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**ESTIMATION OF MIDDLE DISTILLATE FUEL
PROPERTIES BY FT-IR AND
CHEMOMETRICS
Part I. CALIBRATIONS AND VALIDATIONS**

**INTERIM REPORT
TFLRF No. 321**

By

**G.E. Fodor
S.A. Hutzler**

**U.S. Army TARDEC Fuels and Lubricants Research Facility (SwRI)
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San Antonio, Texas**

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13. ABSTRACT (Maximum 200 words) As an alternative to established laboratory protocols, an FT-IR method was developed for the rapid, simultaneous determination of several pertinent fuel properties, using less than 2 mL of sample. The method uses a single wavelength range of baseline uncorrected, raw FT-IR spectra. Neither baseline correction nor use of restricted wavelength regions improved the models. Using training sets comprised of over 500 middle distillate fuel samples, models were built to derive pertinent fuel properties from FT-IR spectra. These models showed that a single calibration training set may properly model several properties of fuels collected during different climatic seasons. cursory experiments suggest that properties of diesel and turbine fuels may be more accurately derived by FT-IR if separate calibration models are used for each class of fuel. At the present state of development, the following fuel property values may be confidently measured by FT-IR: gravity, API°; density, g/mL; kinematic viscosity, cSt, at 40°C; boiling point at 50%; cetane index; carbon, wt %; hydrogen, wt%; carbon-to-hydrogen ratio, C/H; heat of combustion, MJ/kg; monocyclic aromatics, wt%; dicyclic aromatics, wt%; polycyclic aromatics, wt%; and total aromatics, wt%. Cetane number correlations between ASTM D 613 and the FT-IR derived data are limited by the precision of the benchmark data. For comparison, several calibration models were developed from near-infrared (near-IR) and FT-IR spectra. It was demonstrated that, as expected, FT-IR provides better models for the determination of fuel property values.				
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EXECUTIVE SUMMARY

Objectives: The objective of this study is to demonstrate the feasibility of replacing traditional analytical procedures for determining individual diesel fuel properties with a rapid and reliable measurement using midband Fourier transform infrared (FT-IR) spectroscopy that can measure several of these properties simultaneously. Further studies compared the FT-IR method with near-infrared procedures.

Accomplishments: As an alternative to established laboratory protocols, an FT-IR method was developed for the rapid, simultaneous determination of several pertinent fuel properties, using less than 2 mL of sample. The method uses a single wavelength range of baseline uncorrected, raw FT-IR spectra. Neither baseline correction nor use of restricted wavelength regions improved the models.

The following fuel property values may be confidently measured by FT-IR: gravity, API°; density, g/mL; kinematic viscosity, cSt, at 40°C; boiling point at 50%; cetane index; carbon, wt%; hydrogen, wt%; carbon-to-hydrogen ratio, C/H; heat of combustion, MJ/kg; monocyclic aromatics, wt%; dicyclic aromatics, wt%; polycyclic aromatics, wt%; and total aromatics, wt%. Estimation of several other properties is also possible where ultimate precision is not needed.

For comparison, several calibration models were developed from near-infrared and FT-IR spectra. It was demonstrated that FT-IR provides better models for the determination of fuel property values.

Future studies will investigate ways to improve the precision and scope of the method and standardization transfer requirements between FT-IR instruments.

Military Impact: This method may be used to measure several important fuel properties not only in the laboratory but also in remote field areas of operation.

FOREWORD/ACKNOWLEDGMENTS

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I. OBJECTIVE

The objective of this study is to demonstrate the feasibility of replacing traditional analytical procedures for determining several pertinent diesel fuel properties with a rapid and reliable measurement using midband Fourier transform infrared (FT-IR) spectroscopy that can measure several fuel properties simultaneously. Further studies compare the FT-IR method with near-infrared (near-IR) procedures, and will investigate standardization transfer requirements between FT-IR instruments.

II. INTRODUCTION AND BACKGROUND

Most routine evaluations in petroleum analytical laboratories are performed using standard test methods established by the American Society for Testing and Materials (ASTM), the Institute of Petroleum (IP), the military, or other agencies. While these methods may be accurate and enjoy a high degree of acceptance, they also have some disadvantages. For example, they may require relatively large sample sizes, they may use toxic or environmentally dangerous chemicals, or they may be cumbersome and time consuming.

Several articles have described the use of near-IR spectroscopy to determine gasoline^{1,2,3,4} and middle distillate fuel properties.⁵ The usual operating range for near-IR spectroscopy is between the wavelengths of 0.9-2.5 μm , while FT-IR spectra are generally obtained at wavelengths between 2.5-25 μm , corresponding to the 4000-400 cm^{-1} wavenumber region. A near-IR spectrum is the result of overtones and combination bands of the fundamental frequencies associated with molecular vibrations of certain functional groups that produce the directly measurable region of FT-IR. Since FT-IR spectroscopy is based on the measurement of characteristic fundamental resonances, it produces specific, usually sharp, well-defined peaks at substantially increased extinction coefficients. As expected, studies^{6,7} indicated that FT-IR-derived data of middle distillate fuels for aromatic hydrocarbon contents, carbon-to-hydrogen ratio, refractive index, heat of combustion, cetane index, viscosity, and density were superior to those derived by near-IR.⁵ Similar results were obtained for the analysis of gasolines by FT-IR.⁸

Natural requirements for developing FT-IR spectroscopic correlations are that data be calibrated to those fuel properties that are due to chemical structural features associated with active infrared resonance bands, and that measured fuel property values must relate linearly to spectral intensities, that is, they must obey Beer's law. To illustrate some of the composition versus physical property relationships, some generally accepted arguments may be considered. For any given fuel type, high specific gravity is associated with increased concentrations of aromatic or naphthenic hydrocarbons; low specific gravity is associated with elevated concentrations of open-chained paraffinic hydrocarbons. The heat of combustion of a fuel is influenced by its oxidation state, that is, the carbon-to-hydrogen ratio, also implied in the aromatic hydrocarbon concentration, of the fuel. Increased aromatic hydrocarbon concentrations increase octane number and decrease cetane number. The cloud point of a fuel is decreased by increasing the *iso* to *normal* paraffin ratio and increasing aromatic hydrocarbon content in the fuel. Thus, several fuel properties are determined by the presence of special atomic groups: the branching of saturated hydrocarbons, the ratio of *normal* to

iso paraffins, the aromatic hydrocarbon content (including the type and degree of substitution on the aromatic rings), and the ratio of these various constituents. All these chemical features are reflected to some degree in the IR spectra of compounds.⁹ Additionally, the various sulfur, nitrogen, and oxygen containing functional groups also have well-defined characteristic resonance bands in the studied frequency range that, within experimental limits, allow their qualitative and quantitative assessment. The presence and quantity of certain fuel additives may also be determined.

The purpose of this study was to (1) evaluate the applicability of the methodology developed in our earlier work⁶⁻⁸ to a wide range of middle distillate fuels (2) evaluate the comparative merits of near-IR and midband FT-IR spectroscopies, and (3) develop the basis for calibration file transfer.

III. TECHNICAL APPROACH

A large number of fuels with varied compositional and performance parameters were collected during three seasonal periods and analyzed by ASTM methods to serve as "gold standard" or "benchmark" procedures in the calibration or training sets for establishing calibration models.

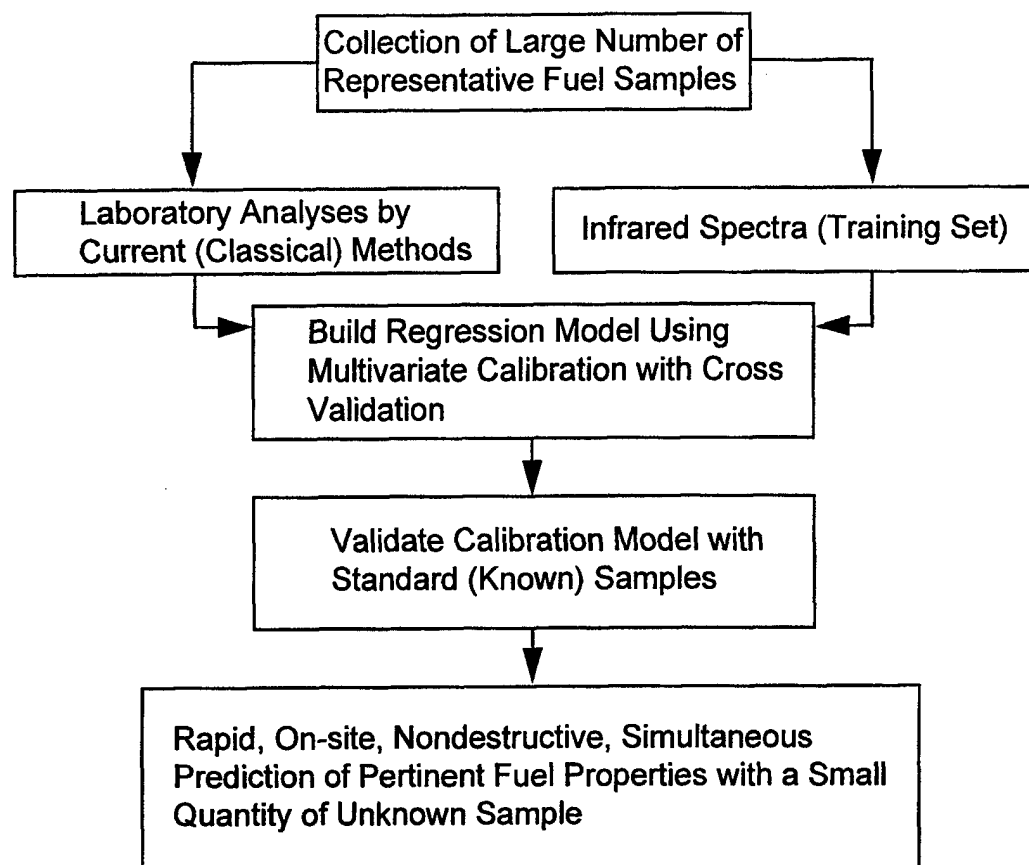
An FT-IR spectrum was collected for each fuel on each of two FT-IR instruments, and these spectra were correlated with the corresponding selected fuel property values. Results of the calibration sets were then validated using fuel samples that were not part of the calibration sets. Effects of changes in calibration parameters and spectral manipulations were also explored.

Beyond proving that several pertinent fuel properties may be simultaneously determined from their FT-IR spectra, the following issues need to be addressed:

- A. Are separate calibrations needed for samples collected during different seasonal periods, or may these various samples be combined into a single calibration file?
- B. May calibration files be developed for a combination of turbine and diesel fuels, or must these fuels use different calibration models?
- C. May calibration files be directly transferred from one instrument to another, or must special calibration transfer programs be developed?
- D. Will calibration transfer programs retain sufficient predictive precision, or must separate calibrations be developed for each instrument?
- E. Comparison of FT-IR and near-IR calibration models

In addition, two commercial chemometric software packages were investigated to determine their applicability to the current project. The software applications were evaluated in terms of their capabilities and their adaptability to integrated laboratory and field applications. In this report we summarize the results of experiments relating to items (A), (B) and (E) only. Items (C) and (D) will be the subject of a future report.

The following simplified flow diagram illustrates the process involved in the development of the correlation models for fuel properties from FT-IR spectra:



IV. SELECTION OF FUELS

Test fuels were collected during winter, spring, and summer months from commercial filling stations in all 50 states of the U.S.A. and at various military installations within the United States and around the world. This approach yielded fuel samples that were as diverse as possible and included the broadest range of values for the properties of interest. Fuel types included commercial, low sulfur grades No. 2-D and No. 1-D diesel fuels (ASTM D 975), military diesel fuels (VV-F-800, now replaced by CID A-A-52557), aviation kerosenes JP-5 (MIL-T-5624N) and JP-8 (MIL-T-83133C). The majority of the JP-5 samples were acquired from sources within the continental United States, while most of the JP-8 samples originated outside the continental U.S.A. The seasonal fuel sets and their combinations are identified as:

- Fuel Set A: collected during December
- Fuel Set B: collected during March
- Fuel Set C: collected during July
- Fuel Set D: combination of Fuel Sets A, B, and C.

To select the calibration and validation samples, each fuel set (A, B, and C) was randomly shuffled. The first 20 percent of each randomized set was then designated as the validation set while the remaining samples were specified as the calibration set. The calibration and validation samples for

the combined fuel set, D, were selected by combining the calibration and validation samples, respectively, from sets A, B, and C.

Table 1 identifies the laboratory benchmark (mainly ASTM) analytical procedures used to obtain the calibration data on the fuels. Repeatability and reproducibility of the listed procedures are summarized in Table 2. The minimum, maximum, average, and the range of the more promising fuel property values for Fuel Sets A, B, C, and D are summarized in Table 3. While over 33 fuel properties were measured on each fuel, some of these properties did not correlate with FT-IR spectra.

V. EXPERIMENTAL

Spectroscopic data were collected for all fuels on a Nicolet Model 510 (Instrument A) and on a Perkin Elmer System 2000 (Instrument B) FT-IR spectrometer. Both units were equipped with deuterated triglycine sulfate (DTGS) detectors and covered horizontal attenuated total reflectance (HATR) zinc selenide (ZnSe) sample cells (Spectra-Tech Model No. 0012-436T). The crystal angle of the HATR cells is 45° , with 12 internal reflections through the sample. The nominal depth of beam penetration at 1000 cm^{-1} is $2\text{ }\mu\text{m}$. Due to substantial differences in scanning speeds, the average of 32 FT-IR scans at a resolution of 4 cm^{-1} was collected for each fuel on Instrument A, while only an average of 16 scans at a resolution of 4 cm^{-1} was collected on Instrument B. The peak-to-peak signal-to-noise ratios of the two instruments are:

Instrument ID	wavenumber, cm^{-1}			
	2200-2150	2150-2100	2100-2050	2050-2000
Instrument A	3540	5660	5440	4160
Instrument B	7860	9650	5510	4170

To avoid problems associated with ZnSe cutoff frequencies, each spectrum was truncated to the wavenumber region of $4000\text{--}650\text{ cm}^{-1}$. No spectral region was excluded from building the calibration models.

The use of transmission cells was investigated, as these cells were expected to produce superior results; however, cells even at the shortest available path length of about 0.015 mm produced spectral overload, with absorbance values in the non-linear range. The use of fully closed "circle" or "tunnel" ZnSe ATR cells was also explored; however, obtaining reproducible baselines and cross-contamination free spectra of successive samples was found to be unacceptably time consuming. Due to the speed of the analysis, sample integrity was not believed to have been excessively compromised by the use of covered HATR cells.

Near-IR spectra were collected on an acousto-optic tunable filter (AOTF) near-infrared fuel analyzer procured from Brimrose Corporation of America. Scans were collected with a fiber-optic

transreflectance immersion probe with an effective pathlength of 1 cm. Each spectrum was acquired as an average of 32 scans run in the 1000-1600 nm region with a 2 nm increment.

VI. CHEMOMETRICS

A. General

Models relating selected fuel properties to FT-IR spectra were developed using chemometrics, a science that applies statistical and mathematical methods to chemical and physical data. Spectroscopic data were correlated to fuel property values with a partial least squares (PLS) regression. The PLS algorithm was run from either PLS_Toolbox™ (Eigenvector Research, Inc.) within the MATLAB™ framework, or the PLSplus program within the GRAMS/386™ and GRAMS/32™ (Galactic Industries Corporation) software package.

The PLS method creates a simplified representation of the spectroscopic data by a process known as spectral decomposition. Good summary treatises of PLS were published by Geladi and Kowalski,¹⁰ Martens and Naes,¹¹ and Haaland and Thomas.^{12, 13} A precursor to the PLS technique, which is closely linked to the bilinear framework used in PLS, is the latent root regression analysis, formulated in the 1970s by Webster, Gunst, and Mason.¹⁴ The PLS approach is based on a bilinear modeling method. The PLS algorithm initially calculates the concentration, or property value, weighted average spectrum of all the spectra of the fuels in the calibration matrix. An important feature of PLS is that the concentration, or property value, information is used during the decomposition process. Instead of decomposing the spectral data and the concentration data separately, they are done simultaneously. The result is a set of scores (scaling constants) and a set of eigenvectors (a.k.a., loading vectors or factors) for both the spectral data and the concentration data. After each factor is calculated, its contribution is removed from the original data. The decomposition is then repeated until all of the desired factors have been calculated. Assuming that a relationship exists between the spectral scores and the concentration scores, a calibration model is constructed.

To optimize a calibration model, a computationally intensive procedure known as cross-validation can be carried out. In the cross-validation procedure, a given number of samples are removed from the calibration data set. A calibration model, calculated from the remaining samples in the training set, is then used to predict the property value of the removed samples. The residual errors, or the difference between the predicted and known concentration values, are squared and summed to determine the prediction error. The cross-validation process is repeated until each sample has been left out once. The information obtained from a cross validation is then used to select the number of factors to correctly model the fuel property. The result is a refined regression model that is useful in predicting the property of the unknown fuel. Also, since the samples that are removed are true unknowns to the calibration model, the predictive power of the model is revealed. After a calibration model is established, it must be tested by independent validation experiments, in which the calibration model is applied to similar fuels that were not part of the calibration training set. The

predicted property values may then be compared with those derived by the established ASTM procedures.

It is critical to establish the correct number of factors to be used in the correlation files, as the predicted fuel property values calculated from the model depend on the number of factors used in the model. Too few factors will not adequately model the system, while too many factors will introduce noise vectors in the calibration. These noise vectors will result in less than optimum prediction for samples outside the calibration set. The PLSplus program by Galactic Industries provides data for selecting the appropriate factor by plotting the predicted residual error sum of squares (PRESS, defined under statistics) versus the factor.

The factor may be selected for (a) the point at which the PRESS value is at a minimum, (b) the point at which the curve indicates that further increase in factors should have negligible effects (a rather arbitrary choice), or (c) a compromise as recommended by Haaland and Thomas.¹² These authors advise the use of F-statistic to arrive at the best compromise in factors. The F-statistic can be calculated as the ratio of the minimum PRESS value to all PRESS values corresponding to fewer factors. As the difference between the minimum PRESS and other PRESS values becomes smaller, the probability that each additional factor provides significant improvement to the model decreases. Haaland and Thomas empirically determined that the optimum number of factors should be at the first PRESS value at which the F-statistic probability, p , drops to or below 0.75. While initially we used factors corresponding to an F-statistic probability of 0.5 (coinciding with a maximum for the squared correlation coefficients), to avoid possible overfitting we standardized by using the compromise value of 0.75 as suggested by Haaland and Thomas.

Some authorities claim that the number of factors in a calibration file must be held to "10 or less." We conducted several experiments in which we reduced the number of factors from those recommended by the Haaland and Thomas criterion to "10 or less." Invariably, results of the validation experiments deteriorated. We hypothesize that some properties of a compositionally complex system, such as a petroleum derived fuel, may not be sufficiently modeled by a model having only 10 or less factors. Therefore, to allow for better modeling while still keeping the time required to develop the calibration models reasonably short, we increased the maximum number of allowed factors to 20.

B. Statistics

The following are definitions that will be used throughout the remainder of the text. They are all similar in that they attempt to measure the average error in a given model. In the following equations, y_i' is the predicted value of sample i for a given property, y_i is the known value of the property for that sample, and n is the number of samples in the calibration set.

1. Predicted Residual Error Sum of Squares, PRESS measures how well the calibration model predicts the property value as each factor is added, defined as:

$$\sum_{i=1}^n (y_i' - y_i)^2 \quad \text{Equation 1}$$

The following equation defines the other error terms used in the report:

$$\sqrt{\frac{\sum_{i=1}^n (y_i' - y_i)^2}{n}} \quad \text{Equation 2}$$

2. Standard Error of Prediction for Cross-Validation, SEP(CV) is the measure of a model's ability to predict property values of new samples that are not a part of the calibration model.^{15, 16} The samples that are removed during each rotation in a cross-validation are subsequently validated using a model built from the remainder of the samples.
3. Standard Error of Calibration, SEC is similar to SEP(CV) with the exception that all samples being predicted were also used in the building of the calibration set.^{15, 16} For this reason, the error in the calibration will usually appear to be lower when using SEC because each sample being predicted has a complimentary sample in the calibration. SEP(CV) is more rigorous and gives a better indication of the robustness of the calibration.
4. Standard Error of Prediction, SEP. After a final calibration model has been constructed, the model is again validated using a separate set of validation samples that were not part of the original calibration set and therefore not included in the model.^{15, 16}
5. Square of the Multiple Correlation Coefficient, R². The multiple correlation coefficient, R², is a measure of how well a linear model fits a given set of data, and has a value between 0 and 1. If the estimated and known values are very similar, then a good fit has been achieved and R² will be close to 1. A poor fit will give an R² closer to 0. R² can be interpreted as the total variability in y (fuel property values) that is explained by x (spectral data). However, caution must be taken in using R² because a large value for R² does not necessarily mean a good fit between the model and the data.¹⁷ In Equation 3, for a given fuel property and sample, y_i is the known value, y_i' is the predicted value, y'' is the mean of all y_i, and n is the number of samples.

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_i - y'_i)^2}{\sum_{i=1}^n (y_i - \bar{y})^2} \quad \text{Equation 3}$$

C. Data Preprocessing

Three data pretreatments were tried in an attempt to optimize the calibration models.

1. Mean-centered spectra are generated by subtracting the mean of the entire data set from each individual spectrum. The mean of a mean-centered data set is zero. As a result, the spectra are now centered around the mean of the data set. In general, mean-centering enhances the concentration differences between samples resulting in more accurate calibration models.
2. First difference spectra are generated by calculating the arithmetic difference between the individual points of the raw spectral data. For a spectrum with n points, the first difference spectrum would be [(X₂-X₁), (X₃-X₂), ... (X_n-X_{n-1})]. The resultant first difference spectrum would contain n-1 points. Using first difference preprocessing has the effect of correcting any baseline offsets in the spectra. Mid-IR spectra typically do not suffer from substantial baseline shifts, unlike near-IR spectra where baseline offsets are commonplace and where baseline correction is typically essential.
3. Second difference spectra are generated by taking the first difference of a first difference spectrum. The result is a spectrum with n-2 points. Using second difference preprocessing corrects both the baseline offset as well as any sloping of the baseline.

The first and second derivatives may be approximated as the first and second differences. However, the Savitsky-Golay and Gap methods¹⁶ are more exact derivatives. There are disadvantages inherent to all of these methods. Difference spectra are often more noisy because the method does not incorporate any smoothing capability. The Savitsky-Golay and Gap methods are often more complicated to use because they require additional parameters for the calculation. Future investigations might look at the effects that these alternate methods may have on the accuracy of the calibration.

VII. PLSplus CALIBRATIONS

Fuel property values for all of the fuels in the calibration sets were entered into a master file to develop the first, or preliminary, correlations. In this preliminary operation we identified possible property value outliers in each category. When feasible, the identified outliers were reanalyzed to confirm or correct the data. When reanalysis was not feasible, the outlier fuel was excluded from

the second calibration in which each fuel property was individually correlated with the corresponding spectra. As new outliers may be identified in each successive calibration, their exclusion could yield improved, or apparently improved, calibration models. However, while in some studies up to 10 percent of the total number of samples are excluded, in our studies only those outliers we identified in the preliminary calibrations were excluded in the subsequent calibrations for individual fuel properties. This resulted in outliers numbering less than 5 percent of the total number of samples in their respective fuel set. Table 4 shows the number of samples used (a) in each fuel set, (b) in the precalibration runs, (c) in the individual fuel property calibrations, and (d) for validation.

The effects of several precalibration and calibration parameters were explored, including mean-centering of spectra, mean-centering of first and second difference spectra, varying the number of cross-validation rotation samples from 5 to 50, and excluding featureless (baseline) regions of spectra. The number of data points in each spectrum was reduced within the 4000 to 650 cm^{-1} region to 869 for spectra from Instrument A and to 838 for spectra from Instrument B.

Using mean-centered spectra from Instrument A and Fuel Set D, some preliminary models were constructed for the following eight fuel properties: density (D 4052); kinematic viscosity, cSt, at 40°C (D 445); cetane number (D 613); two cetane indexes (D 976 and D 4737); carbon-to-hydrogen ratio (D 5291); net heat of combustion (D 240); and total aromatic hydrocarbon contents (D 5196). After the property value outliers were excluded from these preliminary calibration models using factors at $p \leq 0.75$, individual calibration models were developed for each of these eight properties using (a) mean-centered, (b) mean-centered first difference spectra, and (c) mean-centered second difference spectra generated on both Instruments A and B. Calibration and validation results are summarized in Tables 5-8. Calibration and validation experiments gave variable results. Calibration results, judged by the SEP(CV) and R^2 values, gave best results if only mean-centered spectra were used, with mean-centered first difference pretreatment providing nearly identical results. Normally, mean-centered second difference spectra gave inferior results. Validation experiments gave much wider variations in the result. However, generally, mean-centered or mean-centered first difference spectra gave better results than mean-centered second difference spectra.

Effects of increasing the number of samples in the cross-validation rotation from 5 to 50 were explored using mean-centered and mean-centered second difference spectra. These calibration data, summarized in Table 9, indicate that sample rotation of 5 samples produced better statistics than sample rotation of 50 under either preprocessing modes. Validation results are summarized in Tables 10 and 11 for the mean-centered and mean-centered second difference spectra. These data indicate that sample rotation of 50, or approximately 10 percent of the total sample population, may provide essentially identical results as using 5 samples in each rotation, and substantially reduce the time required to build the calibration model. These observations stress the need to verify the performance of calibration models using validation samples.

It was also shown that exclusion of featureless baseline segments of the raw, baseline uncorrected spectra within the single spectral region of 4000 - 650 cm^{-1} decreased the computer time required

to build the calibration model. However, it did not appreciably affect the calibrations' R^2 and SEP(CV) values.

To foster reasonable simplicity and comparable results in this study, it was decided to carry out calibrations on mean-centered baseline uncorrected spectra within the single spectral region of 4000-650 cm^{-1} , using cross validations with a sample rotation of five fuels. While we showed that further individualized refinements may be carried out, achieving such further refinements are beyond the present scope of this project.

The three sets of fuels samples, collected during three different climatic seasons, provided comparison in calibration results among the various fuel sets and their combinations. While minor seasonal adjustments were noted in density values, most seasonal variations in fuel composition were masked by geographic regional effects, as shown in Table 3. For this reason, only summary results are presented for the individual Fuel Sets A, B, and C, while providing more details from the studies for composite Fuel Set D, based on spectra generated in FT-IR Instruments A and B.

Results for each set of calibration experiments are summarized in tables noting (a) property name, (b) number of samples in the calibration set, (c) number of factors (F) used, (d) SEP(CV), and (e) R^2 , using (1) factors at minimum PRESS and (2) factors as recommended by Haaland and Thomas, *i.e.*, the number of factors at the first PRESS value at which the F-ratio probability (p) dropped to or below 0.75.

In evaluating the significance and usefulness of the calibration data, both the data error, as expressed by SEP(CV) and data scatter or "goodness of fit", *i.e.*, the squared correlation coefficient, R^2 , must fall within acceptable limits. However, the listed SEP(CV) error data may not be directly compared to the precision statements of the benchmark ASTM or ISO methods that are summarized in Table 2. The presented calibration data relate to the quality of correlations of data derived between the historically accepted benchmark methods and the spectral data, but do not reflect the repeatability or reproducibility of the FT-IR method, as no such data have been produced in this study. However, both data sets relate to the boundaries for the accepted or the achievable values. Comparing the SEP(CV) data from the FT-IR correlations and the precision statements of the standard methods in such a light provides a cursory measure of acceptability of the FT-IR data.

The squared correlation coefficients, R^2 , between the benchmark data and the FT-IR values include the scatter of both methods. Since the FT-IR spectra are correlated to the data derived by the benchmark methods, without further treatment the FT-IR data cannot be better than that on which they are based. Any uncertainty in the benchmark data will be part of the FT-IR data. For instance, as density (an FT-IR correlatable fuel property) may be determined with precision, the derived FT-IR data are also defined to a high degree of precision. However, cetane number (as determined by an engine test using ASTM D 613 procedures) has a substantial error band, as shown in Table 3. The data scatter in such imprecise benchmark methods was carried into the FT-IR correlations, as reflected by the R^2 data of between 0.7 and 0.8 and by the SEP(CV) values, ranging up to about 2 cetane number units. For the range of values of our data sets, the repeatability and reproducibility

figures for the determination of the heat of combustion, ASTM D 240 specifies 56 and 172 Btu/lb. The SEP(CV) values of the FT-IR data were found to be between 16 and 26 Btu/lb. Such apparent reversal in the expected values obviously does not imply improved accuracy of the derived data over that of the calibration data. However, it indicates enhanced precision in the FT-IR data due possibly to reduction of the subjective elements during FT-IR operations.

Two methods were used to evaluate correlations between the benchmark and the new method. A simple way of assessing the agreement between the standard and new measuring techniques considers the residual errors (arithmetic differences) between the benchmark and new methods. Another procedure uses the limits of agreement.^{18, 19} This method is recommended for cases where the benchmark methods may not give highly accurate true values. Since a number of procedures used in petroleum laboratories are empirical methods, or they rely on imprecise engine tests, this criterion is generally applicable. To generate the limits of agreement between the old (accepted) and new (FT-IR) methods, the residual error is plotted against the average value of the two methods, and the standard deviation is evaluated. The correction procedure proposed by Faber and Kowalsky²⁰, which may yield a lower, more realistic estimate of the true prediction error, will be the subject of a future report.

In the validation tables, data are presented from the benchmark measurements, the derived FT-IR values, and their residual errors. For each data set, the minimum, maximum and average of these values, as well as the sample standard deviation for the residual data, are given.

A. Calibration Data Based on Spectra from Instruments A and B for Fuel Sets A, B, and C

Summaries of the calibration data, based on spectra obtained on Instrument A for Fuel Sets A, B, and C, are shown in Tables 12-14. These data were evaluated by examination of the SEP (CV) and R^2 data pairs. Two density data are shown, as calibrated to both the original manual method (ASTM D 1298), and automatic (ASTM D 4052) method. While both calibrations provided excellent results, data based upon ASTM D 4052 provided slightly improved correlations. Acceptable-to-excellent correlations were also established for kinematic viscosity, boiling point at 50%, cetane number, cetane indexes, carbon and hydrogen contents, carbon-to-hydrogen ratio, net heat of combustion, and monocyclic-, dicyclic-, polycyclic- and total aromatic hydrocarbon concentrations. FT-IR provides intermediate grade correlations with boiling points at 10, 90 and 95%, and final boiling point, usable only for non-critical applications. While the SEP(CV) values for cloud-, freeze-, and pour points were lower than the ASTM defined reproducibility values, the corresponding low R^2 data discourage their use in critical applications. Large SEP(CV) and low R^2 data for flash point, initial boiling point and lubricity values, as determined by the high frequency reciprocating rig, HFRR (ISO/CF-12156 and the to-be-published ASTM D 6079), indicate that FT-IR may not give acceptable values. Data for water (ASTM D 1744), gum (ASTM D 381) and sulfur (ASTM D 4294) contents, and lubricity by the BOCLE (ASTM D 5001) and its modification (the U.S. Army scuffing load methods) gave such poor correlations during preliminary analyses that further calibrations to these properties were abandoned.

Summaries of the calibration data, based on spectra obtained on Instrument B for Fuel Sets A, B, and C, are shown in Tables 15-17. Conclusions are essentially identical to those reached for Instrument A.

The marginally better correlation derived from Instrument B is probably due to the instrument's higher signal-to-noise ratio, S/N. The S/N values were 5,660 vs. 9,650 in the wavenumber range of 2,150-2,100 cm^{-1} for Instruments A and B, respectively. The higher S/N ratio of Instrument B appear to be due to the lower mirror velocity.

B. Validations for Fuel Sets A, B, and C

Separate calibration models were obtained for each fuel property under consideration. The factors in these calibration models were selected at the first PRESS values at which the F-statistic probability (p) dropped to or below 0.75. Using selected batches of these calibration models, calculations were made to obtain a number of fuel property values for each fuel of the validation set that comprised an independent group of analyzed fuels.

Validation summaries, using spectra obtained on Instrument A, are summarized in Tables 18-20, while those for Instrument B are given in Tables 21-23. Each of these summary tables show the minimum, maximum, and average of the individual fuel property values for all fuels of the validation sets as measured by the appropriate ASTM procedures, as calculated from the FT-IR spectra and the residual error between these two corresponding values. Additionally, the sample standard deviations for the column of the individual residual errors are also included.

Comparison of the SEP(CV) and R^2 values for the three fuel sets in Tables 12-17, and in Tables 18-23 indicate similar error and scatter data, encouraging the evaluation of the effects of combining the three seasonal fuel sets into one unified training set.

C. Calibrations on Spectra from Instrument A for Combination Fuel Set D

Fuel Set D comprised 547 samples. After preliminary calibrations, fuel property value outliers (using factors at $p \leq 0.75$) were excluded and separate training sets were constructed for each fuel property. Results of these calibrations, summarized in Table 24, indicate that the combined set of fuels gave similar or marginally better SEP(CV) and R^2 values in comparison with the separate fuel sets.

Comparison of the benchmark ASTM values and the FT-IR derived values within this training set are shown in Figures 1-27. Such graphs are presented for the following fuel properties: API gravity and density based on ASTM D 1298 and D 4052; kinematic viscosity at 40°C in cSt; flash-, cloud-, freeze-, and pour points; initial boiling point; boiling points at 10-, 50-, 90-, and 95%; final boiling point; cetane number determined according to ASTM D 613; cetane indexes according to ASTM D 976 and 4737; hydrogen and carbon contents; carbon-to-hydrogen ratio; net heat of combustion in units of MJ/kg and in Btu/lb; aromatic hydrocarbons in monocyclic-, dicyclic-, and polycyclic structures; and total aromatic hydrocarbons.

