Modified FID for Determination of Formaldehyde in Consumer Products

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Introduction

Formaldehyde is a hazardous compound commonly found in treated wood, caulk, hair products, and a variety of other substances (soil near burial grounds, glue, plywood, fiberboard insulation, car exhaust, etc.). OHSA established an exposure limit of 0.75 ppm on average over an 8-hour work day. Measuring compounds like formaldehyde and formic acid by gas chromatography is difficult due to the low response in the flame ionization detector. Historically, formaldehyde determination required the addition of a mass spectrometer (MS) or photo ionization detector (PID). There are also HPLC methods, but those require the use of post column derivatization to detect the formaldehyde.

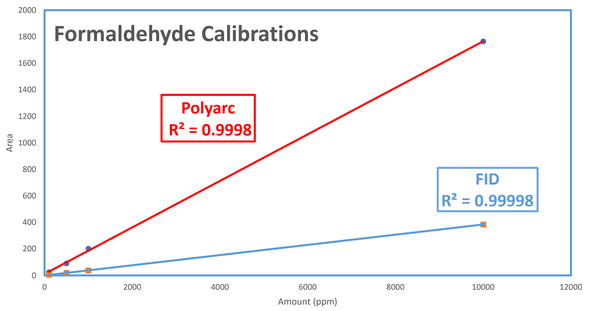
In this study, we investigate the use of the Polyarc® two-stage reactor in-line with a flame ionization detector to measure the presence of formic acid and formaldehyde. The first stage of the reactor oxidizes all the carbon-containing compounds into carbon dioxide. The second stage employs a proprietary catalyst in a hydrogen-rich environment to convert the carbon dioxide into methane, which is then easily detected by the FID. We investigate the use of this detection scheme to measure low levels of formaldehyde and formic acid in a variety of consumer products.

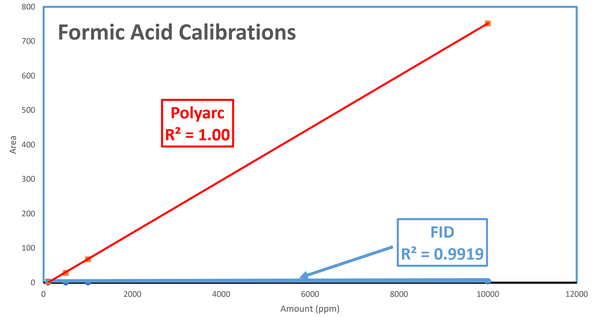
Experimental Conditions

Parameter	Conditions
GC	Agilent 7890B
Column	30 m x 0.25 mm x 0.25 µm DB-5
Temperature Program	Isothermal at 60°C
Flow Mode	Constant Flow
Linear Velocity	32 cm/sec
Carrier Gas	Helium
Septum Purge	3 mL/min
Injection Mode	Split 10:1 (Headspace Injections)
	Split 50:1 (Liquid Injections)
Injection Volume	0.5 mL (Headspace Injections)
	1 µL (Liquid Injections)
Inlet Temperature	250°C
Detector Temperature	250°C
Polyarc® Temperature	400°C
Polyarc® Flow H_2	35 mL/min
Polyarc® Flow Air	2.5 mL/min

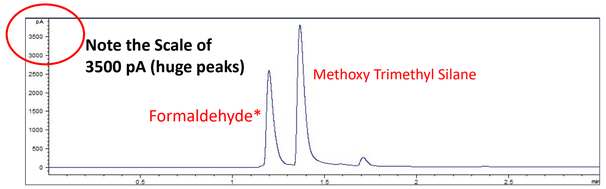
The hydrogen used in this study was generated on demand by a Parker Hannifin H2PEMPD-1300 hydrogen generator (1300 mL/min at 175 psi).

Calibration Curves on FID and Polyarc® (Liquid Injections)





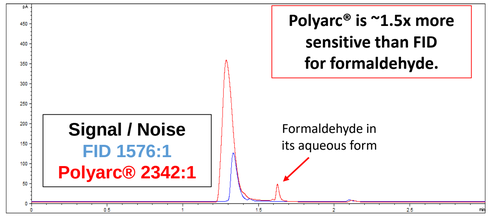
Silicone Caulk (Headspace)



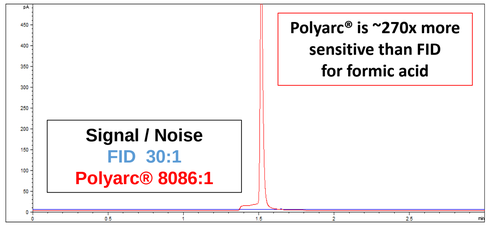
** Formaldehyde exists in different forms in aqueous solutions. This is the peak generated when injecting an aqueous formaldehyde standard.*

Results

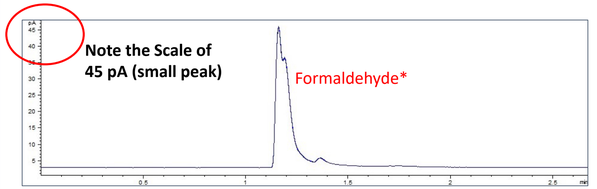
Formaldehyde Sensitivity Polyarc® vs FID (10,000ppm)



Formic Acid Sensitivity Polyarc® vs FID (10,000ppm)



Hair Relaxer (Headspace)



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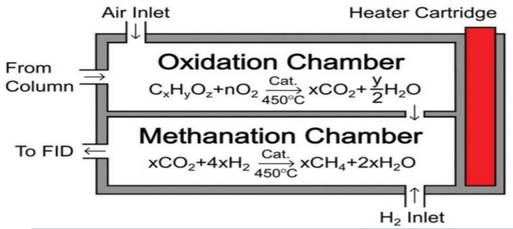
Polyarc® Reaction Chemistry



Courtesy of: activated research company®

Conclusion

The Polyarc® was determined to be an effective tool for analyzing formaldehyde and formic acid. Compared to an FID, the Polyarc® had a 1.5 fold increase in sensitivity for formaldehyde and a 270 fold increase in sensitivity for formic acid. Historically, formaldehyde has been difficult to analyze by GC since formaldehyde lacks enough C-H bonds to produce a significant signal from an FID. To mitigate that problem, the Polyarc® first oxidizes all of the incoming hydrocarbons into carbon dioxide and water by adding oxygen (air), heat, and a catalyst. The subsequent CO2 is then hydrogenated to methane with hydrogen, heat, and a propriety catalyst. The net result is that every carbon is converted to methane, thereby maximizing each compound's response in the FID. This is especially important for carbon-containing compounds with little to no C-H bonds and should work well for compounds such as CO, CO2, CS2, formaldehyde, formic acid, methanol, etc. It should also be noted that installation of the Polyarc® system was very straightforward, and the operation is simple and robust.

Acknowledgements

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