

Quantification of Corrosion Inhibitor/Lubricity Improver In Military Fuels using Infrared Spectroscopy

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Spring 2013

Report Documentation Page

Form Approved
OMB No. 0704-0188

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1. REPORT DATE 26 APR 2013	2. REPORT TYPE Technical Report	3. DATES COVERED 12-05-2012 to 23-04-2013	
4. TITLE AND SUBTITLE Quantification of Corrosion Inhibitor/Lubricity Improver In Military Fuels using Infrared Spectroscopy		5a. CONTRACT NUMBER	
		5b. GRANT NUMBER	
		5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Christine Larkin		5d. PROJECT NUMBER	
		5e. TASK NUMBER	
		5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Department of Chemistry and Environmental Science, Lake Superior State University, 650 W Easterday Ave, Sault Ste. Marie, Mi, 49783		8. PERFORMING ORGANIZATION REPORT NUMBER ; #23814	
		10. SPONSOR/MONITOR'S ACRONYM(S) TARDEC	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army TARDEC, 6501 East Eleven Mile Rd, Warren, Mi, 48397-5000		11. SPONSOR/MONITOR'S REPORT NUMBER(S) #23814	
		12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited	
13. SUPPLEMENTARY NOTES			
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15. SUBJECT TERMS			
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified	Public Release
			18. NUMBER OF PAGES 28
			19a. NAME OF RESPONSIBLE PERSON

Abstract

The overall objective of this project was to evaluate the feasibility of utilizing infrared spectrometry to measure Corrosion Inhibitor/Lubricity Improver (CI/LI) additive in military fuels. Four methodologies were evaluated, but only one methodology was found to be somewhat effective. The Direct Sample, Direct Sample with Standard Addition, and Concentrated Sample methodologies were ineffective. The Concentrated Sample with Standard Addition methodology was effective at correlating concentration and transmittance or absorbance within a single additive brand, but the correlation was not universally applicable across all CI/LI additive brands. It was also found that the absorbance variance of blank fuel samples completely encompassed the measurements of fuel with additives in them. This indicates that the instrument would be unable to accurately assess the concentration of CI/LI additive in a fuel sample of unknown CI/LI concentration. For this technology to be feasible, a different calibration curve would be needed for each commercial additive brand that the Army uses and it would only be capable of measuring additive concentrations as additive is being added to fuel or for the verification of additive injection equipment.

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1. Introduction

The objective of this effort was to evaluate the feasibility of utilizing infrared spectroscopy for the quantification of Corrosion Inhibitor/Lubricity Improver (CI/LI) additive in the military's kerosene based fuels.¹ Analysis of fuel additive concentrations is critical to the Army to ensure the proper additive levels during fuel distribution and in the additive injection processes. The Army would like to develop a lightweight portable instrument with the capability of rapidly analyzing military fuel to measure CI/LI additive concentrations. The Army's goal is to use the device for testing fuel samples and/or monitoring fuels for correct additive levels to ensure the proper function of fuels².

Under the "single fuel on the battlefield" concept, mandated by Department of Defense Directive 4140.25 and AR-70-12, primary fuel support for land-based air and group forces in all theaters of operation is to be accomplished using a single kerosene-type fuel.³⁻⁴ This fuel is JP-8.⁵⁻⁶ JP-8 is identical to the standard commercial aviation fuel, JA-1, save for the addition of the military fuel additive package. This package contains static dissipater additive (SDA), corrosion inhibitor/lubricity improver (CI/LI), and fuel system icing inhibitor (FSII). It may also contain antioxidant (AO) and metal deactivators (MDA).⁶ CI/LI serves both to protect the engine metals from destruction via corrosion and to prevent pump wear due to friction caused by low-lubricity fuels. The Army requires that the concentration of CI/LI in fuel be not less than 6 ppm, but not more than 36 ppm.¹ Six different commercial formulations of Corrosion Inhibitor/Lubricity Improver additives meeting the military's performance specification MIL-PRF-25017 were available for performing the instrument evaluation.⁷ These are listed in Table 1.

Source Name	Manufacture Designation
GE BETZ, INC.	SPEC-AID 8Q22
BAKER PETROLITE CORPORATION	TOLAD 351
BAKER PETROLITE CORPORATION	TOLAD 4410
INNOSPEC FUEL SPECIALTIES LLC	DCI-6A
AFTON CHEMICAL CORPORATION	HITEC 580
NALCO COMPANY	NALCO EXXON 5403

Table 1: CI/LI additives qualified under MIL-PRF-25017 used in method development.

The active ingredient of CI/LI additives is dilinoleic acid, a dimer of linoleic acid, which contains a polar carboxylic acid functionality that adheres to metal surfaces. The dilinoleic acid compound is shown in Figure 1. This forms a thin protective surface film of additive, thereby improving lubricity and inhibiting corrosion. It is the carboxylic acid carbonyl group that allows for infrared spectroscopy monitoring of concentration. The carboxylic acid C=O stretch absorbs infrared radiation at 5.84 μm (1712 cm^{-1}) wavelength.

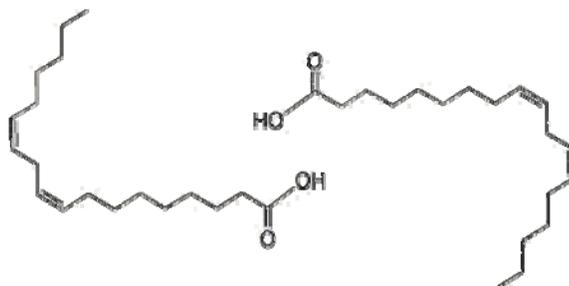


Figure 1: Dilinoleic Acid Chemical Structure

However, this absorption is small and often affected by the composition of the fuel the additive is used in. Therefore, to accurately measure the CI/LI additive in fuel, a differential spectrum must be produced. The developed technology implemented an extraction step that allowed IR measurement of the sample fuel with the CI/LI additive and without it. This allows the generation of a differential spectrum with the CI/LI extracted measurement serving as the blank. The absorbance of the dilinoleic acid carbonyl functionality increases with increase in CI/LI concentration as seen in Figure 2. The success of this technology is dependent on the absorbance being linearly correlated to CI/LI additive concentration.

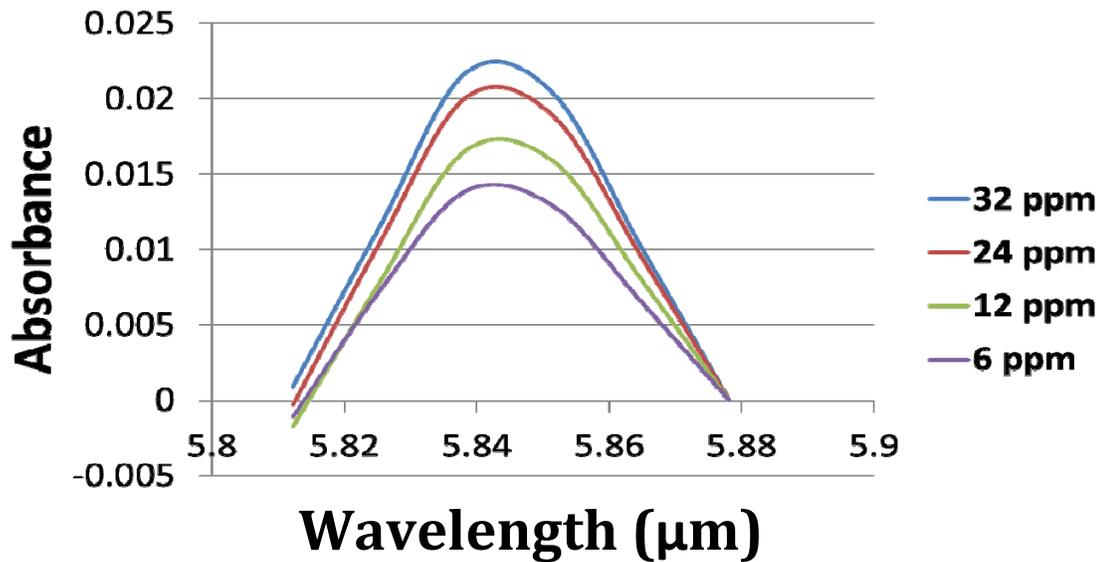


Figure 2: Differential Spectra of CI/LI additive 5.84 μm (1712 cm^{-1}) carbonyl absorbance peak for 6, 12, 24, and 32 ppm standards

2. Objective

The objective of this effort was to evaluate the feasibility of utilizing infrared spectrometry for the quantification of Corrosion Inhibitor/Lubricity Improver (CI/LI) additive in the military's kerosene based fuels.

3. Methods

The four different sampling methods tested included: (1) direct sample, (2) direct sample with standard addition, (3) concentrated sample, and (4) concentrated sample with standard addition. These methodologies are summarized in Figure 3. The fourth methodology, concentrated sample with standard addition, was verified using an in-house Varian 640 FTIR spectrometer. The instrument used for evaluation recorded transmittance, which is logarithmically related to absorbance. The in-house Varian 640 FTIR recorded absorbance. These methods were tested using duplicate trials of 0 ppm, 3 ppm, 6 ppm, 12 ppm, 24 ppm, and 32 ppm additive concentrations to generate a standard curve. Different additive brands and

different base fuels were used in this evaluation to test their effects on instrument accuracy. Instrument accuracy was assessed by calculating a peak height and a least squared linear regression analysis.

Methodology	Sample Preparation
Direct Sample	3 mL sample
Direct Sample with Standard Addition	3 mL sample + 0.20 mL 800 ppm standard
Concentrated Sample	15 mL sample concentrated to 3 mL
Concentrated Sample with Standard Addition	15 mL sample + 1 mL 800 ppm standard concentrated to 3 mL

Figure 3: Methodology Summary

Direct Sample

In the direct sample, 3mL of fuel with the previously identified CI/LI concentrations were directly pipetted into the instrument. Half of the sample underwent extraction and a differential spectrometry spectrum was generated by the instrument. Three different CI/LI additive brands were analyzed including: DCI-A6, HITEC 580, and NALCO EXXON 5804. All samples used PQ-1572-11 as the base fuel.

Direct Sample with Standard Addition

In the direct sample with standard addition, 0.20 mL of an 800 ppm CI/LI standard was added to each 3 mL fuel sample with the previous identified CI/LI concentrations. The entirety of this mixed sample was then directly pipetted into the instrument for extraction and measurement. The intent behind the standard addition was to increase the signal strength to thereby increase the visibility of the dilinoleic acid carboxylic acid carbonyl stretch to the

instruments detector. Again, three different CI/LI concentration were analyzed including DCI-A6, HITEC 580, and NALCO EXXON 5403 and all samples used PQ-1572-11 as the base fuel.