The best correlations were demonstrated for density, kinematic viscosity, boiling point at 50% evaporated volume, both cetane indexes, heat of combustion, and aromatic hydrocarbon components. Cetane number correlations between ASTM D 613 and the FT-IR derived data seem to be as good as the precision of our benchmark data allowed.

The FT-IR derived data on flash-, cloud-, freeze-, and pour points provided lower quality correlations that are not suitable for critical applications. Correlations to flash point and initial boiling point were shown to be poor. Correlations with sulfur and water contents, and lubricity data according to the BOCLE, scuffing load, and HFRR methods, gave unacceptable results as indicated by the SEP(CV) and R^2 values, as illustrated in Figures 28-29 for the sulfur and HFRR data pairs.

The poor quality of correlations with flash point may be due to detectability limitations at the low concentrations of the most volatile hydrocarbons that define flash point. The problem with the determination of initial boiling point, and boiling point at 10%, may be due to imprecise observation of these data points, occurring where the concentration vs. boiling-point curve changes rapidly. The fact that many sulfur bonds produce weak infrared bands, coupled with low sulfur concentrations in the test fuels, yield imprecise results. The most probable reason for the lack of acceptable correlations between lubricity data and FT-IR may also be linked to detectability limits, as the various (known and unknown) polar components that are responsible for the fuels' lubricity are only present at low parts per million concentrations.

Fuel Set D included about 25 turbine fuels, having cloud and freeze points at or below -30°C and pour points at or below -50°C . When these fuels were excluded from the cloud-, freeze-, and pour point data sets, thus constructing training sets comprising only diesel fuels, the SEP(CV) values improved. Corresponding increases in R^2 values were not observed due to a change from a two-cluster to a single-cluster data set. When outliers of these modified training sets were removed, further data improvements were observed. These data are also given in Table 24. Graphical presentation of the ASTM vs. FT-IR data from these series of calculations is shown in Figures 30-32.

While it is unfortunate that the common calibration model for both turbine and diesel fuels gives less accurate measurement of these cold temperature related properties, it is still encouraging that within the diesel fuel range, acceptable values may be obtained from FT-IR spectroscopy. Conceivably, a substantially increased number of turbine fuels in the training set would yield improved correlations between the ASTM and FT-IR derived data pairs, as suggested in References 6 and 7.

D. Calibrations on Spectra from Instrument B for Combination Fuel Set D

In analogous experiments to those performed on spectra obtained on Instrument A, calibrations were also carried out on spectra generated on Instrument B. The results of these calibrations are summarized in Table 25. As in Table 24, cloud-, freeze-, and pour point data were also included for the exclusively diesel fuel set, after the turbine fuels were excluded from the training set. Calibrations restricted to diesel fuels for the other fuel properties have not yet been performed.

Comparison of the data in Tables 24 and 25 reveal that while all data are similar, the SEP(CV) and R^2 values are more favorable when they were derived from spectra obtained on Instrument B. To provide a more complete picture, some of the poorer quality data are also presented, *e.g.*, those for flash point, initial boiling point, carbon- and sulfur contents, and the lubricity data according to the HFRR measurements.

Comparison of data measured by the ASTM methods, with the FT-IR derived data within this training set, is graphically illustrated in Figures 33-64. Essentially identical conclusions may be drawn from experiments based on spectra from either Instrument A or Instrument B.

E. Validations on Fuel Set D

Validation of the calibration models, based on spectra from Instruments A and B, for each selected fuel property was performed using 137 fuel samples. These samples were not part of the calibration training set.

As noted before, for each validation sample, the considered data are the benchmark (ASTM) results, the derived FT-IR values and their residual errors. The validation data for property values derived from spectra from Instruments A and B are summarized in Tables 26 and 27, respectively. In these tables, from each of the data sets we show only the minimum, maximum and average values, and the sample standard deviation for the residual errors.

A summary of the validations of the calibration models restricted to diesel fuels for cloud-, freeze-, and pour points is given in Table 28. It is demonstrated that excluding the turbine fuels from the calibration training sets and the validation set of samples, the FT-IR data gave better agreement with the ASTM data. When the outliers were excluded from the training set comprising exclusively diesel fuels, further improvement was noted in the agreement between the two methods of measurements as shown in the respective SEP(CV) values in Tables 24 and 25.

F. Evaluation of Validation Error

The validation samples were analyzed by the same reference methods that were used for the analysis of the calibration samples. Therefore, statistically the same percent of the sample set may contain erroneous or outlier data. While we excluded several outliers from the calibration fuel set, no outlier detection method was used on the validation samples. Therefore, no outliers were excluded from this set.

1. Residual error of the validation experiments are shown in Tables 26-28. These tables show the minimum, the maximum, and the average deviations between the two sets of measurements. However, they do not reflect the frequency of larger deviations from the mean. More detailed evaluation of the data is available by examination of barcharts showing sample-to-sample residual errors between the ASTM and FT-IR data. Since these barcharts are essentially identical as derived from either of the two FT-IR instruments, only those barcharts based on spectra from

Instrument B are presented in Figures 65-92. Residual errors for the fuel set restricted to diesel fuels are illustrated in Figures 93-95.

Residual error bands at two standard deviations (2σ) were determined for the FT-IR determination of each of the considered fuel properties. These data, based on spectra obtained on Instrument B using Fuel Set D, are summarized in Table 29.

2. Limits of agreement plots are shown in Figures 96-126, indicating the limits corresponding to 1 and 2 standard deviations. Table 29 summarizes the validation errors in the determination of the various fuel properties in Fuel Set D, using Instrument B. All error data shown refer to 2 standard deviations (2σ) for the residual errors and for the limits of agreement. Additionally, the number of samples outside the 2σ limits from the 137 validation samples are also included.

Cursory evaluation of the validation error bands may be done by comparing them to the precision statements of the benchmark ASTM tests summarized in Table 2. If the "1 in 20" general ASTM outlier rule is applied for the FT-IR data, six to seven outliers may be "allowed." Note, however, that several of the stated 2σ limits of agreement are outside the range of acceptable precision, as summarized in Table 29. Accordingly, at the present state of development, the following fuel property values may be confidently measured by FT-IR:

gravity, API°	density, g/ml	kinematic viscosity, cSt, 40°C
boiling point, 50%	cetane index	carbon, wt%
hydrogen, wt%	C/H	heat of combustion, MJ/kg
monocyclic aromatics, wt%	dicyclic aromatics, wt%	polycyclic aromatics, wt%
total aromatics, wt%		

Validations of the calibration model for cetane number (based on data from the engine test specified in ASTM D 613) gave limits of agreement at 2σ of 4.7, which is outside the reproducibility range of the specified 2.5-3.3 cetane numbers. It is expected that agreement between these two methods may be substantially improved through more precise calibration data.

VIII. COMPARISON OF FT-IR AND NEAR-IR

A comparison was made of several calibration models developed from near-IR and FT-IR spectra to either demonstrate essential equivalency of the results provided by the two methods or show superiority of one method over the other. For this work, models of a restricted set of fuel properties were developed using identical calibration and validation fuel sets. The selected fuel properties included those that gave superior, average, and relatively poor correlations using the FT-IR frequency region. The selected eight fuel properties were:

density, as correlated to data from ASTM D 4052;
kinematic viscosity, cSt, at 40°C, as correlated to data from ASTM D 445;
cetane number, as correlated to data from ASTM D 613;

calculated cetane index, as correlated to data from ASTM D 976;
calculated cetane index, as correlated to data from ASTM D 4737;
carbon-to-hydrogen ratio;
net heat of combustion, as correlated to data from ASTM D 240;
total aromatic hydrocarbon content, as correlated to data from ASTM D 5186.

The FT-IR spectra were taken on Instrument B (PE/S2000), while the near-IR spectra were obtained on a Brimrose AOTF spectrometer.

Calibration models were made according to the established two-step procedure. Fuel set D was used for these calibration experiments. In the preliminary calibrations all eight fuel properties were included in a common training set to identify the outliers. The outlier fuels were excluded from calibrations to the individual fuel properties. The number of fuels in the preliminary training set was 547, while the training sets used for the individual fuel properties consisted of between 529 and 538 fuels. Calibration preprocessing of the FT-IR calibrations consisted of mean centering the spectra. Preprocessing of the near-IR set comprised taking first difference of the mean centered spectra, to comply with unpublished recommended results of a related large study conducted at the TARDEC Fuels and Lubricants Research Facility, located at Southwest Research Institute.

Results of the individual calibrations from both the near-IR and FT-IR experiments are summarized in Table 30, using data corresponding to factors selected at $p \leq 0.75$. From the up to 20 PRESS factors allowed during the calibrations in both spectral sets, FT-IR required a higher number of factors than near-IR, both at minimum PRESS values and at $p \leq 0.75$. Both SEP(CV) and R^2 values were found to be more favorable when FT-IR was used.

Near-IR and FT-IR spectra were collected on each of the 137 fuel samples that comprised the validation set. Using the appropriate calibration models, fuel properties were calculated from the corresponding spectra. The summary of the FT-IR and the near-IR results are shown in Tables 31 (an abstract of Table 27) and 32, respectively. While validation results gave essentially identical statistics for the determination of the heat of combustion values, for each of the other seven properties, FT-IR gave more precise results.

IX. MATLAB CALIBRATIONS

For comparison purposes, a series of models were constructed in MATLAB™ using PLS_Toolbox™. The incentive for generating calibrations in MATLAB was driven by its reported flexibility, its advanced options, *i.e.*, a wide selection of calibration techniques and calibration transfer, and the ability to extract the final regression coefficients for each model and incorporate them into a custom application.

Models constructed for this study were the same as those selected for the comparison of FT-IR and near-IR studies: density (D 4052); kinematic viscosity, cSt, at 40°C (D 445); cetane number (D 613); two cetane indexes (D 976 and D 4737); carbon-to-hydrogen ratio (D 5291); net heat of combustion

(D 240); and total aromatic hydrocarbons (D 5196). As described above, the models were built with spectra from both Instrument A and B, Fuel Set D, using (a) mean-centered, (b) mean-centered first difference, and (c) mean-centered second difference preprocessing. For Instrument A, the number of data points was reduced to 869 from 1738 by using every other wavelength, and Instrument B was reduced to 838 from 3351 by using every fourth wavelength. PLS regressions were performed on each property for each type of preprocessing. Ideally, similar results should be obtained from each software package with minor changes resulting from custom modifications made by each developer. The two packages differ in the type of statistics that are presented to the user, the way in which outliers are detected, and the way by which factors are selected. Therefore, the samples identified as outliers in each package could also vary. For these reasons, direct comparison of the results offered by the two computer programs is limited.

Discussions with the developers of PLS_Toolbox™ resulted in some changes to the calibration parameters. A leave-one-out cross validation was found to be rather time consuming, requiring approximately 2 hours of computer time for each property when calculating 20 factors. The suggestion was made by the developers of PLS_Toolbox™ that the number of times that the data is split should be equal to the square root of the number of samples up to a maximum of ten; thus, for a 500 sample calibration set, the number of splits would be ten, which is equivalent to a leave-50-out cross validation. Because a large number of samples are being removed during each rotation, there is the possibility that a small group of unique fuels are being estimated that have no complimentary samples in the calibration set. Therefore, the results might appear worse than those obtained from a leave-one-out cross validation. This could affect the final calibration only if the unique fuels are removed as outliers, reducing the robustness of the calibration.

To aid in the selection of the appropriate number of factors for the model, PLS_Toolbox™ provides the user with a PRESS plot and a table showing the percent variance captured by the model. The table reveals the X- and Y-block contributions of each factor to the model, based on the amount of variation that they captured. The X-block variables are the predictor variables (*i.e.* absorbance at various wavelengths), while the Y-block variables are the predicted variables (*i.e.* fuel property values).¹⁵ The number of factors were chosen as the highest numbered factor whose percent variance was greater than or equal to 1.0 for the Y-block variables while still being less than or equal to the factor which gave the global minimum in the PRESS plot. Otherwise, the factor which corresponded to the minimum in the PRESS plot was selected. The reasoning here is that the Y-block variables have at least 1% variation due to error in the benchmark methods. For example, Table 33 shows percent variance captured for density. Based on the table, seven factors, accounting for 91.32% of the total variation in the Y-block, were chosen. Because factors eight through ten contribute substantially less to the model, adding these additional factors could cause the model to become overfit. The following discussion and plots will continue to use this seven-factor density model as an example.

Once the number of factors for the model are selected, the user is presented with plots of Predicted vs. Actual (Figure 127) and Leverage vs. Studentized Residuals (Figure 128), which are useful for outlier detection. A large studentized residual (>3) indicates that a sample could have an incorrect property value. On the other hand, a high leverage indicates that a sample has a large influence on

the model, perhaps because it is unique in some way or the spectrum was run incorrectly. Two other plots that are useful for outlier detection are that of Q vs. Sample Number (Figure 129) and T^2 vs. Sample Number (Figure 130). Q measures the amount of variation a sample has outside of the factors defined for the model. Stated otherwise, it measures the amount of non-deterministic variation left over after the deterministic variation has been removed.¹⁵ T^2 measures the distance of the sample from the multivariate mean (*i.e.* within the model). The plots clearly indicate those samples that have unusual values of Q and T^2 , thereby identifying them as possible outliers.

Summary statistics of the final calibration sets for Instruments A and B are shown in Tables 34 and 35, respectively. For each property and type of preprocessing, the tables show the minimum and maximum for the calibration and outlier sets and the number of outliers removed during each calibration. These statistics are useful to identify the range that each calibration covers and where the outlier set has samples that lie at either extreme of the fuel property range. Caution is warranted in handling samples with extreme property values. Samples that lie at the extremes of a calibration set are often (and incorrectly) identified as outliers and removed from the set. Samples with extreme property values are easily identifiable as they will often appear as high leverage samples in the Leverage vs Studentized Residuals plot. Tables 33-36 contain the validation statistics for both instruments A and B.

A. Calibrations for Instrument A, Fuel Set D

The SEP(CV) and SEC for the Instrument A calibrations are presented in Table 37. Also shown are the number of factors used to build each calibration model. Notice that there is very little difference among the various calibrations when run with different types of preprocessing. For first-difference/mean-centered preprocessing only two properties, cetane number and kinematic viscosity, show an improvement over mean-centered preprocessing as judged by the SEP(CV). For second-difference preprocessing, only cetane index (D 4737) shows an improvement over the other two types of preprocessing. However, because the improvements over mean-centered spectra are small, further modification of the spectra with 1st and 2nd differences may be unnecessary (in agreement with results obtained using the Galactic PLSplus program). The SEP(CV) for the remainder of the properties indicate that mean-centered spectra are best. The validation phase of the experiment will help to further clarify the best parameters for property estimation using Instrument A.

B. Calibrations for Instrument B, Fuel Set D

The SEP(CV) and SEC for Instrument B are presented in Table 38. Calibration statistics for Instrument B indicate that cetane number and kinematic viscosity are best estimated when using a 2nd difference preprocessing as opposed to 1st difference preprocessing in Instrument A. Aromatics appear to be slightly better with first difference preprocessing, while density and C/H ratio are best when using only mean-centered spectra. Cetane indexes (D 976 and D 4737) and heat of combustion gave identical results for all three types of preprocessing. Again, the use of validation statistics, namely SEP and R^2 , should give us a clear indication of the best data pretreatment.

C. Validations for Instrument A and B, Fuel Set D

In order to determine the ability of the new models to estimate the properties of unknown fuel samples, validation experiments were carried out using the validation set of fuels. From the results of the validations, the SEP and R^2 were calculated for Instrument A (Table 39) and Instrument B (Table 40). For each instrument, the SEP and R^2 pairs for each property are bolded indicating the best type of preprocessing for that particular property. For both Instruments A and B, a high degree of correlation was found between the SEP and R^2 values. Therefore, any decisions made concerning the best preprocessing method can be made with confidence.

Unequivocally, the results for density, C/H ratio, and heat of combustion were best when using mean-centered spectra from either instrument. First difference spectra gave the best results for aromatics. The remainder of the properties showed significant variation between Instruments A and B. In several cases, the SEP was identical for two or more types of preprocessing, so the final decision was made based on the pretreatment that gave the highest R^2 value.

Currently, we are not using any statistical methods for determining outliers in the validation set while running under PLS_Toolbox. Therefore, no samples have been removed. However, future investigations will attempt to utilize some outlier detection routines in the validation phase of the experiments. This should improve the overall results of the validation statistics (SEP and R^2), but will have little or no effect on the calibration itself. Findings from this study indicate that while PLSplus is more user friendly and much simpler to operate, PLS_Toolbox is more powerful because of its flexibility and more advanced options (i.e. calibration transfer and wide selection of calibration techniques).

X. SUMMARY AND CONCLUSIONS

As an alternative to established laboratory protocols, an FT-IR method was developed for the rapid, simultaneous determination of several pertinent fuel properties, using less than 2 mL of sample. The method uses a single wavelength range of baseline uncorrected, raw FT-IR spectra. Neither baseline correction nor use of restricted wavelength regions improved the models.

Using training sets composed of over 500 middle distillate fuel samples, models were built to derive pertinent fuel properties from FT-IR spectra. These models showed that a single calibration training set may properly model several properties of fuels collected during different climatic seasons. Cursory experiments suggest that properties of diesel and turbine fuels may be more accurately derived by FT-IR if separate calibration models are used for each class of fuel, i.e., for diesel fuel, and turbine fuel. However, separate calibration models may prove to be unnecessary to model these fuel classes if the number of analyzed turbine fuel would be substantially increased in the training set.

At the present state of development, the following fuel property values may be confidently measured by FT-IR: gravity, API°; density, g/ml; kinematic viscosity, cSt, at 40°C; boiling point at 50%; cetane index; carbon, wt%; hydrogen, wt%; carbon-to-hydrogen ratio, C/H; heat of combustion, MJ/kg;

monocyclic aromatics, wt%; dicyclic aromatics, wt%; polycyclic aromatics, wt%; and total aromatics, wt%. The combined calibration and validation summary data are presented in Table 41, also reproduced here.

Calibration and Validation Summary (Fuel Set D; Instrument B)

Property	Calibration						Validation
	F @ PRESS (min)			F @ p≤0.75			SEP at
	F	SEP (CV)	R ²	F	SEP (CV)	R ²	F@p≤0.75
API gravity (D1298)	20	0.16	0.9959	18	0.16	0.9957	0.21
density (D1298)	18	0.0008	0.9963	18	0.0008	0.9963	0.0009
API gravity (D4052)	20	0.12	0.9977	20	0.12	0.9977	0.18
density (D4052)	20	0.0005	0.9985	19	0.0005	0.9984	0.0006
kin. visc. cSt, 40°C	20	0.066	0.9782	19	0.067	0.9773	0.09
flash point, °C	20	4.3	0.6078	17	4.4	0.5886	6.0
cloud point, °C	20	2.8	0.8693	17	2.8	0.8612	4.5
cloud point, °C (1)	20	1.9	0.7810	16	2.0	0.7690	7.1
cloud point, °C (2)	19	1.7	0.8187	16	1.7	0.8092	7.3
freeze pt, °C	20	3.0	0.8724	16	3.1	0.8659	4.5
freeze pt, °C (1)	20	2.0	0.7678	14	2.1	0.7518	6.8
freeze pt, °C (2)	20	1.7	0.8081	15	1.8	0.7960	7.2
pour pt, °C	20	4.4	0.8216	14	4.5	0.8116	5.1
pour pt, °C (1)	20	4.0	0.6883	14	4.1	0.6692	6.7
pour pt, °C (2)	20	3.7	0.7263	17	3.8	0.7143	7.1
initial bp, °C	19	6.3	0.6760	14	6.5	0.6555	9.8
bp-10, °C	20	4.7	0.8896	15	4.8	0.8852	6.4
bp-50, °C	20	2.4	0.9794	16	2.5	0.9782	4.7
bp-90, °C	20	5.8	0.8710	18	5.9	0.8661	8.0
bp-95, °C	20	7.5	0.8093	18	7.7	0.7989	9.8
final bp, °C	20	7.9	0.7767	16	8.1	0.7639	10.6
cetane number (D613)	18	1.54	0.7920	12	1.58	0.7811	2.4
cetane index (D976)	20	0.62	0.9498	14	0.64	0.9469	1.21
cetane index (D4737)	20	0.60	0.9579	14	0.62	0.9556	1.35
hydrogen, wt%	9	0.075	0.9404	5	0.077	0.9376	0.10
carbon, wt%	5	0.223	0.6637	2	0.228	0.6495	0.26
C/H	9	0.036	0.9603	5	0.037	0.9581	0.0494
heat of comb., MJ/kg	15	0.046	0.9288	7	0.047	0.9248	0.06
heat of comb., Btu/lb	17	19.85	0.9286	7	20.35	0.9249	27
monocyclic ArH, wt%	20	0.336	0.9947	20	0.336	0.9947	0.42
dicyclic ArH, wt%	20	0.243	0.9878	18	0.249	0.9872	0.046
polycyclic ArH, wt%	20	0.201	0.8378	14	0.206	0.8302	0.28
total ArH, wt%	20	0.377	0.9963	18	0.387	0.9961	0.50

Please Note:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

property (1): excluded jet fuel samples from calibrations under property name

property (2): excluded outliers identified under calibrations in property(1)

ArH = aromatic hydrocarbons

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

In comparing the calibration results obtained from each of the two software packages, some notable differences are found. First, for all properties the number of factors used in building the calibration models are significantly greater when run under PLSplus than under PLS_Toolbox. Because the type of statistics presented to the user vary between the two applications, the selection of factors will also vary. Second, because PLSplus is incorporating more factors into its calibration equations, the calibration statistics will normally indicate that it provides better models. As a result, in most cases the SEP values were slightly lower for the models developed by PLSplus than by PLS_Toolbox (Tables 27 and 40, respectively). However, the differences are small enough that calibration models built using either system may be suitable. At the current time, the main advantage that PLS_Toolbox has over PLSplus is its ability to do calibration transfer, which is part of current investigations.

The manufacturers of Instrument B and several other instruments have already incorporated PLSplus into their own software. This makes PLSplus more attractive as a quick solution for integration of instrument control and calibration models. If more sophisticated applications are required, integrating PLS_Toolbox calibrations into higher level programming languages, such as Visual Basic, is a proven technique.

In regard to instrument performance and calibration, it is not our desire to specify the superiority of one instrument manufacturer or software developer over another. The subjective nature of the chemometric methods would make any such statements unjustifiable. Rather, we used Instruments A and B to develop a foundation for calibration transfer, which will be reported in the near future.

For comparison, several calibration models were developed from near-IR and FT-IR spectra. It was demonstrated that, as expected, FT-IR provides better models for the determination of fuel property values.

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TABLE 1. Analytical Procedures to Establish Calibration Data

Fuel Property	Procedure
density, g/mL	ASTM D 1298
density, g/mL	ASTM D 4052
kinematic viscosity, cSt, at 40°C	ASTM D 445
cloud point, °C	ASTM D 2500
freeze point, °C	ASTM D 2386
pour point, °C	ASTM D 97
flash point, °C	ASTM D 93
distillation, °C	ASTM D 86
cetane number	ASTM D 613
calculated cetane index	ASTM D 976
calculated cetane index	ASTM D 4737
carbon, wt%	ASTM D 5291
hydrogen, wt%	ASTM D 5291
carbon-to-hydrogen ratio	ASTM D 5291
heat of combustion, net, MJ/kg	ASTM D 240
heat of combustion, net, Btu/lb	ASTM D 240
monocyclic aromatic hydrocarbons, wt%	ASTM D 5186
dicyclic aromatic hydrocarbons, wt%	ASTM D 5186
polycyclic aromatic hydrocarbons, wt%	ASTM D 5186
total aromatic hydrocarbons, wt%	ASTM D 5186
sulfur, wt%	ASTM D 4294
lubricity by HFRR, mm	ISO/CD 12156 *

*equivalent method was accepted under ASTM D 6079; to be published

TABLE 2. Repeatability and Reproducibility of Standard Procedures

Property	ASTM D	Repeatability	Reproducibility
density, g/mL	1298	0.0005	0.0012
density, g/mL	4052	0.0001	0.0005
kin. visc., cSt, 40°C	445	0.14 @ 3	0.44 @ 3
cloud point, °C	2500	2.0	4.0
freeze point, °C	2386	1.0	2.5
pour point, °C	97	3.0	6.0
flash point, °C	93	2.0	3.5
initial bp, °C	86	3.5	8.5
bp-10, °C	86	1.5-4.5	4.0-9.5
bp-50, °C	86	1.5-4.5	4.0-9.5
bp-90, °C	86	2.0-5.0	4.5-12.0
bp-95, °C	86	2.0-5.0	4.5-12.0
final bp, °C	86	3.5	10.5
cetane number	613	0.6-0.9	2.5-3.3
calc'd cetane index	976	calculated	calculated
calc'd cetane index	4737	calculated	calculated
carbon, wt%	5291	C: 0.98 @ 87%	C: 2.44 @ 87%
hydrogen, wt%	5291	H: 0.42 @ 13%	H: 0.83 @ 13%
C/H	5291	calculated	calculated
heat of comb., net, MJ/kg	240	0.13	0.40
H, Btu/lb	240	55.9	172
1-ArH, wt%	5186	0.7	4.0
2-ArH, wt%	5186	0.7	4.0
poly-ArH, wt%	5186	0.7	4.0
total ArH, wt%	5186	0.7	4.0
sulfur, wt%	4294	0.02	0.04
HFRR, mm	ISO/CD 12156*	0.11 @ 25°C	0.17 @ 25°C

*equivalent method was accepted under ASTM D 6079; to be published.

TABLE 3. General Statistics for Property Values of Fuel Sets

<u>Property</u>	<u>ASTM D</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Average</u>	<u>Range</u>
Fuel Set A (December):					
density, g/mL	1298	0.7880	0.8703	0.8438	0.0823
density, g/mL	4052	0.7871	0.8716	0.8447	0.0845
cloud point, °C	2500	-60.4	-5.6	-16.8	54.8
freeze point, °C	2386	-59.4	-2.0	-13.4	57.4
pour point, °C	97	-75.0	-12.0	-29.9	63.0
kin. visc., cSt, 40°C	445	1.15	3.55	2.51	2.40
initial bp, °C	86	139	214	176	75
bp at 10%, °C	86	162	247	213	84
bp at 50%, °C	86	194	297	258	104
bp at 90%, °C	86	223	337	312	114
bp at 95%, °C	86	231	358	327	126
final bp, °C	86	241	371	341	130
cetane number	613	36.9	61.3	48.5	24.4
calc'd cetane index	976	37.3	57.0	47.0	19.6
calc'd cetane index	4737	37.4	59.6	47.1	22.2
hydrogen, wt%	5291	12.29	14.33	13.10	2.04
carbon to hydrogen ratio	5291	5.9512	7.0968	6.6039	1.1457
heat of comb, net, MJ/kg	240	42.30	43.46	42.69	1.16
heat of comb, net, Btu/lb	240	18185	18684	18354	499
monocyclic aromatics, wt%	5186	9.0	36.2	23.9	27.2
dicyclic aromatics, wt%	5186	0.6	12.8	5.5	12.2
polycyclic aromatics, wt%	5186	0.1	3.4	1.3	3.3
total aromatics, wt%	5186	10.7	44.2	30.7	33.5
Fuel Set B (March):					
density, g/mL	1298	0.7982	0.8687	0.8441	0.0705
density, g/mL	4052	0.7993	0.8692	0.8445	0.0699
cloud point, °C	2500	-60.5	-0.3	-17.1	60.2
freeze point, °C	2386	-54.7	2.4	-13.6	57.1
pour point, °C	97	-78.0	-6.0	-31.9	72.0
kin. visc., cSt, 40°C	445	1.14	4.05	2.48	2.91
initial bp, °C	86	132	210	175	78
bp at 10%, °C	86	158	256	212	98
bp at 50%, °C	86	182	297	257	115
bp at 90%, °C	86	224	340	311	116
bp at 95%, °C	86	230	359	326	129
final bp, °C	86	240	376	340	135
cetane number	613	40.3	60.0	49.0	19.7
calc'd cetane index	976	37.0	59.8	46.6	22.8
calc'd cetane index	4737	39.9	65.7	46.7	25.8
hydrogen, wt%	5291	12.49	14.00	13.10	1.51
carbon to hydrogen ratio	5291	6.1279	6.9664	6.6173	0.8385
heat of comb, net, MJ/kg	240	42.35	43.22	42.68	0.87
heat of comb, net, Btu/lb	240	18205	18579	18350	374
monocyclic aromatics, wt%	5186	11.10	38.9	24.54	27.8
dicyclic aromatics, wt%	5186	0.4	11.6	5.9	11.2
polycyclic aromatics, wt%	5186	0.0	2.3	0.9	2.3
total aromatics, wt%	5186	13.0	47.2	31.2	34.2

TABLE 3. General Statistics for Property Values of Fuel Sets

<u>Property</u>	<u>ASTM D</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Average</u>	<u>Range</u>
Fuel Set C (July):					
density, g/mL	1298	0.7955	0.8719	0.8461	0.0764
density, g/mL	4052	0.7958	0.8728	0.8467	0.077
cloud point, °C	2500	-60.5	2.1	-16.0	62.6
freeze point, °C	2386	-54.7	6.6	-12.3	61.3
pour point, °C	97	-78.0	-3.0	-28.1	75.0
kin. visc., cSt, 40°C	445	1.14	4.05	2.58	2.91
initial bp, °C	86	99	229	176	129
bp at 10%, °C	86	158	256	215	98
bp at 50%, °C	86	182	297	261	115
bp at 90%, °C	86	224	347	314	123
bp at 95%, °C	86	230	375	328	145
final bp, °C	86	240	388	342	148
cetane number	613	40.3	60.0	49.4	19.7
calc'd cetane index	976	37.0	59.8	46.8	22.8
calc'd cetane index	4737	39.9	65.7	46.8	25.8
hydrogen, wt%	5291	12.36	14.30	13.06	1.94
carbon to hydrogen ratio	5291	6.0028	7.0744	6.6423	1.0716
heat of comb, net, MJ/kg	240	42.25	43.41	42.67	1.16
heat of comb, net, Btu/lb	240	18166	18664	18344	498
monocyclic aromatics, wt%	5186	7.7	38.9	24.5	31.2
dicyclic aromatics, wt%	5186	0.4	11.9	6.2	11.5
polycyclic aromatics, wt%	5186	0.0	2.8	1.0	2.8
total aromatics, wt%	5186	8.3	45.8	31.5	37.5
Fuel Set D (Combined fuel sets):					
density, g/mL	1298	0.7880	0.8719	0.8455	0.0839
density, g/mL	4052	0.7871	0.8728	0.8461	0.0857
cloud point, °C	2500	-60.5	2.1	-16.3	62.6
freeze point, °C	2386	-59.4	6.6	-12.7	66.0
pour point, °C	97	-78	-3	-29	75.0
kin. visc., cSt, 40°C	445	1.14	4.05	2.55	2.91
initial bp, °C	86	99	229	176	129
bp at 10%, °C	86	158	256	214	98
bp at 50%, °C	86	182	297	260	115
bp at 90%, °C	86	223	347	313	124
bp at 95%, °C	86	230	375	328	145
final bp, °C	86	240	388	342	148
cetane number	613	36.9	61.3	49.0	24.4
calc'd cetane index	976	37.0	59.8	46.8	22.8
calc'd cetane index	4737	37.4	65.7	46.8	28.3
hydrogen, wt%	5291	12.29	14.33	13.07	2.04
carbon to hydrogen ratio	5291	5.9512	7.0968	6.6331	1.1457
heat of comb, net, MJ/kg	240	42.25	43.46	42.67	1.21
heat of comb, net, Btu/lb	240	18166	18684	18346	518
monocyclic aromatics, wt%	5186	7.7	38.9	24.4	31.2
dicyclic aromatics, wt%	5186	0.4	12.8	6.0	12.4
polycyclic aromatics, wt%	5186	0.0	3.4	1.1	3.4
total aromatics, wt%	5186	8.3	47.2	31.4	38.9

TABLE 4. Number of Fuels Used in Calibration and Validation Experiments

<u>Fuel Set</u>	<u>total no. of fuels</u>	<u>no. fuels in precalibrations</u>	<u>no. fuels in calibrations</u>	<u>no. fuels in validations</u>
A	208	168	159 - 167	40
B	232	186	179 - 185	46
C	245	195	188 - 193	50
D	682	547	528 - 543	137

**TABLE 5. Effects of Preprocessing on Calibration Models
Instrument A**

Mean Centered Spectra

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	540	20	0.0006	0.9974	19	0.0007	0.9973
kvisc. cSt, 40C	536	20	0.080	0.9652	19	0.080	0.9640
cetane no. (D613)	533	16	1.61	0.7732	12	1.64	0.7641
CCI-976	538	18	0.64	0.9469	14	0.65	0.9448
CCI-4737	538	20	0.62	0.9559	15	0.63	0.9539
C/H	539	7	0.0380	0.9548	5	0.0390	0.9532
H, MJ/kg	536	15	0.046	0.9310	8	0.047	0.9273
TArH, wt%	539	20	0.41	0.9958	18	0.41	0.9956

Mean Centered First Difference

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	536	20	0.0006	0.9977	19	0.0006	0.9977
kvisc. cSt, 40C	537	20	0.097	0.9521	14	0.100	0.9495
cetane no. (D613)	532	13	1.63	0.7646	10	1.67	0.7515
CCI-976	536	14	0.67	0.9419	11	0.68	0.9396
CCI-4737	536	18	0.62	0.9559	13	0.63	0.9535
C/H	539	9	0.0396	0.9512	6	0.0404	0.9493
H, MJ/kg	537	12	0.046	0.9289	7	0.047	0.9251
TArH, wt%	540	19	0.44	0.9951	16	0.45	0.9949

Mean Centered Second Difference

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	544	20	0.0010	0.9941	19	0.0010	0.9938
kvisc. cSt, 40C	534	15	0.106	0.9409	13	0.109	0.9383
cetane no. (D613)	535	15	1.68	0.7576	11	1.72	0.7426
CCI-976	537	17	0.70	0.9351	13	0.71	0.9337
CCI-4737	539	17	0.69	0.9483	13	0.71	0.9457
C/H	538	11	0.0415	0.9482	8	0.0419	0.9470
H, MJ/kg	536	11	0.047	0.9253	9	0.048	0.9227
TArH, wt%	542	20	0.57	0.9919	18	0.59	0.9914

