Calibration Curves for PFPH Formaldehyde Hydrazone Using Thermal Desorption

Application Note
Environment

Author:

C. Zawodny

To make the derivatizing reagent, 1000nMoles 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 ensure 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 was allowed to stand undisturbed for at least 2 hours. The 100 ml volumetric contained 100 µg of formaldehyde per ml. Standard 6mm 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 a CDS Autosampler, which was interfaced to a gas chromatograph/mass spectrometer. The PFPH formaldehyde hydrazone derivative was detected using single ion monitoring for the unique molecular ion m/e 210.

Figure1 is a calibration curve of 10 µg to 50µg of the formaldehyde hydrazone derivative. The R2 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 carryoverwas less than 1% at all concentration levels. Figure 3 is a linearity plot of the 2 µg to10 µg level. The R2 for this plot is 0.98.

Instrument Conditions

CDS Autosampler Dynatherm 9300

Valve Oven:	300°C
Transfer Line:	300°C
Tube Heat:	300°C 15 min
Trap Heat:	300°C 10 min

When using an empty thermal desorption tube for "headspace" analysis, it is important to not heat a sample past its melting point.

GC/MS

Column:	CP-Select 624 (30m x 0.25mm x1.4µm)
Carrier:	Helium, 200:1 split
Injector:	220°C
Oven:	40°C for 4 minutes
	7°C/min to 100°C
	8°C/min to225°C, 2 min hold

Solvent Delay: 19.50 Minutes
Mode: Single Ion Selection (m/e 210)

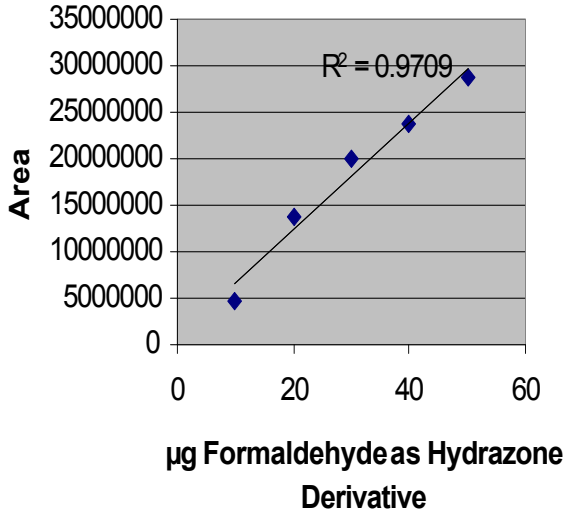

Figure 1. PFPH Formaldehyde Calibration Curve, 10µg to 50µg

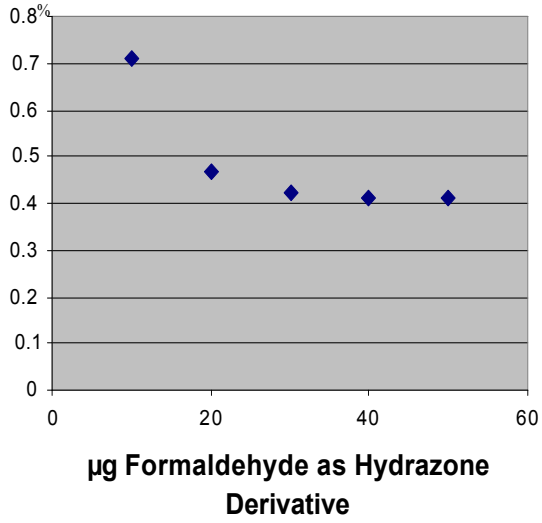

Figure 2. PFPH Formaldehyde Carryover

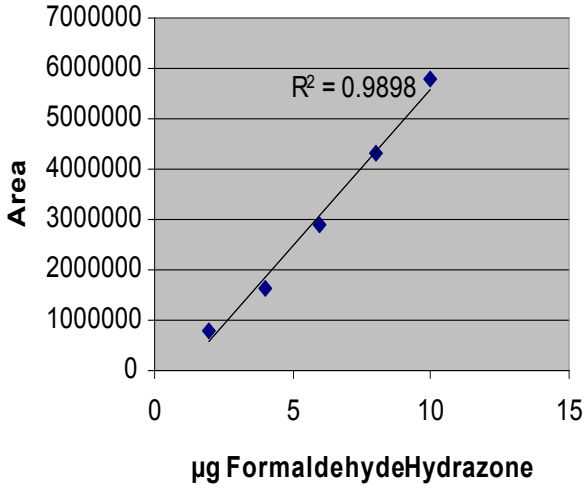

Figure 3. PFPH Formaldehyde Calibration Curve, 2µg to 10µg