Rapid determination of trace organophosphonates used as ionic surfactants in an aqueous system by reactive pyrolysis - GC/MS

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Organophosphonates such as 1-hydroxyethylidene-1,1-diphosphonic acid [HEDP] and bis (hexamethylene) triaminopenta (methylene-phosphonic acid) [BHMT] are used in a number of diverse applications such as industrial water treatment, household and industrial surfactants, pulp and paper processing, oil recovery, etc. Determination of trace organophosphonates in a water system usually involves extraction and/or preconcentration of the target components. The extracts then are often subjected to chromatographic or spectroscopic methods. This conventional methodology is not only cumbersome but often results in poor data quality.

This work presents a novel approach for the rapid and precise determination of trace amounts of organophosphonates in aqueous solution. It is based upon reactive pyrolysis–GC/MS in the presence of tetramethyl ammonium hydroxide (TMAH) [(1)] using the multi-functional micro-furnace pyrolyzer (PY-2020iD, Frontier-Lab, Japan). A 10µL aliquot of water sample containing the trace organophosphonate was injected into the sample cup that is used with the pyrolyzer; 1 µL of 25% of TMAH methanol solution was then added to the cup. Analysis was performed by introducing the sample cup into the furnace which was at 350°C. The heat initiated the hydrolysis followed by methylation of the organophosphonate, and the resulting products were detected by GC/MS to yield the pyrogram.

The reactive pyrolysis of an aqueous solution of HEDP yielded various phosphonate related products. Methyl dimethyl phosphonate and trimethyl phosphonate were clearly observed on the resulting pyrogram. Using a calibration based on an external standard, it was possible to make a rapid and precise determination of the ppm level organophosphate in water system. Precision and accuracy data clearly illustrate the advantages of the reactive pyrolysis method over the conventional methodology currently in use.

Reference:
(1) H.Ohtani, R.Fujii, S.Tsuge, *J. High Resolution Chromatogr.*, **14**, 388-391, 1991

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