Hydrogen as a Carrier Gas in the Analysis of Polar/Non-Polar Compounds Using the Polyarc® System

Application Note

Chemicals

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Abstract

Utilizing the ARC Polyarc® system and FID combination, hydrogen was evaluated as a carrier gas while investigating polar compounds known for potentially poor chromatographic peak shape and response factors. Compounds chosen were n-Decane, n-Octanol, Aniline, m-Cresol, Triethyleneglycol (TEG), Catechol and n-Hexadecane. n-Decane and n-Hexadecane were included for comparison as more ideal compounds for chromatography and response factors. All compounds were treated as external standards between the levels of 0.2 – 5 weight % as carbon. Hydrogen (Air Liquide, Alphagaz1, 99.999% purity) was used as the column carrier gas to evaluate any potential effects on the reactor.

All compounds showed good peak shape and the Polyarc®/FID response factors between components narrowed significantly when compared to just FID response alone. In this study, no adverse effects where observed with hydrogen as the carrier gas. The small amount of hydrogen carrier flow kept the reactor conditioned while one could turn off the air/hydrogen to the reactor mass flow controller as well as the air to the FID during short standby periods.

Introduction

Typically, helium has been used as a carrier gas in gas chromatography for generally all detector systems for many years. Around 2006 there were times when acquiring a constant reliable supply of helium had been difficult and also more expensive. Hydrogen has always been an alternative to helium and generally allows faster analysis times while maintaining chromatographic efficiency.

Some polar compounds can have issues with chromatography systems including columns and inlet liners, which can lead to poor peak shape and response factors. Several compounds were chosen to test using the Polyarc system. Compounds n-Octanol, Aniline, m-Cresol, Triethylene glycol (TEG) and catechol can give challenging chromatography. n-Decane and n-Hexadecane were added to represent more ideal compounds.

A series of standards were prepared and analyzed using FID alone and the Polyarc/FID combination. Plots were determined using wt% compound and carbon as well as comparing response factors of the components.

The column used in this study is an ADEX 325 (alpha-Dex 325) sold by Sigma Aldrich commonly used at our laboratory. It is a chiral GC phase used for positional isomer separation and generally gives good peak shape even for some challenging compounds.

Experimental

GC conditions

Equipment	Agilent 7890A
Front inlet	Split/splitless

Inlet liner	Agilent part number 5190-2295
Inlet Temperature	250 °C
Inlet Mode	100:1 Split
Inlet Pressure	3 psi @ 35cm/sec
Septum purge flow	3 sccm
Oven	135 °C (2.5 minutes) programmed to 175 °C at 10 °C/min (final 3.5 min).
Column	Adex325 (10 m × 0.25 mm × 0.25 µm) (Sigma Aldrich)
Syringe	5 µL
Injection	0.3 µL

Column Flow	0.83 cc/min Hydrogen, constant flow
FID conditions	
Temperature	265 °C
H_2	2 sccm
Air	350 sccm
Makeup	20 sccm (He)
Sampling	50 Hz
Polyarc reactor conditions	
Set point	293 °C
H_2	35 sccm
Air	2.5 sccm

Results and Discussion

Table 1 shows the external standard levels and weight percent ranges. Figure 1 shows a chromatogram of the external standard level Estd2 using the Polyarc/FID combination. The first large peak is the solvent peak. Peaks 5 and 6 tend to show tailing on certain other GC phases, in this case the peak shapes were good on all the standard levels using the ADEX325 and Polyarc system.

