Universal GC/FID Detection with Shimadzu GC 2030

Application Note

Universal carbon quantitation

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Abstract

A Polyarc reactor was installed on a Shimadzu GC 2030 and characterized for performance. A chromatography test mix was shown to give 2.4% average error without the need for response factors. Peak capacity was maintained when comparing GC/FID runs with Polyarc-GC/FID runs for gasoline. The Polyarc improves accuracy more than twenty-fold when quantifying ethanol against heptane as an internal standard.

Introduction

The flame ionization detector (FID) is a powerful detector with a linear dynamic range of over seven orders of magnitude, a limit of detection on the order of a picogram of carbon per second, and a unique carbon selectivity. One of its biggest drawbacks, however, is its varying response to organic compounds that have different degrees of substitutions, such as alcohols, halogens, or other functional groups. This varied response has historically resulted in the need for response factors and targeted calibration standards and led to losses in sensitivity and accuracy.

The Polyarc reactor was designed to address these drawbacks and give a universal carbon response in the FID, allowing for compound-independent single point calibrations and increased responses for some molecules, resulting in lower limits of detection. The Polyarc reactor is a two-stage catalytic microreactor which converts organic compounds to methane for universal FID detection via the following reaction:

Carbon-Containing Compounds + Air + H_2 → Methane (CH_4) + Non-Carbonaceous Byproducts

The Polyarc is positioned as an intermediate between the analytical GC column and the FID. Activated Research Company has partnered with Shimadzu to design a Polyarc enclosure specifically for the GC 2030 to be positioned discretely in the inlet position.

Figure 1. Polyarc installed on a Shimazu GC 2030

Experimental

GC conditions

Front inlet	Split
Inlet temperature	250 °C
Inlet pressure	19.6 psi
Septum purge flow	3.0 sccm
Oven	40 °C (5 min), 10 °C/min to 125 °C, 25 °C/min to 250 °C (2 min)
Column	5% Phenyl (30 m × 0.25 mm × 0.25 µm)
Syringe	10 µL
Injection volume	0.5 µL

FID conditions

Temperature	315 °C
H_2	1.5 sccm (35 sccm with no Polyarc)
Air	350 sccm
Makeup	5 sccm (He)
Sampling rate	20 Hz

Polyarc reactor conditions

Setpoint	293 °C
H_2	35 sccm
Air	2.5 sccm

Results and Discussion

The relative response of two organic compounds measured with a Polyarc/FID can be calculated from the ratio of the number of carbons (i.e., the number of methane molecules produced) with the following equation:

$$C_A = C_s\left(\frac{Area_A}{Area_S}\right)\left(\frac{\#C_S}{\#C_A}\right)\left(\frac{MW_A}{MW_S}\right)$$

where:

C_A = Mass concentration of analyte
C_S = Mass concentration of standard
$\#C_S$ = Number of carbon atoms for standard
$\#C_A$ = Number of carbon atoms for analyte
$Area_A$ = Integrated peak area of the analyte
$Area_S$ = Integrated peak area of the standard
Mw_A = Molecular weight of the analyte
Mw_S = Molecular weight of the standard

A mixture (PA-PTM-R73) containing a variety of molecule types was used to evaluate chromatographic performance. The responses resulted in an average error below 3% (Table 1) and USP tailing factors below 1.2 for all compounds indicating near-gaussian peaks and high performance. The USP tailing factor for aniline, which has the strongest basicity in the mixture, was 1.13 indicating a negligible increase in tailing due to the presence of the Polyarc reactor.

Table 1. Polyarc Test Mix Results

Compound	% Error
Aniline	0.4%
2-Chlorophenol	0.6%
1-Octanol	-4.9%
2-Nonanone	-1.1%
2-Dodecanol (IS)	N/A (IS)
Methyl Laurate	3.1%
n-Heptadecane	2.2%
n-Nonadecane	4.5%
Average	***2.4%***

Next, gasoline was injected with a standard FID (Figure 2) and a Polyarc/FID (Figure 3) to compare chromatographic response and peak resolution. Similar peak shapes and peak capacities were obtained in both, indicating chromatographic resolution was maintained. Higher peak areas were observed for oxygenated molecules including ethanol.

