified during analyses. Experiments were worked out in scan mode (to determine the retention time of compounds) and in MRM mode (to quantify these compounds).

Results and discussion

In both mixtures all compounds were identified, as well as the transitions and energies of collision for each compound. Thanks to these results, a MRM method was created to analyze samples of water containing phenols. Once this method was established, a calibration range for Evian water was performed to draw a base line for calibration. Different volumes from reference mixtures were transferred to 1L water bottles. Then, an extraction was done to get organic phase phenols at the concentration of 1mol/L. This concentrated extract was then silylated. The range was analyzed with the MRM method previously created. Once a chromatogram was obtained (figure 1), it was necessary to assign each peak to a compound and create a table of affectation for the software, thereby allowing the software to trace calibration curves (figure 2). Therefore, in contaminated samples of waters, the concentration of every compound of phenol can be determined.

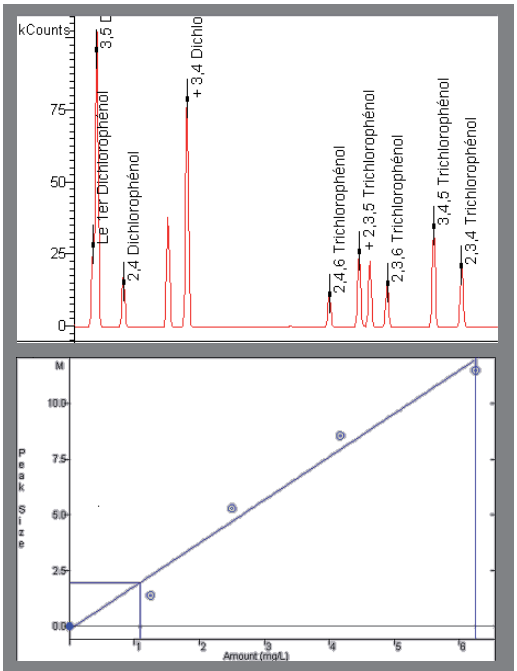

Figure1: Chromatogram of Trichlorophenols and Dichlorophenols silylated (obtained with the MRM method created).
Figure2: Calibration curve of 2,4,6 Trichlorophenol.

Conclusion

TPhenol derivatization has no big impact on using the GC-MS/MS triple quadrupole. Even though chromatographic peaks were more characteristic and compounds were better separated, the derivatization did not increase the sensibility of analysis. However, the conclusions could be different with an Ionic trap. This one is not sensitive to phenols lacking derivatization and allows one to view after derivatization, if sensibility increases or not.

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