

**Table 4** Semi-quantitative results of aqueous soil sample extracts  
(Round Robin test I)

Compound	Technique	Lab.No.**	Concentration (mg/kg) *		
			I	II	III
EMPSA	$\mu$ -LC/FPD	8	80	100	83
EMPSA	TSP-LC-MS	8	50	74	40
EMPSA	$^1\text{H}$ NMR	8	-	130	-
EMPSA	$^{31}\text{P}$ NMR	9	28	24	24
EMPA	$\mu$ -LC/FPD	8	1.3	0.2	3
EMPA	TSP-LC-MS	8	0.4	0.5	0.2
EMPA	$^{31}\text{P}$ NMR	9	2.8	2.4	2.4
MPA	$\mu$ -LC/FPD	8	12	18	10
MPA	TSP-LC-MS	8	24	29	18

\* Not corrected for extraction recovery: -, not analysed.

\*\* See ref. 11; No. 8: data from our laboratory.

### *Round Robin Test II*

Initial experiments with micro-LC-FPD using direct 60-nl injections under the same conditions as described above, indicated the presence of a large quantity of DMP in the strongly alkaline aqueous sample; this is a known hydrolysis product of dichlorvos. Next, the sample was screened by LC on a PRP-1 column. To detect hydrophobic decomposition products, using methanol-water (80:20, 60:40 or and 40:60, v/v) containing 0.7% of glacial acetic acid and 0.5% of 25% ammonia as eluent. Because the FPD cannot be used due to quenching of the analyte signal by methanol [4], the TID in the P mode was used as detector. Under these conditions the large amount of sodium ions present in the sample seriously disturbed the shape of the DMP peak which showed negligible retention on the PRP-1 column.

Off-line pretreatment carried out by passing 100  $\mu$ l of sample through a cation-exchange SCX cartridge (Bond Elut, Analytichem, Harbor City, CA, U.S.A.) to remove the ions considerably improved the DMP peak shape. Both the untreated sample and the sample flushed over the SCX cartridge were