**TABLE 6. Effects of Preprocessing on Validations
Instrument A**

Mean Centered Spectra

summary	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	11.60	12.21	-1.81	5.9512	6.0027	-0.1395	0.7958	0.7984	-0.0026	42.25	42.29	-0.28
Maximum	42.90	43.12	0.97	7.0744	7.0368	0.2773	0.8668	0.8670	0.0018	43.46	43.35	0.10
Average	30.25	30.30	-0.04	6.5875	6.5903	-0.0028	0.8416	0.8416	-0.0000	42.71	42.72	-0.01
St. Deviation	---	---	0.47	---	---	0.0531	---	---	0.0007	---	---	0.06

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	1.14	0.90	-0.22	40.1	41.2	-13.9	37.01	38.62	-5.13	39.05	39.45	-2.59
Maximum	3.92	3.63	0.39	61.3	62.9	5.6	59.05	59.68	7.58	64.86	61.40	9.85
Average	2.42	2.42	0.00	49.1	49.3	-0.2	47.28	47.22	0.06	47.48	47.31	0.17
St. Deviation	---	---	0.10	---	---	2.40	---	---	1.22	---	---	1.31

Mean Centered First Difference

summary	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	11.60	11.95	-2.05	5.9512	5.9711	-0.1483	0.7958	0.7975	-0.0017	42.25	42.27	-0.29
Maximum	42.90	43.07	1.71	7.0744	7.0636	0.2884	0.8668	0.8674	0.0028	43.46	43.38	0.10
Average	30.25	30.25	0.00	6.5875	6.5899	-0.0024	0.8416	0.8416	-0.0000	42.71	42.72	-0.01
St. Deviation	---	---	0.45	---	---	0.0512	---	---	0.0007	---	---	0.06

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	1.14	0.82	-0.29	40.1	43.3	-10.9	37.01	39.67	-5.59	39.05	40.57	-2.66
Maximum	3.92	3.68	0.36	61.3	59.9	6.4	59.05	60.21	7.39	64.86	62.09	9.66
Average	2.42	2.43	-0.01	49.1	49.2	-0.1	47.28	47.24	0.04	47.48	47.39	0.09
St. Deviation	---	---	0.11	---	---	2.35	---	---	1.24	---	---	1.27

Mean Centered Second Difference

summary	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	11.60	11.84	-1.97	5.9512	5.9267	-0.1187	0.7958	0.7949	-0.0028	42.25	42.25	-0.31
Maximum	42.90	43.69	1.26	7.0744	7.0856	0.2835	0.8668	0.8679	0.0037	43.46	43.38	0.14
Average	30.25	30.28	-0.02	6.5875	6.5894	-0.0019	0.8416	0.8417	-0.0002	42.71	42.72	-0.01
St. Deviation	---	---	0.59	---	---	0.0534	---	---	0.0010	---	---	0.07

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	1.14	0.78	-0.29	40.1	43.1	-14.7	37.01	39.93	-6.32	39.05	40.35	-3.54
Maximum	3.92	3.64	0.56	61.3	63.7	5.9	59.05	60.41	6.47	64.86	62.34	9.12
Average	2.42	2.42	0.00	49.1	49.4	-0.3	47.28	47.25	0.03	47.48	47.43	0.06
St. Deviation	---	---	0.13	---	---	2.49	---	---	1.26	---	---	1.24

Notes:

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR

**TABLE 7. Effects of Preprocessing on Calibration Models
Instrument B**

Mean Centered Spectra

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	534	20	0.0005	0.9985	19	0.0005	0.9984
kvisc. cSt, 40C	529	20	0.066	0.9782	19	0.067	0.9773
cetane no. (D613)	533	18	1.54	0.7920	12	1.58	0.7811
CCI-976	536	20	0.62	0.9498	14	0.64	0.9469
CCI-4737	538	20	0.60	0.9579	14	0.62	0.9556
C/H	537	9	0.0360	0.9603	5	0.0370	0.9581
H, MJ/kg	538	15	0.046	0.9288	7	0.047	0.9248
TArH, wt%	536	20	0.38	0.9963	18	0.39	0.9961

Mean Centered First Difference

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	542	20	0.0004	0.9987	20	0.0004	0.9987
kvisc. cSt, 40C	534	20	0.068	0.9766	19	0.068	0.9762
cetane no. (D613)	534	11	1.58	0.7807	9	1.61	0.7705
CCI-976	539	15	0.62	0.9494	12	0.64	0.9465
CCI-4737	538	19	0.60	0.9598	15	0.61	0.9580
C/H	535	9	0.0367	0.9583	6	0.0373	0.9569
H, MJ/kg	538	9	0.047	0.9269	6	0.048	0.9240
TArH, wt%	539	20	0.38	0.9963	18	0.39	0.9962

Mean Centered Second Difference

Property	No. of Samples	F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	537	20	0.0006	0.9979	20	0.0006	0.9979
kvisc. cSt, 40C	535	20	0.077	0.9710	17	0.078	0.9697
cetane no. (D613)	531	13	1.59	0.7781	10	1.62	0.7680
CCI-976	535	17	0.63	0.9461	13	0.64	0.9447
CCI-4737	536	20	0.61	0.9581	17	0.62	0.9560
C/H	539	16	0.0383	0.9552	9	0.0390	0.9536
H, MJ/kg	537	14	0.047	0.9247	8	0.048	0.9212
TArH, wt%	536	20	0.40	0.9960	19	0.40	0.9959

**TABLE 8. Effects of Preprocessing on Validations
Instrument B**

Mean Centered Spectra

summary	Total ArH, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	11.60	12.11	-2.94	5.9512	5.9966	-0.1299	0.7958	0.7992	-0.0034	42.25	42.25	-0.31
maximum	42.90	42.79	1.09	7.0744	7.0670	0.2684	0.8668	0.8670	0.0013	43.46	43.35	0.11
average	30.25	30.28	-0.03	6.5875	6.5905	-0.0030	0.8416	0.8416	-0.0001	42.71	42.72	-0.01
sample std. deviation	----	----	0.50	----	----	0.0495	----	----	0.0006	----	----	0.06

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.78	-0.19	40.1	41.6	-13.5	37.01	38.32	-5.97	39.05	38.62	-3.11
maximum	3.92	3.66	0.36	61.3	62.5	5.5	59.05	58.88	7.71	64.86	60.56	10.04
average	2.42	2.41	0.01	49.1	49.3	-0.3	47.28	47.29	-0.02	47.48	47.35	0.13
sample std. deviation	----	----	0.09	----	----	2.35	----	----	1.21	----	----	1.35

Mean Centered First Difference

summary	Total ArH, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	11.60	12.43	-2.05	5.9512	5.9703	-0.1400	0.7958	0.7982	-0.0025	42.25	42.25	-0.31
maximum	42.90	42.97	1.17	7.0744	7.0598	0.2790	0.8668	0.8671	0.0023	43.46	43.36	0.10
average	30.25	30.25	0.01	6.5875	6.5883	-0.0008	0.8416	0.8416	-0.0001	42.71	42.72	-0.01
sample std. deviation	----	----	0.45	----	----	0.0502	----	----	0.0005	----	----	0.06

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.77	-0.22	40.1	43.2	-11.1	37.01	40.06	-5.50	39.05	40.35	-3.54
maximum	3.92	3.60	0.37	61.3	60.1	5.5	59.05	58.32	7.35	64.86	62.34	9.12
average	2.42	2.41	0.01	49.1	49.3	-0.2	47.28	47.27	0.01	47.48	47.43	0.06
sample std. deviation	----	----	0.09	----	----	2.31	----	----	1.24	----	----	1.24

Mean Centered Second Difference

summary	Total ArH, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	11.60	12.08	-2.10	5.9512	5.9597	-0.1280	0.7958	0.7972	-0.0040	42.25	42.27	-0.41
maximum	42.90	42.97	2.60	7.0744	7.0440	0.2637	0.8668	0.8678	0.0026	43.46	43.37	0.11
average	30.25	30.23	0.03	6.5875	6.5871	0.0004	0.8416	0.8416	-0.0001	42.71	42.72	-0.01
sample std. deviation	----	----	0.52	----	----	0.0522	----	----	0.0008	----	----	0.07

summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.88	-0.24	40.1	41.7	-9.7	37.01	39.1	-5.61	39.05	39.40	-2.31
maximum	3.92	3.72	0.35	61.3	60.3	5.3	59.05	58.9	6.97	64.86	62.36	9.85
average	2.42	2.42	0.00	49.1	49.3	-0.2	47.28	47.3	0.01	47.48	47.40	0.09
sample std. deviation	----	----	0.09	----	----	2.33	----	----	1.17	----	----	1.20

**TABLE 9. Effect of Rotation Sample Number on Calibration
Instrument A**

A. Mean Centered Spectra

Property	No. of Samples	Rotation =5					
		F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	540	20	0.0006	0.9974	19	0.0007	0.9973
kvisc. cSt, 40C	536	20	0.080	0.9652	19	0.080	0.9640
cetane no. (D613)	533	16	1.61	0.7732	12	1.64	0.7641
CCI-976	538	18	0.64	0.9469	14	0.65	0.9448
CCI-4737	538	20	0.62	0.9559	15	0.63	0.9539
C/H	539	7	0.0380	0.9548	5	0.0390	0.9532
H, MJ/kg	536	15	0.046	0.9310	8	0.047	0.9273
TArH, wt%	539	20	0.41	0.9958	18	0.41	0.9956

Property	No. of Samples	Rotation =50					
		F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	544	20	0.0009	0.9955	18	0.0009	0.9952
kvisc. cSt, 40C	534	20	0.103	0.9457	19	0.105	0.9439
cetane no. (D613)	535	16	1.79	0.7265	12	1.83	0.7102
CCI-976	537	16	0.66	0.9433	14	0.67	0.9409
CCI-4737	539	19	0.70	0.9473	16	0.71	0.9457
C/H	538	6	0.0399	0.9520	5	0.0404	0.9507
H, MJ/kg	536	8	0.049	0.9198	6	0.050	0.9172
TArH, wt%	542	20	0.45	0.9951	17	0.46	0.9948

B. Mean Centered Second Difference Spectra

Property	No. of Samples	Rotation =5					
		F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	544	20	0.0010	0.9941	19	0.0010	0.9938
kvisc. cSt, 40C	534	15	0.106	0.9409	13	0.109	0.9383
cetane no. (D613)	535	15	1.68	0.7576	11	1.72	0.7426
CCI-976	537	17	0.70	0.9351	13	0.71	0.9337
CCI-4737	539	17	0.69	0.9483	13	0.71	0.9457
C/H	538	11	0.0415	0.9482	8	0.0419	0.9470
H, MJ/kg	536	11	0.047	0.9253	9	0.048	0.9227
TArH, wt%	542	20	0.57	0.9919	18	0.59	0.9914

Property	No. of Samples	Rotation =50					
		F @ PRESS (min)			F @ p<0.75		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
d-4052	544	20	0.0012	0.9917	17	0.0119	0.9913
kvisc. cSt, 40C	534	19	0.119	0.9268	15	0.122	0.9230
cetane no. (D613)	535	16	1.79	0.7265	12	1.83	0.7102
CCI-976	537	13	0.73	0.9303	12	0.74	0.9284
CCI-4737	539	14	0.74	0.9404	12	0.75	0.9388
C/H	538	10	0.043	0.9443	7	0.044	0.9417
H, MJ/kg	536	12	0.048	0.9233	9	0.049	0.9192
TArH, wt%	542	20	0.64	0.9899	18	0.65	0.9895

**TABLE 10. Effect of Rotation Sample Number on Validation
Mean Centered Spectra from Instrument A**

Rotation = 5

summary	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	11.60	12.21	-1.81	5.9512	6.0027	-0.1395	0.7958	0.7984	-0.0026	42.25	42.29	-0.28
maximum	42.90	43.12	0.97	7.0744	7.0368	0.2773	0.8668	0.8670	0.0018	43.46	43.35	0.10
average	30.25	30.30	-0.04	6.5875	6.5903	-0.0028	0.8416	0.8416	-0.0000	42.71	42.72	-0.01
sample std. deviation	----	----	0.47	----	----	0.0531	----	----	0.0007	----	----	0.06
summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.90	-0.22	40.1	41.2	-13.9	37.01	38.62	-5.13	39.05	39.45	-2.59
maximum	3.92	3.63	0.39	61.3	62.9	5.6	59.05	59.68	7.58	64.86	61.40	9.85
average	2.42	2.42	0.00	49.1	49.3	-0.2	47.28	47.22	0.06	47.48	47.31	0.17
sample std. deviation	----	----	0.10	----	----	2.40	----	----	1.22	----	----	1.31

Rotation = 50

summary	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	11.60	12.15	-1.87	5.9512	5.9985	-0.1291	0.7958	0.7982	-0.0024	42.25	42.28	-0.30
maximum	42.90	43.26	1.00	7.0744	7.0378	0.2795	0.8668	0.8666	0.0017	43.46	43.32	0.14
average	30.25	30.28	-0.02	6.5875	6.5885	-0.0011	0.8416	0.8416	-0.0000	42.71	42.72	-0.01
sample std. deviation	----	----	0.43	----	----	0.0525	----	----	0.0007	----	----	0.06
summary	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.80	-0.21	40.1	41.9	-13.5	37.01	38.90	-5.24	39.05	39.36	-2.57
maximum	3.92	3.64	0.43	61.3	62.5	5.8	59.05	59.54	7.51	64.86	61.60	9.76
average	2.42	2.41	0.01	49.1	49.4	-0.3	47.28	47.21	0.07	47.48	47.34	0.15
sample std. deviation	----	----	0.11	----	----	2.36	----	----	1.24	----	----	1.30

Notes:

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR = residual error

**TABLE 11. Effect of Rotation Sample Number on Validation
Mean Centered Second Difference Spectra from Instrument A**

Rotation = 5

Statistics	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	11.60	11.84	-1.97	5.9512	5.9267	-0.1187	0.7958	0.7949	-0.0028	42.25	42.25	-0.31
Maximum	42.90	43.69	1.26	7.0744	7.0856	0.2835	0.8668	0.8679	0.0037	43.46	43.38	0.14
Average	30.25	30.28	-0.02	6.5875	6.5894	-0.0019	0.8416	0.8417	-0.0002	42.71	42.72	-0.01
St. Deviation	----	----	0.59	----	----	0.0534	----	----	0.0010	----	----	0.07
Kin. viscosity, cSt, at 40C												
Statistics	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	1.14	0.78	-0.29	40.1	43.1	-14.7	37.01	39.93	-6.32	39.05	40.35	-3.54
Maximum	3.92	3.64	0.56	61.3	63.7	5.9	59.05	60.41	6.47	64.86	62.34	9.12
Average	2.42	2.42	0.00	49.1	49.4	-0.3	47.28	47.25	0.03	47.48	47.43	0.06
St. Deviation	----	----	0.13	----	----	2.49	----	----	1.26	----	----	1.24

Rotation = 50

Statistics	Total Aromatics, wt%			Carbon / Hydrogen			Density (D 4052)			Heat of Comb., MJ/kg		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	11.60	11.84	-1.97	5.9512	5.9496	-0.1229	0.7958	0.7956	-0.0032	42.25	42.25	-0.31
Maximum	42.90	43.69	1.26	7.0744	7.0857	0.2679	0.8668	0.8681	0.0037	43.46	43.38	0.14
Average	30.25	30.28	-0.02	6.5875	6.5893	-0.0018	0.8416	0.8418	-0.0002	42.71	42.72	-0.01
St. Deviation	----	----	0.59	----	----	0.0526	----	----	0.0010	----	----	0.07
Kin. viscosity, cSt, at 40C												
Statistics	Kin. viscosity, cSt, at 40C			Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
Minimum	1.14	0.73	-0.31	40.1	42.6	-14.1	37.01	39.73	-6.49	39.05	40.36	-3.85
Maximum	3.92	3.62	0.45	61.3	63.1	5.9	59.05	61.34	6.89	64.86	62.88	9.15
Average	2.42	2.43	-0.00	49.1	49.3	-0.2	47.28	47.24	0.04	47.48	47.42	0.06
St. Deviation	----	----	0.13	----	----	2.50	----	----	1.33	----	----	1.30

Notes:
ASTM = ASTM values
FTIR = FTIR values
A - F = ASTM - FTIR = residual error

TABLE 12. Calibration Summary: Fuel Set A; Instrument A

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	165	11	0.0011	0.9932	9	0.0011	0.9926
density (D4052)	165	14	0.0007	0.9969	12	0.0008	0.9966
kvisc. cSt, 40°C	165	15	0.099	0.9537	13	0.102	0.9505
flash point, °C	164	10	5.1	0.4484	2	5.2	0.4072
cloud point, °C	162	14	2.9	0.8759	13	2.9	0.8744
pour point, °C	163	12	4.8	0.7722	3	5.0	0.7509
freeze point, °C	162	14	2.9	0.8802	13	2.9	0.8770
initial bp, °C	163	12	7.0	0.6051	10	7.2	0.5786
bp-10, °C	166	12	5.6	0.8530	9	5.9	0.8361
bp-50, °C	164	14	3.2	0.9666	10	3.3	0.9641
bp-90, °C	164	14	7.0	0.8504	13	7.1	0.8471
bp-95, °C	164	15	10.0	0.7170	10	10.4	0.6854
final bp, °C	162	15	8.8	0.7624	13	9.2	0.7391
cetane number (D613)	165	11	1.56	0.7418	8	1.64	0.7147
cetane index (D976)	164	15	0.76	0.9154	13	0.80	0.9059
cetane index (D4737)	164	15	0.70	0.9306	11	0.73	0.9235
hydrogen, wt%	165	6	0.107	0.8730	3	0.112	0.8601
C/H	163	7	0.048	0.9269	4	0.048	0.9249
heat of comb., MJ/kg	164	14	0.059	0.8499	10	0.061	0.8338
heat of comb., Btu/lb	164	14	25.25	0.8490	11	25.75	0.8413
monocyclic ArH, wt%	165	15	0.37	0.9926	14	0.38	0.9921
dicyclic ArH, wt%	165	13	0.22	0.9878	11	0.23	0.9870
polycyclic ArH, wt%	164	7	0.28	0.6217	5	0.29	0.5904
total ArH, wt%	164	13	0.49	0.9927	11	0.50	0.9924
HFRR, mm	166	3	0.07	0.6188	2	0.07	0.5867

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 13. Calibration Summary: Fuel Set B; Instrument A

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	181	14	0.0010	0.9942	11	0.0010	0.9938
density (D4052)	183	16	0.0006	0.9976	14	0.0007	0.9975
kin. visc. cSt, 40°C	179	18	0.09	0.9613	16	0.10	0.9576
cloud point, °C	180	16	3.2	0.8819	14	3.2	0.8751
pour point, °C	181	14	5.5	0.7975	11	5.8	0.7766
freeze point, °C	180	18	2.7	0.9094	16	2.8	0.9041
initial bp, °C	181	9	7.3	0.6482	7	7.5	0.6246
bp-10, °C	181	13	4.8	0.9033	10	5.1	0.8932
bp-50, °C	182	16	2.7	0.9795	13	2.8	0.9779
bp-90, °C	181	16	6.5	0.8976	11	6.7	0.8916
bp-95, °C	180	18	7.8	0.8408	14	8.1	0.8312
final bp, °C	179	18	7.9	0.8219	15	8.3	0.8061
cetane number (D613)	183	10	1.94	0.7661	7	2.01	0.7464
cetane index (D976)	179	17	0.58	0.9578	14	0.61	0.9539
cetane index (D4737)	183	15	0.63	0.9539	12	0.64	0.9519
hydrogen, wt%	183	5	0.065	0.9526	3	0.066	0.9508
C/H	182	7	0.031	0.9655	4	0.033	0.9626
heat of comb., MJ/kg	184	12	0.051	0.9126	6	0.053	0.9051
heat of comb., Btu/lb	184	12	21.8	0.9127	6	22.4	0.9072
monocyclic ArH, wt%	186	20	0.413	0.9928	18	0.427	0.9923
dicyclic ArH, wt%	184	18	0.243	0.9863	13	0.250	0.9855
polycyclic ArH, wt%	182	20	0.113	0.9285	17	0.119	0.9208
total ArH, wt%	183	16	0.408	0.9959	14	0.427	0.9955
HFRR, mm	181	16	0.048	0.6762	12	0.050	0.6472

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 14. Calibration Summary: Fuel Set C; Instrument A

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	193	20	0.0008	0.9957	17	0.0008	0.9955
density (D4052)	193	17	0.0008	0.9952	15	0.0008	0.9948
kin. visc. cSt, 40°C	190	13	0.09	0.9395	10	0.09	0.9364
cloud point, °C	191	13	3.4	0.6786	9	3.6	0.6486
pour point, °C	191	12	4.2	0.6992	9	4.4	0.6680
freeze point, °C	191	15	3.2	0.7456	9	3.4	0.7184
initial bp, °C	187	13	7.0	0.5388	10	7.2	0.5033
bp-10, °C	191	13	5.5	0.8108	7	5.7	0.7903
bp-50, °C	190	17	2.8	0.9576	14	2.9	0.9546
bp-90, °C	186	19	4.7	0.6475	18	4.8	0.6393
bp-95, °C	190	15	8.4	0.6054	9	8.8	0.5545
final bp, °C	190	11	9.7	0.4853	6	10.1	0.4332
cetane number (D613)	188	11	1.49	0.7500	8	1.56	0.7246
cetane index (D976)	190	17	0.57	0.9578	13	0.60	0.9547
cetane index (D4737)	190	13	0.62	0.9611	10	0.64	0.9594
carbon, wt%	188	5	0.184	0.7685	3	0.190	0.7546
hydrogen, wt%	191	6	0.065	0.9599	5	0.066	0.9593
C/H	193	8	0.035	0.9685	5	0.035	0.9677
heat of comb., MJ/kg	190	8	0.039	0.9561	6	0.040	0.9533
heat of comb., Btu/lb	189	8	16.38	0.9577	6	16.99	0.9545
monocyclic ArH, wt%	192	20	0.489	0.9891	16	0.503	0.9885
dicyclic ArH, wt%	192	16	0.284	0.9863	11	0.294	0.9852
polycyclic ArH, wt%	191	12	0.206	0.8354	9	0.214	0.8225
total ArH, wt%	190	15	0.430	0.9954	12	0.451	0.9950
sulfur, wt%	188	5	0.008	0.2767	1	0.008	0.1968
HFRR, mm	185	20	0.093	0.4137	4	0.098	0.2887

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 15. Calibration Summary: Fuel Set A; Instrument B

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	165	15	0.0007	0.9966	14	0.0008	0.9964
density (D4052)	167	20	0.0006	0.9978	16	0.0006	0.9976
kin. visc. cSt, 40°C	166	20	0.088	0.9637	14	0.092	0.9600
cloud point, °C	164	14	3.2	0.8766	13	3.3	0.8681
pour point, °C	165	6	5.0	0.7772	4	5.1	0.7687
freeze point, °C	164	14	3.1	0.8874	14	3.1	0.8874
initial bp, °C	162	14	5.9	0.7290	11	6.2	0.6995
bp-10, °C	167	14	5.5	0.8634	10	5.7	0.8500
bp-50, °C	165	18	3.0	0.9720	13	3.1	0.9694
bp-90, °C	163	20	5.1	0.9098	18	5.3	0.9053
bp-95, °C	164	19	7.1	0.8619	17	7.5	0.8464
final bp, °C	164	19	7.7	0.8272	14	7.8	0.8188
cetane number (D613)	165	12	1.48	0.7699	9	1.53	0.7522
cetane index (D976)	164	19	0.64	0.9405	15	0.67	0.9342
cetane index (D4737)	164	16	0.62	0.9454	14	0.64	0.9419
carbon, wt%	159	6	0.28	0.6107	3	0.29	0.5673
hydrogen, wt%	166	4	0.102	0.8834	3	0.102	0.8811
C/H	164	4	0.044	0.9380	3	0.044	0.9365
heat of comb., MJ/kg	164	11	0.054	0.8714	3	0.057	0.8574
heat of comb, Btu/lb	165	13	25.09	0.8505	8	26.36	0.8332
monocyclic ArH, wt%	167	20	0.277	0.9955	19	0.288	0.9952
dicyclic ArH, wt%	167	19	0.184	0.9914	13	0.193	0.9905
polycyclic ArH, wt%	164	12	0.271	0.6547	5	0.282	0.6226
total ArH, wt%	163	15	0.410	0.9948	13	0.423	0.9944
HFRR, mm	163	13	0.061	0.7062	11	0.064	0.6758

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 16. Calibration Summary: Fuel Set B; Instrument B

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	182	13	0.0008	0.9959	10	0.0008	0.9956
density (D4052)	183	14	0.0005	0.9986	12	0.0005	0.9985
kin. visc. cSt, 40°C	180	20	0.066	0.9816	19	0.067	0.9807
cloud point, °C	182	18	2.8	0.9010	17	2.8	0.8957
pour point, °C	183	12	5.8	0.7709	6	6.1	0.7474
freeze point, °C	179	18	2.6	0.9051	17	2.7	0.9015
initial bp, °C	183	8	7.5	0.6472	7	7.6	0.6362
bp-10, °C	181	12	5.0	0.8963	10	5.2	0.8875
bp-50, °C	181	17	2.2	0.9853	15	2.3	0.9847
bp-90, °C	181	17	5.4	0.9133	16	5.6	0.9078
bp-95, °C	180	17	7.4	0.8552	13	7.7	0.8432
final bp, °C	179	19	7.1	0.8390	15	7.4	0.8210
cetane number (D613)	181	10	1.85	0.7850	8	1.91	0.7695
cetane index (D976)	180	18	0.53	0.9613	15	0.55	0.9582
cetane index (D4737)	182	15	0.57	0.9660	12	0.59	0.9632
hydrogen, wt%	184	5	0.066	0.9498	4	0.066	0.9496
carbon, wt%	182	2	0.163	0.7413	2	0.163	0.7413
C/H	183	7	0.032	0.9637	4	0.032	0.9632
heat of comb., MJ/kg	185	6	0.052	0.9057	5	0.055	0.8970
heat of comb., Btu/lb	184	8	21.80	0.9116	5	22.86	0.9028
monocyclic ArH, wt%	181	20	0.295	0.9961	20	0.295	0.9961
dicyclic ArH, wt%	182	20	0.212	0.9896	18	0.219	0.9889
polycyclic ArH, wt%	181	20	0.105	0.9389	18	0.109	0.9342
total ArH, wt%	181	19	0.411	0.9958	15	0.427	0.9954
HFRR, mm	181	16	0.044	0.7206	12	0.046	0.6906

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 17. Calibration Summary: Fuel Set C; Instrument B

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
density (D1298)	192	18	0.0007	0.9971	15	0.0007	0.9968
density (D4052)	192	18	0.0006	0.9975	16	0.0006	0.9974
kin. visc. cSt, 40°C	186	20	0.059	0.9730	16	0.061	0.9704
flash point, °C	192	9	5.1	0.4706	7	5.2	0.4463
cloud point, °C	190	19	2.8	0.8174	16	2.9	0.8072
pour point, °C	189	20	4.0	0.7680	12	4.2	0.7496
freeze point, °C	188	18	2.8	0.8175	16	2.8	0.8083
initial bp, °C	188	14	6.7	0.5829	10	6.9	0.5525
bp-10, °C	192	20	4.6	0.8720	18	4.8	0.8599
bp-50, °C	191	19	2.6	0.9621	15	2.7	0.9600
bp-90, °C	189	18	6.1	0.7782	16	6.3	0.7622
bp-95, °C	190	19	7.6	0.7288	16	7.9	0.7065
final bp, °C	190	19	8.3	0.6987	16	8.7	0.6687
cetane number (D613)	188	12	1.52	0.7489	8	1.57	0.7262
cetane index (D976)	192	13	0.58	0.9577	12	0.59	0.9554
cetane index (D4737)	190	14	0.62	0.9564	12	0.65	0.9531
hydrogen, wt%	189	9	0.059	0.9677	7	0.060	0.9655
carbon, wt%	189	6	0.174	0.7925	3	0.179	0.7804
C/H	192	9	0.032	0.9724	6	0.033	0.9710
heat of comb., MJ/kg	188	9	0.037	0.9607	6	0.038	0.9581
heat of comb., Btu/lb	188	10	16.01	0.9598	6	16.53	0.9572
monocyclic ArH, wt%	191	20	0.416	0.9923	19	0.428	0.9917
dicyclic ArH, wt%	192	18	0.278	0.9872	13	0.282	0.9868
polycyclic ArH, wt%	192	15	0.195	0.8549	10	0.204	0.8408
total ArH, wt%	190	14	0.434	0.9953	11	0.448	0.9950
S, wt%	191	18	0.008	0.3730	11	0.009	0.3233
HFRR, mm	188	17	0.087	0.5040	15	0.090	0.4678

Notes:

No. of Samples*: excluded outliers identified in preliminary calibrations using 547 samples

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 18. Validation Summary: Fuel Set A, Instrument A

summary	density (D 1298)			density (D 4052)			cloud point, C			pour point, C			freeze point, C			kin. viscosity, cSt, at 40C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	0.7986	0.7983	-0.0033	0.7986	0.7986	-0.0032	-60.4	-47.8	-23.9	-75	-62	-20	-58.7	-46.0	-21.4	1.23	1.04	-0.30
maximum	0.8582	0.8576	0.0015	0.8578	0.8584	0.0023	-6.1	8.9	6.0	-12	-3	10	-3.0	10.5	5.8	3.36	3.33	0.33
average	0.8386	0.8387	-0.0001	0.8393	0.8395	-0.0002	-18.2	-17.3	-0.9	-31	-31	0	-14.9	-14.2	-0.8	2.38	2.38	-0.00
sample std. deviation	-----	-----	0.0009	-----	-----	0.0009	-----	-----	5.4	-----	-----	6	-----	-----	5.2	-----	-----	0.12
summary	initial boiling point, C			boiling point at 10%, C			boiling point at 50%, C			boiling point at 90%, C			boiling point at 95%, C			final boiling point, C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	143	118	-16	165	161	-21	196	192	-17	236	241	-53	246	256	-54	256	280	-71
maximum	204	190	54	236	232	29	284	292	29	331	348	22	347	366	18	357	374	17
average	174	170	4	209	209	-0	255	255	0	309	309	-1	324	325	-1	338	338	-0
sample std. deviation	-----	-----	15	-----	-----	20	-----	-----	23	-----	-----	24	-----	-----	24	-----	-----	15
summary	Cetane number (D 613)			Cetane Index (D 976)			Cetane Index (D 4737)			Heat of Comb., MJ/kg			Heat of Comb., BTU/lb			HFRR, mm		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	40.1	40.4	-5.7	37.45	37.28	-3.77	39.05	38.73	-2.49	42.38	42.42	-0.15	18219	18250	-69	0.165	0.126	-0.108
maximum	61.3	59.8	6.3	56.96	58.59	6.80	59.60	59.96	9.16	43.46	43.35	0.15	18694	18636	83	0.715	0.637	0.155
average	49.0	49.1	-0.1	47.84	47.63	0.21	48.08	47.85	0.23	42.73	42.73	0.00	18372	18372	-0	0.290	0.297	-0.007
sample std. deviation	-----	-----	2.2	-----	-----	1.74	-----	-----	1.96	-----	-----	0.07	-----	-----	30	-----	-----	0.056
summary	Hydrogen, wt %			Carbon / Hydrogen			Monocyclic aromatics, wt%			Dicyclic aromatics, wt%			Polycyclic aromatics, wt%			Total aromatics, wt%		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	12.65	12.69	-0.25	5.9512	5.9726	-0.2169	9.0	10.1	-2.8	0.9	1.1	-0.8	0.1	0.4	-0.8	11.6	14.5	-2.9
maximum	14.33	14.30	0.30	6.8308	6.9503	0.1125	29.5	29.3	1.3	10.7	10.7	0.9	3.2	2.2	1.3	38.2	37.8	1.5
average	13.21	13.19	0.01	6.5427	6.5576	-0.0149	22.9	22.9	-0.1	5.3	5.2	0.0	1.3	1.3	0.0	29.4	29.6	-0.2
sample std. deviation	-----	-----	0.10	-----	-----	0.0611	-----	-----	0.7	-----	-----	0.3	-----	-----	0.4	-----	-----	0.9

Notes:
 ASTM = ASTM values
 FTIR = FTIR values
 A - F = ASTM - FTIR = residual error

