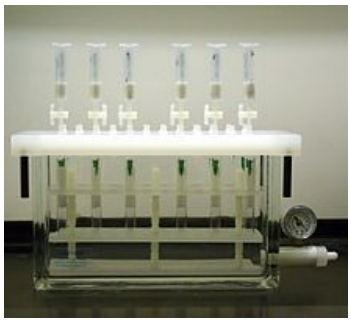
Determination organophosphorus pesticides (OPPs) in environmental water samples by Gas chromatography-Mass spectrometry after solid phase extraction (SPE)

The scope of this experimental project/application is the transfer of knowledge regarding to the analysis of organophosphorus pesticides in environmental water samples by GC-MS after preconcentration using solid phase extraction. This project will include four distinguishable experimental sections:

1) *Sample preparation*: Sample preparation protocol based on solid phase extraction will be carried out in order to preconcentrate the sample prior to its analysis. The theory behind this technique will be demonstrated and discussed through the particular application.

   A typical experimental pathway includes the following general steps:

   - SPE cartridge (OASIS HLB, 200 mg) condition: 3 mL CH_3OH, 3 mL H_2O
   - Loading: 250 mL of sample
   - Washing: 3 ml of mixture H_2O/CH_3OH, 95/5 % v/v
   - Elution: 6 mL CH_3OH
   - Addition of a portion of Na_2SO_4 and filtration through 0.22 um syringe filter
   - GC-MS analysis



2) GC-MS: in this step courses will be provided both on the theoretical and the operational aspects of GC-MS including software handling. The oven will be programmed to hold for 2 min at 80 ^{o}C, ramp to 200 ^{o}C at 15 ^{o}C min^{-1} and hold for 3 min, ramp to 240 ^{o}C at 5 ^{o}C min^{-1} and finally ramp to a temperature of 280 ^{o}C at 15 ^{o}C min^{-1} and hold for 3 min. The carrier flow rate will be 1.0 mL/min while the transfer line temperature will be set at 280 ^{o}C. All separations will be carried out on

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HP-5MS (5% phenyl methyl polysiloxane coated, 30 m x 0.250 um, 0.25 um film thickness) capillary column.

Looking to the MS parameters the temperature of ion source and the quadrupoles will be adjusted to 230 ^{o}C and 150 ^{o}C respectively. The quantification of the analytes will be performed in SIM mode. The retention time and the SIM segments are shown in the following table.

Pesticides	t_R **(min)**	**SIM segment start time (min)**	**Quantification ions**	**Identification ions**
Dichlorvos	7.27	5.00	109	185
Phorate	11.37	10.00	121	75, 260
Diazinon	12.55	12.00	179	199, 304
Ipronbenfos	13.19		204	246, 288
Chlorpyrifos	15.49	15.00	197	258, 314
Chlorfenvinphos	16.85	16.00	267	323, 325
Phenthoate	16.98		274	246, 121
Tetrachlorvinphos	17.79	17.30	329	109, 331
Triazophos	20.78	19.50	161	257, 285
TPP (ISTD)	22.03	21.50	326	325
Phosmet	22.76		160	317
Azinphos-methyl	23.66		160	132
Coumaphos	25.05	24.5	362	226, 210

3) Method validation: method validation parameters including linearity, precision and accuracy, LOD and LOQ will be investigated and discussed. Calibration curves will be constructed analyzing a series of standard solutions in low ppb levels. Statistical evaluation of the calibration curve will be conducted while a set of experiments will be devoted on the investigation of the intra-day reproducibility and to calculate the LOD and LOQ values.

4) Application: the final step involves the analysis of real and/or spiked samples while a significant part of this lecture will be focused on the data interpretation and evaluation.

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