Table 1: Selected Compounds as Wt% Carbon and as Wt% Compound

Compound	MW	CAS#	Wt% as carbon			Wt% as Compound		
			Estd1	Estd2	Estd3	Estd1	Estd2	Estd3
Carbon	12.0107	-	**N/A**					
n-Decane (n-C10)	142.2817	**124-18-5**	**0.29**	**1.13**	**4.24**	**0.343**	**1.34**	**5.02**
n-Octanol (n-C8OH)	130.2279	**72-69-5**	**0.25**	**0.98**	**3.70**	**0.343**	**1.33**	**5.02**
Aniline	93.1265	**62-53-3**	**0.27**	**1.05**	**3.93**	**0.347**	**1.35**	**5.08**
m-Cresol	108.1378	**108-39-4**	**0.29**	**1.14**	**4.27**	**0.375**	**1.46**	**5.49**
Triethylene glycol (TEG)	150.1730	**112-27-6**	**0.17**	**0.66**	**2.47**	**0.352**	**1.37**	**5.15**
Catechol	110.0368	**120-80-9**	**0.23**	**0.89**	**3.33**	**0.347**	**1.35**	**5.09**
n-Hexadecane (nC16)	226.4412	**544-76-3**	**0.30**	**1.17**	**4.40**	**0.354**	**1.38**	**5.19**

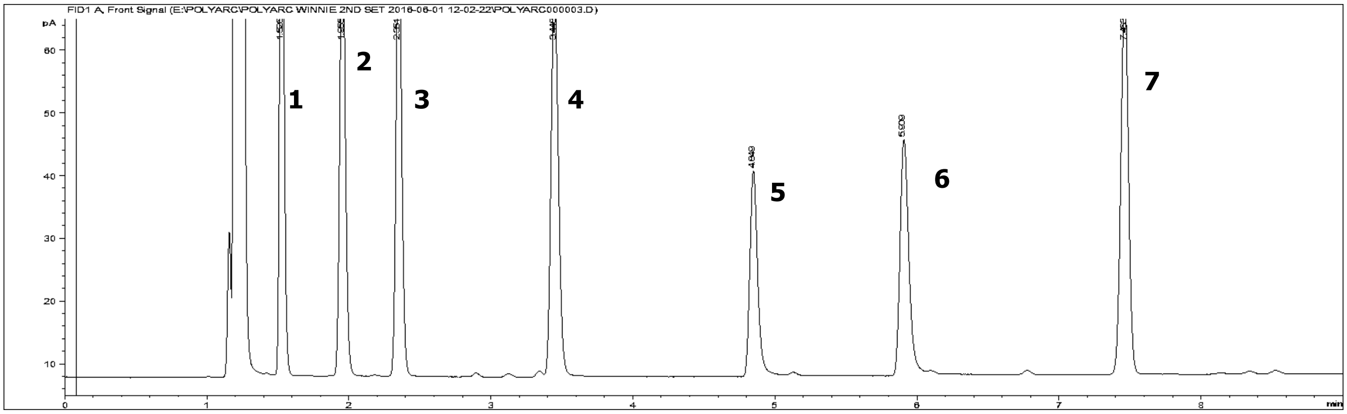

Figure 1: External Standard Level 2: After the solvent peak, 1 = n-Decane (1.13 wt% as C); 2 = n-Octanol (0.98 wt% as C); 3 = Aniline (1.05 wt% as C); 4 = m-Cresol (1.14 wt% as C); 5 = Triethylene glycol (0.66 wt% as C); 6 = Catechol (0.89 wt% as C); 7 = n-Hexadecane (1.17 wt% as C).

In Figures 2 and 3 below, the FID response is plotted "As Compound" and "As Carbon". In Figure 3 the line for TEG "As Compound" was left in the plot for comparison.