TABLE 19. Validation Summary: Fuel Set B, Instrument A

summary	density (D 1298)		density (D 4052)		cloud point, C		pour point, C		freeze point, C		kin. viscosity, cSt, at 40C	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	0.7982	0.8009	-0.0027	0.7993	0.8000	-0.0012	-54.5	-52.7	-11.2	-72.0	-78.0	-11.8
maximum	0.8665	0.8651	0.0021	0.8663	0.8662	0.0010	-8.9	-0.8	10.4	-18.0	-10.4	13.2
average	0.8394	0.8396	-0.0002	0.8399	0.8399	-0.0000	-18.2	-17.2	-1.1	-33.4	-33.2	-0.2
sample std. deviation	-----	-----	0.0010	-----	-----	0.0005	-----	-----	3.9	-----	-----	5.6
summary	initial boiling point, C		boiling point at 10%, C		boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	146.6	136.9	-17.3	158.4	150.9	-13.0	182.2	187.4	-6.8	228.8	229.8	-19.6
maximum	204.3	182.0	26.2	248.6	237.9	10.7	291.2	289.0	6.6	330.3	342.3	10.4
average	172.9	172.1	0.8	207.8	206.9	0.8	253.0	252.6	-0.3	307.5	308.6	-1.1
sample std. deviation	-----	-----	8.2	-----	-----	5.1	-----	-----	2.5	-----	-----	6.7
summary	cetane number (D 613)		cetane index (D 976)		cetane index (D 4737)		heat of comb., MJ/kg		heat of comb., BTU/lb		HFRR, mm	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	41.4	43.1	-9.5	37.0	42.1	-5.1	40.5	42.5	-2.2	42.39	42.37	-0.26
maximum	60.0	58.5	4.4	53.4	53.6	2.4	54.6	55.6	1.3	43.22	43.16	0.08
average	48.8	49.4	-0.6	47.0	47.0	-0.0	47.2	47.1	0.0	42.72	42.73	-0.01
sample std. deviation	-----	-----	2.5	-----	-----	1.1	-----	-----	0.7	-----	-----	0.06
summary	hydrogen, wt %		carbon / hydrogen		monocyclic aromatics, wt%		dicyclic aromatics, wt%		polycyclic aromatics, wt%		total aromatics, wt%	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	12.55	12.50	-0.48	6.1279	6.1533	-0.1313	11.90	11.79	-0.59	0.60	0.65	-0.64
maximum	14.00	13.91	0.22	6.9084	6.9586	0.2603	32.70	32.91	0.98	11.00	11.21	0.57
average	13.17	13.15	0.02	6.5755	6.5855	-0.0100	23.77	23.73	0.04	5.79	5.80	-0.01
sample std. deviation	-----	-----	0.11	-----	-----	0.0548	-----	-----	0.35	-----	-----	0.25

Notes:

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR= residual error

TABLE 20. Validation Summary: Fuel Set C, Instrument A

summary	density (D 1298)		density (D 4052)		kin. viscosity, cSt, at 40C		cloud point, C		pour point, C		freeze point, C	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	ASTM	FTIR	A - F	A - F
minimum	0.7955	0.7994	-0.0039	0.7958	0.7991	-0.0033	0.7958	-40.0	-69	-59	-51.7	-25.4
maximum	0.8114	0.8112	0.0015	0.8668	0.8660	0.0015	2.1	8.9	-3	-6	5.3	12.9
average	0.7955	0.7994	-0.0002	0.8446	0.8449	-0.0003	-16.3	-14.9	-27	-25	-12.6	-11.2
sample std. deviation	-----	-----	0.0009	-----	-----	0.0009	-----	-----	-----	-----	-----	6.4
summary	initial boiling point, C		boiling point at 10%, C		boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	ASTM	FTIR	A - F	A - F
minimum	99.1	102.7	-65.5	166.1	147.3	-10.8	199.0	203.1	241.6	276.5	256.6	-48.4
maximum	228.5	198.2	42.9	253.4	232.9	20.9	279.4	282.9	349.2	354.1	361.9	30.8
average	176.5	174.6	1.9	214.1	212.6	1.5	258.8	259.5	326.1	329.4	340.3	-1.6
sample std. deviation	-----	-----	13.8	-----	-----	7.2	-----	-----	-----	-----	-----	14.6
summary	cetane number (D 613)		cetane index (D 976)		cetane index (D 4737)		heat of comb., MJ/kg		heat of comb., Btu/lb		lubricity by HFRR, mm	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	ASTM	FTIR	ASTM	A - F
minimum	41.1	44.7	-12.7	39.2	41.8	-3.0	40.7	39.6	18166	18177	0.155	-0.279
maximum	58.2	61.7	3.9	59.1	60.1	2.0	64.9	62.6	18594	18599	0.730	0.250
average	49.3	49.7	-0.5	47.0	47.2	-0.2	42.69	42.69	18351	18354	0.286	-0.016
sample std. deviation	-----	-----	2.6	-----	-----	1.0	-----	-----	-----	-----	-----	0.119
summary	hydrogen, wt %		carbon / hydrogen		monocyclic aromatics, wt%		dicyclic aromatics, wt%		polycyclic aromatics, wt%		total aromatics, wt%	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	ASTM	FTIR	ASTM	A - F
minimum	12.36	12.41	-0.45	6.0636	6.1014	-0.0702	0.80	0.18	0.10	0.14	14.99	-1.76
maximum	14.14	13.97	0.17	7.0744	7.0321	0.2640	11.50	11.93	2.20	2.06	42.86	2.02
average	13.08	13.09	-0.01	6.6329	6.6235	0.0093	6.08	5.99	0.99	0.97	30.81	-0.09
sample std. deviation	-----	-----	0.09	-----	-----	0.0497	-----	-----	-----	-----	-----	0.66

Notes:

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR = residual error

TABLE 21. Validation Summary: Fuel Set A, Instrument B

summary	density (D 1298)		density (D 4052)		cloud point, C		pour point, C		freeze point, C		kin. viscosity, cSt, at 40C	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	0.7986	0.7989	-0.0040	0.7986	0.7988	-0.0029	-60.4	-52.2	-11.7	-75.0	-65.4	-18.8
maximum	0.8582	0.8566	0.0030	0.8578	0.8578	0.0031	-6.1	5.6	7.3	-12.0	-5.4	10.7
average	0.8389	0.8390	-0.0001	0.8397	0.8398	-0.0002	-17.4	-17.4	0.0	-30.1	-31.1	0.9
sample std. deviation	----	----	0.0010	----	----	0.0009	----	----	3.5	----	----	5.2
summary	initial boiling point, C		boiling point at 10%, C		boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	142.8	130.2	-16.6	165.4	153.0	-11.6	196.2	185.0	-20.9	245.7	256.2	-13.5
maximum	204.0	192.8	35.6	236.0	232.2	33.5	283.5	295.9	26.0	346.5	351.7	37.6
average	173.7	171.9	1.7	209.1	209.7	-0.5	255.6	256.1	-0.5	325.6	324.0	1.6
sample std. deviation	----	----	10.3	----	----	7.7	----	----	6.9	----	----	10.3
summary	cetane number (D 613)		cetane Index (D 976)		cetane Index (D 4737)		heat of comb., MJ/kg		heat of comb., BTU/lb		HFRR, mm	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	40.10	40.24	-5.21	37.45	36.83	-3.15	42.38	37.95	-1.94	18219	18249	-87
maximum	61.30	60.23	5.02	56.96	58.80	6.17	59.60	59.74	9.44	18684	18635	49
average	49.12	49.32	-0.20	47.99	47.84	0.14	48.23	47.95	0.28	18371	18375	-5
sample std. deviation	----	----	2.10	----	----	1.54	----	----	1.90	----	----	27.1
summary	hydrogen, wt %		carbon / hydrogen		monocyclic A/H, wt%		dicyclic A/H, wt%		polycyclic A/H, wt%		total A/H, wt%	
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	12.65	12.62	-0.23	5.95	5.95	-0.10	9.00	9.29	-1.41	0.10	0.28	-0.78
maximum	14.33	14.23	0.28	6.83	6.89	0.09	29.50	29.61	1.17	3.20	2.23	1.27
average	13.20	13.18	0.02	6.55	6.56	-0.01	22.88	22.89	-0.01	1.31	1.27	0.04
sample std. deviation	----	----	0.11	----	----	0.05	----	----	0.42	----	----	0.38

Notes:
 ASTM = ASTM values
 FTIR = FTIR values
 A - F = ASTM - FTIR = residual error

TABLE 22. Validation Summary: Fuel Set B, Instrument B

summary	density (D 1298)		density (D 4052)		cloud point, C		pour point, C		freeze point, C		kin. viscosity, cSt, at 40C	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	0.7982	0.7999	-0.0035	0.7993	0.7988	-0.0015	-54.5	-54.5	-17.2	-72.0	-76.2	-12.4
maximum	0.8665	0.8661	0.0015	0.8663	0.8672	0.0009	-8.9	-4.0	12.2	-18.0	-9.8	10.2
average	0.8394	0.8398	-0.0004	0.8399	0.8400	-0.0001	-18.2	-17.4	-0.8	-33.4	-34.2	0.8
sample std. deviation	-----	-----	0.0010	-----	-----	0.0005	-----	-----	4.1	-----	-----	5.2
summary	initial boiling point, C		boiling point at 10%, C		boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	146.6	125.4	-16.7	158.4	154.1	-7.6	182.2	185.5	-3.9	245.5	244.4	-1.8
maximum	204.3	183.8	22.6	248.6	240.5	9.4	291.2	292.8	8.1	345.4	360.0	14.1
average	172.9	172.4	0.5	207.8	207.4	0.3	253.0	252.8	0.2	322.5	324.5	-2.0
sample std. deviation	-----	-----	8.4	-----	-----	4.4	-----	-----	2.7	-----	-----	8.9
summary	cetane number (D 613)		cetane Index (D 976)		cetane Index (D 4737)		heat of comb., MJ/kg		heat of comb., BTU/lb		lubricity by HFRR, mm	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	41.4	42.8	-9.1	37.01	41.96	-4.95	40.54	42.35	-1.87	18224	18218	-85.0
maximum	60.0	58.6	3.8	53.44	53.04	2.73	54.64	55.49	1.43	18579	18558	35.0
average	48.8	49.3	-0.5	46.97	47.05	-0.08	47.16	47.15	0.01	18367	18370	-3.6
sample std. deviation	-----	-----	2.4	-----	-----	1.11	-----	-----	0.62	-----	-----	23
summary	hydrogen, wt %		carbon / hydrogen		monocyclic A:H, wt%		dicyclic A:H, wt%		polycyclic A:H, wt%		total A:H, wt%	
	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F	ASTM	FTIR	A-F
minimum	12.55	12.54	-0.49	6.1279	6.1670	-0.1264	11.90	11.04	-0.75	0.60	0.49	-0.25
maximum	14.00	13.87	0.22	6.9084	6.9346	0.2496	32.70	33.11	1.46	11.00	11.25	0.62
average	13.17	13.16	0.02	6.5755	6.5827	-0.0072	23.77	23.71	0.05	5.79	5.78	0.00
sample std. deviation	-----	-----	0.11	-----	-----	0.0531	-----	-----	0.51	-----	-----	0.26

Notes:

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR = residual error

TABLE 23. Validation Summary: Fuel Set C, Instrument B

summary	density (D 1298)			density (D 4052)			flash point, C			cloud point, C			pour point, C			freeze point, C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	0.7955	0.7980	-0.0026	0.7958	0.7979	-0.0026	23.0	21.0	-37.7	-58.6	-46.4	-31.9	-69.0	-61.5	-25.3	-51.7	-43.3	-26.4
maximum	0.8660	0.8657	0.0011	0.8668	0.8670	0.0006	96.0	73.9	22.1	2.1	5.1	8.1	-3.0	-4.1	6.8	5.3	7.2	8.2
average	0.8441	0.8443	-0.0002	0.8446	0.8450	-0.0003	62.9	61.7	1.1	-16.3	-14.8	-1.5	-27.4	-25.2	-2.2	-12.6	-11.4	-1.2
sample std. deviation	----	----	0.0008	----	----	0.0007	----	----	9.5	----	----	5.7	----	----	6.2	----	----	5.1
summary	initial boiling point, C			boiling point at 10%, C			boiling point at 50%, C			boiling point at 90%, C			boiling point at 95%, C			final boiling point, C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	99	98	-65	166	134	-11	199	200	-9	233	251	-33	242	256	-42	257	271	-53
maximum	229	194	48	253	237	34	279	279	8	332	352	15	349	375	22	362	387	27
average	176	174	2	214	212	2	259	260	-1	312	314	-2	326	329	-3	340	343	-3
sample std. deviation	----	----	14	----	----	8	----	----	3	----	----	9	----	----	12	----	----	13
summary	cetane number (D 613)			cetane index (D 976)			cetane index (D 4737)			heat of comb., MJ/kg			heat of comb., Btu/lb			carbon, wt %		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	41.1	45.9	-12.4	39.2	41.7	-3.9	40.7	40.8	-3.1	42.25	42.25	-0.27	18166	18166	-117	85.71	85.74	-0.53
maximum	58.2	61.4	4.1	59.1	59.0	1.8	64.9	61.3	3.6	43.25	43.20	0.10	18594	18571	41	87.44	87.42	0.47
average	49.3	49.8	-0.5	47.0	47.2	-0.2	47.2	47.1	0.1	42.69	42.69	-0.01	18351	18353	-2	86.68	86.66	0.03
sample std. deviation	----	----	2.5	----	----	1.0	----	----	0.9	----	----	0.06	----	----	28	----	----	0.21
summary	monocyclic aromatics, wt %			dicyclic aromatics, wt %			polycyclic aromatics, wt %			total aromatics, wt %			hydrogen, wt %			carbon / hydrogen		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	9.80	12.18	-2.38	0.80	0.31	-1.44	0.10	-0.00	-0.65	15.20	15.09	-1.94	12.36	12.36	-0.45	6.0636	6.1041	-0.0828
maximum	33.00	33.08	2.70	11.50	11.95	3.87	2.20	2.33	1.19	42.90	43.20	1.70	14.14	14.02	0.13	7.0744	7.0622	0.2660
average	23.80	23.80	0.00	6.08	6.04	0.05	0.99	1.01	-0.02	30.72	30.82	-0.09	13.08	13.09	-0.01	6.6329	6.6259	0.0069
sample std. deviation	----	----	0.60	----	----	0.64	----	----	0.26	----	----	0.62	----	----	0.09	----	----	0.0481
summary	kin. viscosity, cSt, at 40C			sulfur, wt %			scuff load, g			lubricity by HFRR, mm			Notes:					
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM = ASTM values FTIR = FTIR values A - F = ASTM - FTIR = residual error					
minimum	1.23	0.63	-0.13	0.000	0.000	-0.029	1000	2287	-2445	0.155	0.047	-0.308						
maximum	3.42	3.33	0.60	0.080	0.073	0.012	4900	4748	1664	0.730	0.728	0.381						
average	2.52	2.49	0.03	0.031	0.033	-0.002	3508	3257	251	0.286	0.295	-0.009						
sample std. deviation	----	----	0.13	----	----	0.008	----	----	796	----	----	0.139						

Notes:
 ASTM = ASTM values
 FTIR = FTIR values
 A - F = ASTM - FTIR = residual error

TABLE 24. Calibration Summary: Fuel Set D; Instrument A

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
API gravity (D1298)	538	20	0.19	0.9945	18	0.19	0.9943
density (D1298)	537	20	0.0009	0.9953	19	0.0009	0.9952
API gravity (D4052)	539	20	0.15	0.9964	19	0.15	0.9962
density (D4052)	540	20	0.0006	0.9974	19	0.0007	0.9973
kin. visc. cSt, 40°C	536	20	0.08	0.9652	19	0.08	0.9640
flash point, °C	534	11	4.7	0.5368	9	4.8	0.5203
cloud point, °C	533	19	3.1	0.8466	16	3.1	0.8430
cloud point, °C (1)	515	16	2.1	0.7263	13	2.2	0.7110
cloud point, °C (2)	505	14	1.9	0.7685	12	2.0	0.7542
freeze point, °C	530	18	3.0	0.8524	14	3.1	0.8447
freeze point, °C (1)	513	15	2.1	0.7334	13	2.2	0.7223
freeze point, °C (2)	503	13	1.9	0.7709	13	1.9	0.7599
pour point, °C	534	16	4.6	0.8173	12	4.7	0.8083
pour point, °C (1)	512	15	4.4	0.6422	10	4.5	0.6179
pour point, °C (2)	504	15	4.0	0.6577	10	4.2	0.6362
initial bp, °C	527	12	6.7	0.6282	10	6.7	0.6165
bp-10, °C	537	18	5.3	0.8627	13	5.4	0.8567
bp-50, °C	537	20	2.6	0.9747	17	2.7	0.9735
bp-90, °C	536	20	6.2	0.8648	17	6.3	0.8606
bp-95, °C	535	19	7.9	0.8028	16	8.1	0.7932
final bp, °C	535	20	8.3	0.7708	17	8.3	0.7652
cetane number (D613)	533	16	1.61	0.7732	12	1.64	0.7641
cetane index (D976)	538	18	0.64	0.9469	14	0.65	0.9448
cetane index (D4737)	538	20	0.62	0.9559	15	0.63	0.9539
hydrogen, wt%	535	6	0.076	0.9383	5	0.077	0.9372
carbon, wt%	534	7	0.233	0.6303	3	0.239	0.6100
C/H	539	7	0.038	0.9548	5	0.039	0.9532
heat of comb., MJ/kg	536	15	0.046	0.9310	8	0.047	0.9273
heat of comb., Btu/lb	536	16	19.88	0.9290	7	20.45	0.9247
monocyclic ArH, wt%	542	20	0.353	0.9942	18	0.362	0.9939
dicyclic ArH, wt%	535	18	0.233	0.9887	14	0.240	0.9880
polycyclic ArH, wt%	533	18	0.199	0.8389	13	0.203	0.8321
total ArH, wt%	539	20	0.407	0.9958	18	0.414	0.9956
S, wt%	533	20	0.013	0.3004	5	0.014	0.2095
HFRR, mm	528	20	0.072	0.4743	13	0.074	0.4420

Notes:

No. of Samples*: excluded outliers identified in preliminary calibrations using 547 samples

property (1): excluded jet fuel samples from calibrations under property name

property (2): excluded outliers identified under calibrations in property(1)

ArH = acoustic hydrocarbons

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 25. Calibration Summary: Fuel Set D; Instrument B

Property	No. of Samples	F @ PRESS (min)			F @ $p \leq 0.75$		
		F	SEP(CV)	R ²	F	SEP(CV)	R ²
API gravity (D1298)	535	20	0.16	0.9959	18	0.16	0.9957
density (D1298)	538	18	0.0008	0.9963	18	0.0008	0.9963
API gravity (D4052)	536	20	0.12	0.9977	20	0.12	0.9977
density (D4052)	534	20	0.0005	0.9985	19	0.0005	0.9984
kin. visc. cSt, 40°C	529	20	0.066	0.9782	19	0.067	0.9773
flash point, °C	532	20	4.3	0.6078	17	4.4	0.5886
cloud point, °C	532	20	2.8	0.8693	17	2.8	0.8612
cloud point, °C (1)	516	20	1.9	0.7810	16	2.0	0.7690
cloud point, °C (2)	507	19	1.7	0.8187	16	1.7	0.8092
freeze pt, °C	535	20	3.0	0.8724	16	3.1	0.8659
freeze pt, °C (1)	515	20	2.0	0.7678	14	2.1	0.7518
freeze pt, °C (2)	504	20	1.7	0.8081	15	1.8	0.7960
pour pt, °C	533	20	4.4	0.8216	14	4.5	0.8116
pour pt, °C (1)	513	20	4.0	0.6883	14	4.1	0.6692
pour pt, °C (2)	505	20	3.7	0.7263	17	3.8	0.7143
initial bp, °C	531	19	6.3	0.6760	14	6.5	0.6555
bp-10, °C	534	20	4.7	0.8896	15	4.8	0.8852
bp-50, °C	535	20	2.4	0.9794	16	2.5	0.9782
bp-90, °C	536	20	5.8	0.8710	18	5.9	0.8661
bp-95, °C	535	20	7.5	0.8093	18	7.7	0.7989
final bp, °C	534	20	7.9	0.7767	16	8.1	0.7639
cetane number (D613)	533	18	1.54	0.7920	12	1.58	0.7811
cetane index (D976)	536	20	0.62	0.9498	14	0.64	0.9469
cetane index (D4737)	538	20	0.60	0.9579	14	0.62	0.9556
hydrogen, wt%	536	9	0.075	0.9404	5	0.077	0.9376
carbon, wt%	534	5	0.223	0.6637	2	0.228	0.6495
C/H	537	9	0.036	0.9603	5	0.037	0.9581
heat of comb., MJ/kg	538	15	0.046	0.9288	7	0.047	0.9248
heat of comb., Btu/lb	538	17	19.85	0.9286	7	20.35	0.9249
monocyclic ArH, wt%	543	20	0.336	0.9947	20	0.336	0.9947
dicyclic ArH, wt%	536	20	0.243	0.9878	18	0.249	0.9872
polycyclic ArH, wt%	536	20	0.201	0.8378	14	0.206	0.8302
ArH, wt%	536	20	0.377	0.9963	18	0.387	0.9961
S, wt%	536	20	0.012	0.4258	15	0.012	0.3822
HFRR, mm	533	19	0.075	0.4862	14	0.077	0.4518

Notes:

No. of Samples* : excluded outliers identified in preliminary calibrations using 547 samples

property (1): excluded jet fuel samples from calibrations under property name

property (2): excluded outliers identified under calibrations in property(1)

ArH = aromatic hydrocarbons

F = factors = terms in equation to model property

p = F-statistic probability

SEP(CV) = standard error of prediction, cross validated

R² = squared correlation coefficient

TABLE 26. Validation Summary: Fuel Set D, Instrument A

summary	API gravity (D 1298)		API gravity (D 4052)		density (D 1298)		density (D 4052)		kin. viscosity, cSt, at 40C		flash point, C				
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F			
minimum	31.80	31.81	-0.52	31.64	31.59	-0.49	0.7955	0.7981	-0.0040	1.14	0.90	23.0	27.8		
maximum	46.30	45.47	0.85	46.22	45.34	0.88	0.8665	0.8660	0.0022	3.92	3.63	96.0	72.4		
average	36.73	36.70	0.03	36.60	36.59	0.02	0.8410	0.8411	-0.0001	2.42	2.42	61.2	61.1		
sample std. dev.	---	---	0.21	---	---	0.19	---	---	0.0009	---	---	---	---		
SEP	---	---	0.21	---	---	0.19	---	---	0.0007	---	---	---	7.0		
summary	initial boiling point, C		boiling point at 10%, C		boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C				
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F			
minimum	99.1	113.2	-60.1	158.4	146.1	-11.8	182.2	189.0	-11.5	228.8	234.0	-34.6	243.4	-45.3	
maximum	228.5	190.0	41.6	253.4	238.2	33.2	291.2	288.8	30.3	332.1	345.9	23.6	363.4	25.2	
average	174.4	173.5	0.9	210.5	210.0	0.5	256.0	255.8	0.2	310.1	309.8	0.3	324.7	324.2	
sample std. dev.	---	---	10.4	---	---	6.7	---	---	4.4	---	---	7.8	---	9.8	
SEP	---	---	10.4	---	---	6.7	---	---	4.3	---	---	7.7	---	9.8	
summary	cetane number (D 613)		cetane index (D 976)		cetane index (D 4737)		heat of comb., MJ/kg		heat of comb., BTU/lb		carbon / hydrogen				
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F			
minimum	40.1	41.2	-13.9	37.01	38.62	-5.13	39.05	39.45	-2.59	42.25	42.29	-0.28	18166	18183	-122
maximum	61.3	62.9	5.6	59.05	59.68	7.58	64.86	61.40	9.85	43.46	43.35	0.10	18684	18635	49
average	49.1	49.3	-0.2	47.28	47.22	0.06	47.48	47.31	0.17	42.71	42.72	-0.01	18362	18367	-4
sample std. dev.	---	---	2.4	---	---	1.22	---	---	1.31	---	---	0.06	---	---	27
SEP	---	---	2.4	---	---	1.22	---	---	1.32	---	---	0.06	---	---	27
summary	carbon, wt %		hydrogen, wt %		monocyclic aromatics, wt%		dicyclic aromatics, wt%		polycyclic aromatics, wt%		total aromatics, wt%				
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F			
minimum	84.95	85.67	-1.07	12.36	12.38	-0.46	9.00	9.01	-0.95	0.60	0.52	-1.50	0.00	0.04	-0.71
maximum	87.44	87.24	0.49	14.33	14.15	0.35	33.00	33.25	2.35	11.50	12.33	3.85	3.20	2.26	1.02
average	86.53	86.55	-0.02	13.15	13.14	0.01	23.52	23.49	0.03	5.76	5.76	-0.00	1.05	1.04	0.01
sample std. dev.	---	---	0.28	---	---	0.11	---	---	0.38	---	---	0.46	---	---	0.27
SEP	---	---	0.28	---	---	0.11	---	---	0.38	---	---	0.46	---	---	0.27
summary	cloud point, C		cloud point (a), C		freeze point, C		freeze point (a), C		pour point, C		pour point (a), C				
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F			
minimum	-60.4	-51.2	-19.7	-28.9	-26.3	-9.2	-58.7	-49.2	-17.9	-24.8	-23.1	-8.8	-75.0	-75.1	-14.1
maximum	2.1	13.5	12.2	2.1	6.4	8.2	5.3	14.6	15.7	5.3	10.1	8.6	-3.0	5.1	13.4
average	-17.3	-17.2	-0.0	-14.4	-14.3	-0.1	-13.8	-13.7	-0.0	-10.8	-10.8	-0.1	-30.2	-30.3	0.1
sample std. dev.	---	---	4.3	---	---	3.0	---	---	4.6	---	---	3.0	---	---	4.8
SEP	---	---	4.3	---	---	3.0	---	---	4.6	---	---	3.0	---	---	4.8

Notes:
property (a) = excluded jet fuel samples and outliers from calibrations under property name
ASTM = ASTM values
FTIR = FTIR values
A - F = ASTM - FTIR = residual error

sample std. dev. = sample standard deviation
SEP = standard error of prediction

TABLE 27. Validation Summary: Fuel Set D, Instrument B

summary	API Gravity (D 1298)		API Gravity (D 4052)		density (D 1298)		density (D 4052)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	31.80	31.82	0.000	31.64	31.56	-0.28	0.7955	0.7992	-0.0038
maximum	46.30	45.23	1.318	46.22	45.22	1.00	0.8665	0.8659	0.0020
average	36.73	36.69	0.042	36.60	36.58	0.03	0.8410	0.8411	-0.0001
sample std. deviation	---	---	0.139	---	---	0.17	---	---	-0.0009
SEP	---	---	0.206	---	---	0.18	---	---	0.0009
summary	kin. viscosity, cSt, at 40C		flash point, C		initial boiling point, C		boiling point at 10%, C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	1.14	0.78	-0.19	23.0	32.6	-24.0	99.1	113.2	-56.9
maximum	3.92	3.66	0.36	96.0	80.7	18.1	228.5	201.3	32.4
average	2.42	2.41	0.01	61.2	61.4	-0.3	174.4	174.1	0.4
sample std. deviation	---	---	0.09	---	---	6.0	---	---	9.8
SEP	---	---	0.09	---	---	6.0	---	---	9.8
summary	boiling point at 50%, C		boiling point at 90%, C		boiling point at 95%, C		final boiling point, C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	182.2	191.9	-9.9	228.8	238.9	-35.5	241.6	246.9	-42.1
maximum	291.2	290.7	32.4	332.1	339.9	21.8	349.2	357.7	34.8
average	256.0	255.7	0.2	310.1	310.6	-0.5	324.7	325.0	-0.3
sample std. deviation	---	---	4.7	---	---	8.0	---	---	9.8
SEP	---	---	4.7	---	---	8.0	---	---	9.8
summary	cetane number (D 613)		cetane index (D 976)		cetane index (D 4737)		carbon, wt %		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	40.1	41.6	-13.5	37.01	38.32	-5.97	39.05	38.62	-3.11
maximum	61.3	62.5	5.5	59.05	58.88	7.71	64.86	60.56	10.04
average	49.1	49.3	-0.3	47.28	47.29	-0.02	47.48	47.35	0.13
sample std. deviation	---	---	2.3	---	---	1.21	---	---	1.35
SEP	---	---	2.4	---	---	1.21	---	---	1.35
summary	hydrogen, wt %		carbon / hydrogen		heat of comb., MJ/kg		heat of comb., BTU/lb		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	12.36	12.34	-0.48	5.9512	5.9966	-0.1299	42.25	42.25	-0.31
maximum	14.33	14.15	0.33	7.0744	7.0670	0.2684	43.46	43.35	0.11
average	13.15	13.14	0.01	6.5875	6.5905	-0.0030	42.71	42.72	-0.01
sample std. deviation	---	---	0.10	---	---	0.0495	---	---	0.06
SEP	---	---	0.10	---	---	0.0494	---	---	0.06
summary	monocyclic AroH, wt%		dicyclic AroH, wt%		polycyclic AroH, wt%		total AroH, wt%		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	9.00	8.96	-2.62	0.60	0.18	-1.51	0.00	-0.07	-0.63
maximum	33.00	32.95	2.48	11.50	11.74	3.97	3.20	2.25	1.30
average	23.52	23.49	0.03	5.76	5.75	0.01	1.05	1.04	0.01
sample std. deviation	---	---	0.42	---	---	0.46	---	---	0.28
SEP	---	---	0.42	---	---	0.46	---	---	0.28
minimum	11.60	12.11	-2.94	11.60	12.11	-2.94	11.60	12.11	-2.94
maximum	42.90	42.79	1.09	42.90	42.79	1.09	42.90	42.79	1.09
average	30.25	30.28	-0.03	30.25	30.28	-0.03	30.25	30.28	-0.03
sample std. deviation	---	---	0.50	---	---	0.50	---	---	0.50
SEP	---	---	0.50	---	---	0.50	---	---	0.50

Notes:
 A - F = ASTM - FTIR = residual error
 SEP = standard error of prediction
 sample std. deviation = sample standard deviation

**TABLE 28. Validation Summary of Calibrations for Cold Temperature Properties
Fuel Set D, Instrument B**

summary	cloud point, C			cloud point, C (1)			cloud point, C (2)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	-60.4	-56.9	-20.4	-28.9	-24.5	-14.7	-28.9	-24.5	-14.3
maximum	2.1	8.7	13.5	2.1	2.9	5.3	2.1	2.5	6.3
average	-17.3	-16.9	-0.4	-14.4	-14.2	-0.2	-14.4	-14.2	-0.2
sample std. dev.	----	----	4.5	----	----	3.0	----	----	2.9
SEP	----	----	4.5	----	----	3.0	----	----	2.9

summary	freeze point, C			freeze point, C (1)			freeze point, C (2)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	-58.7	-55.6	-18.0	-24.8	-25.2	-8.3	-24.8	-22.4	-10.0
maximum	5.3	11.5	21.1	5.3	5.9	6.1	5.3	6.9	6.0
average	-13.8	-13.6	-0.1	-10.8	-10.8	-0.1	-10.8	-10.7	-0.2
sample std. dev.	----	----	4.5	----	----	2.5	----	----	2.7
SEP	----	----	4.5	----	----	2.5	----	----	2.7

summary	pour point, C			pour point, C (1)			pour point, C (2)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	-75.0	-76.3	-17.9	-48.0	-48.7	-12.9	-48.0	-52.6	-12.1
maximum	-3.0	-1.7	11.8	-3.0	-3.5	9.2	-3.0	1.2	7.3
average	-30.2	-29.9	-0.3	-27.1	-26.8	-0.4	-27.1	-26.9	-0.3
sample std. dev.	----	----	5.1	----	----	4.5	----	----	4.3
SEP	----	----	5.1	----	----	4.5	----	----	4.3

Notes:

property: excluded only outliers identified in preliminary calibrations

property (1): excluded turbine fuels from "property" set

property (2): excluded outliers from "property (1) set

ASTM = ASTM values

FTIR = FTIR values

A - F = ASTM - FTIR = residual error

sample std. dev. = sample standard deviation

SEP = standad error of prediction

**TABLE 29. Errors in Calculation of Fuel Properties from FT-IR Spectra;
Fuel Set D, Instrument B**

Property	Residual error, 2σ	LoA, 2σ	Outside ^a LoA 2σ
API gravity (D1298)	0.40	0.30	6
API gravity (D4052)	0.34	0.24	6
density (D1298)	0.0018	0.0015	5
density (D4052)	0.0012	0.0010	8
kin. viscosity, cSt, 40°C	0.18	0.16	9
flash point, °C	12.0	11.7	6
cloud point, °C	9.0	8.0	11
freeze point, °C	9.0	7.7	9
pour point, °C	10.2	9.9	10
initial bp, °C	19.6	19.6	4
bp-10, °C	12.8	12.7	5
bp-50, °C	9.3	9.3	5
bp-90, °C	16.0	16.0	7
bp-95, °C	19.6	19.6	7
final bp, °C	21.2	21.2	4
cetane number (D613)	4.7	4.7	3
cetane index (D976)	2.42	2.43	3
cetane index (D4737)	2.70	2.70	5
hydrogen, wt%	0.20	0.20	4
carbon, wt%	0.52	0.51	4
carbon-to-hydrogen ratio	0.099	0.099	4
net heat of comb., MJ/kg	0.12	0.126	5
net heat of comb., Btu/lb	53.8	53.8	5
monocyclic ArH, wt%	0.84	0.83	2
dicyclic ArH, wt%	0.92	0.92	3
polycyclic ArH, wt%	0.56	0.56	7
total ArH, wt%	1.00	1.00	3

validation set comprises 137 samples

residual error = ASTM - FTIR values

LoA = limits of agreement

2σ = 2 standard deviations

a = number of samples outside the 2σ limits of agreement

TABLE 30. Comparison of calibrations by near-IR and FT-IR
Fuel Set D; F @ p<0.75

property	FT-IR (Instrument B) *			
	samples ***	F	SEP (CV)	R ²
density (D4052)	534	19	0.0005	0.9984
kinematic viscosity, cSt, 40C	529	19	0.067	0.9773
cetane no. (D613)	533	12	1.58	0.7811
calc'd cetane index (D976)	536	14	0.64	0.9469
calc'd cetane index (D4737)	538	14	0.62	0.9556
C/H	537	5	0.037	0.9581
heat of comb., net, MJ/kg	538	7	0.047	0.9248
total aromatics, wt%	536	18	0.387	0.9961

	near-IR (Unit 7) **			
	samples ***	F	SEP (CV)	R ²
density (D4052)	537	12	0.0015	0.9865
kin. viscosity, cSt, 40C	536	11	0.137	0.9048
cetane no. (D613)	535	5	2.03	0.6353
calc'd cetane index (D976)	537	10	0.80	0.9147
calc'd cetane index (D4737)	539	10	0.80	0.9271
C/H	539	7	0.040	0.9516
heat of comb., net, MJ/kg	539	6	0.051	0.9127
total aromatics, wt%	538	11	0.700	0.9873

Notes:

