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Calibration Curves for PFPH Formaldehyde Hydrazone using Thermal Desorption

To make the derivatizing reagent, 1000 nMoles of pentafluorophenyl hydrazine (Aldrich 156388) was added to a 500 ml volumetric flask and dissolved in a suitable amount of anhydrous methanol. Slight sonication may be necessary to insure complete dissolution. The standard solution was prepared by adding 10 milligrams of 37% formaldehyde solution (Aldrich 252549) to a 100ml volumetric flask, which is then brought to volume with the methanolic PFPH. The flask should be allowed to stand undisturbed for at least 2 hours. The 100 ml volumetric contains 100 µg of formaldehyde per ml. Standard 6 mm thermal desorption tubes packed with Tenax were quantitatively loaded with a series of concentrations ranging from 10 µg to 50 µg (in 10 µg increments) and a series from 2 µg to 10 µg (in 2 µg increments) repectively, using a Dynatherm Model 60 Tube conditioner with spiking station. The samples were then thermally desorbed using the CDS 9300 Autosampler, which was interfaced to a gas chromatograph/mass spectrometer. The PFPH formaldehyde hydrazone derivative is detected using single ion monitoring for the unique molecular ion m/e 210.

Figure1 is a calibration curve of 10 µg to 50 µg of the formaldehyde hydrazone derivative. The R^2 for this linearity plot is 0.97. Figure 2 is a plot of percent carryover of the formaldehyde hydrazone derivative from this analysis. Note that carryover is less than 1% at all concentration levels. Figure 3 is a linearity plot of the 2 µg to10 µg level. The R^2 for this plot is 0.98.

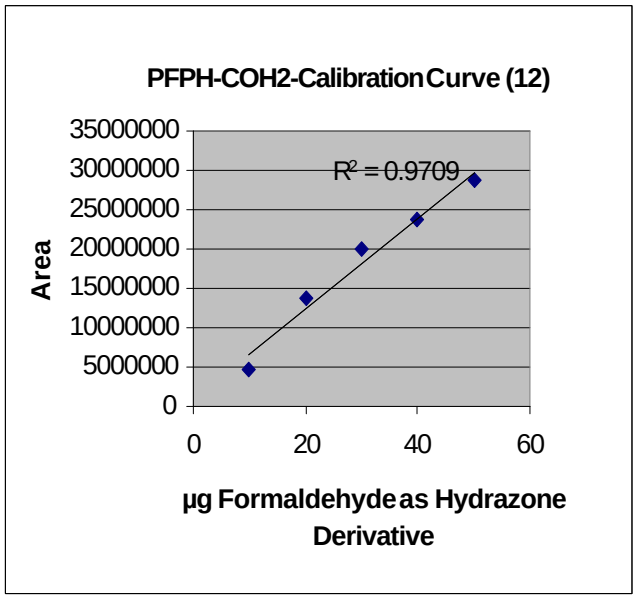

Figure 1.

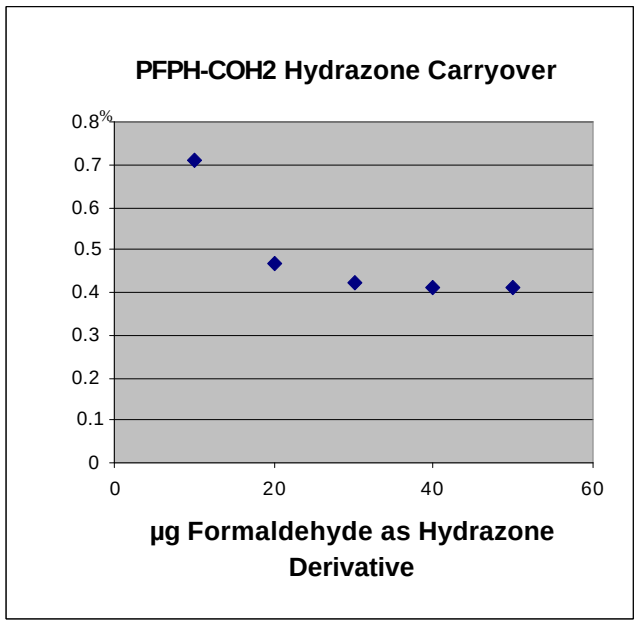

Figure 2.

CDS 9300 TDA Conditions:

Valve Oven:	300°C	
Transfer Line:	300°C	
Tube Idle:	40°C	
Dry Tube:	40°C	0.00 Minutes
Tube Heat:	300°C	15.00 Minutes
Tube Cool:		0.00 Minutes
Trap Idle:	40°C	
Trap Heat:	300°C	10.00 Minutes
Interconnect Line:	300°C	
Sample Saver Idle:	45°C	
Sample Saver Heat:	0°C	0.00 Minutes

GC Conditions:

Column: CP Select 624, 30mm x 0.25mm x 1.4µm

GC Program:
40°C for 4 Min,
7°C/min to100°C,
8°C/min to225°C, 2 min hold

Column Flow:	1ml/min
Split:	200
Solvent Delay:	19.50 Minutes

Mass Spectrometer:

Ion trap
Mode: Single Ion Selection (m/e 210)

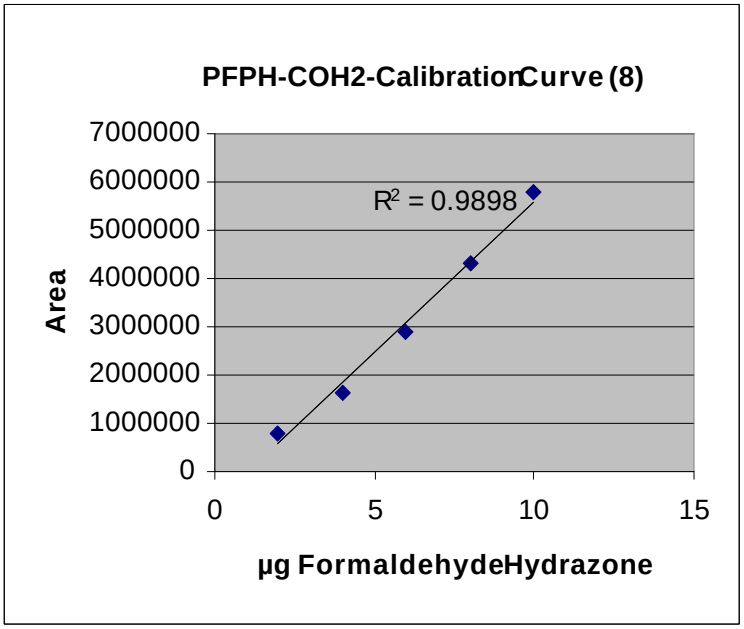

Figure 3.

Further Information:

HO and YU, Environ. Sci Technol, 2004, 38, 862-870.

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