Concentrated Sample

In the concentrated sample, the CI/LI additive was concentrated by a ratio of 5:1 and then 3 mL was pipetted into the instrument for extraction and measurement. Only one additive brand was analyzed – SPEC-AID 8Q22 – and a different set of CI/LI concentrations was used. These concentrations included 0 ppm, 10 ppm, 14 ppm, 20 ppm, 24 ppm, 30 ppm, and 100 ppm. All samples used PQ-1573-11 as the base fuel.

Concentrated Sample with Standard Addition

The method essentially combined the direct sample with standard addition and the concentrated sample methodologies. In the concentrated sample with standard addition, 1 mL of 800 ppm CI/LI standard was added to each sample. This mixture was then concentrated by a 5:1 ratio and pipetted into the instrument for extraction and measurement. Four different CI/LI brands and four different base fuels were used including DCI-6A in PQ-1572-11, NALCO EXXON 5403 in PQ 1572-11, TOLAD 351 in PQ-1576-11, and TOLAD 4410 in two different base fuels -- PQ-1575-11 and PQ-1573-11.

FTIR Concentrated Sample with Standard Addition

In the FTIR samples, the concentrated sample with standard addition methodology was followed, except that both the extracted and un-extracted samples were collected before spectrometric analysis in the developed instrument. Instead, both samples were evaluated using an in-house Varian 640 FTIR. Data was recorded in absorbance rather than transmission. This verification method was not performed in duplicate for each of the CI/LI concentrations, rather

only one measurement was taken for each concentration. Six sample sets were analyzed using this methodology including HITEC 580 in PQ-1577-11, TOLAD 351 in FL-14394-12, NALCO EXXON 5403 in FL-14380-12, SPEC-AID 8Q22 in FL-14394-12, TOLAD 4410 in PQ-1574-11, and TOLAD 4410 in FL-14377-12.

Measurement

The transmittance or absorbance of the CI/LI concentrations was measured using a peak height detection metric based on the height of the dilinoleic acid C=O stretch absorbance peak at the 5.85 μm (1712 cm^{-1}) wavelength as seen below:

$$\frac{(T_{5.84\ \mu\text{m}\ \text{unfiltered}}/T_{5.84\ \mu\text{m}\ \text{filtered}})^2}{(T_{5.47\ \mu\text{m}\ \text{unfiltered}}/T_{5.47\ \mu\text{m}\ \text{filtered}})(T_{6.00\ \mu\text{m}\ \text{unfiltered}}/T_{6.00\ \mu\text{m}\ \text{filtered}})}$$

The peak height detection metric was calculated with transmittance values for the developed instrument and with absorbance values for the in-house FTIR. The transmittance or absorbance of the CI/LI additive at 3, 6, 12, 24, and 32 ppm was then plotted against its respective concentration to generate a standard curve. A method of least squares linear regression was utilized to determine how effectively the detection metric was correlated to CI/LI concentration. The closer the coefficient of determination, R^2 , was to 1 the more linearity the standard curve line exhibited and the better the transmittance or absorbance was correlated to its concentration. A better correlation indicated a better ability to effectively quantify CI/LI additive using the developed infrared spectroscopy technology.

4. Results

Direct Sample

The linear regressions performed with the data developed from this evaluation showed little correlation between the concentration and detection metric of the absorbance. The coefficients of determination, R^2 , are .2204, .268, and .1194 respectively as seen in Figure 4, Figure 5, and Figure 7.

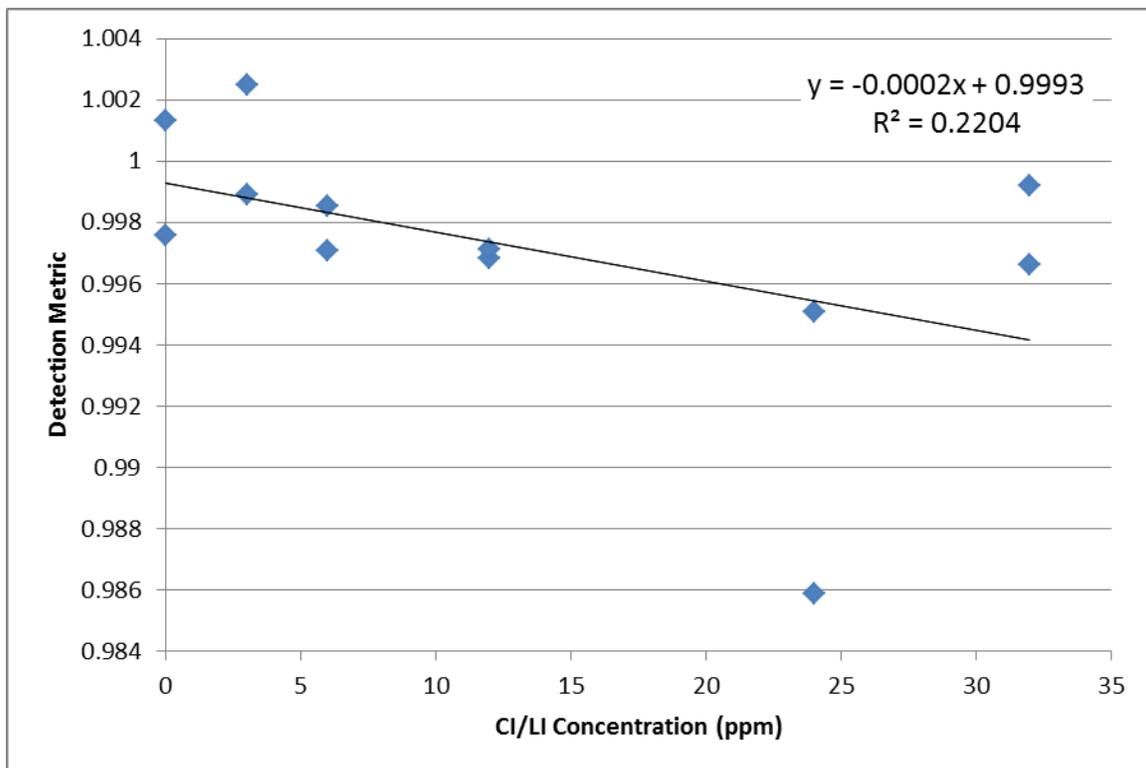


Figure 4: Least squares linear regression graph of direct sample analysis of DCI-A6 CI/LI additive in PQ-1572-11 base fuel

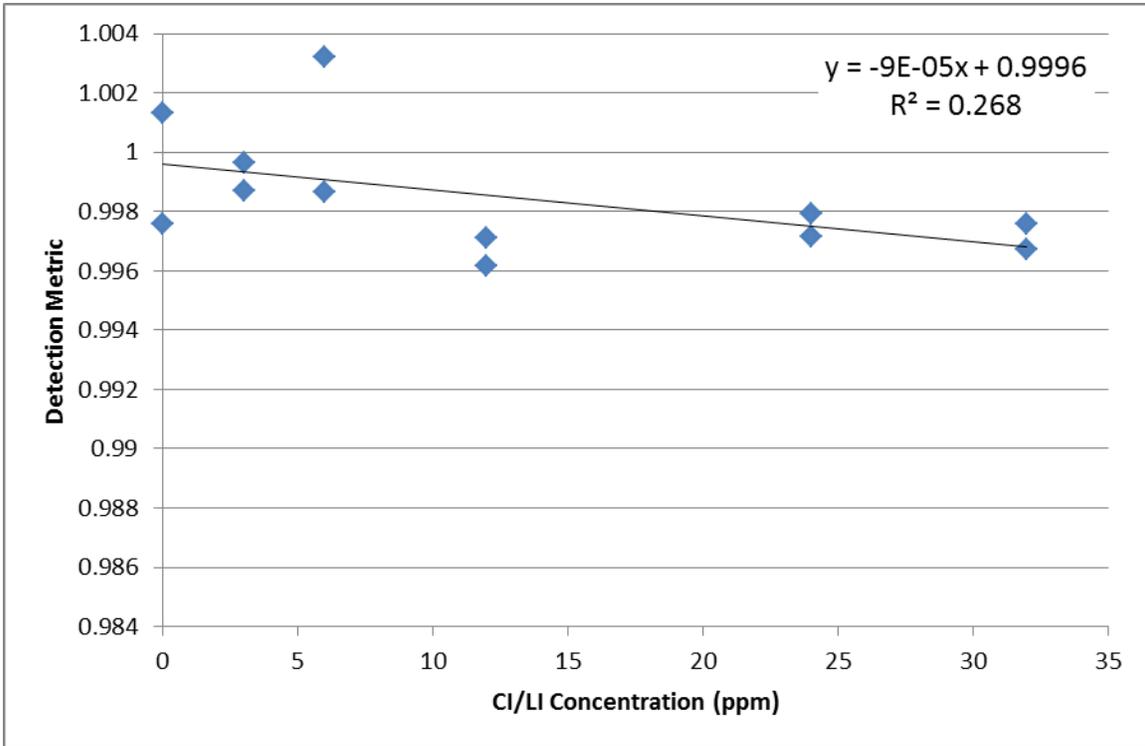


Figure 5: Least squares linear regression graph of direct sample analysis of HITEC 580 CI/LI additive in PQ-1572-11 base fuel

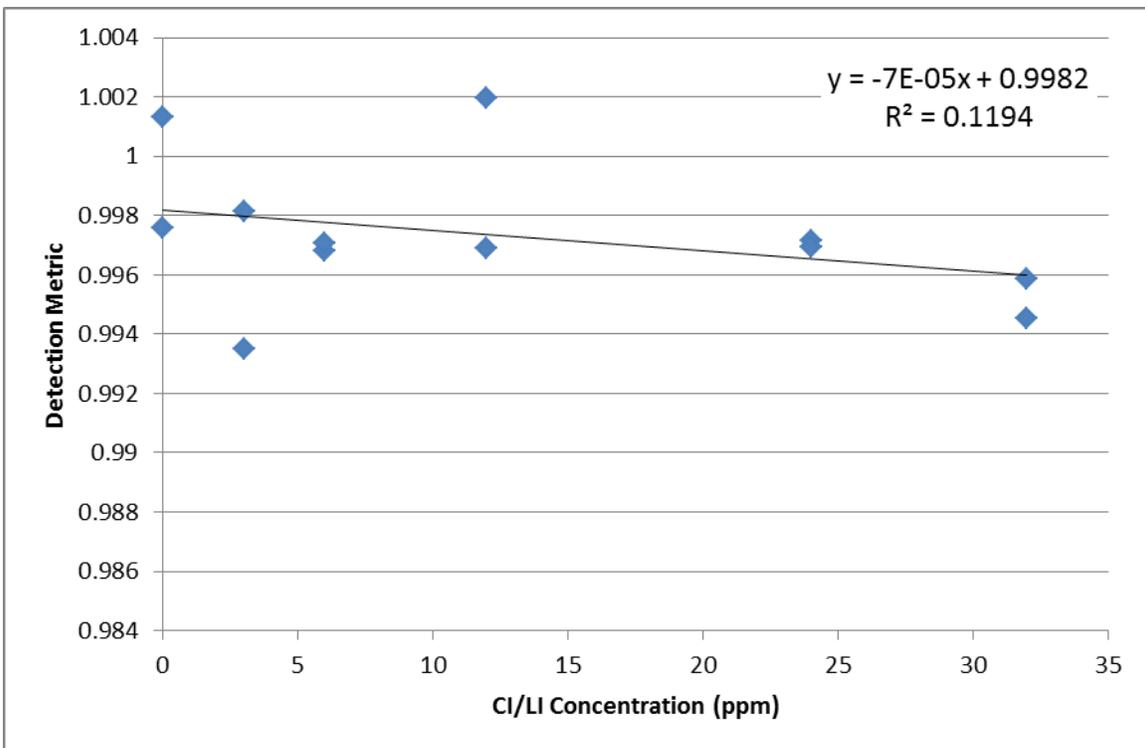


Figure 6: Least squares linear regression graph of direct sample analysis of NALCO EXXON 5403 CI/LI additive in PQ-1572-11 base fuel