* FT-IR spectra preprocessed by mean centering

** near-IR spectra preprocessed by taking mean centered first difference

Samples*** : No. of calibration samples (excluded outliers from preliminary calibration set)

F = factors = terms in equation to model property

SEP(CV) = cross validated standard error of prediction

R² = squared correlation coefficient

**TABLE 31. Validation Summary of FT-IR Calibration Models
Fuel Set D; Instrument B**

summary	heat of comb., MJ/kg			cetane number (D 613)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	42.25	42.25	-0.31	40.1	41.6	-13.54
maximum	43.46	43.35	0.11	61.3	62.5	5.50
average	42.71	42.72	-0.01	49.1	49.3	-0.28
sample st. deviation	----	----	0.06	----	----	2.35
SEP	----	----	0.06	----	----	2.35

summary	density (D 4052)			kin. viscosity, cSt, at 40C		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	0.7958	0.7992	-0.0034	1.14	0.78	-0.19
maximum	0.8668	0.8670	0.0013	3.92	3.66	0.36
average	0.8416	0.8416	-0.0001	2.42	2.41	0.01
sample st. deviation	----	----	0.0006	----	----	0.09
SEP	----	----	0.0006	----	----	0.09

summary	cetane index (D 976)			cetane index (D 4737)		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	37.01	38.32	-5.97	39.05	38.62	-3.11
maximum	59.05	58.88	7.71	64.86	60.56	10.04
average	47.28	47.29	-0.02	47.48	47.35	0.13
sample st. deviation	----	----	1.21	----	----	1.35
SEP	----	----	1.21	----	----	1.35

summary	carbon / hydrogen			total aromatics, wt%		
	ASTM	FTIR	A - F	ASTM	FTIR	A - F
minimum	5.9512	5.9966	-0.1299	11.60	12.11	-2.94
maximum	7.0744	7.0670	0.2684	42.90	42.79	1.09
average	6.5875	6.5905	-0.0030	30.25	30.28	-0.03
sample st. deviation	----	----	0.0495	----	----	0.50
SEP	----	----	0.0494	----	----	0.50

Notes:

ASTM = ASTM values

FTIR = calculated FTIR values

NIR = calculated near-IR values

A - F = ASTM - FTIR = residual error

sample st. dev. = sample standard deviation

SEP = standard error of prediction

**TABLE 32. Validation Summary of Near-IR Calibration Models
Fuel Set D; Near-IR Unit No. 7**

summary	heat of comb., MJ/kg			cetane number (D 613)		
	ASTM	NIR	A - NIR	ASTM	NIR	A - NIR
minimum	42.25	42.27	-0.28	40.1	42.7	-17.18
maximum	43.46	43.38	0.12	61.3	66.2	5.58
average	42.71	42.72	-0.01	49.1	49.4	-0.36
sample st. dev.	----	----	0.06	----	----	2.76
SEP	----	----	0.06	----	----	2.77

summary	density (D 4052)			kin. viscosity, cSt, at 40C		
	ASTM	NIR	A - NIR	ASTM	NIR	A - NIR
minimum	0.7958	0.7983	-0.0051	1.14	0.55	-0.32
maximum	0.8668	0.8699	0.0036	3.92	3.64	0.68
average	0.8416	0.8420	-0.0004	2.42	2.44	-0.01
sample st. dev.	----	----	0.0016	----	----	0.16
SEP	----	----	0.0016	----	----	0.16

summary	cetane index (D 976)			cetane index (D 4737)		
	ASTM	NIR	A - NIR	ASTM	NIR	A - NIR
minimum	37.01	37.17	-7.83	39.05	37.10	-4.72
maximum	59.05	62.82	8.01	64.86	64.18	10.28
average	47.28	47.33	-0.05	47.48	47.40	0.08
sample st. dev.	----	----	1.42	----	----	1.42
SEP	----	----	1.41	----	----	1.41

summary	carbon / hydrogen			total aromatics, wt%		
	ASTM	NIR	A - NIR	ASTM	NIR	A - NIR
minimum	5.9512	5.9656	-0.1233	11.60	11.66	-2.34
maximum	7.0744	7.0463	0.2665	42.90	44.24	1.73
average	6.5875	6.5883	-0.0009	30.25	30.33	-0.08
sample st. dev.	----	----	0.0535	----	----	0.80
SEP	----	----	0.0533	----	----	0.80

Notes:

ASTM = ASTM values

FTIR = calculated FTIR values

NIR = calculated near-IR values

A - NIR = ASTM - NIR = residual error, NIR

sample st. dev. = sample standard deviation

SEP = standad error of prediction

**TABLE 33. Percent Variance Captured by PLS
Model for Density (D4052)**

# factors	X-Block		Y-Block	
	This factor	Total	This factor	Total
1	37.43	37.43	41.19	41.19
2	35.42	72.85	20.48	61.67
3	10.76	83.61	10.02	71.69
4	8.27	91.88	3.25	74.94
5	3.59	95.47	6.49	81.42
6	2.04	97.51	7.32	88.74
7	0.21	97.72	2.58	91.32
8	0.48	98.2	0.68	92.01
9	0.26	98.46	0.89	92.89
10	0.13	98.6	0.73	93.63

TABLE 34. Summary Statistics of the Calibration and Outlier Sets for Instrument A

property	Mean-Centered				1st Difference/Mean-Centered				2nd Difference/Mean-Centered				
	Calibration Set		Outliers		Calibration Set		Outliers		Calibration Set		Outliers		
	min	max	min	# outliers	min	max	min	# outliers	min	max	min	# outliers	
density (D4052)	0.7871	0.8728	0.8015	0.8592	9	0.7871	0.8728	8	0.7871	0.8728	0.8015	0.8592	7
cetane number (D613)	39.1	59.9	36.9	59.7	7	39.1	59.9	8	36.9	59.9	40.7	59.7	7
total ArH, wt%	10.7	47.2	8.3	35.4	10	8.3	47.2	11	10.7	47.2	8.3	40.7	13
kin. visc., cSt, 40 C	1.15	3.93	1.3	4.05	9	1.2	3.93	3	1.2	3.93	1.15	4.05	12
C/H	6.0028	7.0968	6.3436	6.7619	8	6.0028	7.0968	3	6.0028	7.0968	6.3169	6.7619	7
cetane index (D976)	37.3	59.8	40.8	57	10	38.4	59.8	11	37.3	57.6	40.8	59.8	13
cetane index (D4737)	37.4	60.1	40.7	65.7	10	39.9	60.1	11	37.4	60.1	40.7	65.7	12
heat of comb., MJ/kg	42.29	43.41	42.35	43.01	17	42.29	43.41	17	42.29	43.21	42.35	43.41	15

TABLE 35: Summary Statistics of the Calibration and Outlier Sets for Instrument B

property	Mean-Centered				1st Difference/Mean-Centered				2nd Difference/Mean-Centered						
	Calibration Set		Outliers		Calibration Set		Outliers		Calibration Set		Outliers				
	min	max	min	max	# outliers	min	max	# outliers	min	max	min	max	# outliers		
density (D4052)	0.8062	0.8728	0.7871	0.8677	14	0.7871	0.8728	6	0.8104	0.8592	0.7871	0.8728	0.8015	0.8592	13
cetane number (D613)	36.9	59.9	40.7	56.7	10	39.1	59.9	12	36.9	59.7	36.9	59.9	40.7	59.7	16
total ArH, wt%	11.8	47.2	8.3	38.6	14	13.7	47.2	14	8.3	35.4	13.7	47.2	8.3	35.4	15
kin. visc., cSt, 40 C	1.15	3.94	1.3	4.05	10	1.15	3.94	9	1.2	4.05	1.15	3.93	1.2	4.05	12
C/H	6.0028	7.0968	6.3436	6.9952	9	6.0028	7.0968	8	6.2894	6.9952	6.0028	7.0968	6.2415	6.9676	12
cetane index (D976)	37.3	59.8	40.8	57	15	37.3	59.8	14	40.8	57	37.3	59.8	40.8	57	14
cetane index (D4737)	37.4	60.1	40.7	65.7	15	39.9	60.1	14	37.4	65.7	37.4	60.1	40.2	65.7	13
heat of comb., MJ/kg	42.3	43.21	42.29	43.41	16	42.29	43.21	15	42.35	43.41	42.29	43.21	42.35	43.41	14

**TABLE 36. Summary Statistics of the Validation Set
for Instruments A and B**

property	Validation Set	
	<i>min</i>	<i>max</i>
density (D4052)	0.7958	0.8668
cetane number (D613)	40.1	61.3
total ArH, wt%	11.6	42.9
kin. visc., cSt, 40 C	1.14	3.92
C/H	5.9512	7.0744
cetane index (D976)	37.0	59.1
cetane index (D4737)	39.1	64.9
heat of comb., MJ/kg	42.25	43.46

TABLE 37. Calibration Results for Instrument A

Property	Mean-Centered			1st Difference/Mean-Centered			2nd Difference/Mean-Centered		
	Factors	SEP(CV)	SEC	Factors	SEP(CV)	SEC	Factors	SEP(CV)	SEC
density (D4052)	5	0.0017	0.0017	4	0.0018	0.0018	4	0.0024	0.0022
cetane number (D613)	10	1.9	1.7	13	1.8	1.5	11	1.8	1.6
total ArH, wt%	5	0.9	0.9	4	0.9	0.8	4	1.2	1.1
kin. visc., cSt, 40 C	7	0.13	0.12	9	0.11	0.09	8	0.13	0.11
C/H	5	0.0407	0.0397	3	0.0480	0.0472	4	0.0475	0.0456
cetane index (D976)	7	0.8	0.8	5	0.9	0.8	6	0.9	0.8
cetane index (D4737)	6	0.9	0.8	5	0.9	0.8	8	0.8	0.7
heat of comb., MJ/kg	5	0.05	0.05	4	0.05	0.05	4	0.05	0.05

TABLE 38. Calibration Results for Instrument B

Property	Mean-Centered			1st Difference/Mean-Centered			2nd Difference/Mean-Centered		
	Factors	SEP(CV)	SEC	Factors	SEP(CV)	SEC	Factors	SEP(CV)	SEC
density (D4052)	8	0.0022	0.0020	5	0.0026	0.0025	6	0.0028	0.0025
cetane number (D613)	12	1.9	1.7	9	1.9	1.7	11	1.7	1.5
total ArH, wt%	5	1.2	1.2	4	1.1	1.0	4	1.2	1.2
kin. visc., cSt, 40 C	9	0.15	0.13	9	0.12	0.10	10	0.11	0.10
C/H	6	0.0460	0.0450	4	0.0500	0.0478	3	0.0572	0.0543
cetane index (D976)	8	0.8	0.8	9	0.8	0.7	8	0.8	0.7
cetane index (D4737)	9	0.8	0.7	9	0.8	0.7	8	0.8	0.8
heat of comb., MJ/kg	6	0.06	0.05	4	0.06	0.05	6	0.06	0.05

TABLE 39. Validation Results for Instrument A

Property	Mean-Centered		1st Difference/Mean-Centered		2nd Difference/Mean-Centered	
	R ²	SEP	R ²	SEP	R ²	SEP
density (D4052)	0.9847	0.0018	0.9839	0.0019	0.9801	0.0021
cetane number (D613)	0.5241	<u>2.4</u>	0.5695	<u>2.4</u>	0.5373	<u>2.4</u>
total ArH, wt%	0.9763	1.0	0.9823	0.8	0.9716	1.1
kin. visc., cSt, 40 C	0.9360	0.12	0.9286	0.13	0.9143	0.14
C/H	0.9490	0.0444	0.9331	0.0509	0.9372	0.0493
cetane index (D976)	0.8565	<u>1.3</u>	0.8630	<u>1.3</u>	0.8434	<u>1.3</u>
cetane index (D4737)	0.8783	1.3	0.8838	1.3	0.8910	<u>1.2</u>
heat of comb., MJ/kg	0.9108	<u>0.06</u>	0.9045	<u>0.06</u>	0.8910	0.07

TABLE 40. Validation Results for Instrument B

Property	Mean-Centered		1st Difference/Mean-Centered		2nd Difference/Mean-Centered	
	R ²	SEP	R ²	SEP	R ²	SEP
density (D4052)	0.9772	0.0023	0.9691	0.0026	0.9593	0.0030
cetane number (D613)	0.5718	<u>2.3</u>	0.5438	2.4	0.5515	2.4
total ArH, wt%	0.9570	1.3	0.9634	1.2	0.9529	1.4
kin. visc., cSt, 40 C	0.9306	0.13	0.9488	0.11	0.9408	0.12
C/H	0.9320	0.0513	0.9297	0.0522	0.8935	0.0642
cetane index (D976)	0.8510	1.3	0.8582	1.3	0.8668	1.2
cetane index (D4737)	0.8820	<u>1.3</u>	0.8763	<u>1.3</u>	0.8803	<u>1.3</u>
heat of comb., MJ/kg	0.8925	<u>0.07</u>	0.8765	<u>0.07</u>	0.8897	<u>0.07</u>

TABLE 41. Calibration and Validation Summary
(Fuel Set D; Instrument B)

Property	Calibration						Validation
	F @ PRESS (min)			F @ p≤0.75			SEP at
	F	SEP(CV)	R ²	F	SEP(CV)	R ²	F @ p≤0.75
API gravity (D1298)	20	0.16	0.9959	18	0.16	0.9957	0.21
density (D1298)	18	0.0008	0.9963	18	0.0008	0.9963	0.0009
API gravity (D4052)	20	0.12	0.9977	20	0.12	0.9977	0.18
density (D4052)	20	0.0005	0.9985	19	0.0005	0.9984	0.0006
kin. visc. cSt, 40°C	20	0.066	0.9782	19	0.067	0.9773	0.09
flash point, °C	20	4.3	0.6078	17	4.4	0.5886	6.0
cloud point, °C	20	2.8	0.8693	17	2.8	0.8612	4.5
cloud point, °C (1)	20	1.9	0.7810	16	2.0	0.7690	7.1
cloud point, °C (2)	19	1.7	0.8187	16	1.7	0.8092	7.3
freeze pt, °C	20	3.0	0.8724	16	3.1	0.8659	4.5
freeze pt, °C (1)	20	2.0	0.7678	14	2.1	0.7518	6.8
freeze pt, °C (2)	20	1.7	0.8081	15	1.8	0.7960	7.2
pour pt, °C	20	4.4	0.8216	14	4.5	0.8116	5.1
pour pt, °C (1)	20	4.0	0.6883	14	4.1	0.6692	6.7
pour pt, °C (2)	20	3.7	0.7263	17	3.8	0.7143	7.1
initial bp, °C	19	6.3	0.6760	14	6.5	0.6555	9.8
bp-10, °C	20	4.7	0.8896	15	4.8	0.8852	6.4
bp-50, °C	20	2.4	0.9794	16	2.5	0.9782	4.7
bp-90, °C	20	5.8	0.8710	18	5.9	0.8661	8.0
bp-95, °C	20	7.5	0.8093	18	7.7	0.7989	9.8
final bp, °C	20	7.9	0.7767	16	8.1	0.7639	10.6
cetane number (D613)	18	1.54	0.7920	12	1.58	0.7811	2.4
cetane index (D976)	20	0.62	0.9498	14	0.64	0.9469	1.21
cetane index (D4737)	20	0.60	0.9579	14	0.62	0.9556	1.35
hydrogen, wt%	9	0.075	0.9404	5	0.077	0.9376	0.10
carbon, wt%	5	0.223	0.6637	2	0.228	0.6495	0.26
C/H	9	0.036	0.9603	5	0.037	0.9581	0.0494
heat of comb., MJ/kg	15	0.046	0.9288	7	0.047	0.9248	0.06
heat of comb., Btu/lb	17	19.85	0.9286	7	20.35	0.9249	27
monocyclic ArH, wt%	20	0.336	0.9947	20	0.336	0.9947	0.42
dicyclic ArH, wt%	20	0.243	0.9878	18	0.249	0.9872	0.046
polycyclic ArH, wt%	20	0.201	0.8378	14	0.206	0.8302	0.28
total ArH, wt%	20	0.377	0.9963	18	0.387	0.9961	0.50

No. of Samples: excluded outliers identified in preliminary calibrations using 547 samples

property (1): excluded jet fuel samples from calibrations under property name

property (2): excluded outliers identified under calibrations in property(1)

ArH: aromatic hydrocarbons

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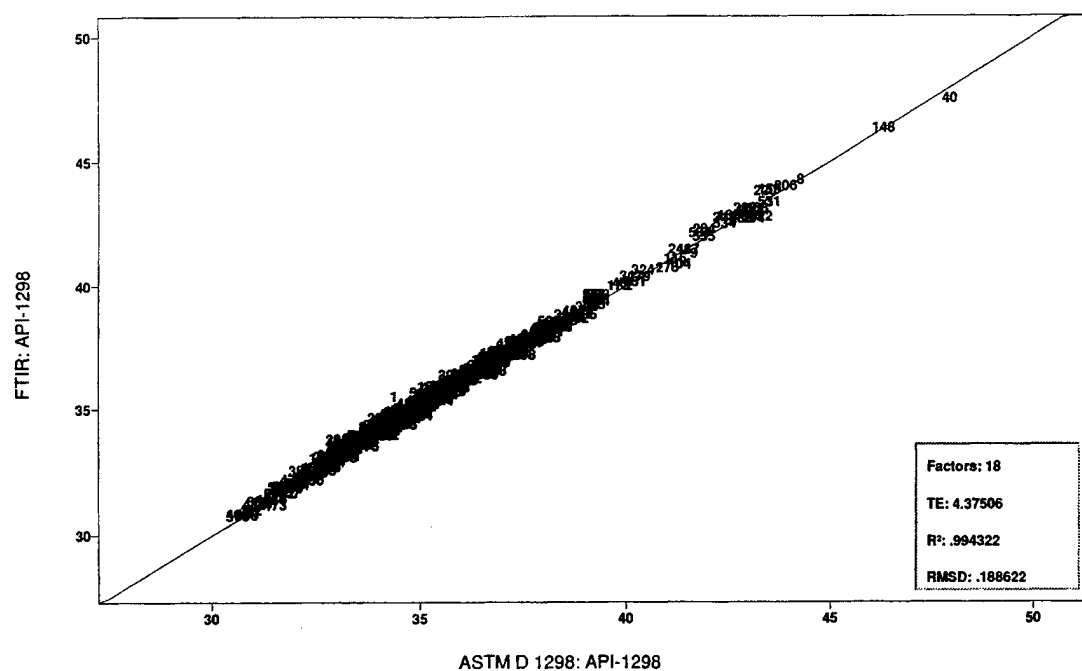


