

cartridge material emphasizes the need to use a direct methodology. Finally, using micro-LC-TID on a PRP-1 column with methanol-water (40 : 60, v/v) as eluent, IMPA was detected (60-nl injection; data not shown) and quantitated at the 10 ppm level by means of standard addition.

Next the sample was screened for the presence of polar hydrolysis products by means of micro-LC-FPD, using the PRP-X100 column and aqueous eluents containing 0-40% methanol and 0.5% formic acid. The FPD was used instead of the TID because of its better sensitivity when using low (< 50%) methanol concentrations in the eluent. A 450 mm x 0.32 mm i.d. PRP-X100 column was used to obtain sufficient separation for the structurally closely related polar organophosphates and phosphonates. The large amount of sodium hydroxide present in the sample ruled out the use of preconcentration (overloading of the ion-exchange precolumn). In order to increase sensitivity the use of relatively large volume injections (1-10 μ l) was investigated. Rather

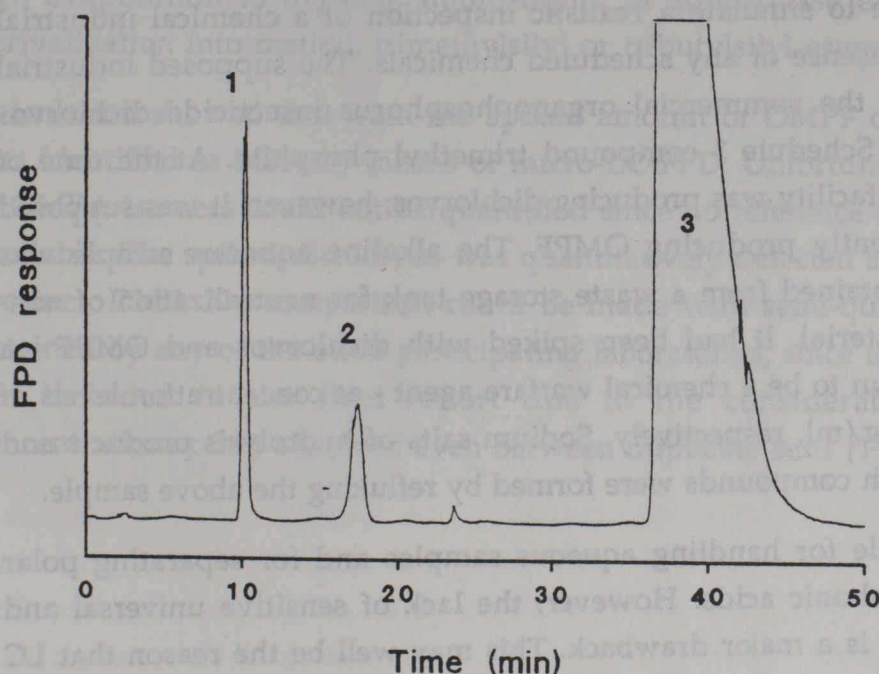


Fig. 10. Micro-LC-FPD of aqueous Round Robin II sample. Column, 450 mm x 0.32 mm i.d. packed with 10- μ m PRP-X100. Eluent, 0.5% formic acid in water; flow-rate, 15 μ l/min. Injection volume, 2 μ l. Peaks: 1, MPA; 2, MMP; 3, DMP.