

Determination of trace organophosphonate surfactant in aqueous media using Reactive Pyrolysis-GC/MS

[Background] The determination of organophosphonates, which are used as surfactants and as scale inhibitors, usually involves ion chromatography, capillary electrophoresis, and ICP. However, when analyzing a trace amount, cumbersome pretreatments such as extraction and concentration are required. Therefore, the development of simple and sensitive analytical methodology is of interest.

Reactive pyrolysis-GC/MS using tetramethyl ammonium hydroxide (TMAH) is a simple method for characterising polymers with ester groups and polar functionalities. This note shows the determination of 1-hydroxyethylidene-1,1-diphosphonic acid (HEDP, Fig. 1) in an aqueous media using reactive pyrolysis (RxPy) -GC/MS.

[Experimental] A Multi-Shot pyrolyzer (EGA/PY-3030D), which was directly interfaced to the split/splitless injector of a GC/MS system, was used. 10 µL of a 10 ppm aqueous solution of HEDP was placed in a sample cup (id=4 mm, height=8 mm), then an excess of TMAH (1 µL of 25 wt% methanol solution) was added. It was then subjected to RxPy-GC/MS analysis at 350°C.

[Results] The reactive pyrolysis of HEDP forms trimethylphosphate (TMP) as the main component (Fig.2); a small amount of dimethylphosphonate (DMP, Fig.3) is observed. The precision of the m/z 110 extracted ion chromatogram was 7.4%RSD (n=3), which is good and suggests that HEDP can be determined using the TMP peak. Also the detection limit was estimated to be 0.3 ppm (S/N=3), which is sensitive enough for the practical analysis of organophosphonates in aqueous media using RxPY-GC/MS.

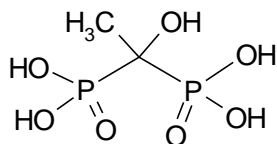


Fig.1 1-Hydroxyethylidene-1,1-diphosphonic acid (HEDP)

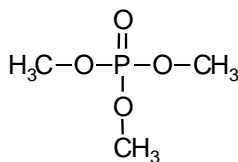


Fig.2 Trimethyl phosphate (TMP)

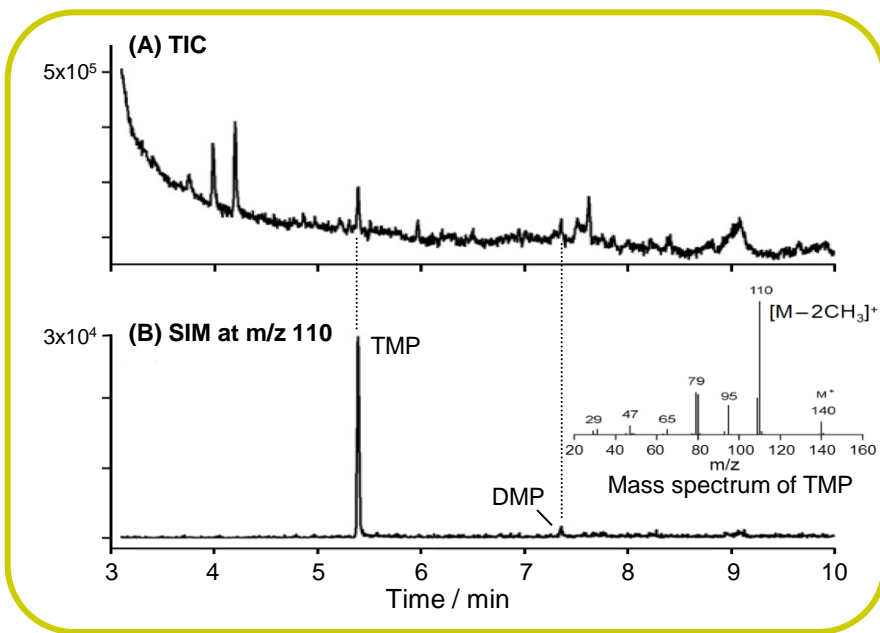


Fig.3 Chromatogram of aqueous solution of HEDP (10 ppm) obtained by reactive pyrolysis-GC/MS

Pyrolyzer furnace temp.: 350°C, GC oven temp.: 60-200°C (20°C/min, 3 min hold)
Separation column: Ultra ALLOY®- 5, (5% phenyl 95% dimethylpolysiloxane, L=30 m, id=0.25 mm, df=1 µm)

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Keywords : Organophosphonate, scale inhibitor, reactive pyrolysis, GC/MS, TMAH

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, UA-5

Applications : Scale inhibitor, Pulp and paper industry

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