**Figure 1. Calibration for API Gravity: ASTM D 1298 vs FT-IR
Fuel Set D, Instrument A**

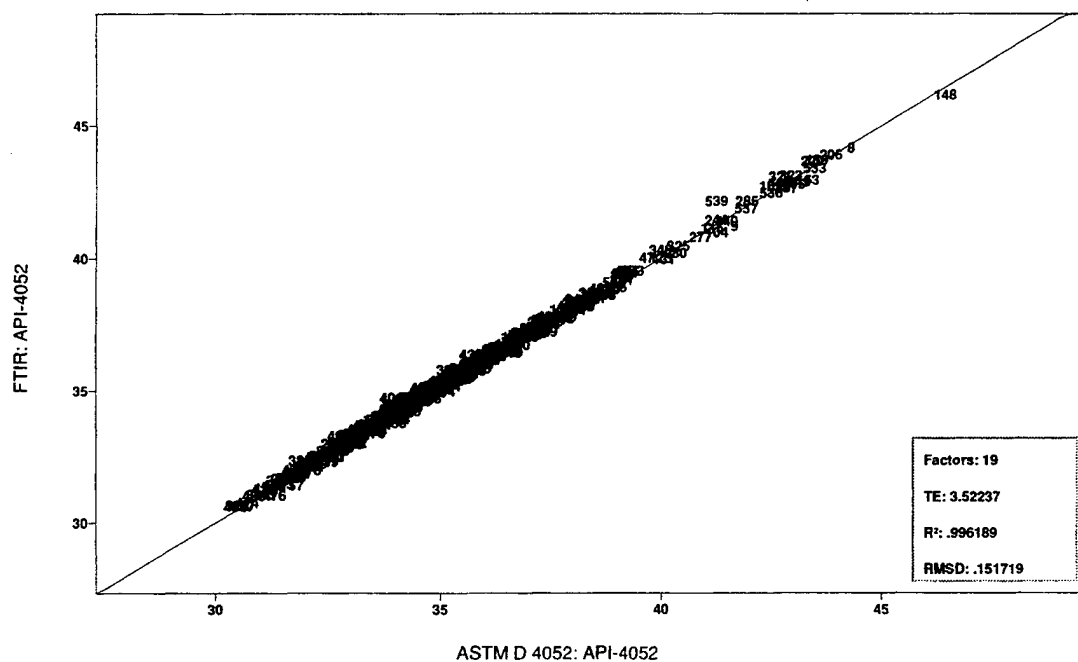


**Figure 2. Calibration for API Gravity: ASTM D 4052 vs FT-IR
Fuel Set D, Instrument A**

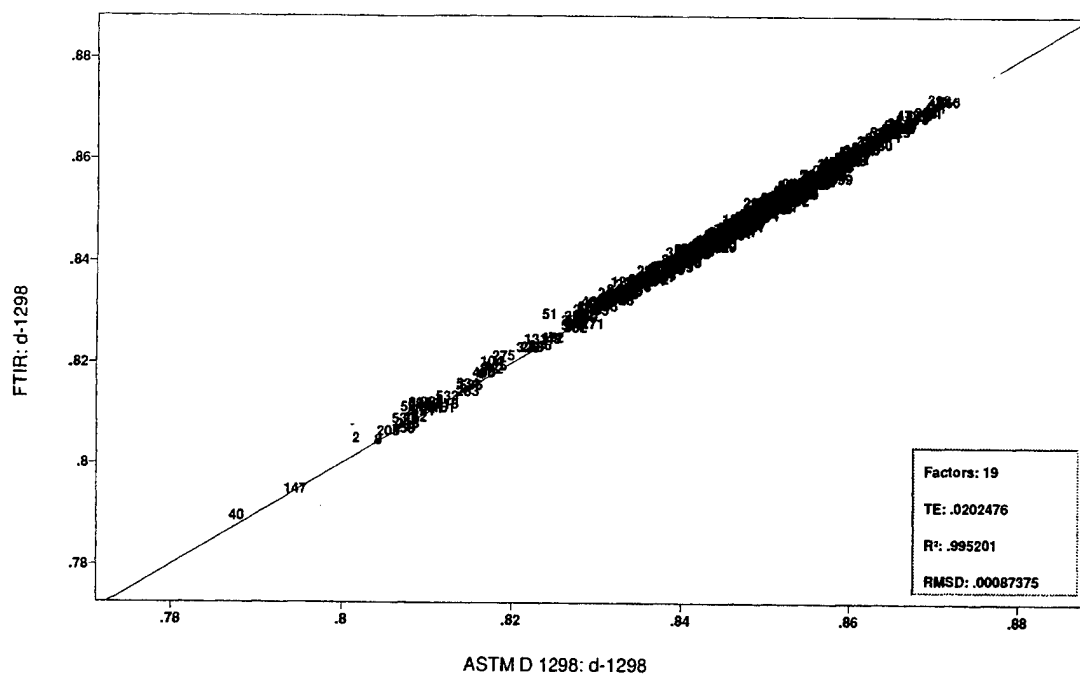


Figure 3. Calibration for Density: ASTM D 1298 vs FT-IR
Fuel Set D, Instrument A

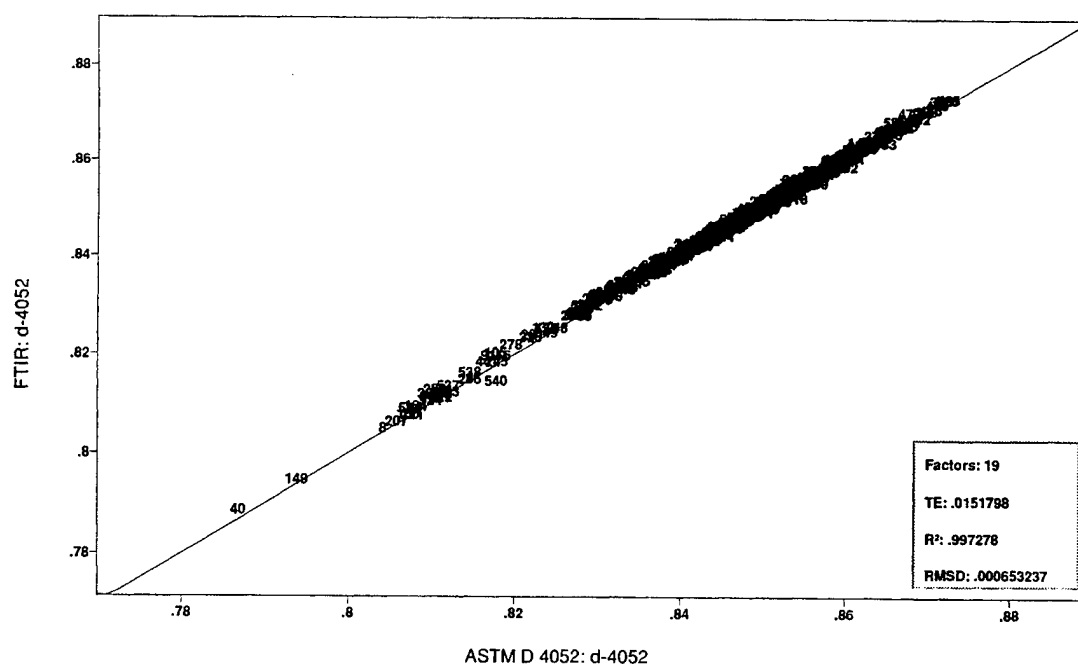


Figure 4. Calibration for Density: ASTM D 4052 vs FT-IR
Fuel Set D, Instrument A

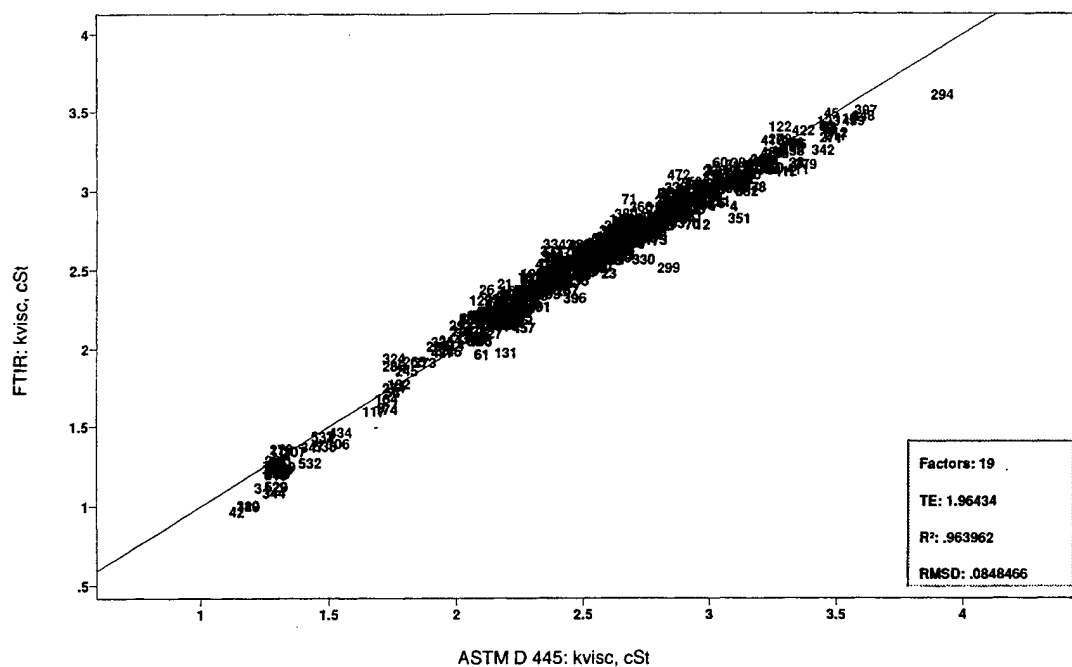


**Figure 5. Calibration for Kinematic Viscosity, cSt, at 40°C: ASTM D 445 vs FT-IR
Fuel Set D, Instrument A**

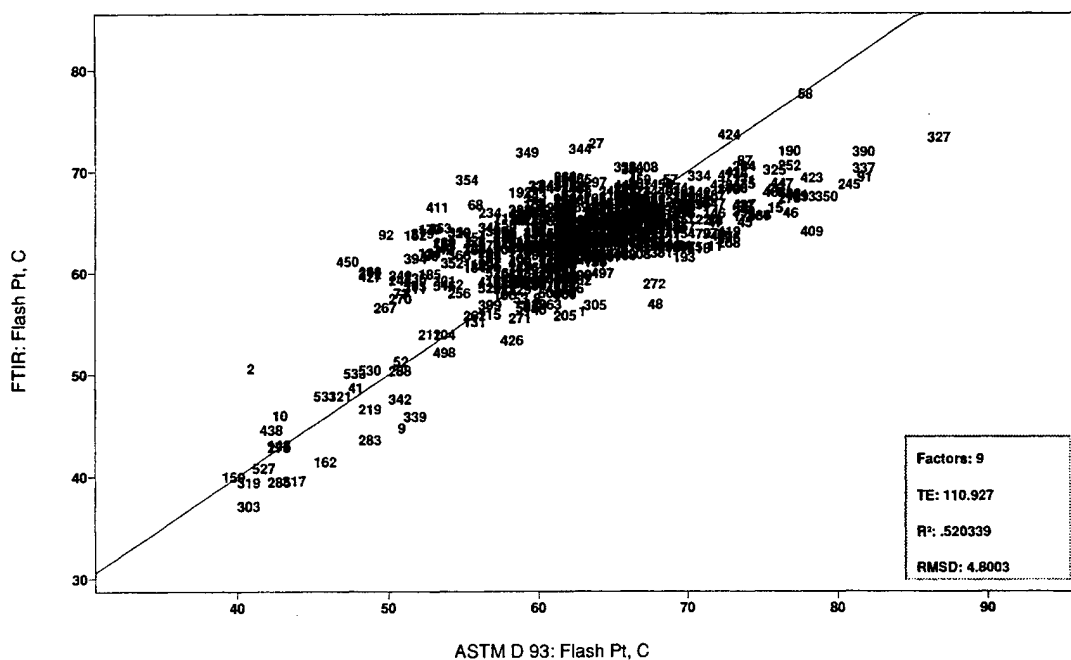


**Figure 6. Calibration for Flash Point: ASTM D 93 vs FT-IR
Fuel Set D, Instrument A**

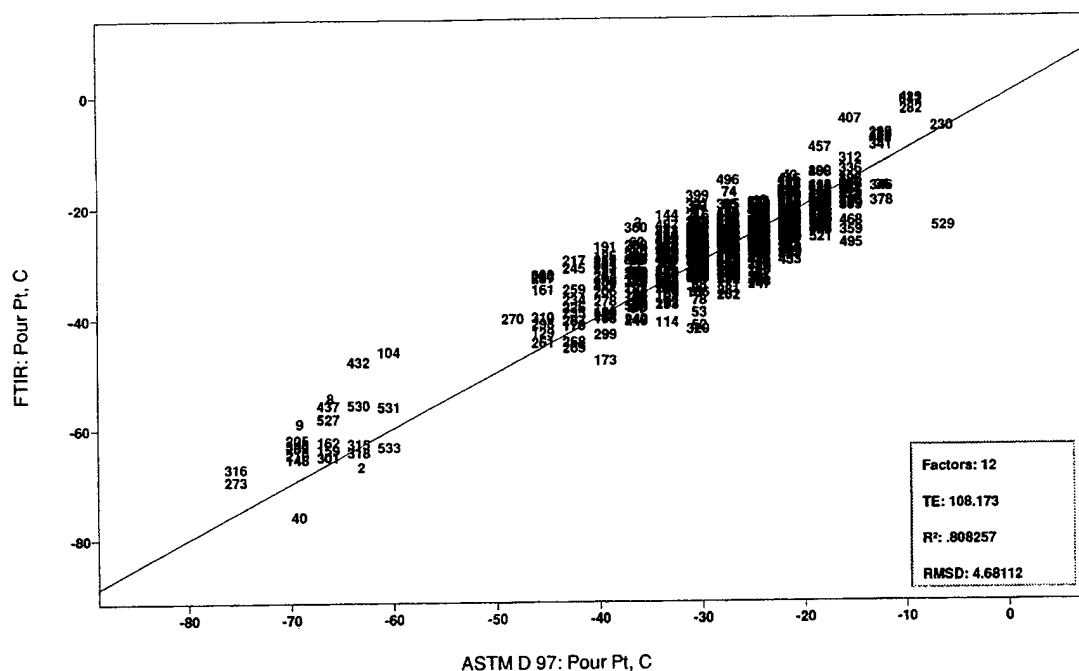


**Figure 9. Calibration for Pour Point: ASTM D 97 vs FT-IR
Fuel Set D, Instrument A**

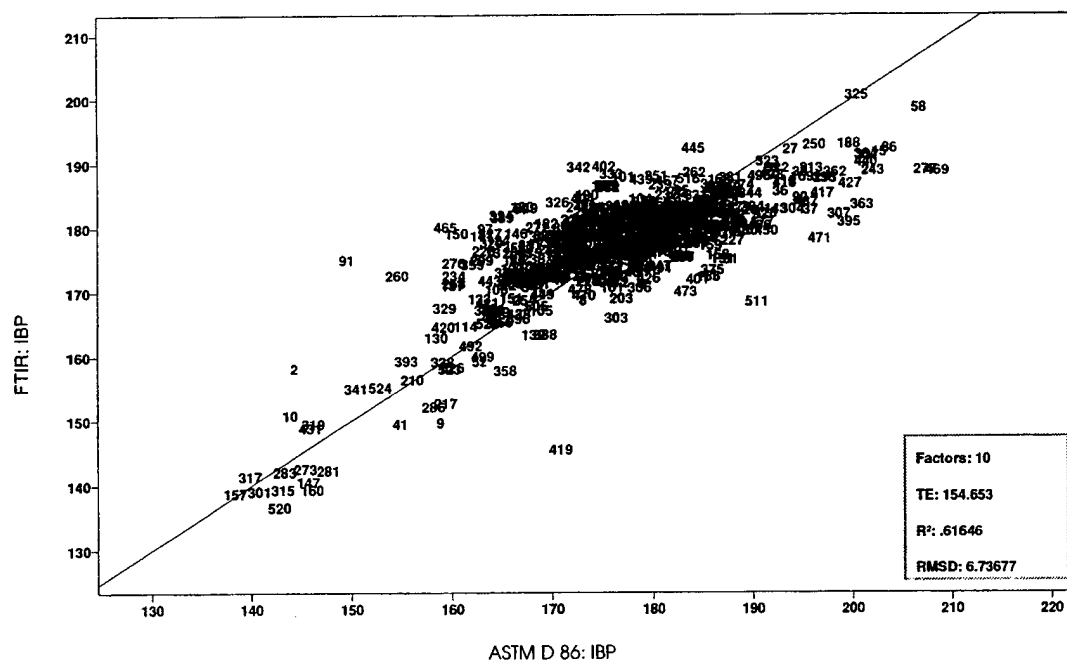


**Figure 10. Calibration for Initial Boiling Point: ASTM D 86 vs FT-IR
Fuel Set D, Instrument A**

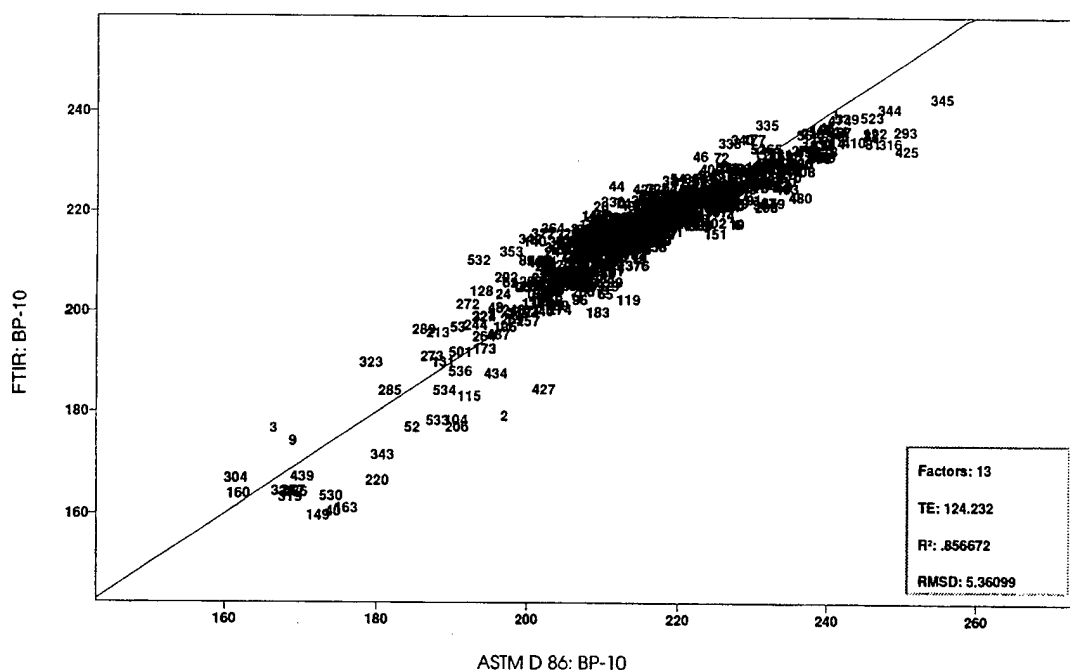


**Figure 11. Calibration for Boiling Point at 10%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument A**

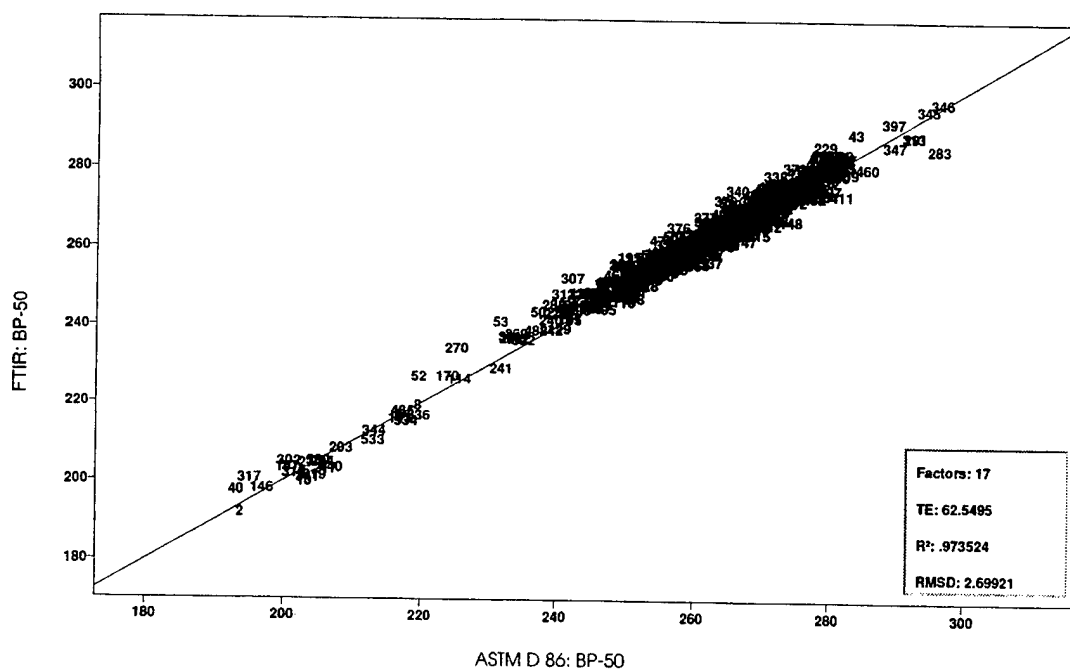


**Figure 12. Calibration for Boiling Point at 50%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument A**

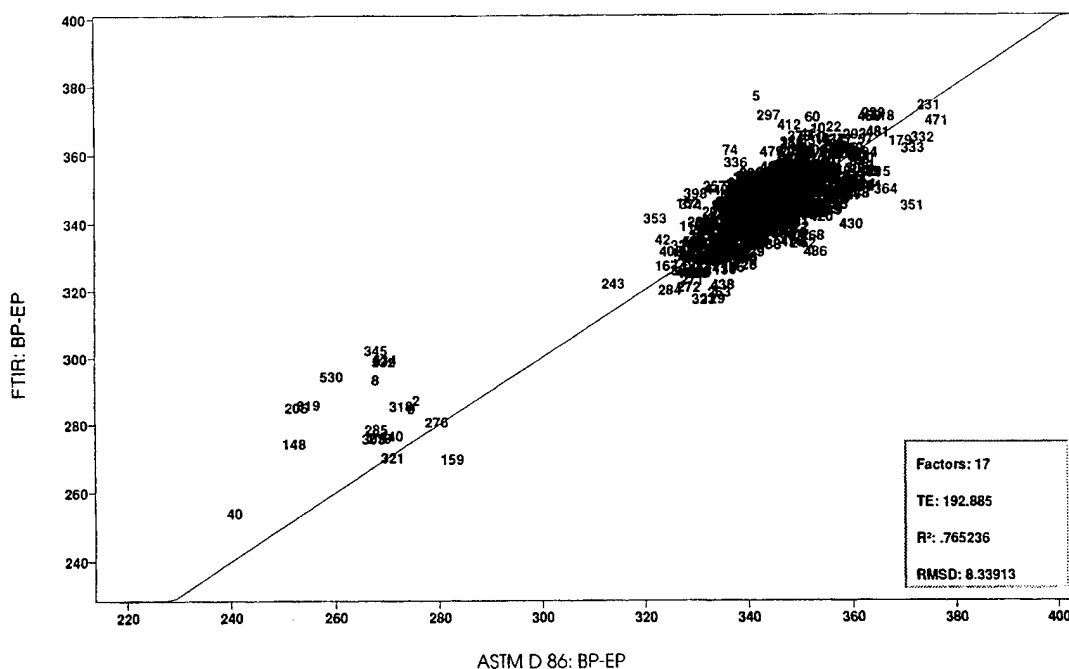


**Figure 15. Calibration for Final Boiling Point at: ASTM D 86 vs FT-IR
Fuel Set D, Instrument A**

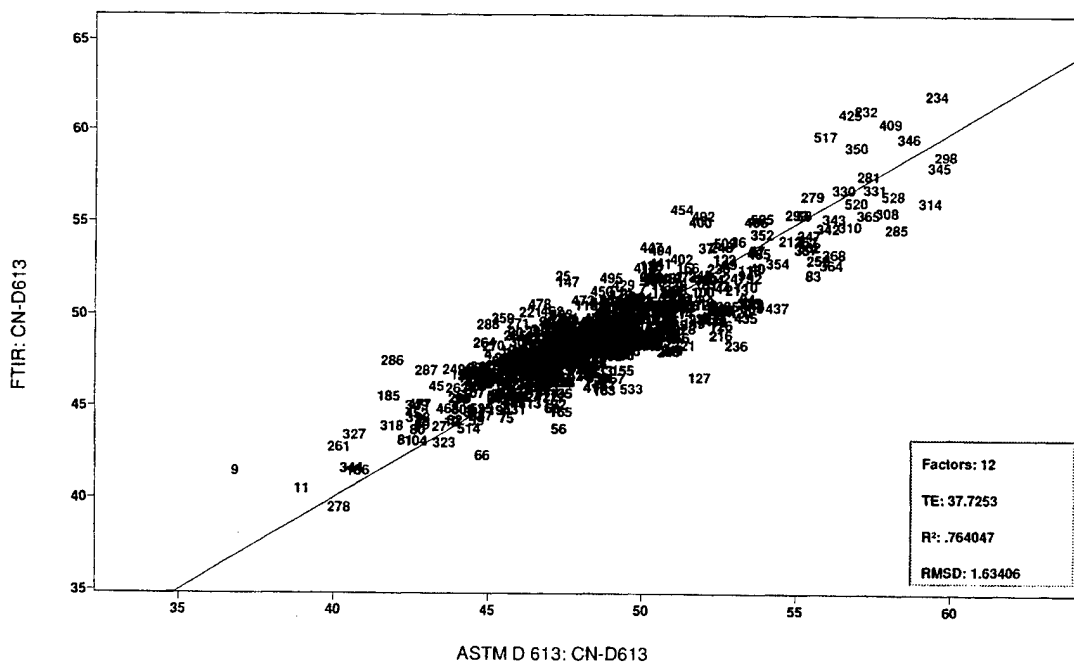


**Figure 16. Calibration for Cetane Number: ASTM D 613 vs FT-IR
Fuel Set D, Instrument A**

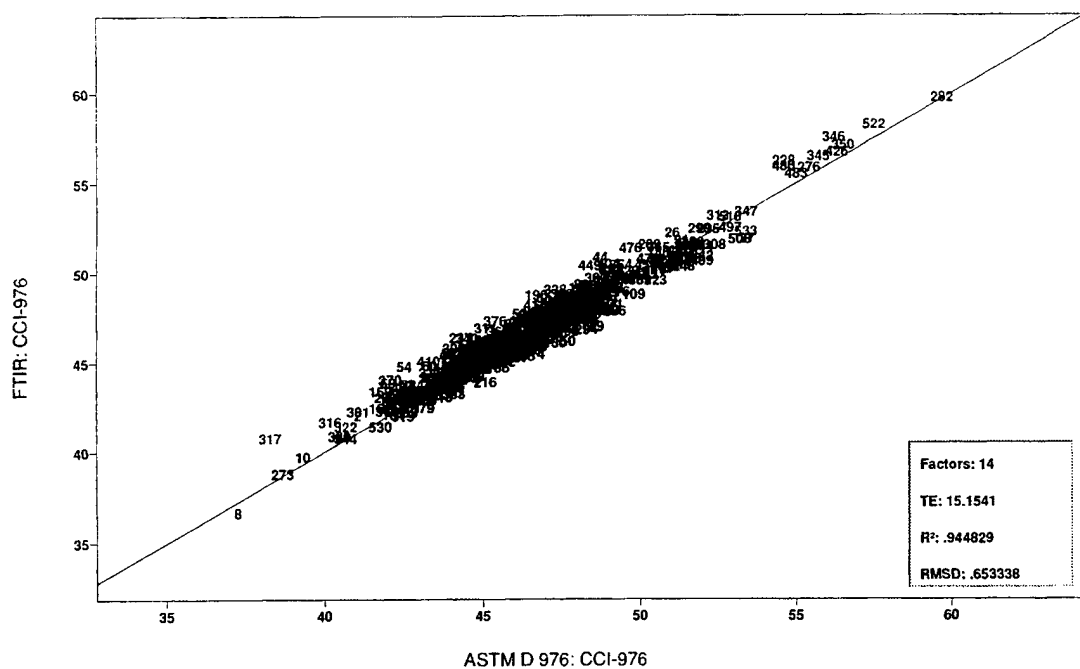


Figure 17. Calibration for Calculated Cetane Index: ASTM D 976 vs FT-IR Fuel Set D, Instrument A

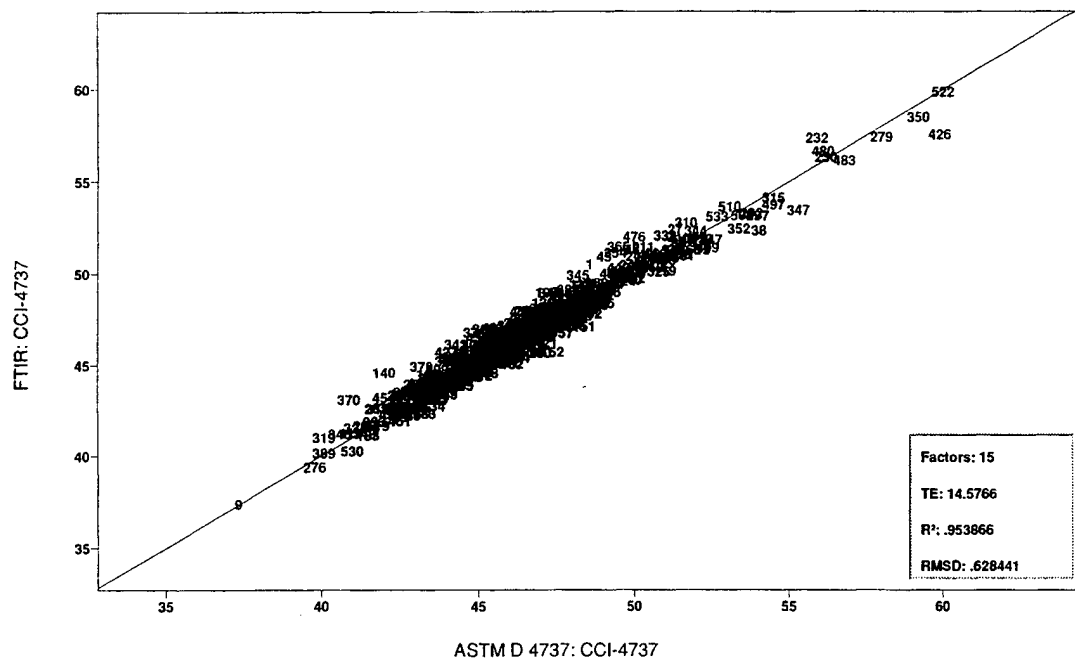


Figure 18. Calibration for Calculated Cetane Index: ASTM D 4737 vs FT-IR Fuel Set D, Instrument A

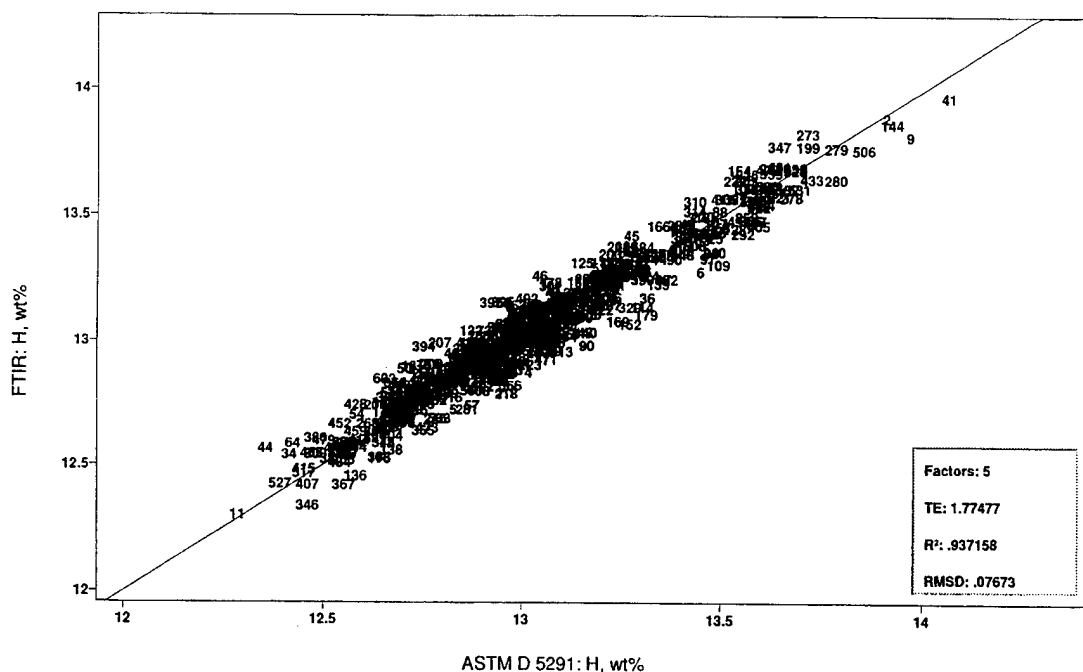


**Figure 19. Calibration for Hydrogen Content: ASTM D 5291 vs FT-IR
Fuel Set D, Instrument A**

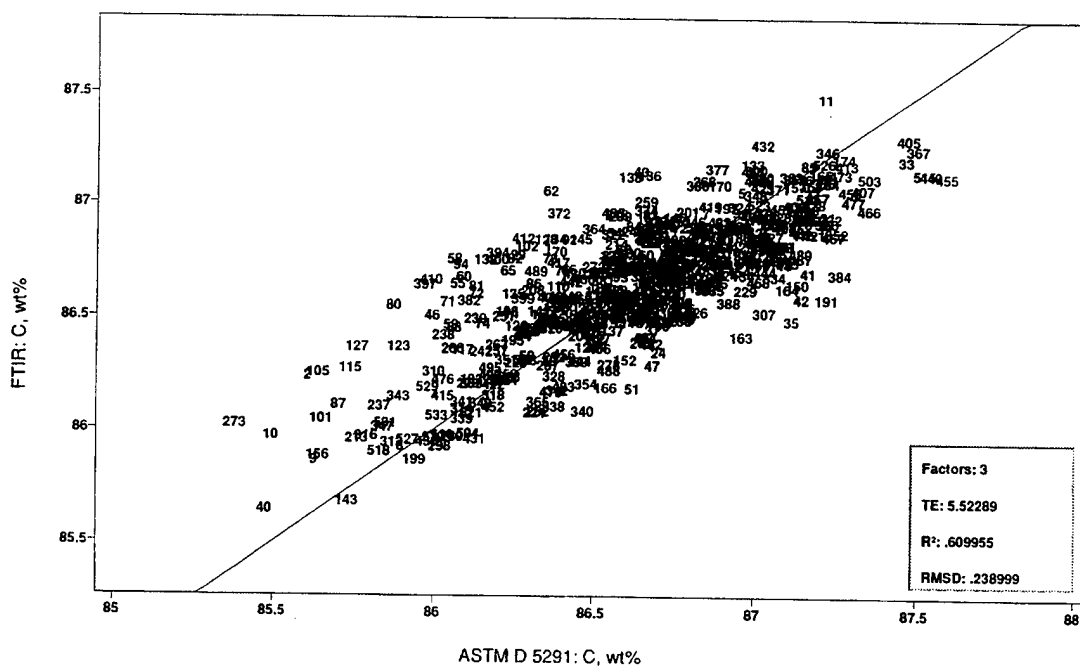


**Figure 20. Calibration for Carbon Content: ASTM D 5291 vs FT-IR
Fuel Set D, Instrument A**

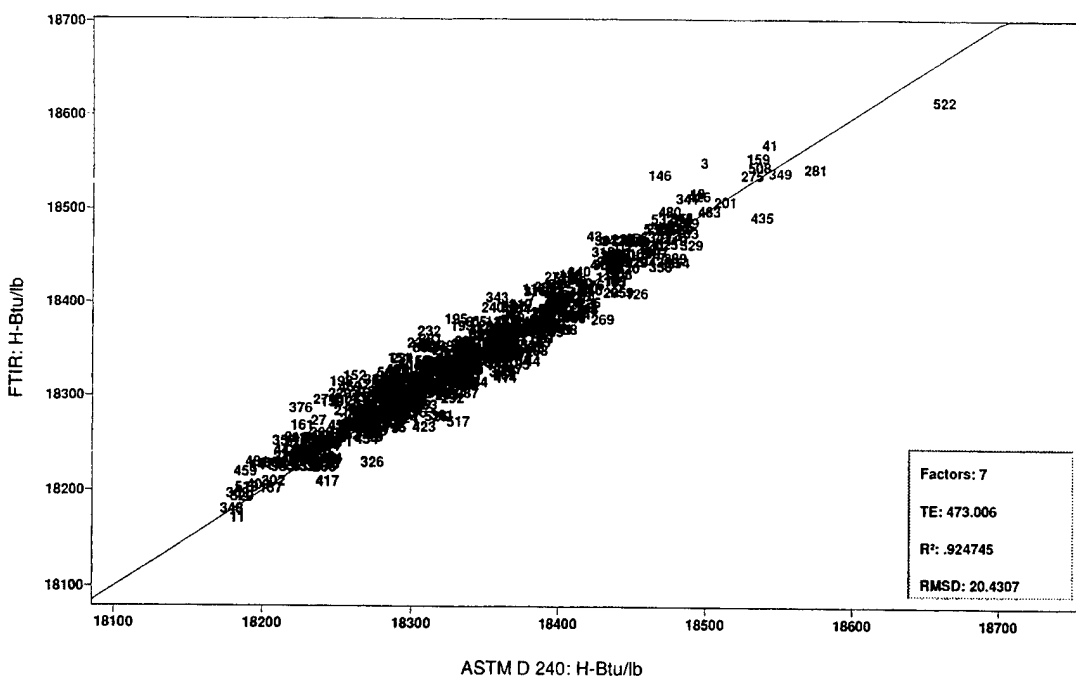


**Figure 23. Calibration for Net Heat of Combustion, Btu/lb: ASTM D 240 vs FT-IR
Fuel Set D, Instrument A**

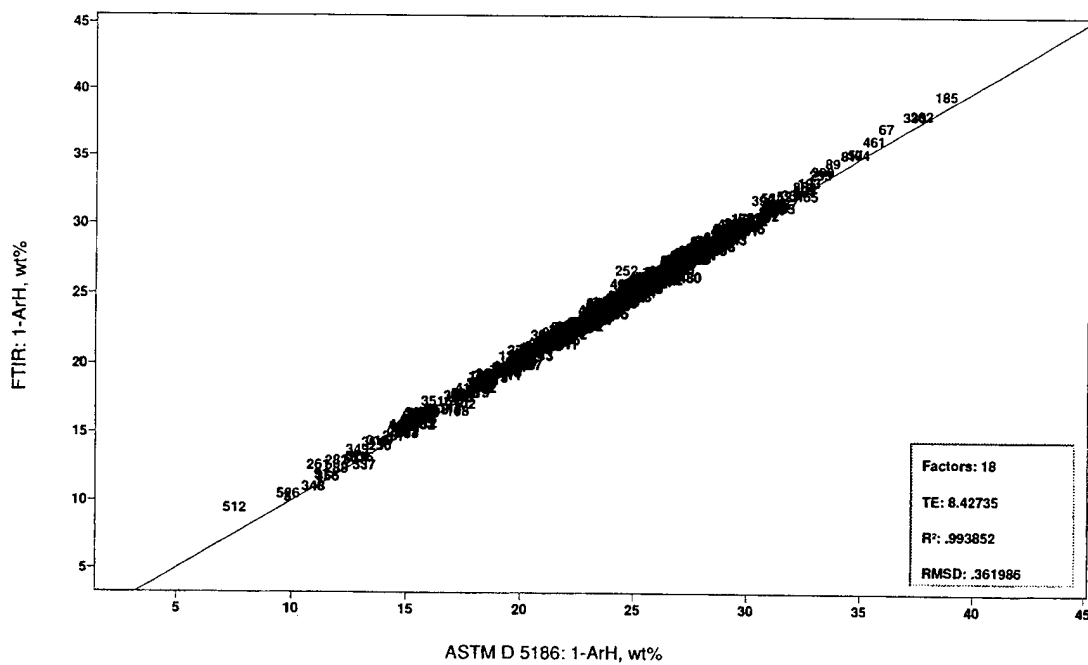


**Figure 24. Calibration for Monocyclic Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument A**

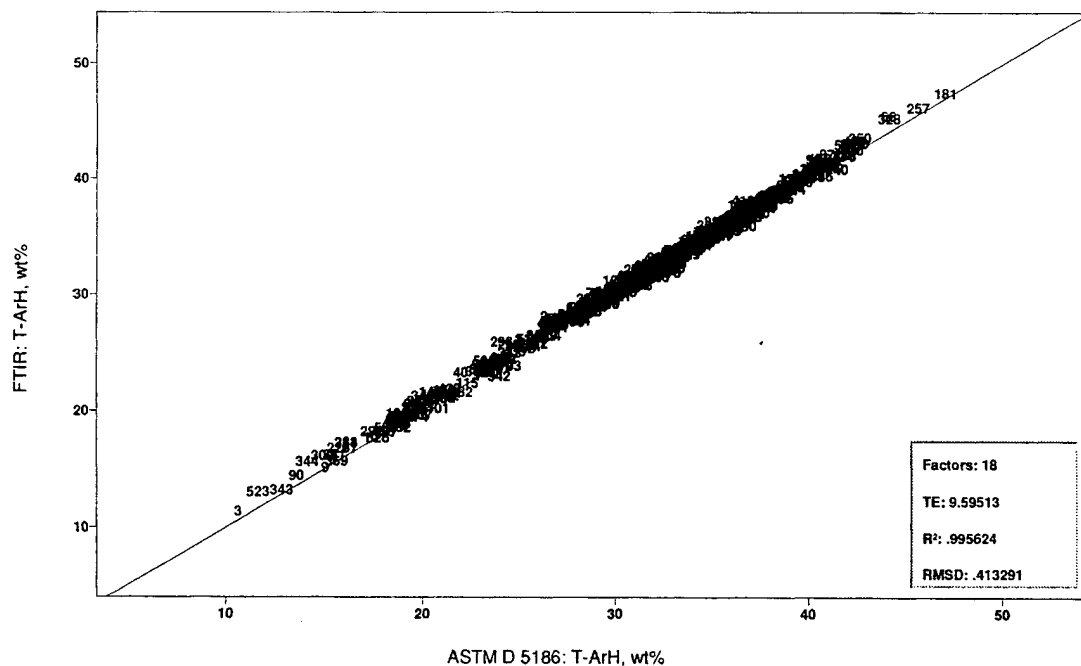


**Figure 27. Calibration for Total Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument A**

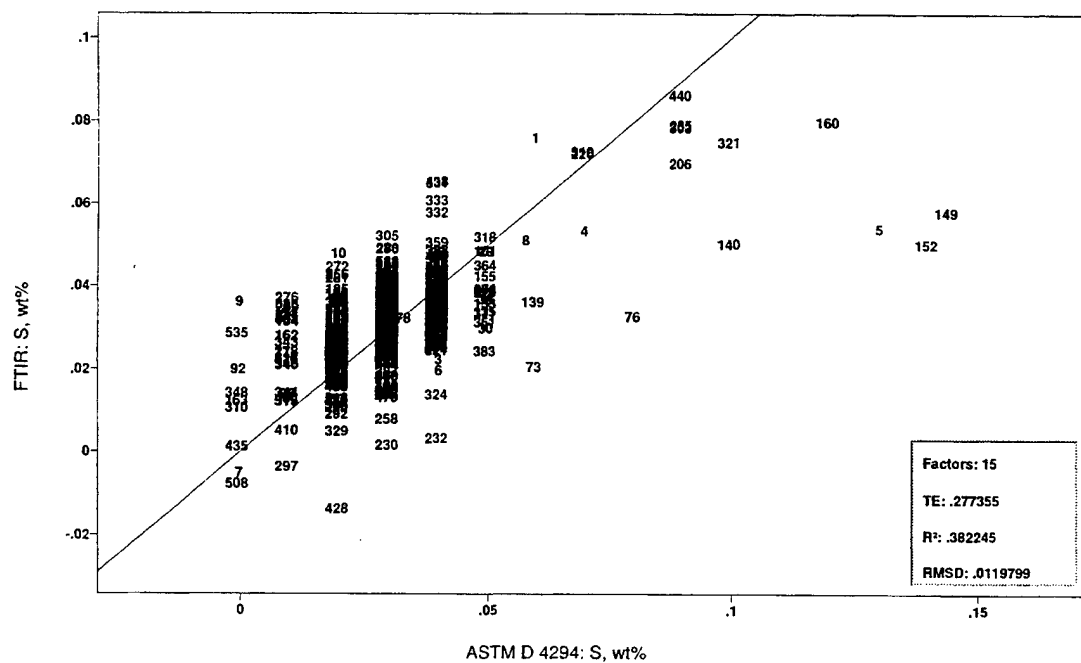


**Figure 28. Calibration for Sulfur Content: ASTM D 4294 vs FT-IR
Fuel Set D, Instrument A**

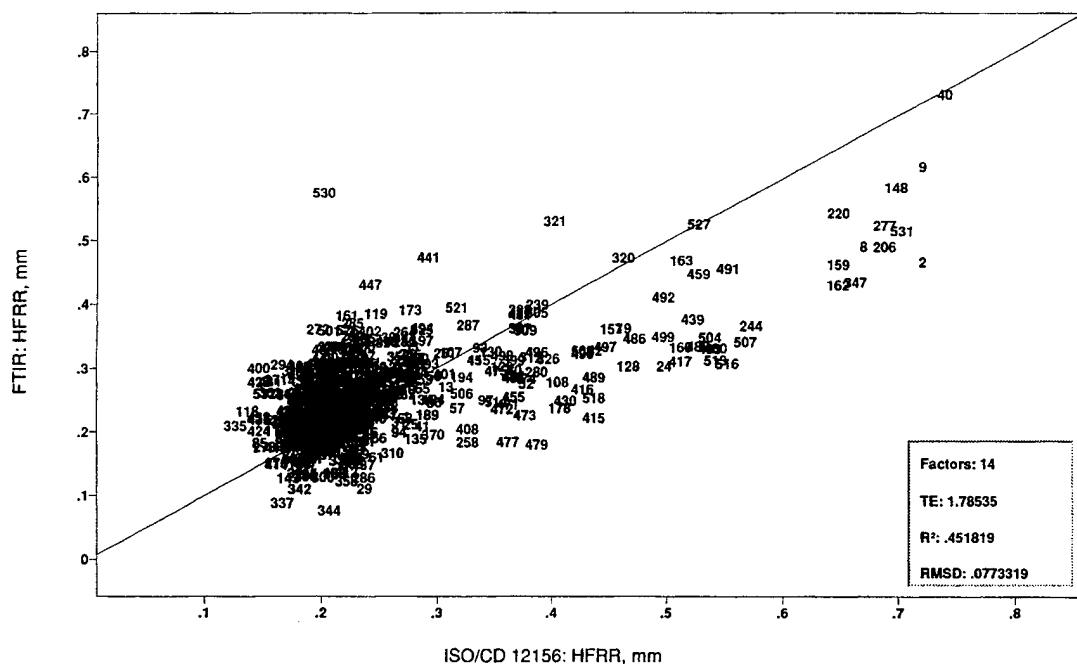


**Figure 29. Calibration for Lubricity by HFRR: ISO/CD 12156 vs FT-IR
Fuel Set D, Instrument A**

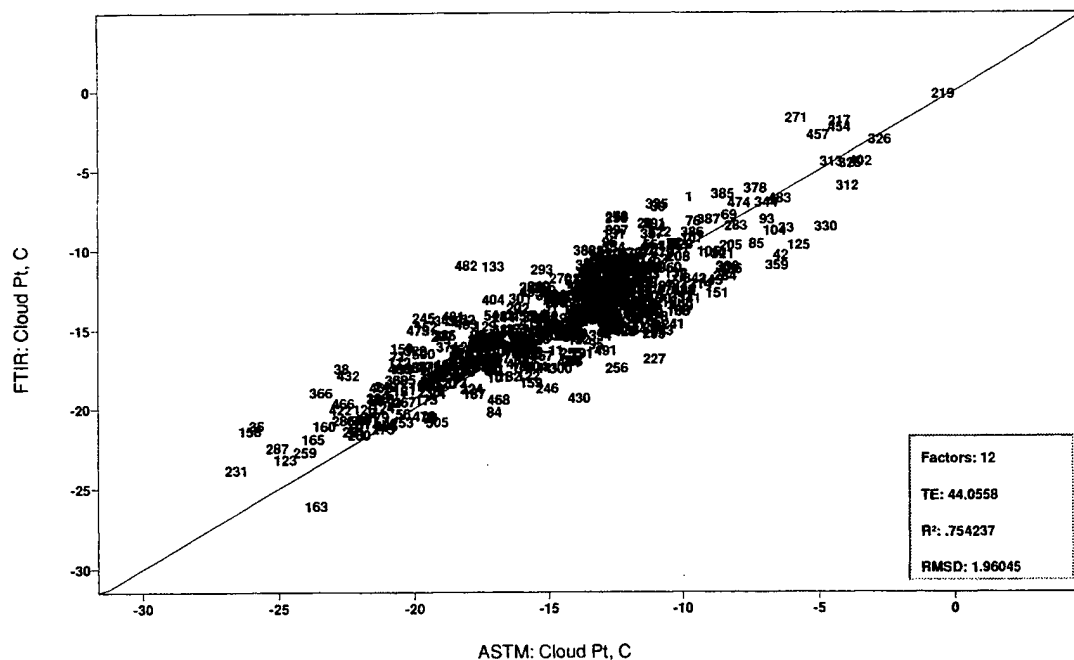


**Figure 30. Calibration for Cloud Point for Diesel Fuels: ASTM D 2500 vs FT-IR
Fuel Set D, Instrument A**

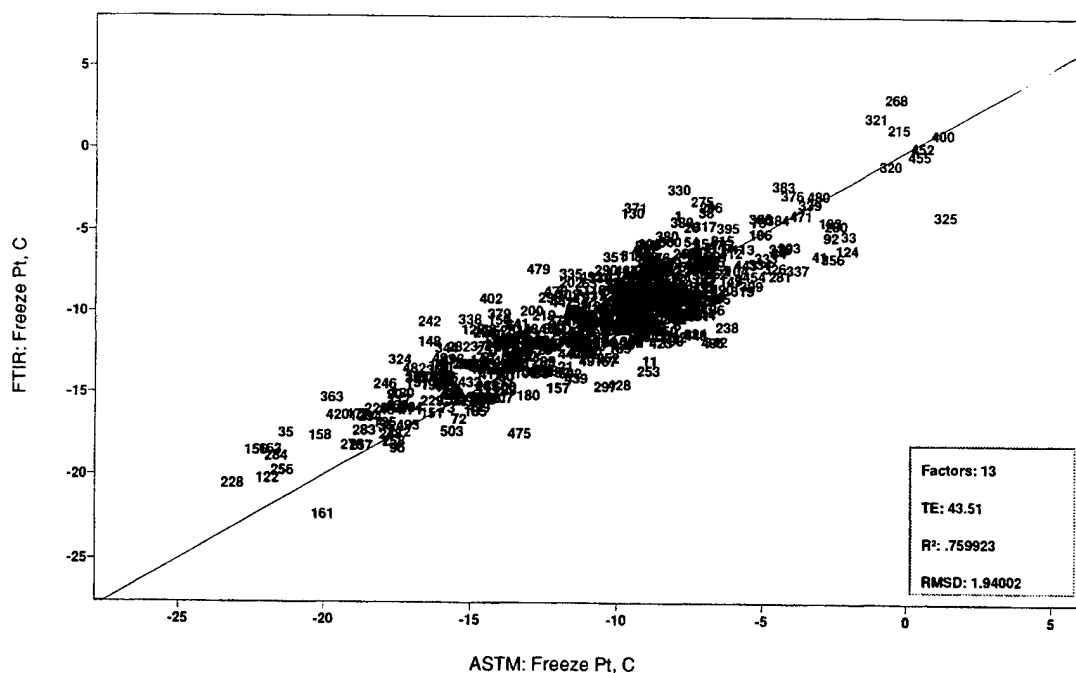


Figure 31. Calibration for Freeze Point for Diesel Fuels: ASTM D 2386 vs FT-IR Fuel Set D, Instrument A

