

Experimental

Materials

All solvents were of HPLC-grade quality; they were supplied by Merck (Darmstadt, FRG). The 10 μm styrene-divinylbenzene copolymer PRP-1 and copolymer anion-exchanger PRP-X100 (Hamilton, Reno, NE, USA) were used as packing material for LC. Phosphorous acid was obtained from Merck.

The degradation products of nerve agents - methylphosphonic acid (MPA), ethyl methylphosphonic acid (EMPA), isopropyl methylphosphonic acid (IMPA) and pinacolyl methylphosphonic acid (PMPA), - the nerve agent sarin, the insecticide degradation products dimethyl phosphoric acid (DMP), monomethyl phosphoric acid (MMP) and diethyl phosphoric acid (DEP) as well as mustard (also known as mustard gas) and sesquimustard were synthesized in our laboratory and gave satisfactory elemental analyses and infrared, NMR, and MS data.

The various fused silica connection tubings (0.02-0.3 mm i.d.) were supplied by Chrompack (Middelburg, The Netherlands).

Set-up of the System

The set-up of the micro-LC-FPD system is shown in **Figure 1**. The eluent pump (I) is connected to a six-port Valco valve (A) which is provided with a 10 μl sample loop, and is connected to a second Valco valve (B) which contains an internal 60 nl sample loop and which is connected to the micro-LC column. With this set-up, it is possible to inject 1-10 μl of samples containing low ($\mu\text{g}/\text{ml}$) analyte concentrations (Valve A) and also - without changing the system - 60 nl of more concentrated (mg/ml) solutions used to study column focusing effects (Valve B).

A second micro pump (II) is directly connected to the interface (C) to deliver a make-up flow of water. The use of a make-up flow is essential to reduce the extra-column peak broadening of extremely narrow (compressed) peaks in the connective capillary tubing between the micro-LC column and the detector. Because the FPD is a mass-sensitive detector, its response is proportional to the