Direct Sample with Standard Addition

Again, the linear regressions performed with the data generated from this evaluation showed little correlation between the concentration and detection metric of the absorbance. The coefficients of determination, R^2 , are 0.0043, .0.0741, and 0.2596 respectively as seen in Figure 7, Figure 8, and Figure 9.

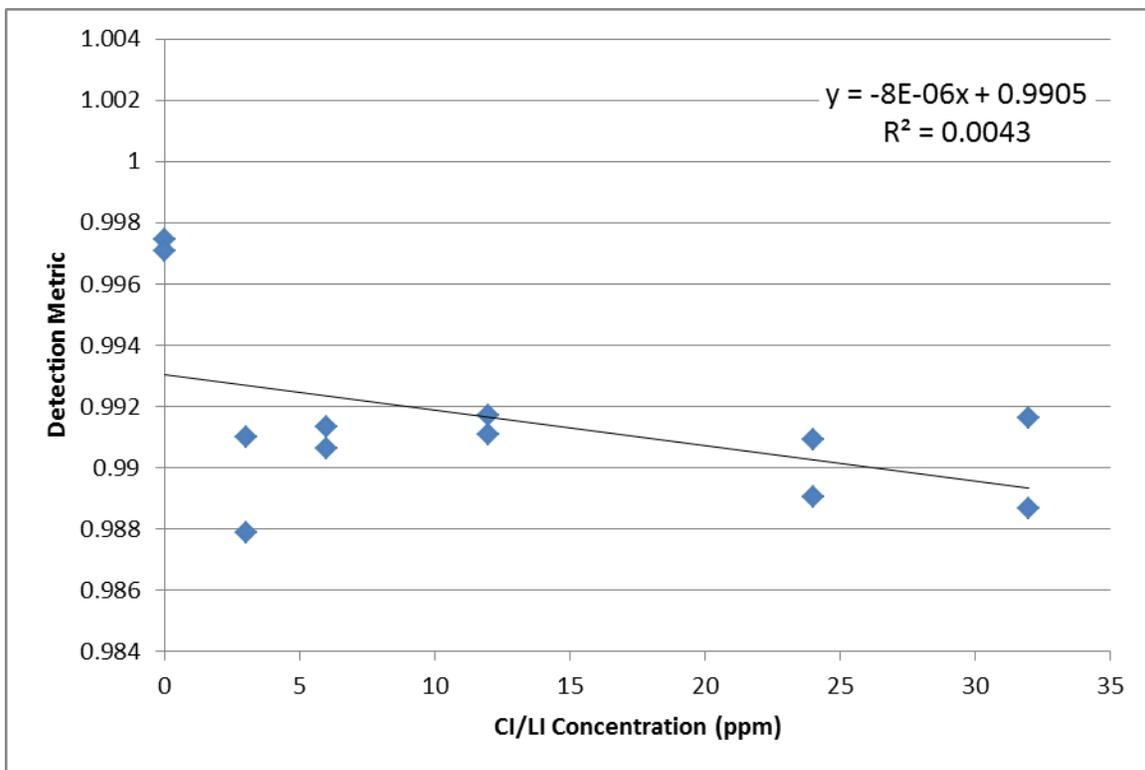


Figure 7: Least squares linear regression graph of direct sample with standard addition of DCI-A6 CI/LI additive in PQ-1572-11 base fuel

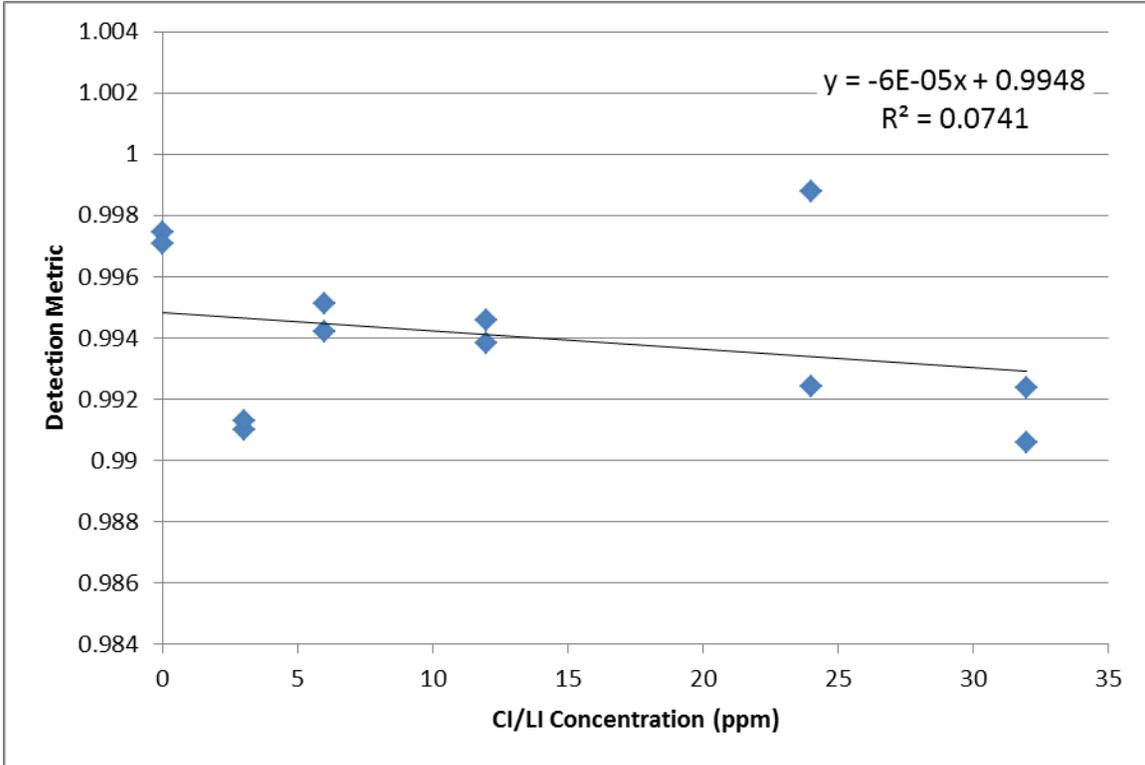


Figure 8: Least squares linear regression graph of direct sample with standard addition of HITEC 580 CI/LI additive in PQ-1572-11 base fuel

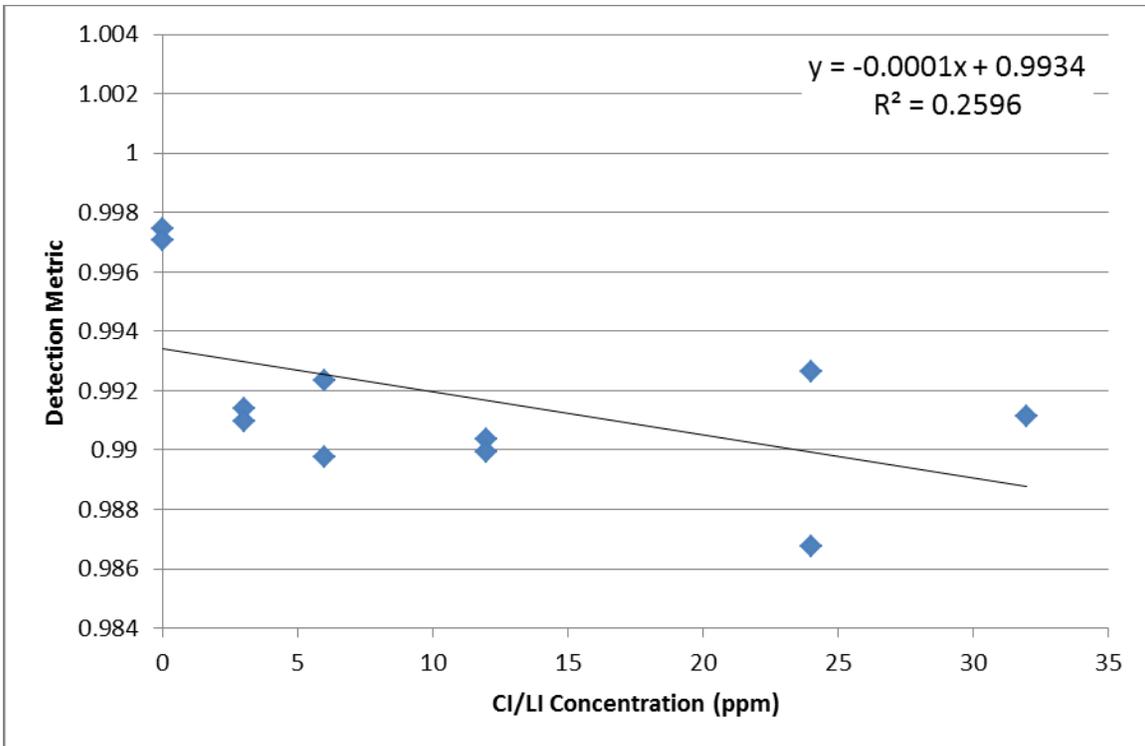


Figure 9: Least squares linear regression graph of direct sample with standard addition of NALCO EXXON 5403 CI/LI additive in PQ-1572-11 base fuel

Concentrated Sample

Previous work had indicated successful correlation of CI/LI additive and detection metric using this methodology when a 100 ppm CI/LI standard was included in the analysis. This was repeated and the coefficient of determination, R^2 , was found to be 0.8706 as seen in Figure 10. However, the range of acceptable CI/LI additive concentration is 6-36 ppm and when the 100 ppm standard was removed from analysis, the coefficient of determination, R^2 , decreased to 0.1309 as seen in Figure 11. On this linear regression graph, it was noticed that the variance of the 0 ppm detection metric points completely encompassed the measurements of fuel with additives in them.