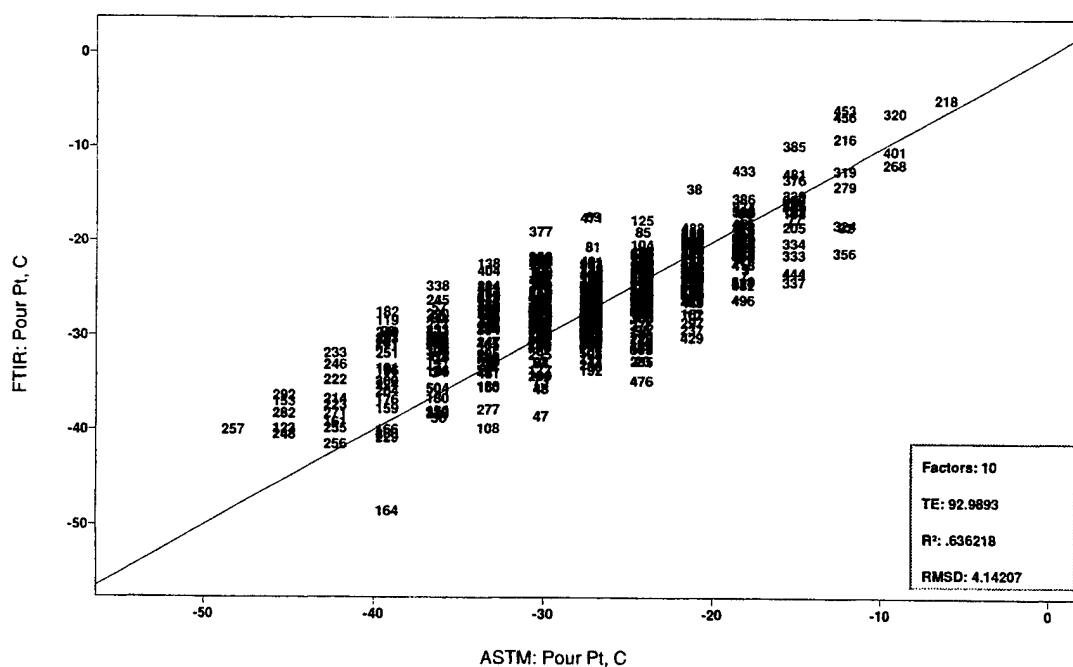


Figure 32. Calibration for Pour Point for Diesel Fuels: ASTM D 97 vs FT-IR Fuel Set D, Instrument A

