Secondary Confirmation of Calibration Standards

Application Note

Light Gases

Author

Andrew Jones
Activated Research Company
7561 Corporate Way, Eden Prairie, MN 55344
Andrew.Jones@activatedresearch.com

Abstract

A method is proposed for the secondary validation of calibration standards using a GC equipped with a microreactor and FID. The linear response of the system is validated with several certified reference standards including CO, CO_2, C1-C8 hydrocarbons, alcohols, aldehydes and chlorofluorocarbons. Equivalent per carbon responses provided secondary validation that gravimetrically prepared standards were correct.

Introduction

Gaseous mixtures of standards are used in a variety of industries to calibrate analysis equipment and provide retention indices for proper compound identification. It is desirable to have a secondary method to validate the concentrations of compounds in the standard for the purpose of quality control, but also to qualify expired standards which may have changed composition due to reactivity or adsorption.

Experimental

GC conditions

Front inlet	Split/Splitless
Carrier Gas	Helium
Inlet temperature	200 °C
Inlet pressure	18.7 psi
Septum purge flow	3 sccm
Oven	50 °C (5 min), 10 °C/min to 180 °C (1 min)
Column	DB-624 (60 m × 0.25 mm × # µm)
Sample Loop	2 mL

FID conditions

Temperature	350 °C
H_2	2 sccm
Air	450 sccm
Makeup	25 sccm (N_2)

Polyarc® reactor conditions

Setpoint	293 °C
H_2	50 sccm
Air	3 sccm

Results and Discussion

Figure 1. FID response curve for various molecules from separate injections of CO, CO_2, C1-C8 hydrocarbons, alcohols, aldehydes and chlorofluorocarbons. Every molecule tested responds proportionally to the amount of carbon injected, with the same response factor.

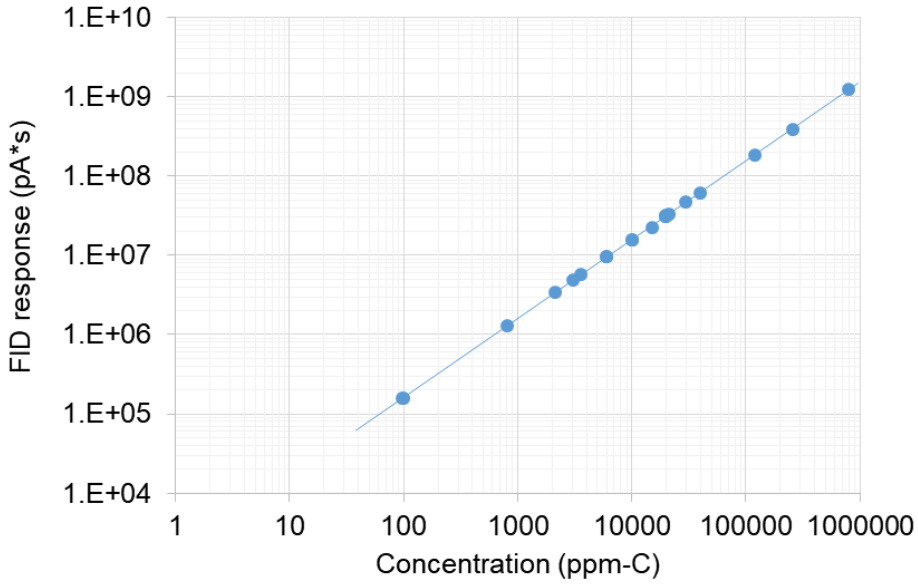

Table 1. Gravimetric and measured concentrations of various analytes from Figure 1.

Compound	Gravimetric Conc. (ppm-C)	Measured Conc. (ppm-C)	Error
n-octane	800	811	1.4%
n-heptane	2107	2132	1.2%
hexane	3600	3621	0.6%
n-pentane	9995	10038	0.4%
isopentane	9995	10080	0.8%
n-butane	39984	39369	1.5%
isobutane	19992	19545	2.2%
propane	120120	119958	0.1%
ethane	260000	252308	3.0%
methane	803997	813810	1.2%
CO	100	96	3.6%
CO	15021	14275	5.0%
CO2	97	98	0.7%
Benzene	29988	30284	1.0%
Toluene	20993	21434	2.1%
Hexane	6030	6136	1.8%
Cyclohexane	6030	6102	1.2%
Ethane	19922	19894	0.1%
Ethylene oxide	19650	20298	3.3%
Acetaldehyde	3042	3049	0.2%

Conclusions

The Polyarc/FID provides an accurate and easy method for the secondary validation of standards. The uniform and linear response of all analytes on a carbon basis indicate a complete recovery and conversion to CH_4 for all analytes in the microreactor. This method is generally applicable to all carbon-containing molecules, except CF_4 and C_2F_6, due to thermodynamic limitations on their combustion properties. This method is expected to save in both the time and cost for the production of standards as well as provide a tool for the qualification of expired or tenuous mixtures.

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, www.activatedresearch.com.

Activated Research Company shall not be liable for errors contained herein, or for incidental or consequential damages in connection with the furnishing, performance, or use of this material.

Information, descriptions, and specifications in this publication are subject to change without notice.

Printed in the USA
July 5, 2018
PA-APP-1814