on the use of phosphorous trichloride. Detection of the presence of phosphorous acid can therefore give a clue regarding the origin of the sample.

Micro-LC-FPD was carried out on a PRP-X100 column using 0.5% formic acid in water as eluent. When diluted in water, independent of the pH of the sample, mustard hydrolyses relatively fast (half-life *ca.* 5 min, ref. 20). In the present instance, the samples were dissolved in water-acetonitrile (97.5 : 2.5, v/v), sonificated for one hour and centrifuged to obtain a concentration of 1-2 mg/ml of the basic hydrolysis product of mustard, thiodiglycol:

$$\underset{\textit{mustard}}{Cl\text{-}C_2H_4\text{-}S\text{-}C_2H_4\text{-}Cl} + 2H_2O \rightarrow \underset{\textit{thiodiglycol}}{HO\text{-}C_2H_4\text{-}S\text{-}C_2H_4\text{-}OH} + 2HCl$$

In order to effect peak compression for phosphorous acid and thiodiglycol, hydrochloric acid (0.01%) and n-butanol (9 vol.%) were added to the sample. Subsequently, 15 µl were injected onto the micro-LC column.

**Figure 6** shows the results obtained with a hydrolysed mustard sample (mustard : water, 1 : 10, v/v), with and without spiking with 200 ppb of phosphorous acid. The low detection limit of 10 ppb observed for phosphorous acid is rather gratifying. It can be achieved only because of the strong displacement of the major, non-ionic, sample constituents by n-butanol. Obviously, the present sample does not contain phosphorous acid. For the rest, large peaks show up in the 4-10 min retention time range. Peaks no.1 and no.2 can be assigned to thiodiglycol and 1,2-bis(2-hydroxyethylthio)ethane, respectively (assignment based on retention time data). Peak no.2 probably is the hydrolysis product of sesquimustard (1,2-bis(2-chloroethylthio)ethane), which was found to be one of the major impurities present in some of the original samples as identified by $^{1}H$ NMR, $^{13}C$ NMR and GC-MS. The unretained peak no.3 may well be (largely) due to thiodiglycol sulphoxide. Confirmation studies by means of LC-TSP-MS are presently in progress.