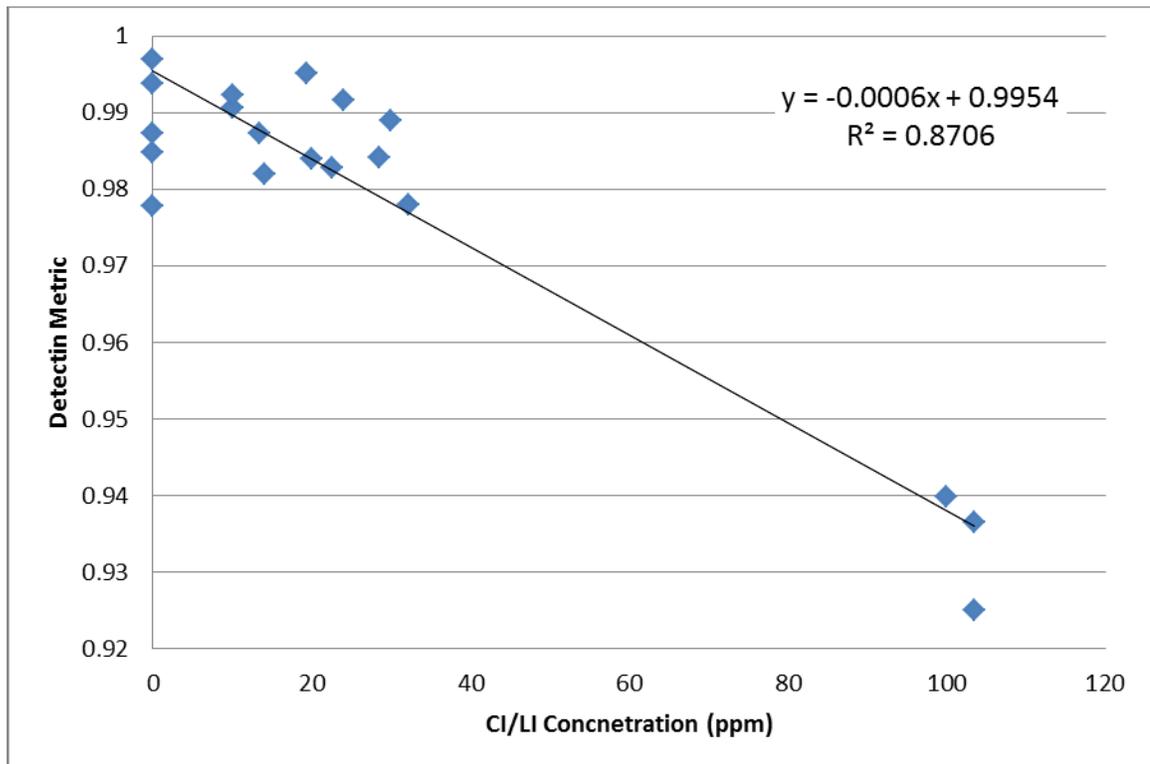


Figure 10: Least squares linear regression graph of concentrated sample of SPEC-AID 8Q22 CI/LI additive with 100 ppm SPEC-AID 8Q22 CI/LI standard in PQ-1573-11 base fuel

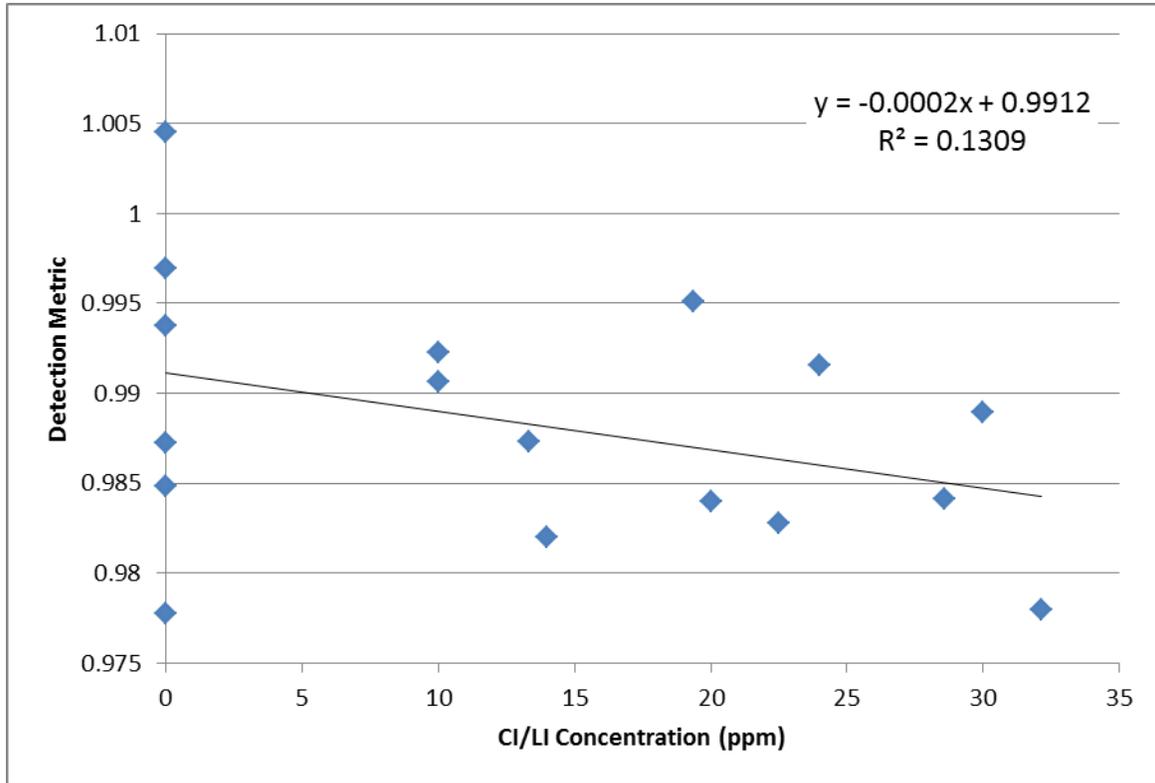


Figure 11: Least squares linear regression graph of concentrated sample of SPEC-AID 8Q22 CI/LI additive without 100 ppm SPEC-AID 8Q22 CI/LI standard in PQ-1573-11 base fuel

Concentrated Sample with Standard Addition

This methodology demonstrated significant correlation between the CI/LI concentration and detection metric. The coefficients of determination, R^2 , are 0.943, 0.9008, 0.9041, 0.8713, 0.7883, and .8699 respectively as seen in Figure 12, Figure 13, Figure 14, Figure 15, Figure 16, and Figure 17. When all six sample sets were combined in the same linear regression the coefficient of determination, R^2 , is 0.637 as seen in Figure 18.

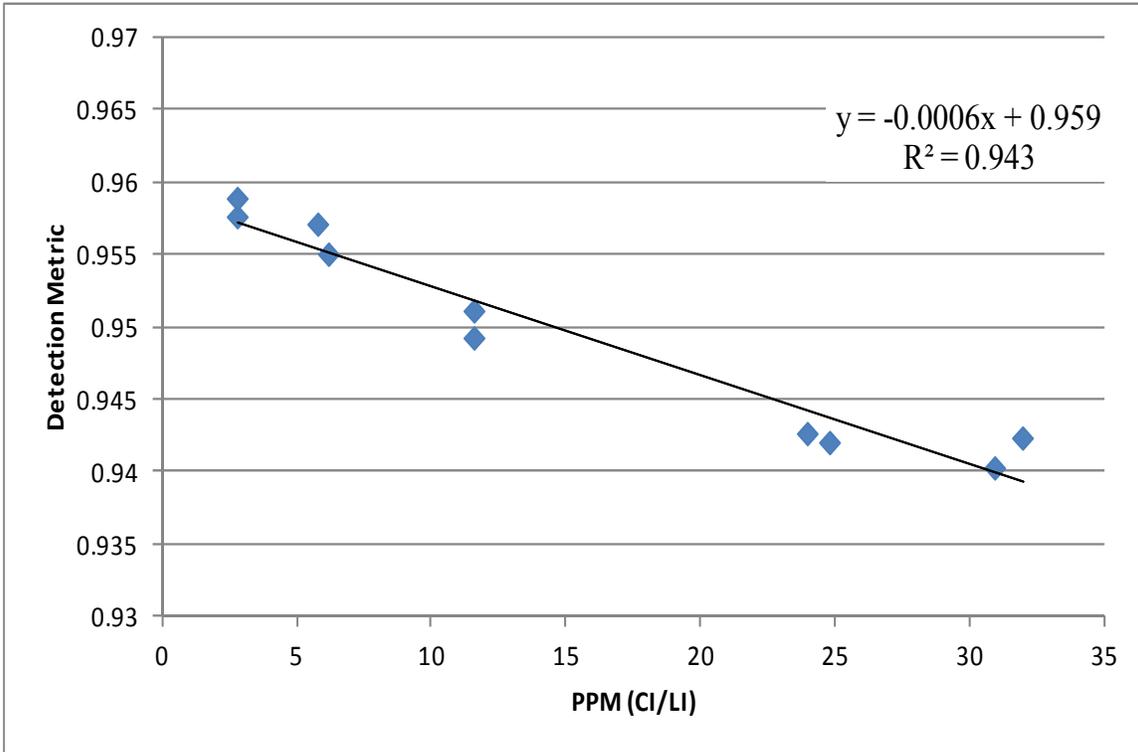


Figure 12: Least squares linear regression graph of concentrated sample with standard addition of DCI-6A CI/LI additive in PQ-1572-11 base fuel