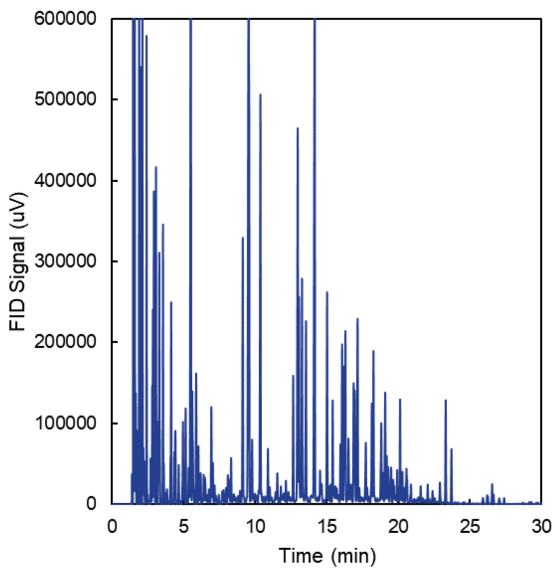

Figure 2. GC/FID Gasoline results

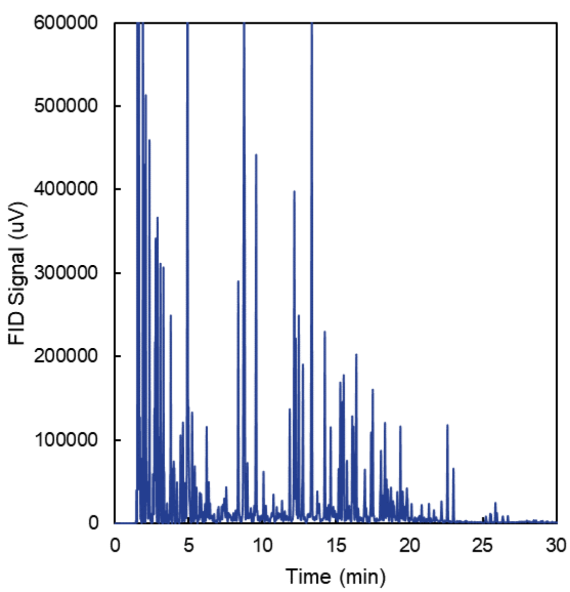

Figure 3. GC/Polyarc-FID Gasoline Results

The response of ethanol is compared to the response of heptane in Figure 4. Ethanol responses per carbon were 22% lower than heptane using a standard FID, due to the different response of the FID to oxygenated compounds. However, ethanol responses per carbon were nearly identical (within 1%) to those of heptane with the Polyarc/FID due to complete conversion to methane of both ethanol and heptane.

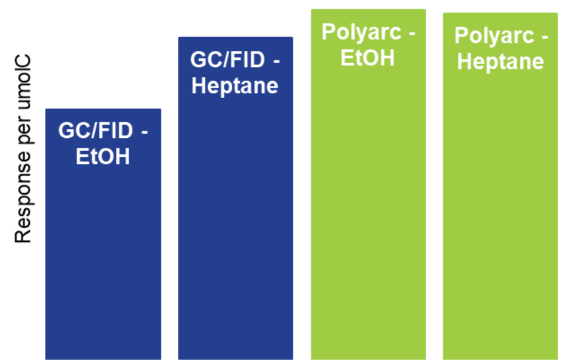

Figure 4. Quantitation Comparison

Conclusions

The Polyarc improves the quantitative accuracy of oxygenates on the Shimadzu GC 2030 with negligible influence on peak shape, resolution, and peak capacity. Compound-independent calibration with the Shimadzu Polyarc/FID allows scientists to quantify complex mixtures with a single internal or external calibration standard.

Contact Us

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

Please visit their website for details and additional technical literature.

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