

Evaluation of Round Robin Test I. After completion of the test it was revealed that the soil samples had been spiked (level, 100 mg/kg) with VX and three of its hydrolysis products, viz. EMPA, EMPSA and the non-phosphorus-containing 2-diisopropylamino-ethanethiol.

As was discussed above two hydrolysis products, EMPA and EMPSA, were detected, while VX and 2-diisopropyl-aminoethanethiol were not observed. As regards VX, the present method was developed for the acidic hydrolysis products and is not suitable for the determination of the intact agents because these are not sufficiently retained on the anion-exchanger in the precolumn. As regards 2-diisopropyl-aminoethanethiol, lack of the phosphorus moiety prevents sufficiently sensitive detection. Actually, none of the ten participating laboratories reported finding this compound. It was concluded that, probably, the compound is converted into the corresponding disulphide or is too strongly bound to the soil [10].

From the ten laboratories which participated in the Round Robin test, seven were able to identify one or more of the acidic hydrolysis products. However, apart from our group only one other laboratory (no. 9 in Table 4) gave semi-quantitative results, which illustrates the problems involved in determining highly polar compounds in a soil matrix. The semi-quantitative results and analytical methods used are summarized in Table 4. Next to micro-LC-FPD more sophisticated techniques such as ^1H - and ^{31}P -NMR and high-resolution MS were used. With micro-LC-FPD, 80-100% of the added EMPSA was found in the soil sample which compares favourably with the results obtained by means of LC-MS and NMR. EMPA was found at a very low level of 0.2-3 ppm which agrees rather well with the LC-MS (0.2-0.5 ppm) and NMR (2.4-2.8 ppm) data. Probably, EMPA is degraded in the soil to MPA which was also found in the samples although it had not been added as a spike. Because EMPSA was recovered essentially quantitatively (80-100%), the MPA found in the soil must result from conversion of the spiked EMPA. In other words, with micro-LC-FPD 50-100% of the EMPA spiked to the soil was found indirectly, as was calculated from the MPA recovery (see Table 4) after correction for its molecular weight and low extraction efficiency. From results obtained with, e.g., GC-MS [10], it can be concluded that the major part of the spiked VX was still present intact in the soil.