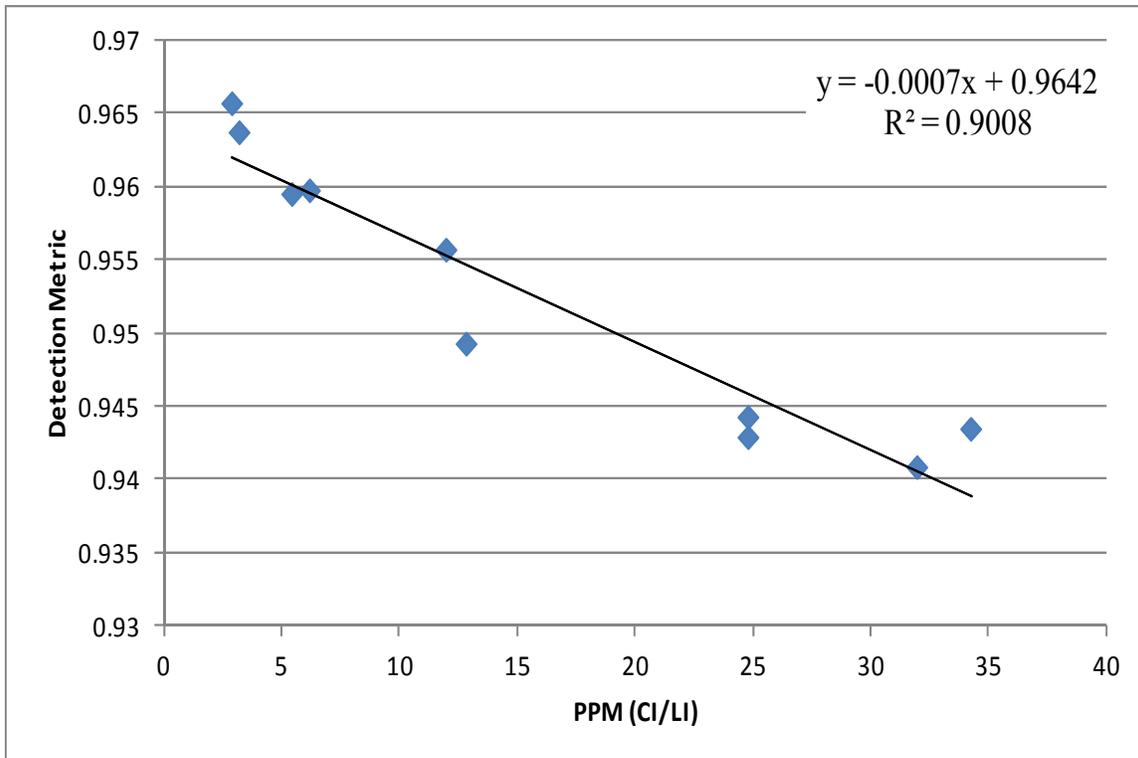


Figure 13: Least squares linear regression graph of concentrated sample with standard addition of NALCO EXXON 4503 CI/LI additive in PQ-1572-11 base fuel

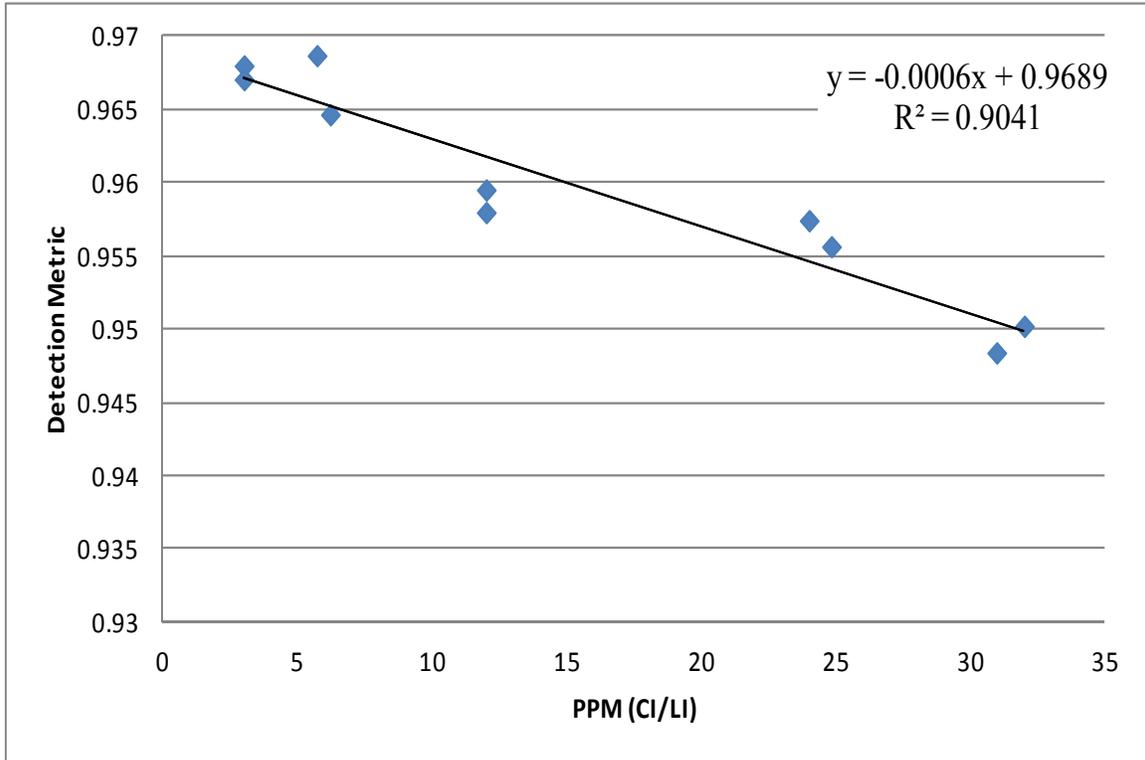


Figure 14: Least squares linear regression graph of concentrated sample with standard addition of TOLAD 351 CI/LI additive in PQ-1576-11 base fuel

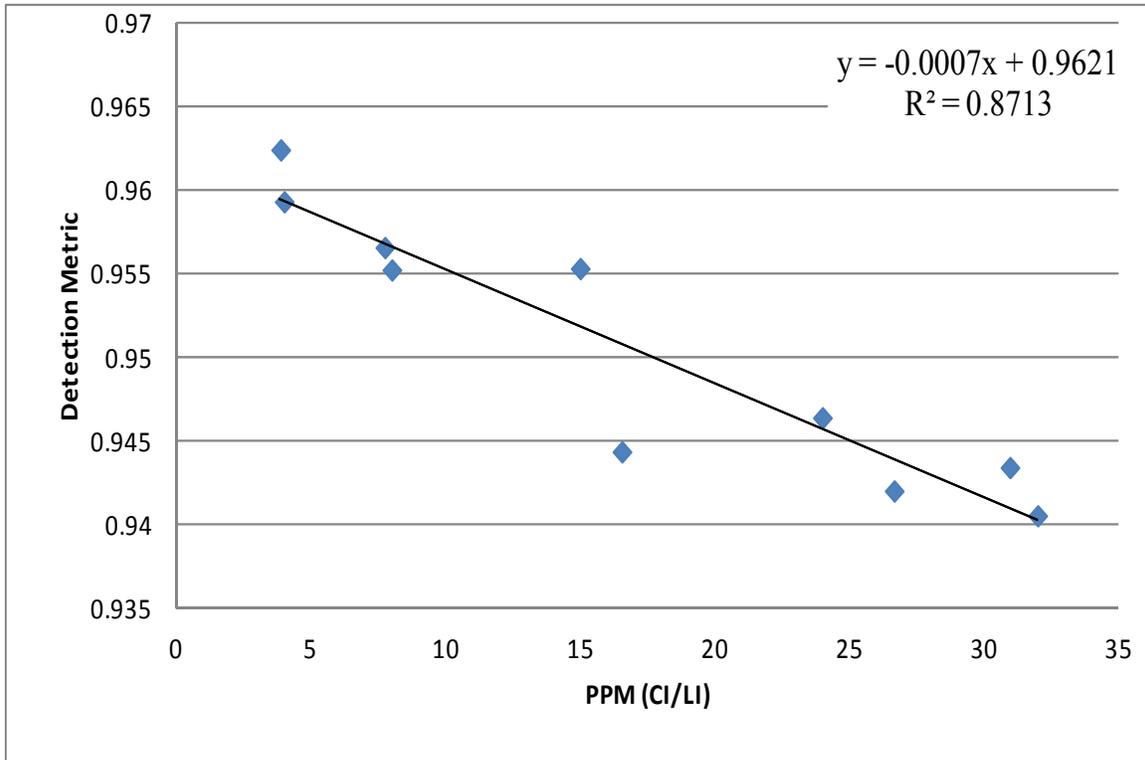


Figure 15: Least squares linear regression graph of concentrated sample with standard addition of TOLAD 4410 CI/LI additive in PQ-1575-11 base fuel

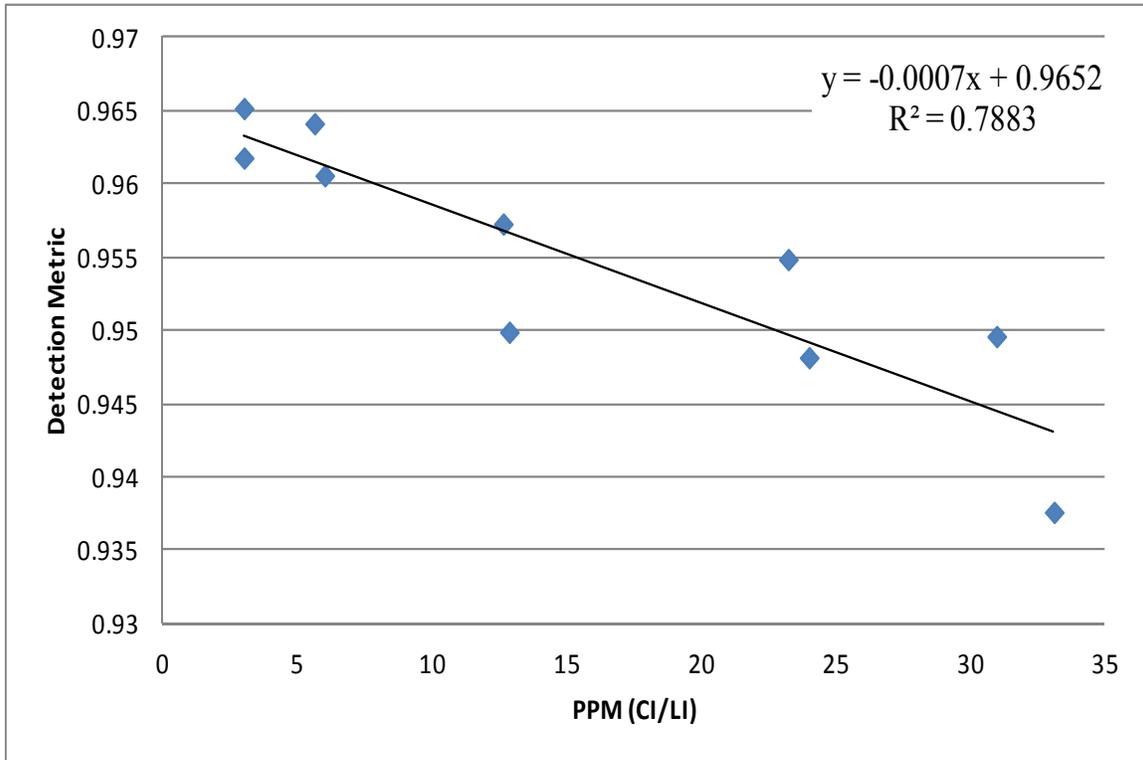


Figure 16: Least squares linear regression graph of concentrated sample with standard addition of TOLAD 4410 CI/LI additive in PQ-1575-11 base fuel