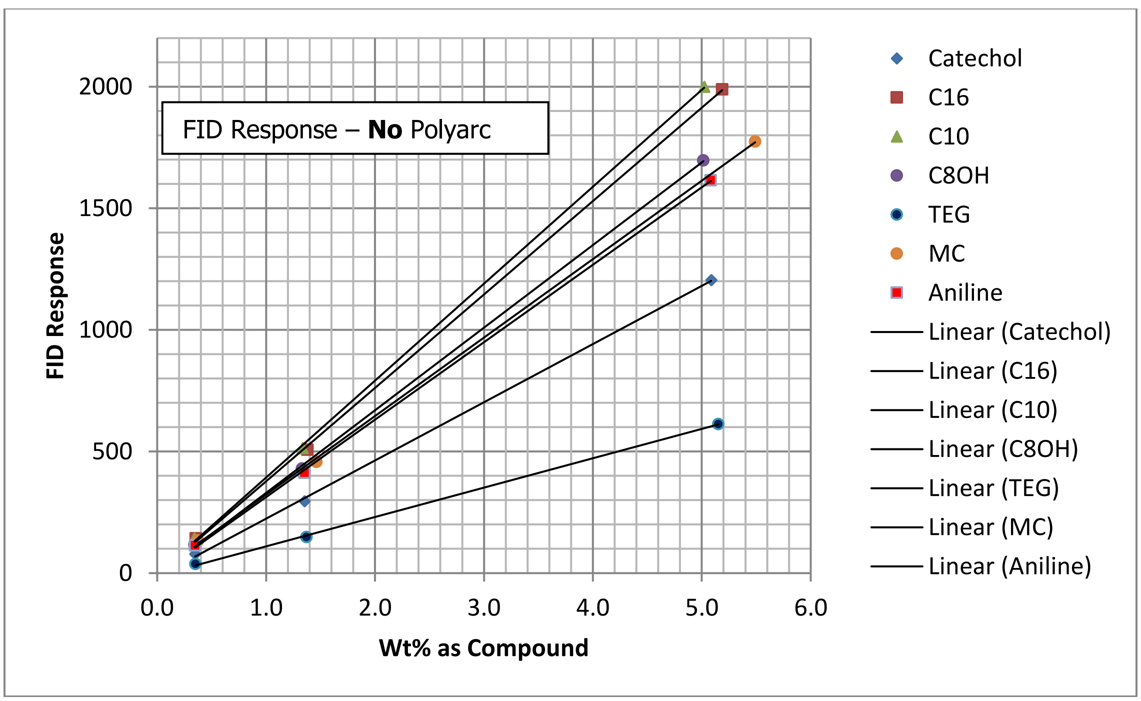

Figure 2: Plot of Components as Showing FID Response vs "**Wt% as Compound**"

Figure 3 shows that even when the FID alone response is plotted "As Carbon" the calibration curves tend to be closer together even without the Polyarc system.

