

Experiment 215 GC-MS method

The duplicate plate destined for GC analysis was transferred to a dry box (nitrogen atmosphere) and 60 μ L of derivitization reagent (BSTFA in anhydrous pyridine containing butylated hydroxytoluene as a derivitization standard) was added to each dried sample. Process blanks, solvent blanks (SB, 100% derivative reagent) and blank blanks were processed identically. Samples were sealed using crimp-caps, and the plate was removed from the dry box, vortexed (2000 rpm, 2 min), and incubated (60°C, 30 min) on a heat block. The GC plate was allowed to cool to RT, then transferred to the GC autosampler. GC analyses were done on an Agilent 7890A-5975C inert XL MSD GCMS instrument. Samples were injected (1 μ L, split 10:1, 250C) onto a HP-5MS 5% phenyl-methyl Silox (30 m x 250 μ M x 0.25 μ M) column and eluted (1 mL/min H₂, 60C(0.5 min hold), 10 C/min to 80C, 50 C/min to 325C, 4.5 min at 325C for 11.9 min total run time), transferred to the MSD unit (280C), ionized (EI, 70V), and scanned from 800-50 m/z after a solvent delay of 3 min (source 230C, quad at 150C). Data was analyzed using Agilent CHEMSTATION software.