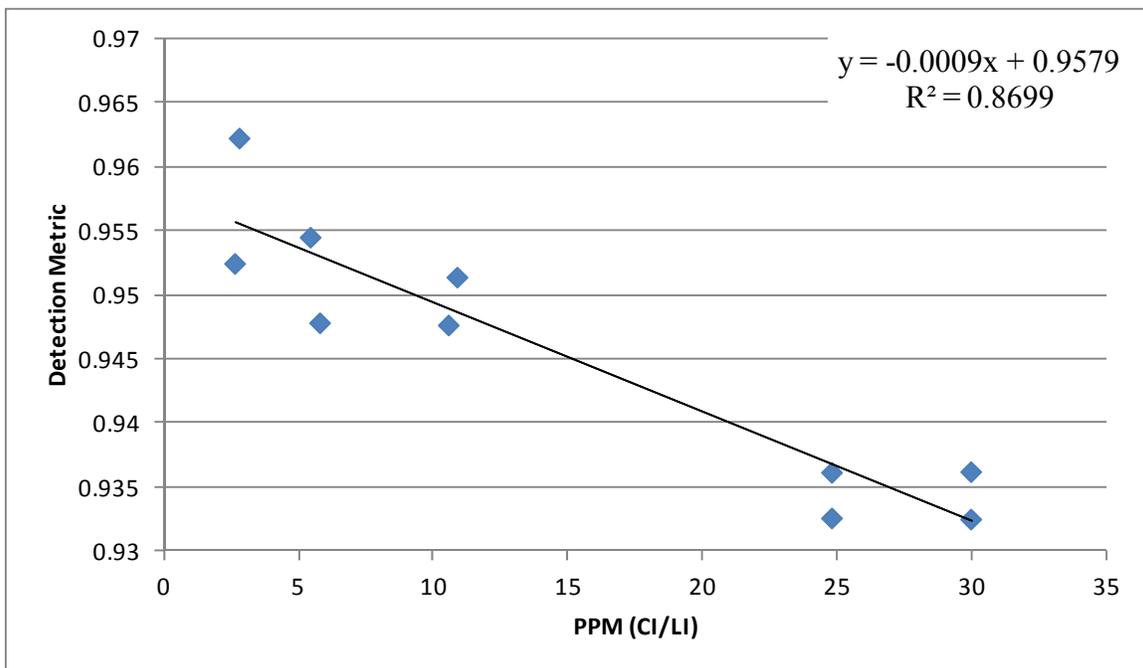


Figure 17: Least squares linear regression graph of concentrated sample with standard addition of TOLAD 4410 CI/LI additive in PQ-1573-11 base fuel

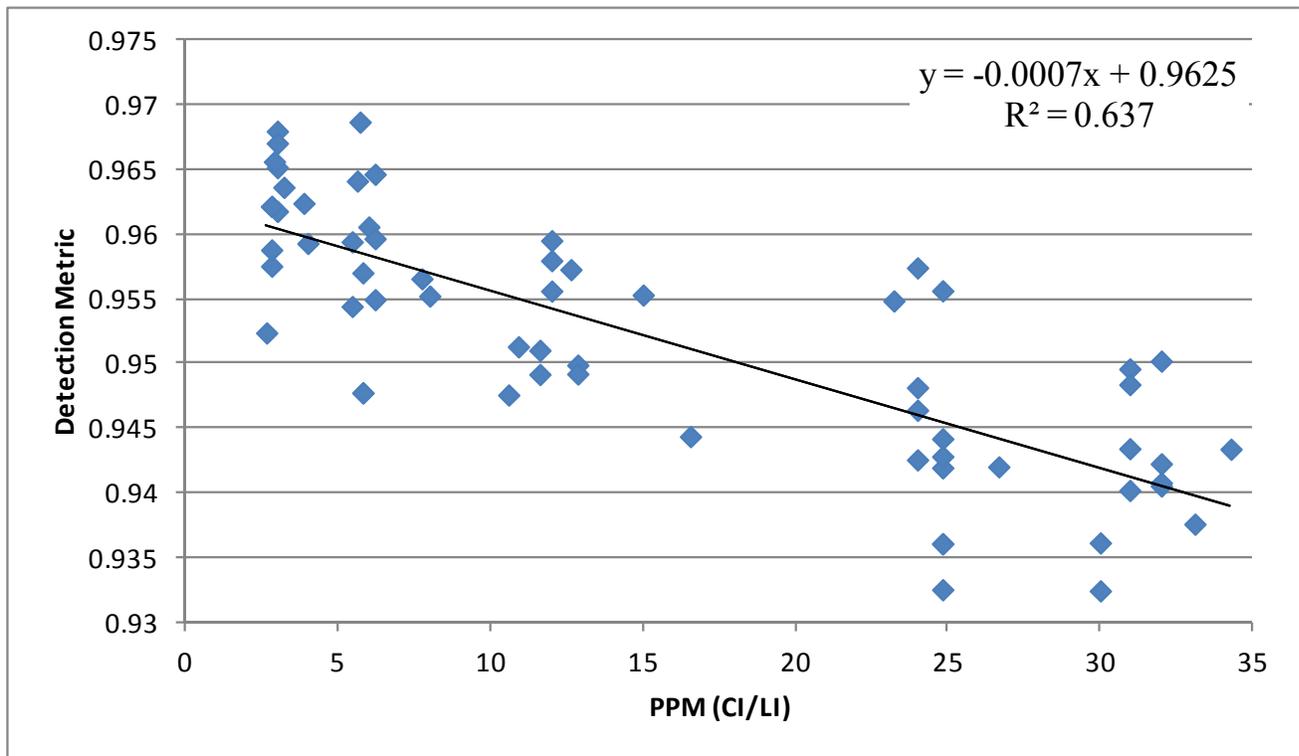


Figure 18: Combined least squares linear regression graph of all concentrated sample with standard addition additive brands data points

FTIR Concentrated Sample with Standard Addition

This methodology also demonstrated significant correlation between the CI/LI concentration and detection metric. The slopes generated in these analyses are in the opposite direction of those generated using the developed instrument and therefore positive because the FTIR recorded absorbance rather than transmittance. The coefficients of determination, R^2 , are 0.9922, 0.8631, 0.9971, 0.8987, 0.9711, and 0.9398 respectively as seen in Figure 19, Figure 20, Figure 21, Figure 22, Figure 23, and Figure 24. When all six sample sets were combined in the same linear regression the coefficient of determination, R^2 , is 0.338 as seen in Figure 25.

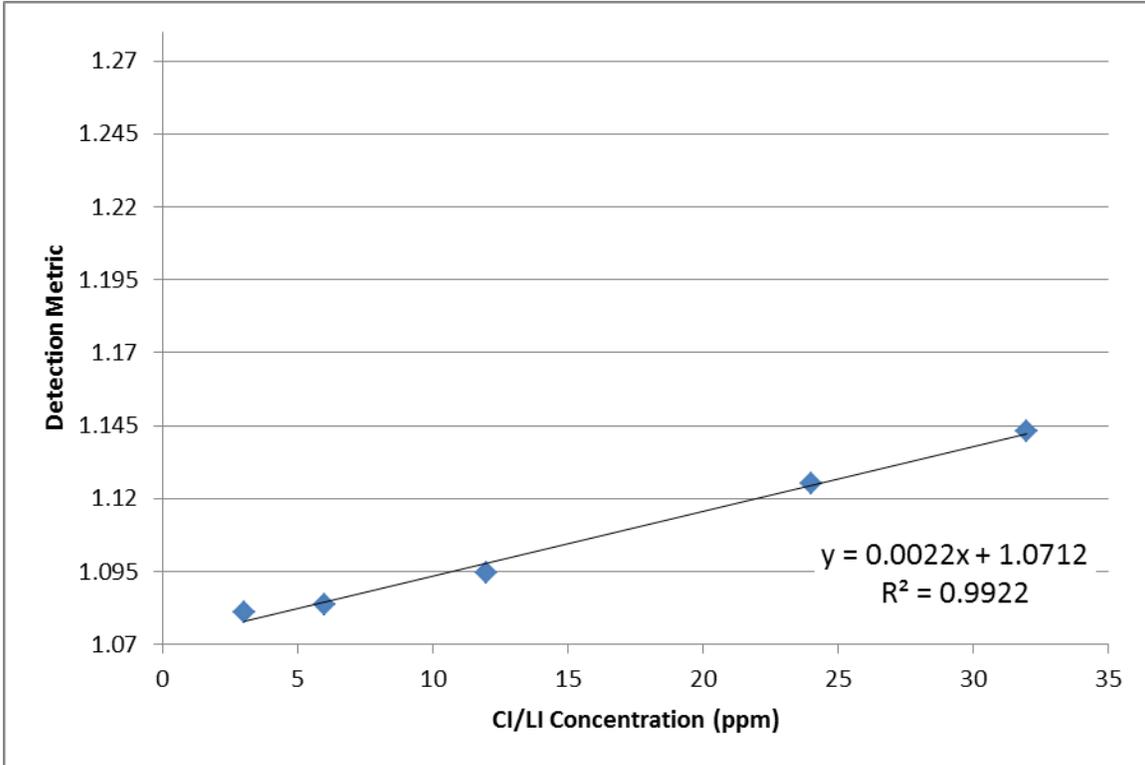


Figure 19: Least squares linear regression graph of FTIR concentrated sample with standard addition of HITEC 580 CI/LI additive in PQ-1577-11 base fuel