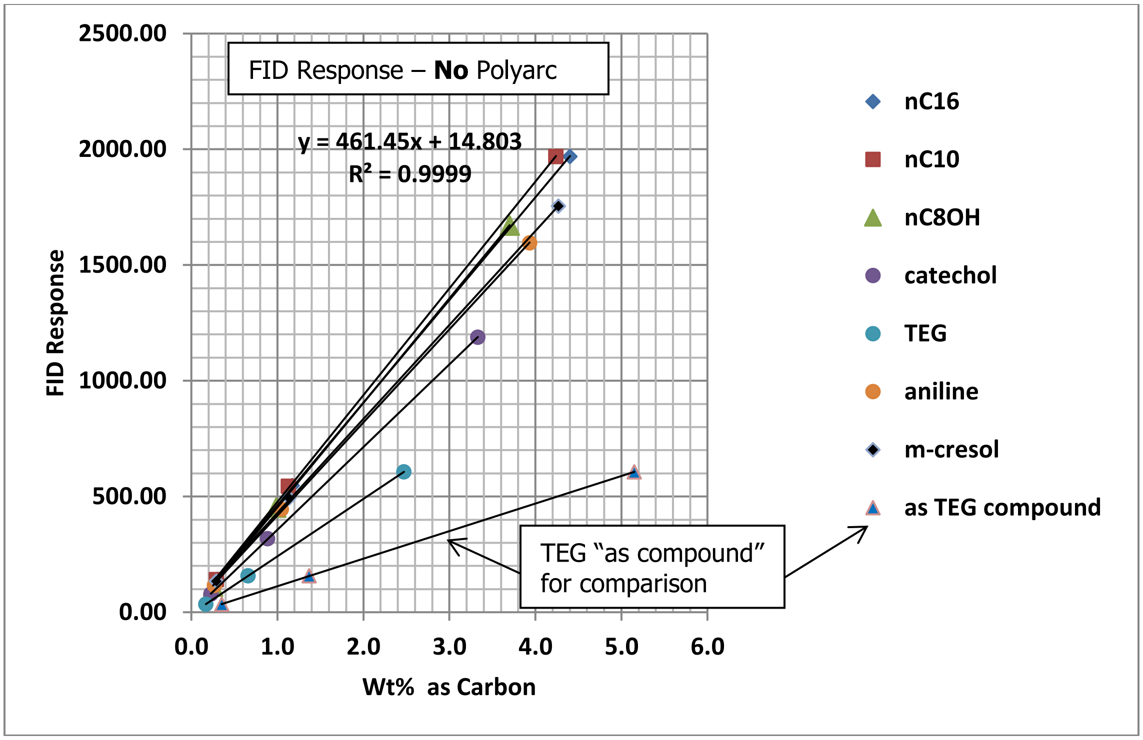

Figure 3: Plot Showing FID Response as "**Wt% as Carbon**"

Figure 4 shows the calibration curves when using the Polyarc/FID combination come closer together as one would expect since the FID is now seeing all compounds as methane.

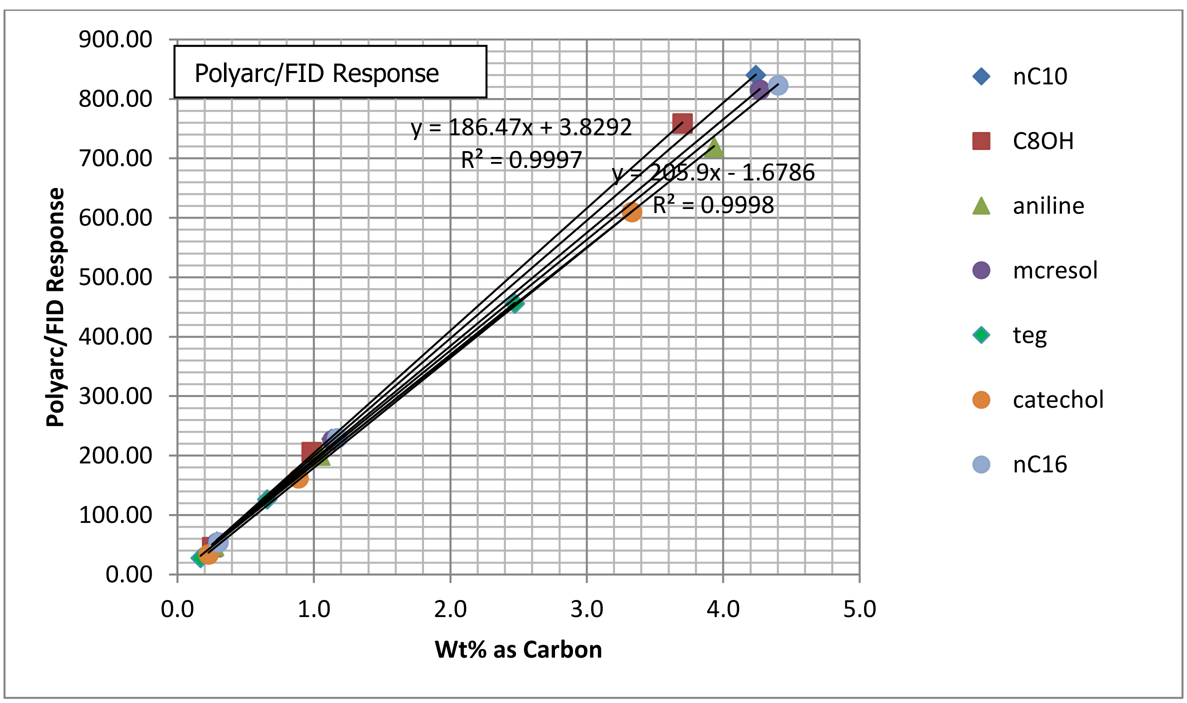

Figure 4: Polyarc/FID Response vs "**Wt% as Carbon**"

Figure 5 shows all the points plotted together and corrected for density differences during injection when used as external standards. Although the points do not show a perfect R^2, using the Polyarc/FID combination shows that the responses for dissimilar compounds come closer together. Figure 5 shows that the Polyarc/FID is probably more forgiving on unknown components when analyzing complex mixtures by area % than FID alone.

