

ANALYTICAL PROCEDURE FOR PHOSPHOROUS

Extraction Procedure

Approximately 5 g portions of sample were extracted with 10 ml aliquots of toluene on a gyratory shaker at 400 rpm for 20 minutes. A surrogate, 1,3-dichlorobenzene, was added to achieve a level of 100 ug/L in the final 10.0 ml extract. Before analysis, 10.0 uL of 10000 ug/L 1,4-dichlorobenzene-d₄ was added to achieve a concentration of 100ug/L in the extract.

Analysis Procedure

The sample extracts were analysed by GC/MS using an HP 5995C Gas Chromatograph/ Mass Spectrometer interfaced with a HP-1000 RTE-6/VM computer. Sample injections were performed with an HP-7673A autoinjector.

Instrument Conditions

Column	Restek Rtx-5 (crossbonded SE-54) 30 m x 0.32 mm ID, 0.50 u film thickness
Injector Temperature	290° C
Transfer Temperature	290° C
Source Temperature	240° C
Analyser Temperature	240° C
Temperature Program	30° C for 3 min 15° C/min to 70° C, hold for 0.2 min 8° C/min to 145 C, hold for 1 min
Splitless Injection	60 sec split time
Injection Volume	2.0 uL

The ions were monitored between 9.50 and 11.50 minutes. Ions monitored were:

Compound	Ions	Quantified Ion
1,4-dichlorobenzene-d ₄	78, 150, 152	150
1,3-dichlorobenzene	112, 146, 148	146
Phosphorous	62, 93, 124	124

The GC/MS system was calibrated by analysing a 6 point calibration range of a phosphorous standard at 5, 10, 25, 100, 250, and 500 ng/ml in toluene. The Relative Standard Deviation should be +/- 10 percent. Before analysis each day, the system must a) pass DFTPP tuning, and b) pass a continuing calibration check by analysing a 100 ng/ml daily standard for phosphorous in which the difference between the result and the standard was less than 25%.

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