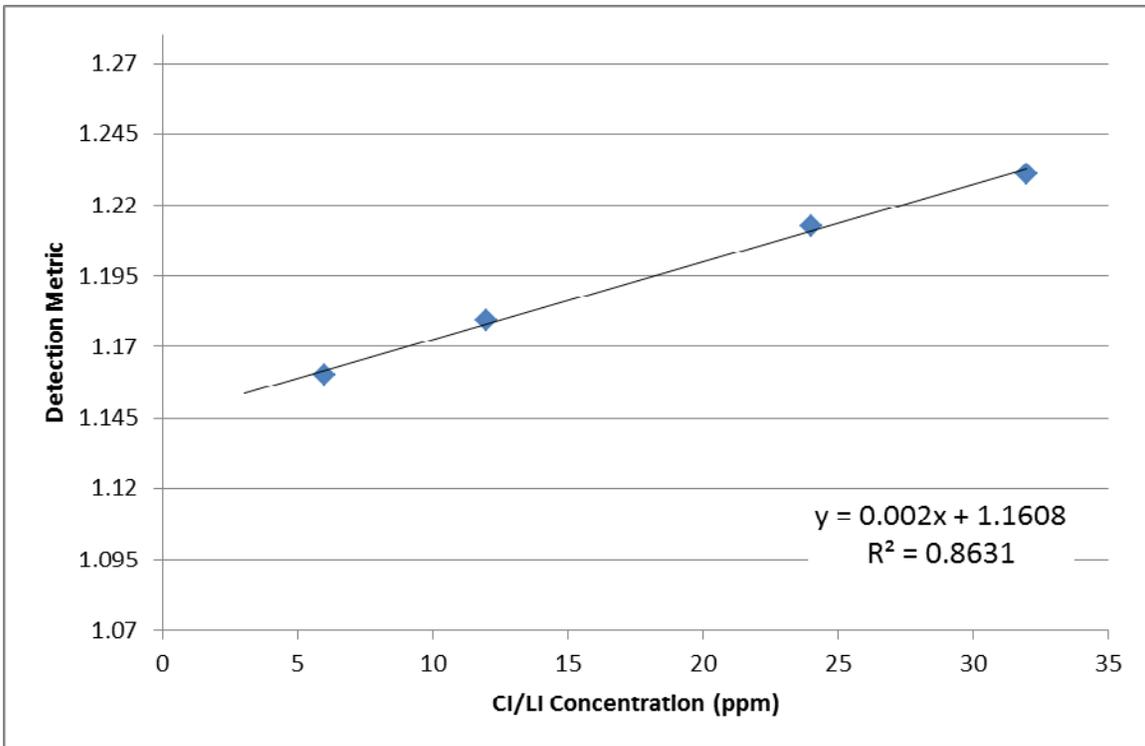


Figure 20: Least squares linear regression graph of FTIR concentrated sample with standard addition of TOLAD 351 CI/LI additive in FL-14394-12 base fuel

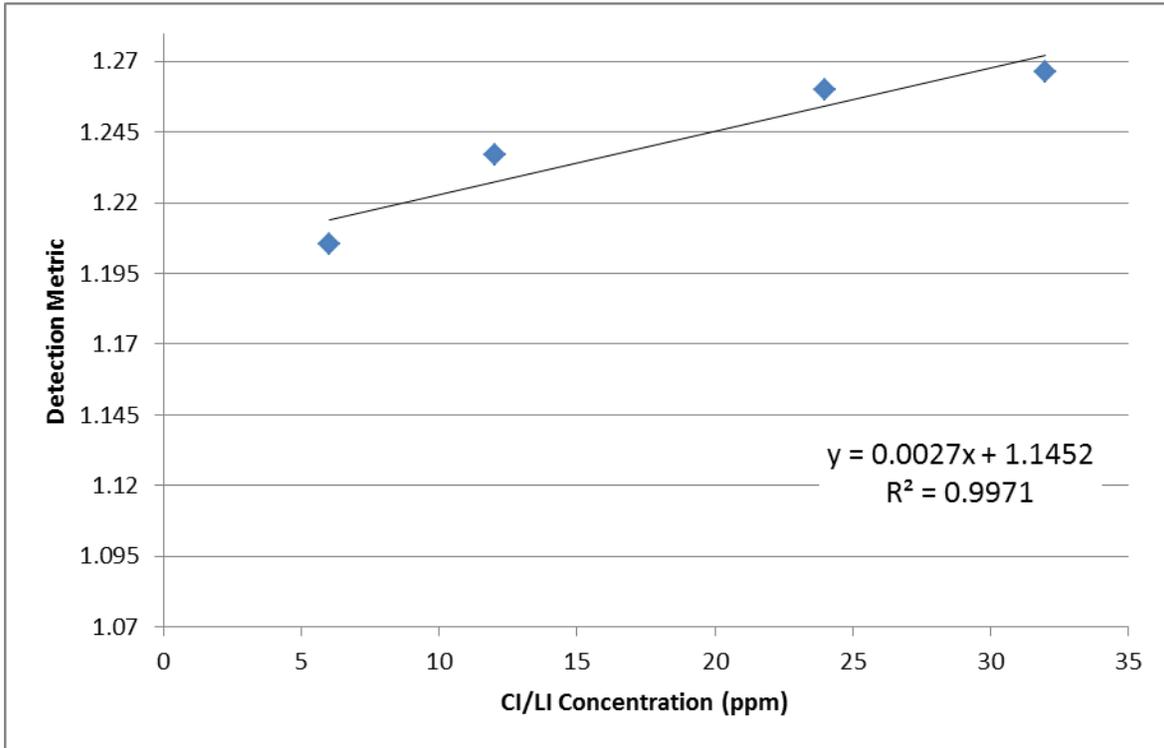


Figure 21: Least squares linear regression graph of FTIR concentrated sample with standard addition of NALCO EXXON 5403 CI/LI additive in FL-14380-12 base fuel

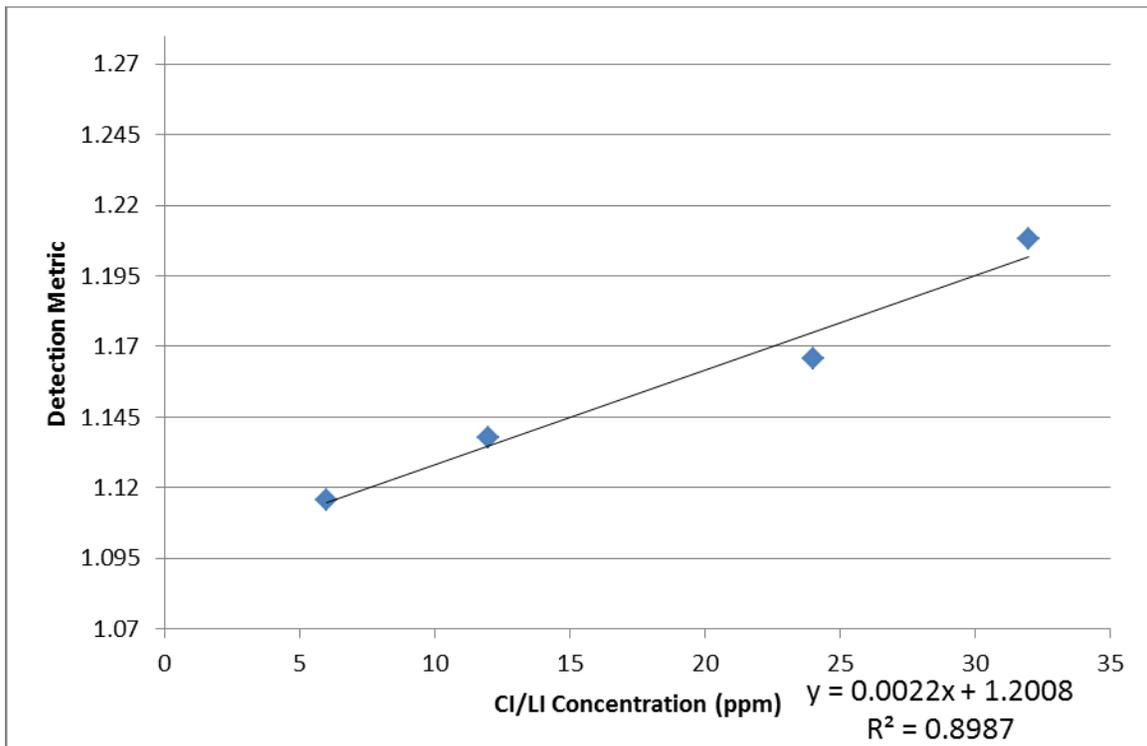


Figure 22: Least squares linear regression graph of FTIR concentrated sample with standard addition of SPEC-AID 8Q22 CI/LI additive in FL-14394-12 base fuel

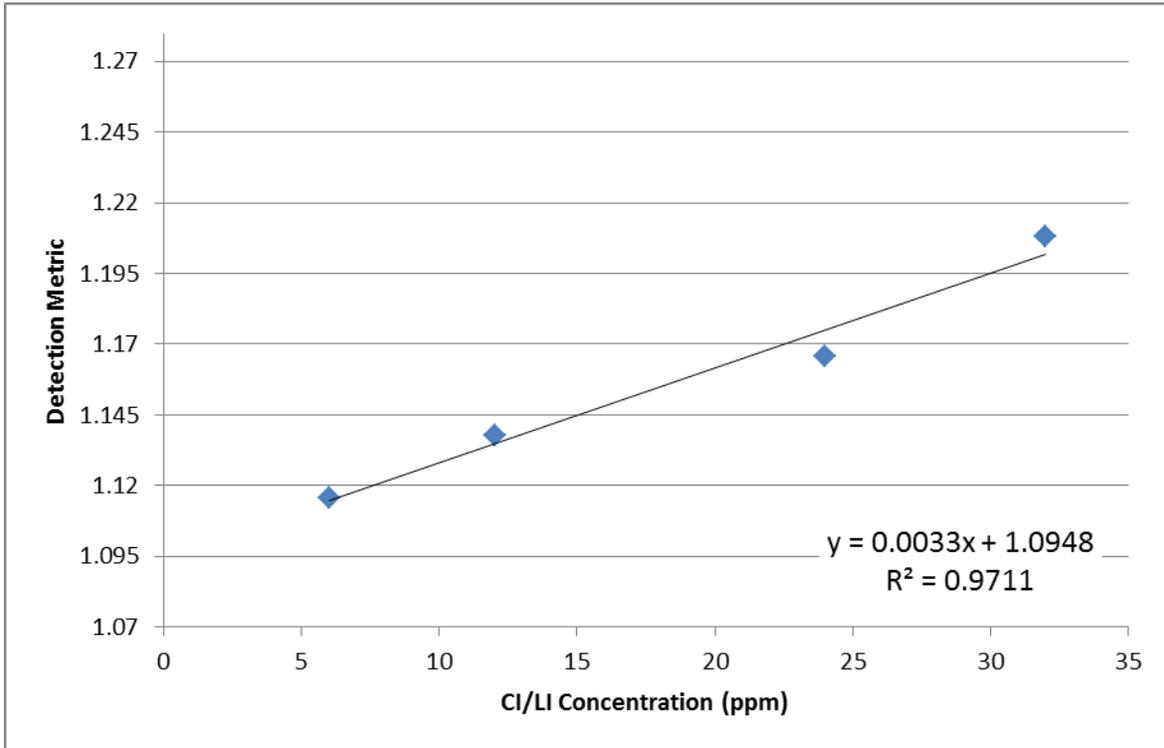


Figure 23: Least squares linear regression graph of FTIR concentrated sample with standard addition of TOLAD 4410 CI/LI additive in PQ-1574-11 base fuel