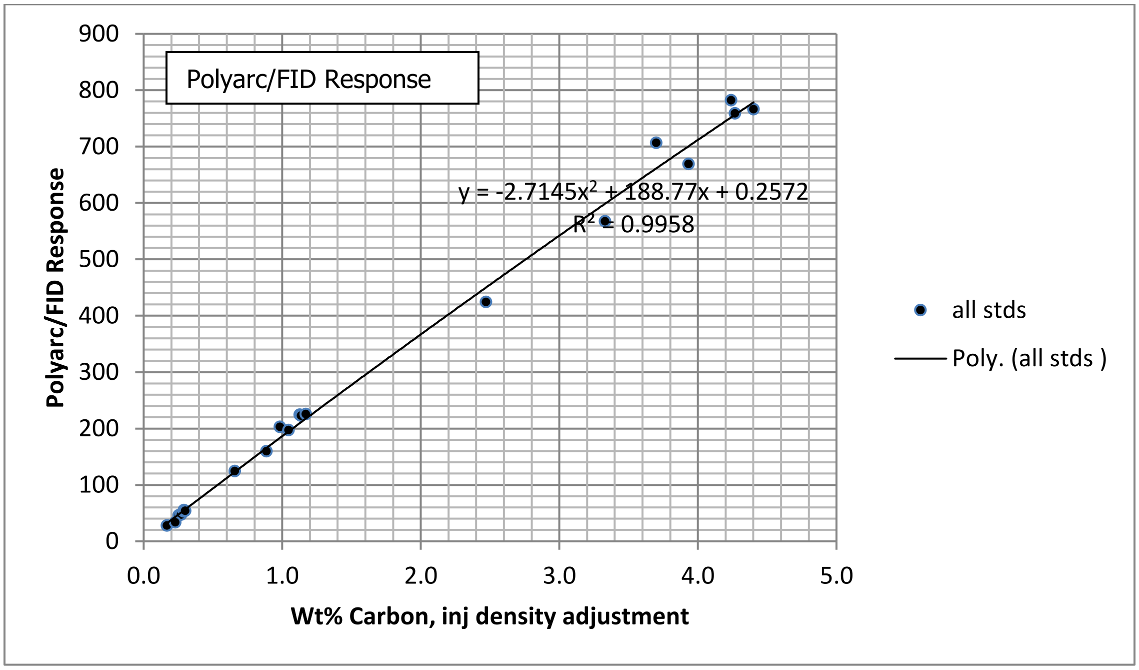

Figure 5: All points plotted adjusted for density corrections

Table 2: Comparison of Estd Response Factors

ESTD	Avg RF x 10^{-3} FID RF As cmpd	FID RF As carbon	Polyarc/FID As carbon
n-Decane	2.509	2.118	**5.249**
n-Octanol	2.999	2.213	**5.208**
Aniline	3.155	2.441	**5.632**
m-Cresol	3.035	2.359	**5.415**
Triethylene glycol	9.054	4.345	**5.739**
Catechol	4.414	2.891	**6.077**
n-Hexadecane	2.607	2.213	**5.513**
Avg	4.597	2.654	**5.548**
Avg Dev.	1.581	0.551	**0.230**
Range	6.546	2.227	**0.869**

Table 2 shows the differences in response factors when calculated "As compound", "As Carbon" for the FID alone and "As Carbon" for the Polyarc/FID combination. The values in each of the columns are averaged response factors calculated for each external standard level for each compound. The last column with values for the Polyarc/FID shows how close the responses are to each other as compared to FID alone.

Conclusions

1. Hydrogen can be used as a carrier for the Polyarc system. Although this study tested only one flow (~1cc/min), further studies could include higher flows to determine any possible affects.

2. With the closeness of the response factors, the data from the Polyarc/FID in Table 2 shows that area% per carbon of known components and unknowns would tend to be more accurate than with FID alone.

3. Inlet discrimination and absorptive interactions between a component and the GC stationary phase can still be problematic. This is probably why the components in the calibration plots start showing more scatter at higher concentrations.

Contact Us

For more information or to purchase a Polyarc system, please contact Activated Research Company at 612-787-2721 or contact@activatedresearch.com.

Please visit their website for details and additional technical literature.

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