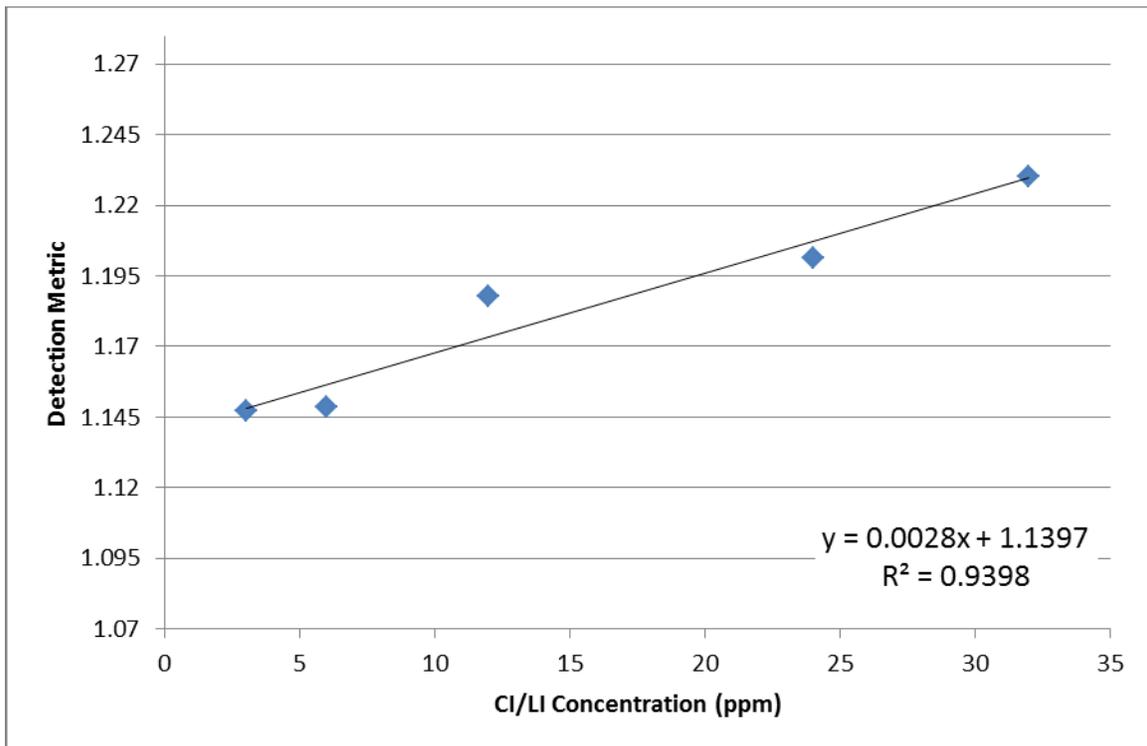


Figure 24: Least squares linear regression graph of FTIR concentrated sample with standard addition of TOLAD 4410 CI/LI additive in FL-14377-12 base fuel

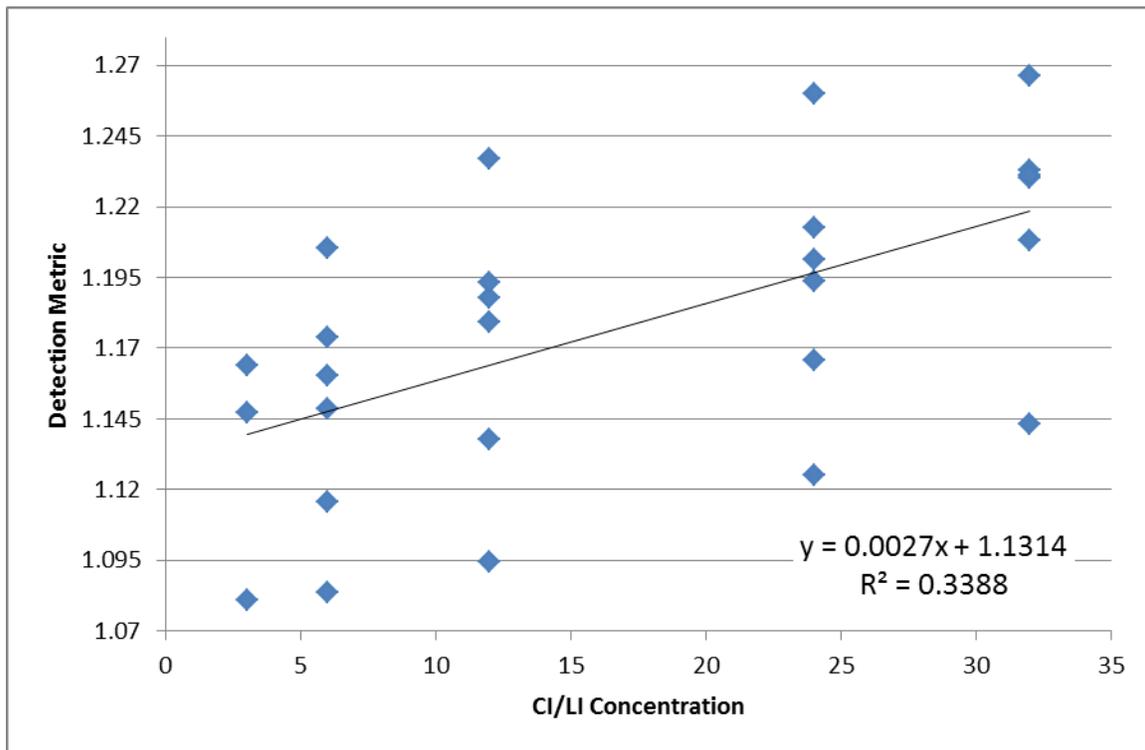


Figure 25: Combined least squares linear regression graph of all FTIR concentrated sample with standard addition additive brands data points

5. Discussion

The objective of this effort was to determine if infrared spectrometry can be used to measure Corrosion Inhibitor/Lubricity Improve in the military's kerosene based fuels in the field. Four different methodologies were tested. The first was a direct sampling methodology. This methodology produced standard curves with coefficients of determination ranging from 0.1194 to 0.2204. The second was a direct sampling methodology with standard addition. This resulted in coefficients of determination ranging from 0.0043 to 0.2596. Coefficients of determination for all methodologies are summarized in Figure 27. Neither the direct sampling methodology nor the direct sampling methodology with standard addition demonstrated a correlation between CI/LI additive concentration and IR absorbance. Therefore both of these methodologies are unable to

measure concentration of CI/LI additive in fuel.

Prior work had indicated some level of success with the concentrated sample methodology using an additional concentration of 100 ppm CI/LI additive in the standard curve linear regression. The inclusion of a 100 ppm standard in the linear regression analysis using the concentrated sample methodology produced a coefficient of determination, R^2 , of 0.8706. However, the acceptable range for CI/LI additive in military fuel ranges from 6-36 ppm CI/LI and the 100 ppm standard was deemed essentially an outlying concentration and removed from analysis. This generated a coefficient of determination, R^2 , of 0.1309. Again, this demonstrated very little correlation between CI/LI additive concentration and IR absorbance. Another clear issue with this data was that the detection metric variance for the 0 ppm fuel standard completely encompassed the measurements of fuels with the additive present. This indicates that the instrument is unable to measure the concentration of unknown fuels. It would therefore only be capable of measuring additive concentration as additive is being added to fuels and for the verification of additive injection equipment.

The concentrated sample methodology produced coefficients of determination between 0.778 and 0.943. This did exhibit the correlation between IR absorbance and CI/LI additive needed effectively quantify CI/LI additive in fuel. However, trials using all CI/LI additive brands in all base fuels were included in the same linear regression the linearity decreased to a coefficient of determination of 0.637. This indicates that a universal standard curve for all CI/LI additive brands is not achievable. This would be prohibitive of developing a single rapid method to measure CI/LI additive in the field using the instrument.

During testing, the concentrated sample with standard addition methodology was repeated, but a Varian 640 FTIR was used to verify instrument performance. This produced coefficients of

determination ranging from 0.8631 to 0.997 and verified that the correlation between IR absorbance and CI/LI additive could be used as an effective quantification method. However, when all FTIR trials were combined into the sample linear regression, the coefficient of determination decreased to 0.3388. This supports the necessity of individual standard curves for each commercial CI/LI additive brand that the military uses and the inapplicability of a universal standard curve for all CI/LI additive brands.

In conclusion, IR spectroscopy is a feasible method to quantify CI/LI additive in military fuels, but the logistical and practical limitations of being unable to measure unknown fuel samples and needing a separate calibration curve for each CI/LI additive brand formulation will severely limit its implementation.

Direct Sample	R²
DCI-A6 in PQ-1572-11	0.2204
HITEC 580 in PQ-1572-11	0.268
NALCO EXXON 5403 in PQ-1572-11	0.1194
Direct Sample with Standard Addition	
DCI-A6 in PQ-1572-11	0.0043
HITEC 580 in PQ-1572-11	0.0741
NALCO EXXON 5403 in PQ-1572-11	0.2596
Concentrated Sample	
SPEC-AID 8Q22 in PQ-1573-11 with 100 ppm standard	0.8706
SPEC-AID 8Q22 in PQ-1573-11 without 100 ppm standard	0.1309
Concentrated Sample with Standard Addition	
DCI-6A in PQ-1572-11	0.943
NALCO EXXON 5403 in PQ-1572-11	0.9008
TOLAD 351 in PQ-1576-11	0.9041
TOLAD 4410 in PQ-1575-11	0.8713
TOLAD 4410 in PQ-1575-11	0.7883
TOLAD 4410 in PQ-1573-11	0.8699
Combined	0.637
FTIR Concentrated Sample with Standard Addition	
HITEC 580 in PQ-1577-11	0.9922
TOLAD 351 in FL-14394-12	0.8631
NALCO EXXON 5403 in FL-14380-12	0.9971
SPEC-AID 8Q22 in FL-14394-12	0.8987
TOLAD 4410 in PQ-1574-11	0.9711
TOLAD 4410 in FL-14377-12	0.9398
Combined	0.3388

Figure 26: Summary of Coefficients of Determination (R^2)

6. References

1. Performance Specification Inhibitor, Corrosion/Lubricity Improver, Fuel Soluble (NATO S-1747). 2011. MIL-PRF-25017H w/Amendment 1.
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7. Acknowledgements

I would like to thank the U.S. Army Tank Automotive Research Development and Engineering Center and the Force Projection Technology Team for allowing me to work with them this summer. I would especially like to thank my summer hire sponsor Joel Schmitigal for organizing this project and guiding me with my work. I would also like to thank Dr. Werner for his editing help.