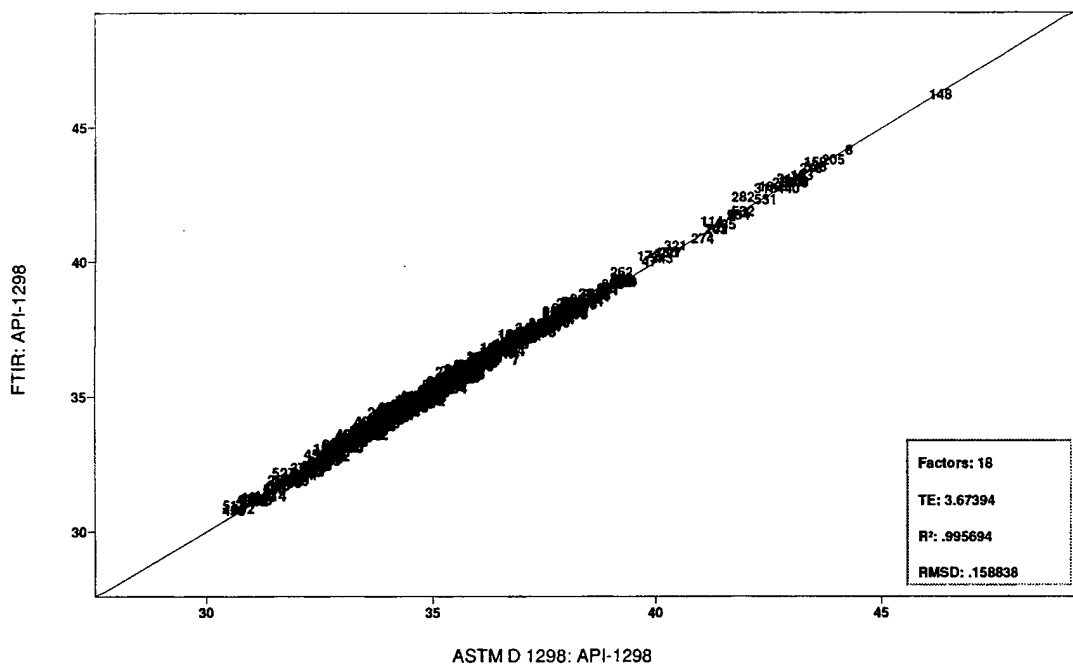


**Figure 33. Calibration for API Gravity: ASTM D 1298 vs FT-IR
Fuel Set D, Instrument B**

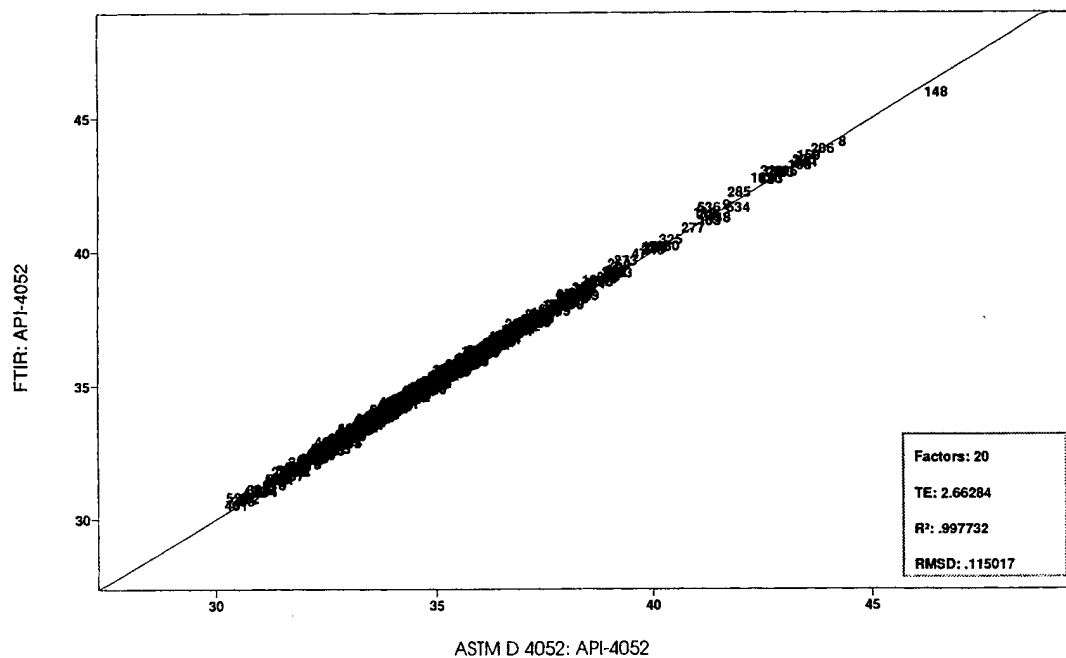


**Figure 34. Calibration for API Gravity: ASTM D 4052 vs FT-IR
Fuel Set D, Instrument B**

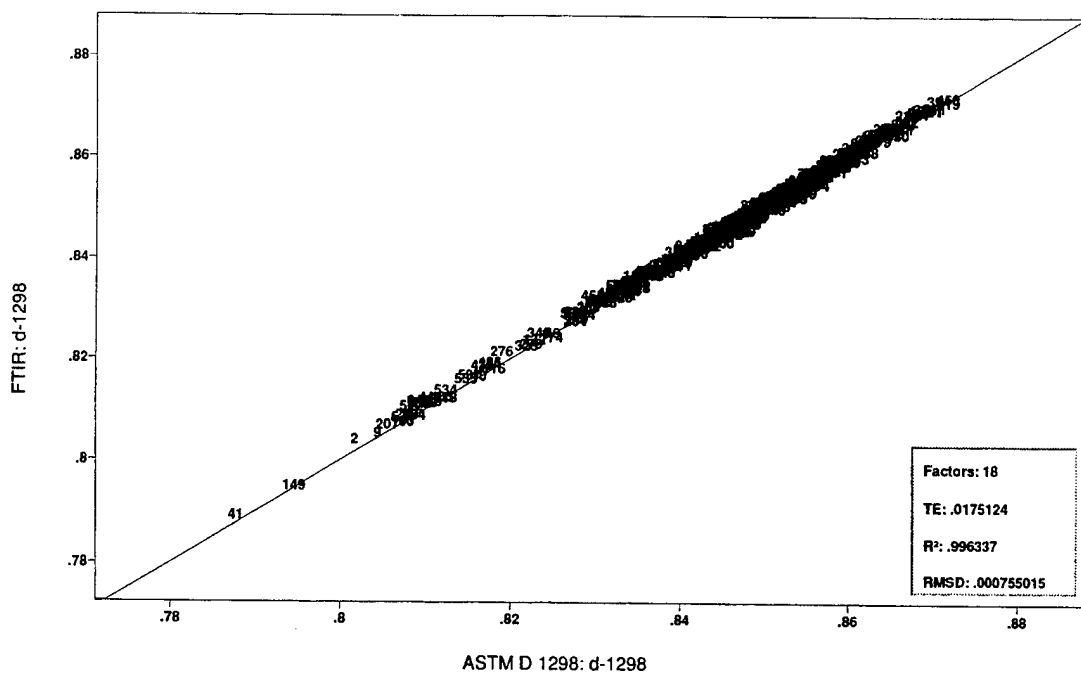


**Figure 35. Calibration for Density: ASTM D 1298 vs FT-IR
Fuel Set D, Instrument B**

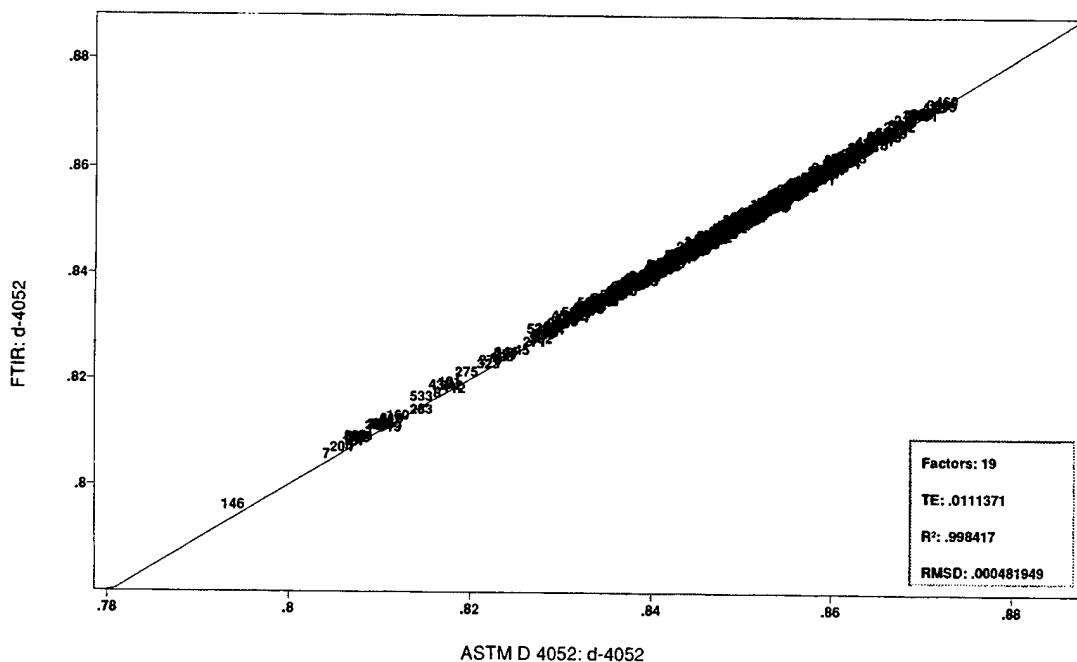


**Figure 36. Calibration for Density: ASTM D 4052 vs FT-IR
Fuel Set D, Instrument B**

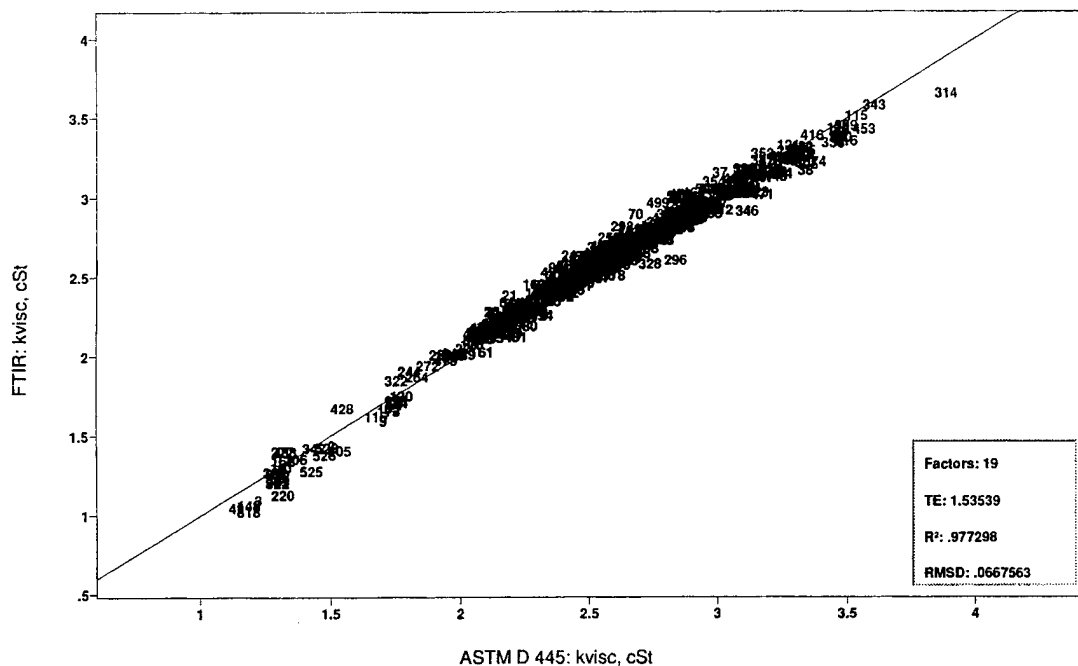


**Figure 37. Calibration for Kinematic Viscosity, cSt, at 40°C: ASTM D 445 vs FT-IR
Fuel Set D, Instrument B**

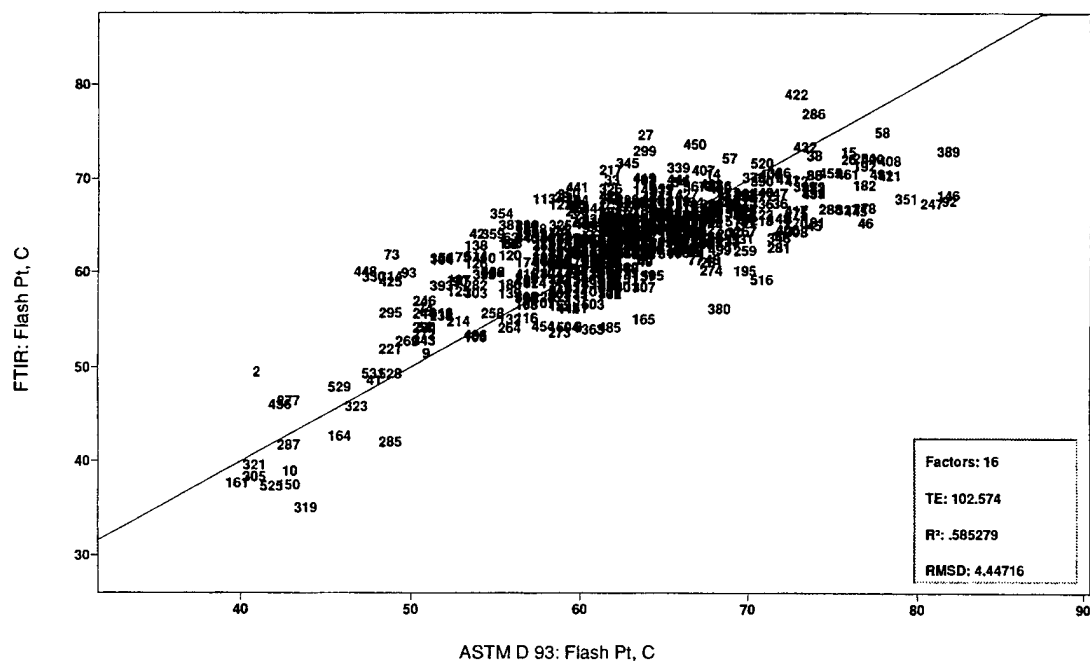


**Figure 38. Calibration for Flash Point: ASTM D 93 vs FT-IR
Fuel Set D, Instrument B**

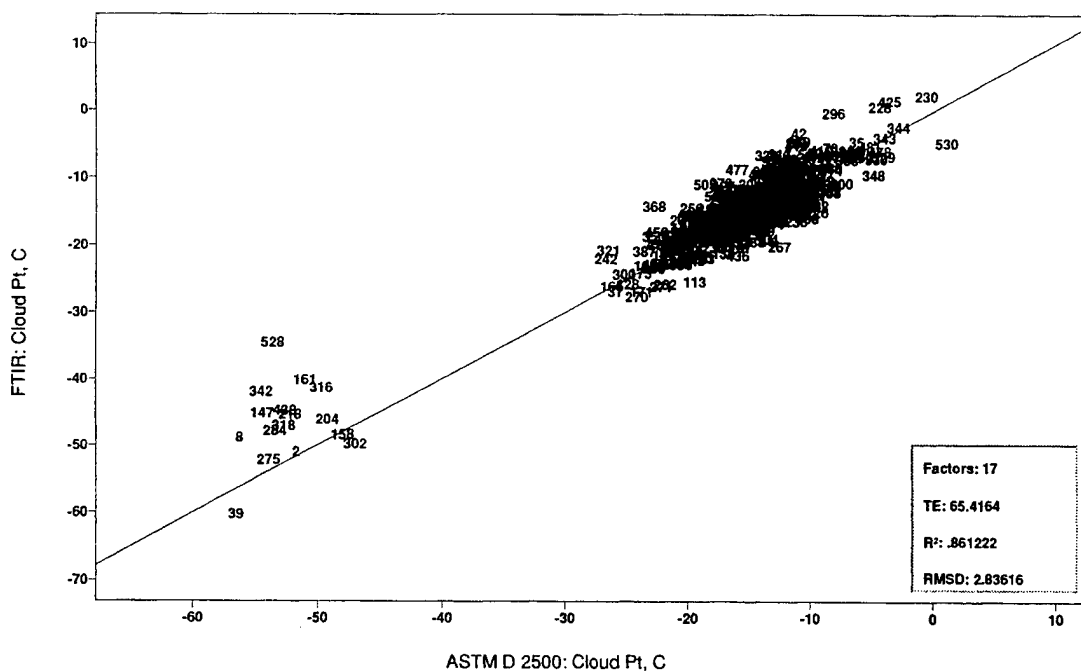


**Figure 39. Calibration for Cloud Point: ASTM D 2500 vs FT-IR
Fuel Set D, Instrument B**

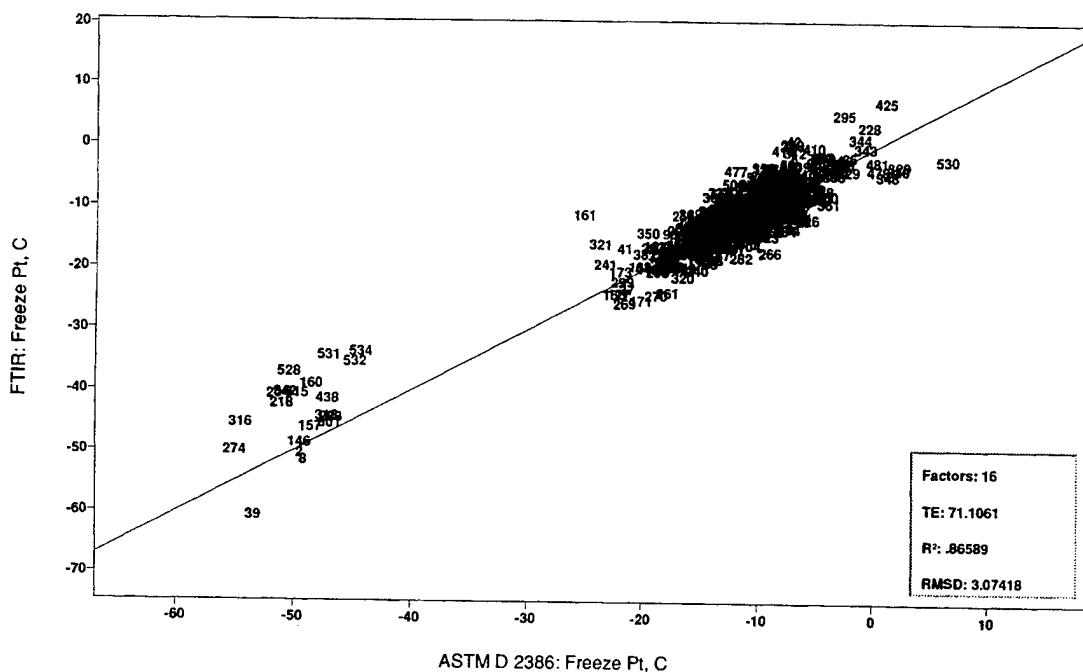


**Figure 40. Calibration for Freeze Point: ASTM D 2386 vs FT-IR
Fuel Set D, Instrument B**

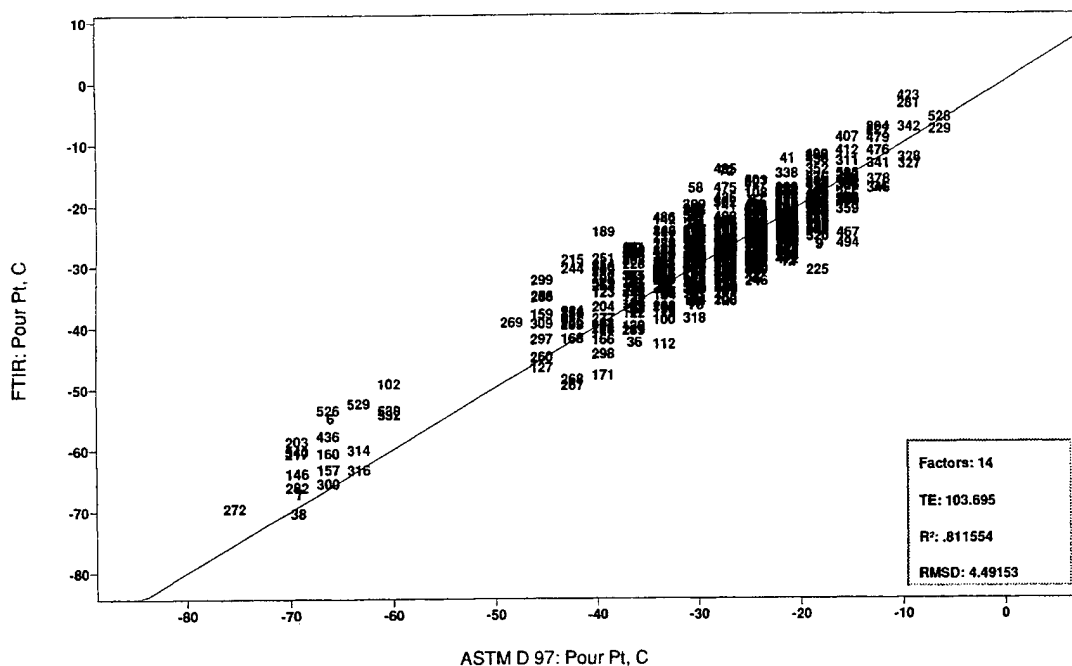


**Figure 41. Calibration for Pour Point: ASTM D 97 vs FT-IR
Fuel Set D, Instrument B**

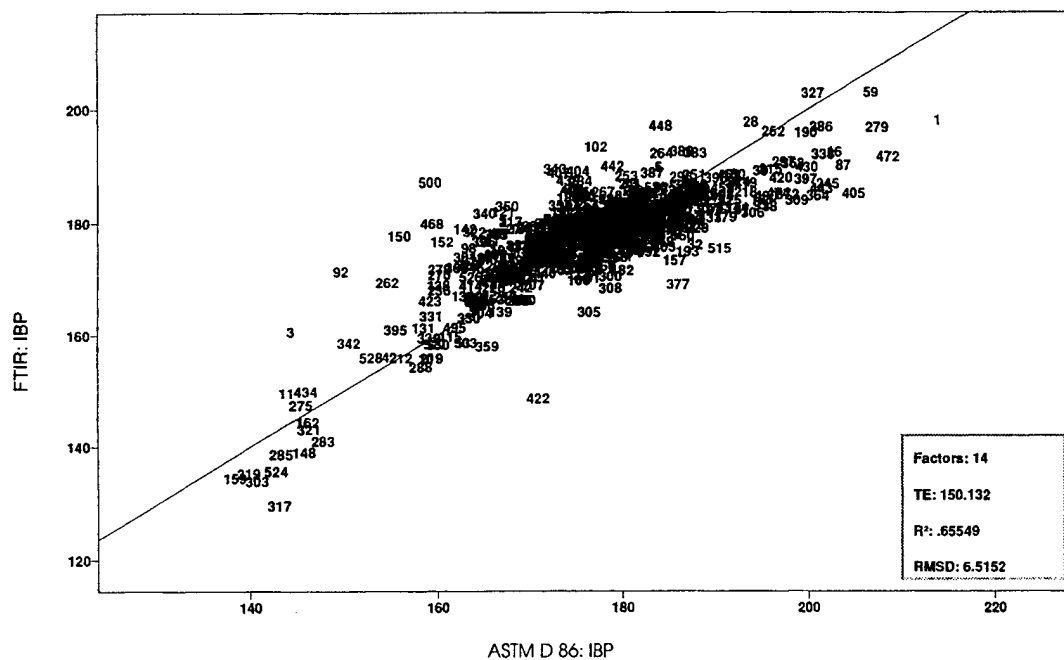


**Figure 42. Calibration for Initial Boiling Point: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

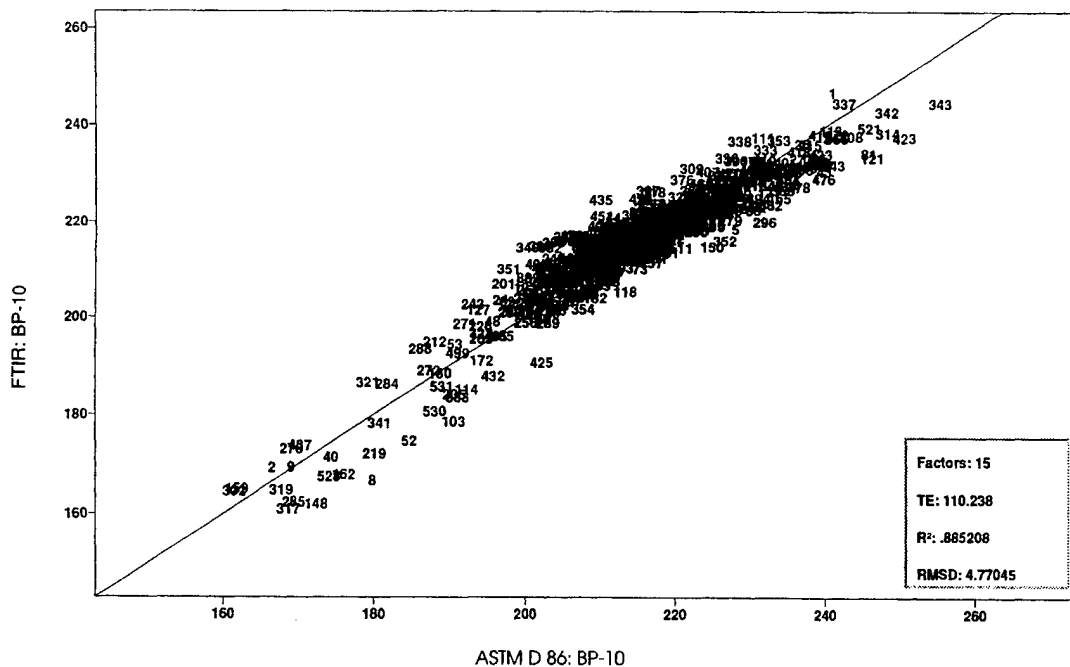


**Figure 43. Calibration for Boiling Point at 10%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

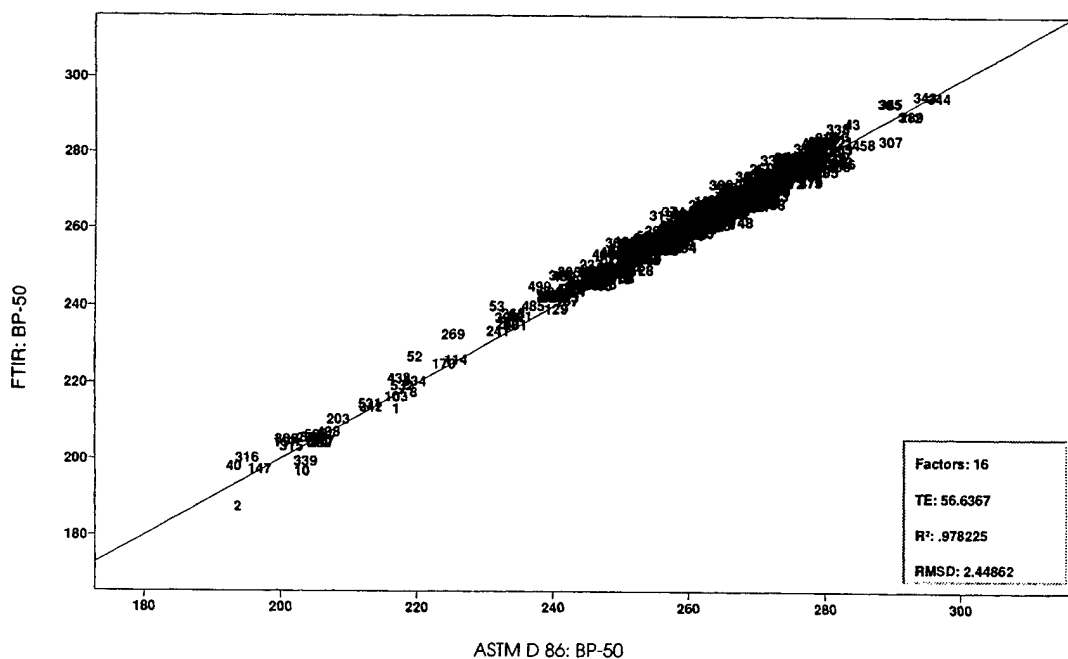


**Figure 44. Calibration for Boiling Point at 50%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

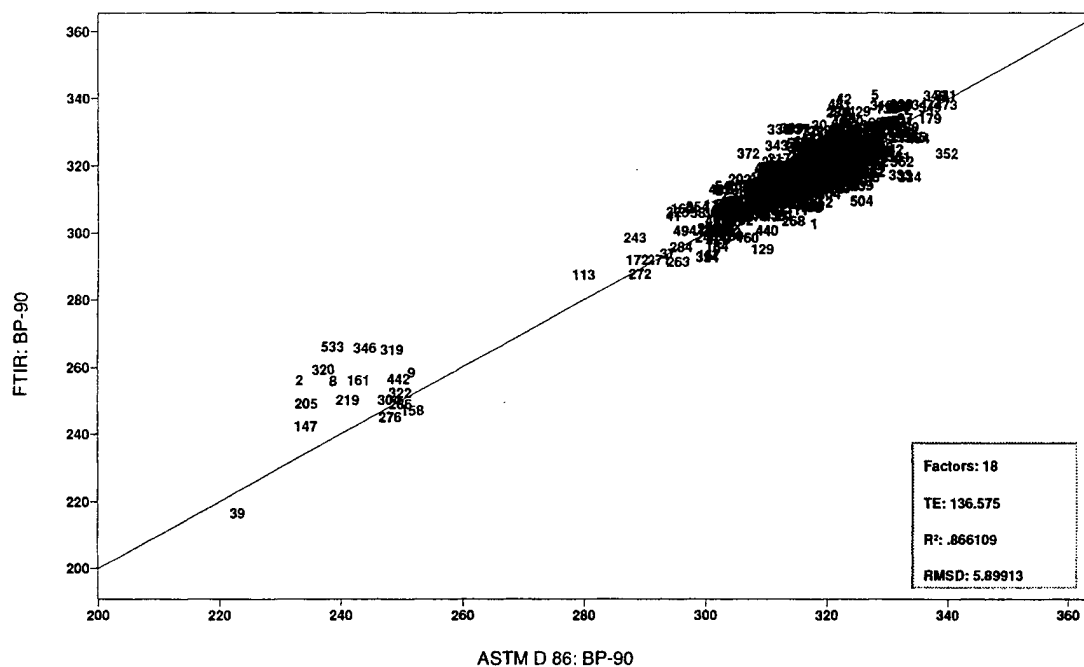


**Figure 45. Calibration for Boiling Point at 90%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

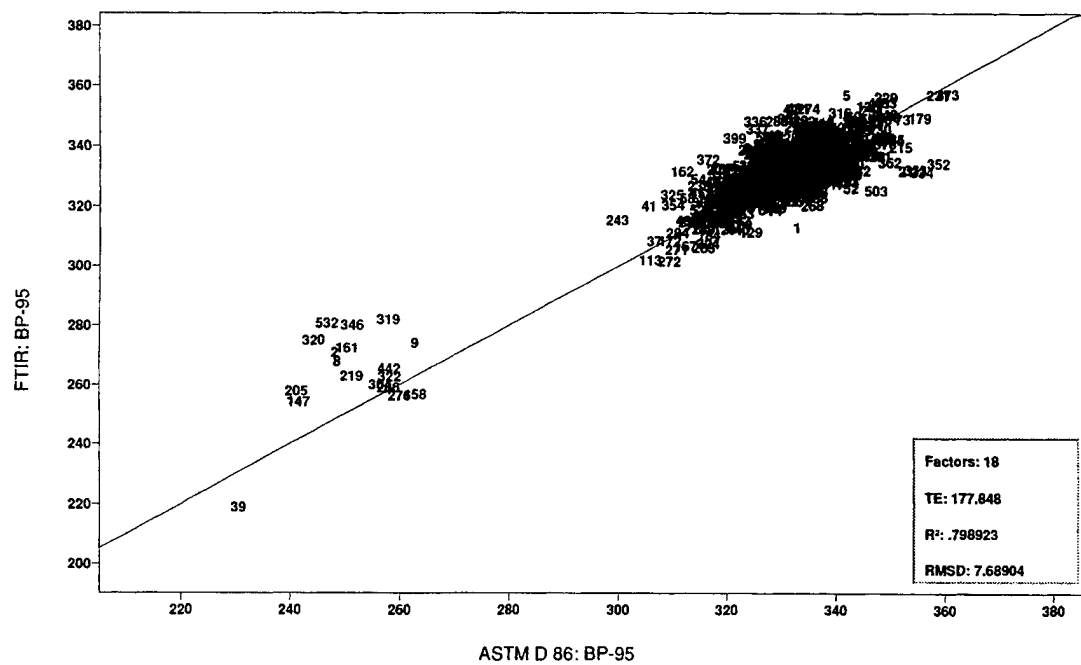


**Figure 46. Calibration for Boiling Point at 95%: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

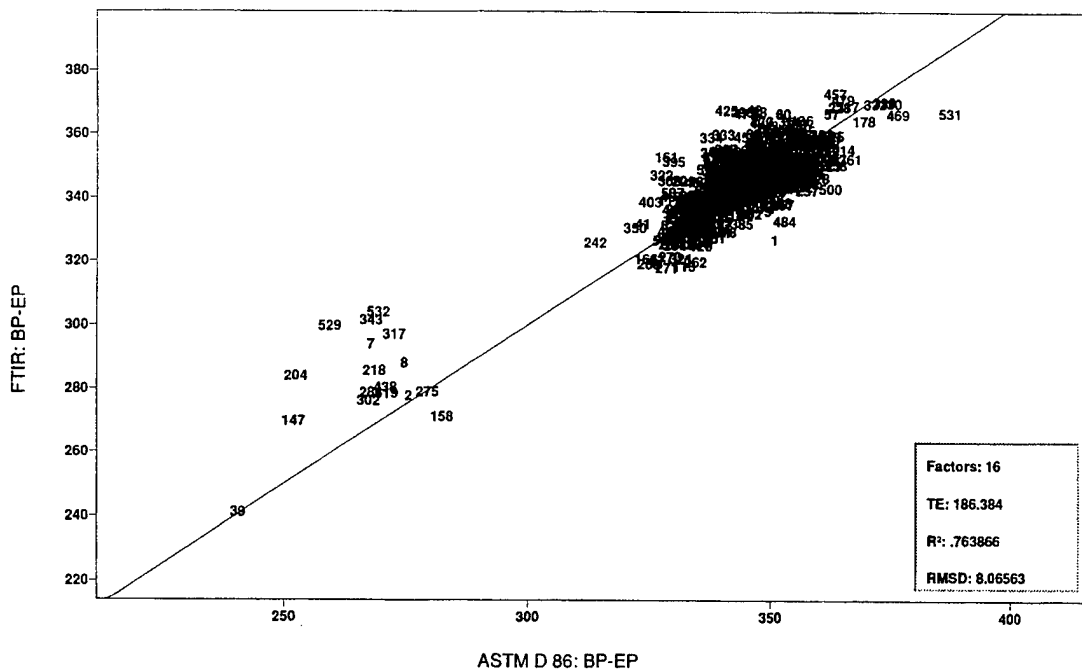


**Figure 47. Calibration for Final Boiling Point: ASTM D 86 vs FT-IR
Fuel Set D, Instrument B**

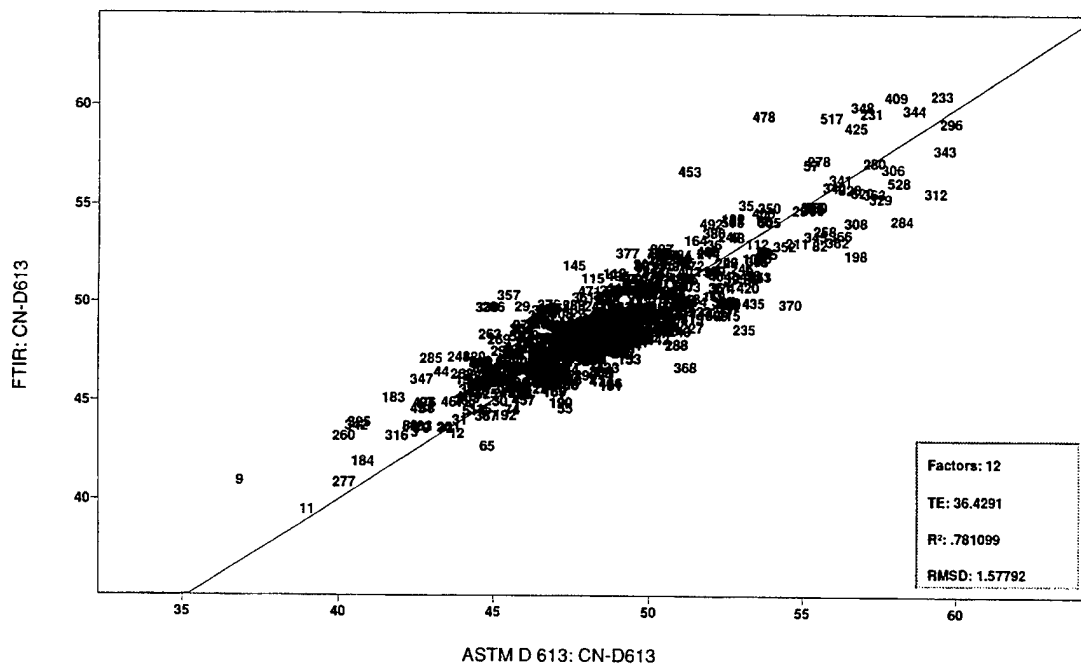


**Figure 48. Calibration for Cetane Number: ASTM D 613 vs FT-IR
Fuel Set D, Instrument B**

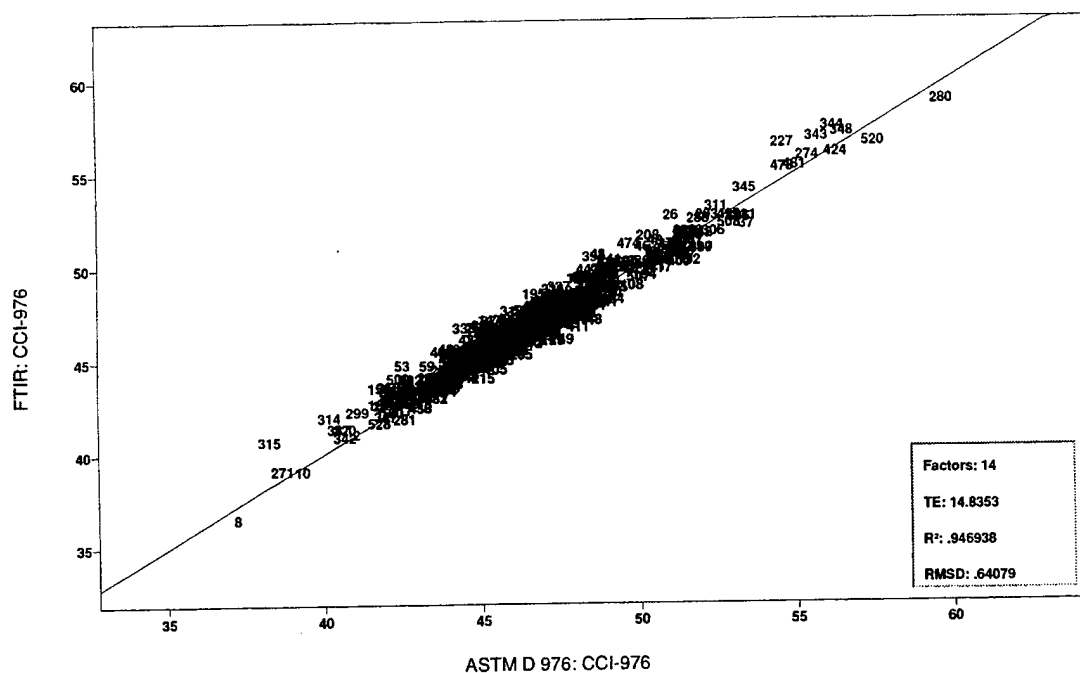


**Figure 49. Calibration for Calculated Cetane Index: ASTM D 976 vs FT-IR
Fuel Set D, Instrument B**

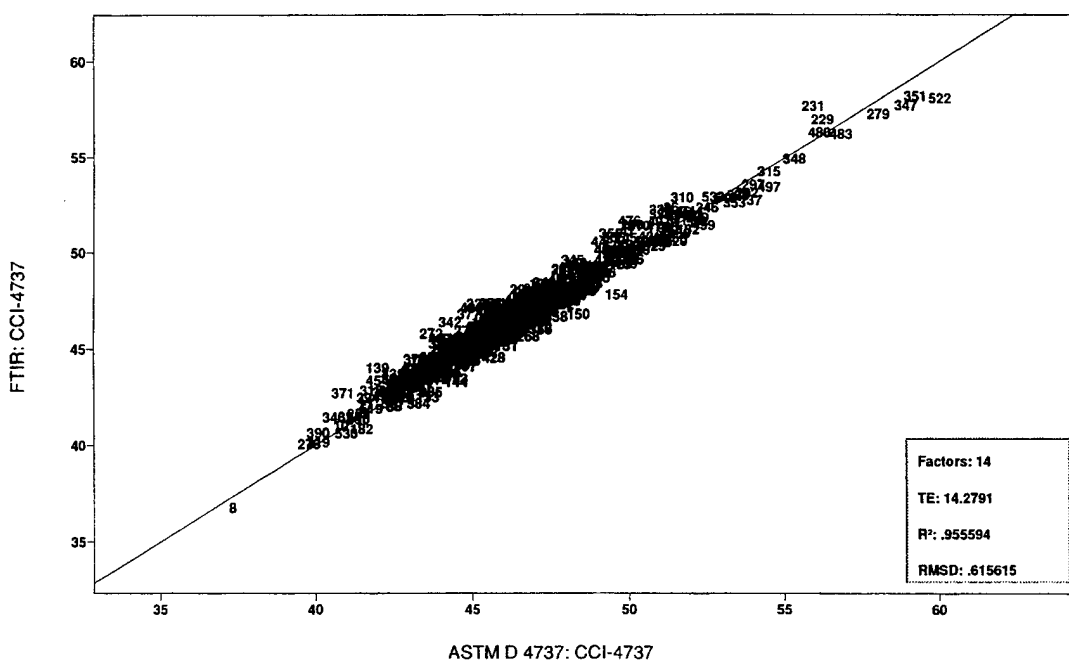


**Figure 50. Calibration for Calculated Cetane Index: ASTM D 4737 vs FT-IR
Fuel Set D, Instrument B**

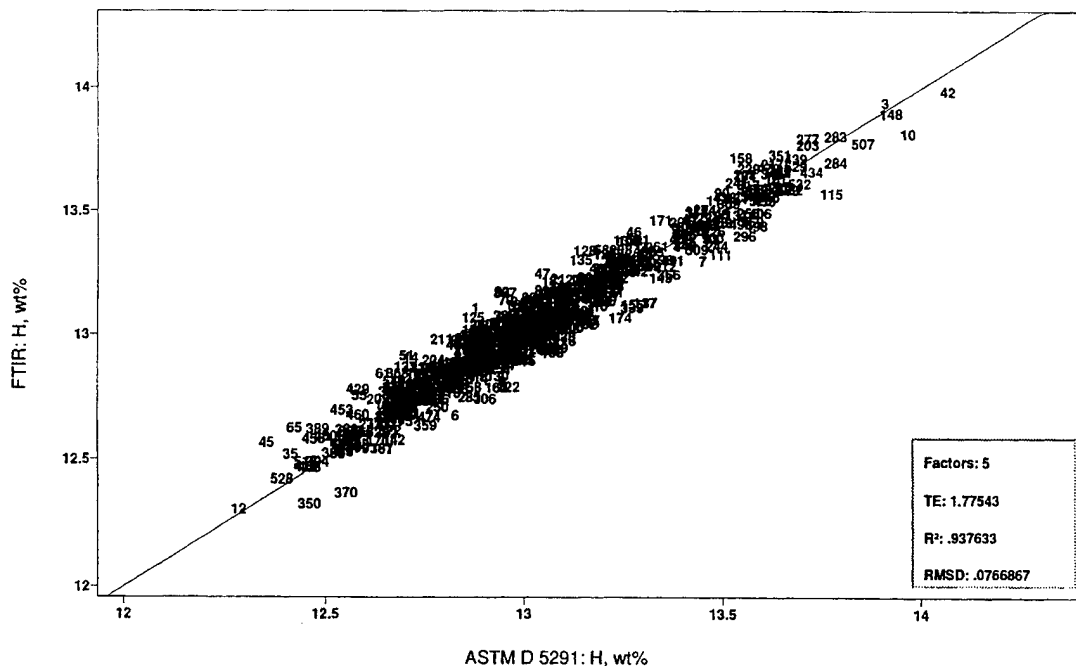


**Figure 51. Calibration for Hydrogen Content: ASTM D 5291 vs FT-IR
Fuel Set D, Instrument B**

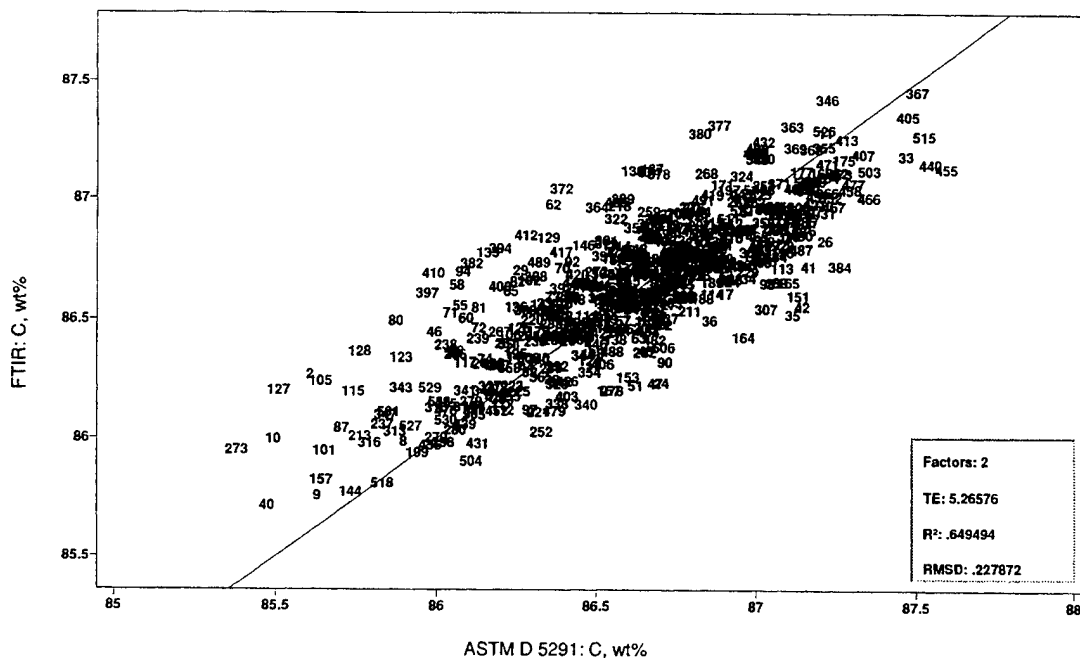


**Figure 52. Calibration for Carbon Content: ASTM D 5291 vs FT-IR
Fuel Set D, Instrument B**

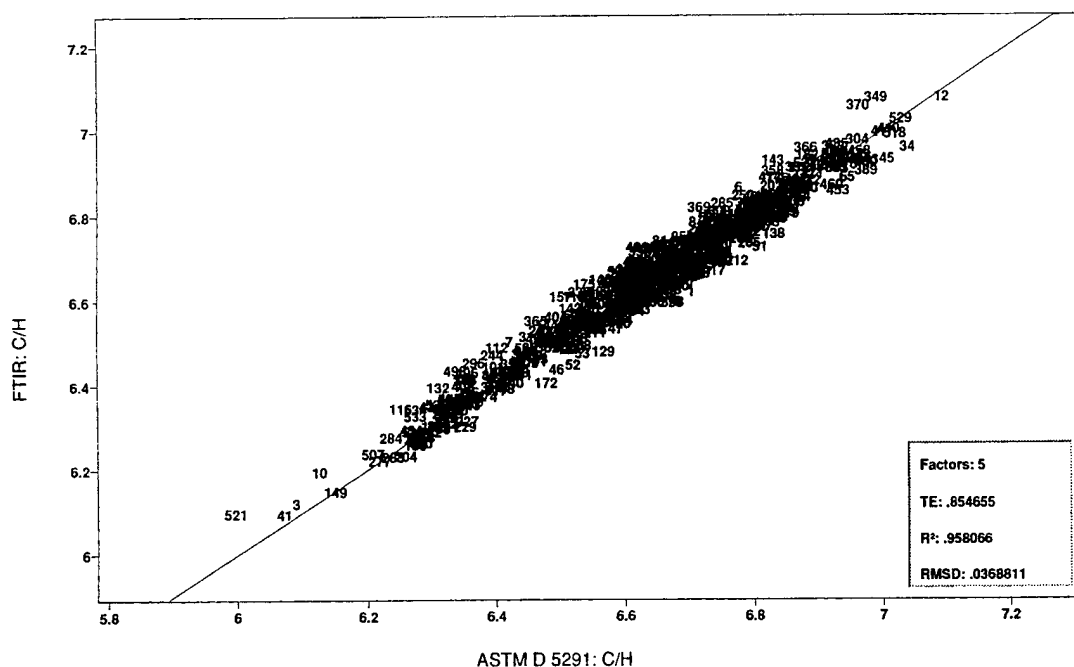


**Figure 53. Calibration for Carbon to Hydrogen Ratio: ASTM D 5291 vs FT-IR
Fuel Set D, Instrument B**

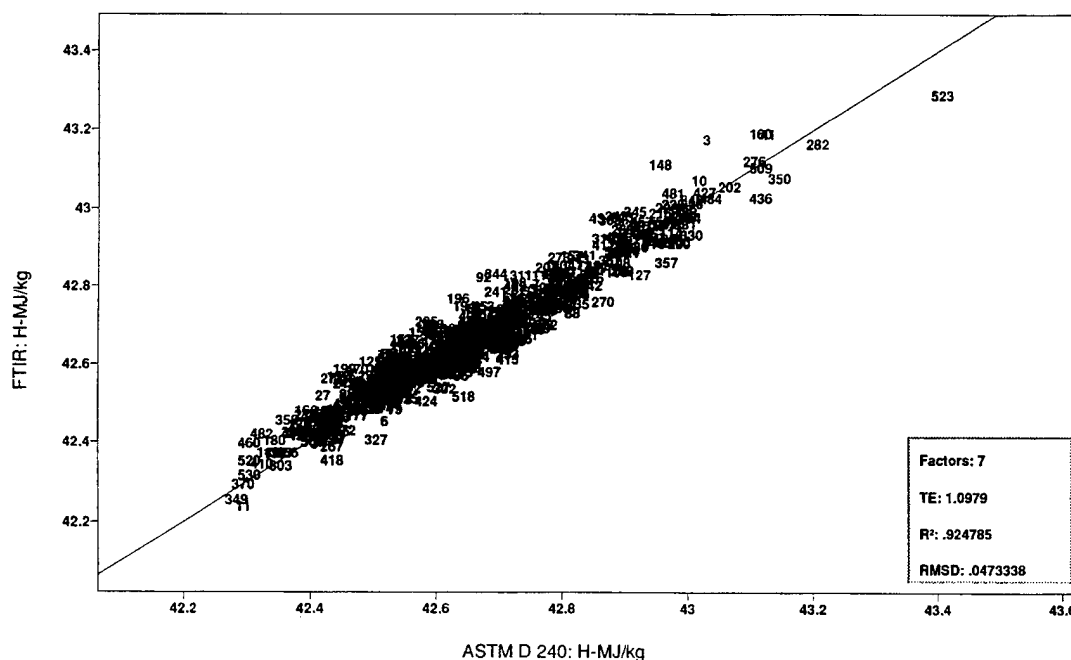


**Figure 54. Calibration for Net Heat of Combustion, MJ/kg: ASTM D 240 vs FT-IR
Fuel Set D, Instrument B**

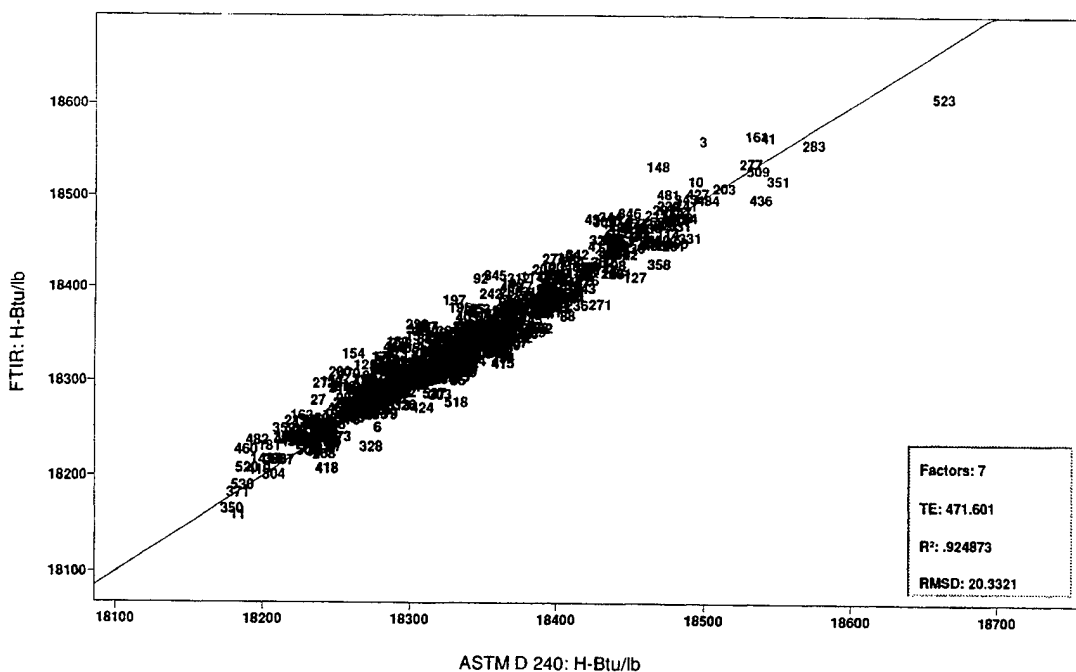


**Figure 55. Calibration for Net Heat of Combustion, Btu/lb: ASTM D 240 vs FT-IR
Fuel Set D, Instrument B**

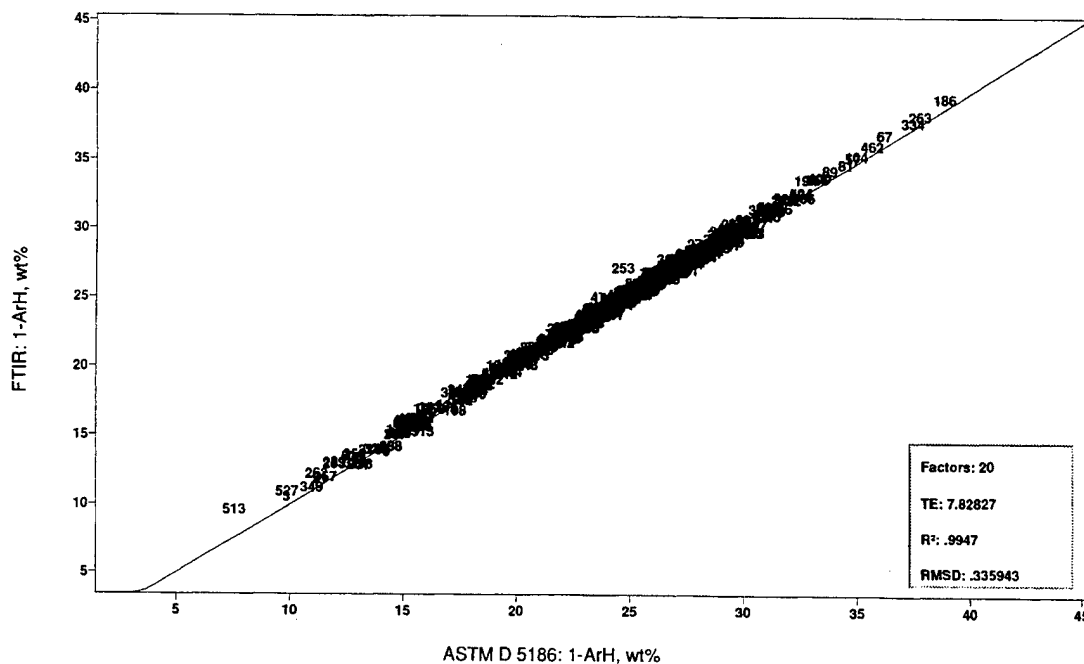


**Figure 56. Calibration for Monocyclic Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument B**

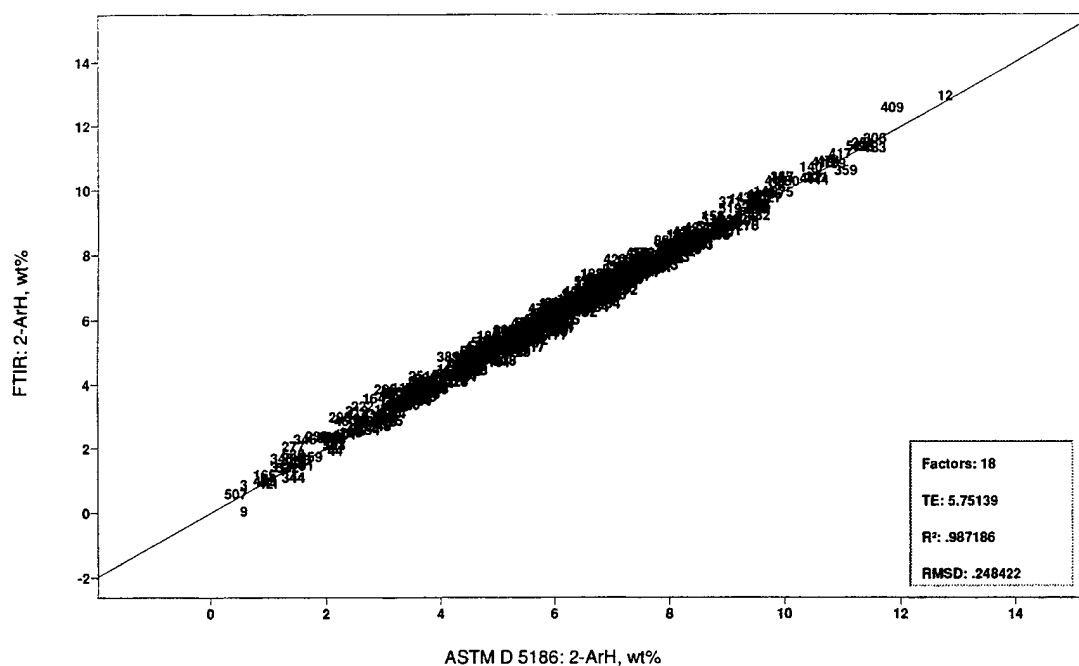


**Figure 57. Calibration for Dicyclic Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument B**

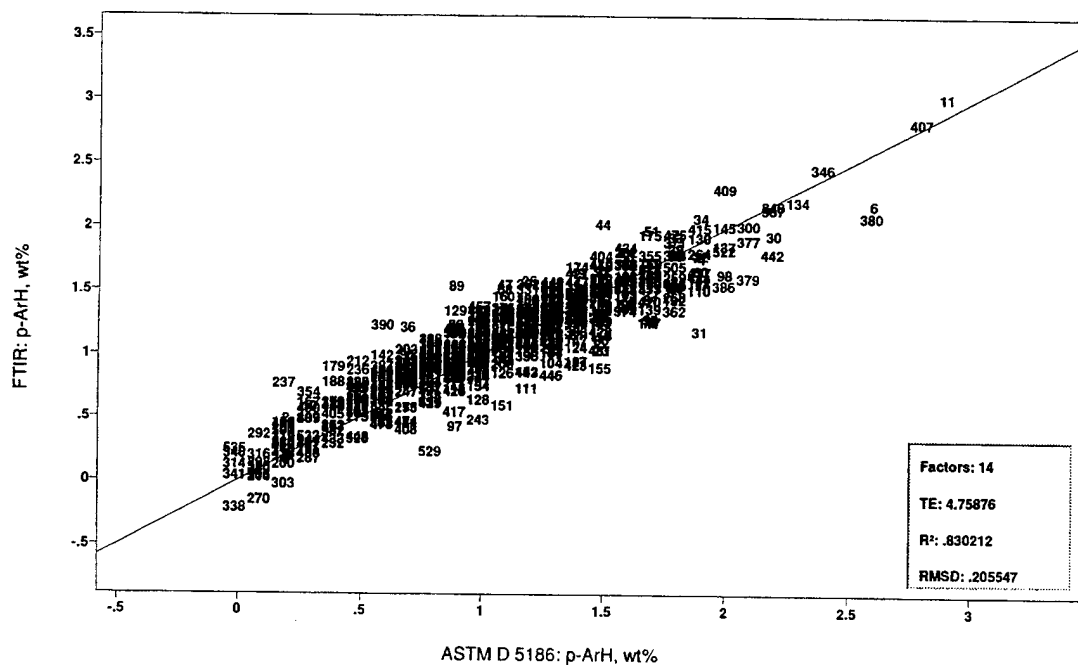


**Figure 58. Calibration for Polycyclic Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument B**

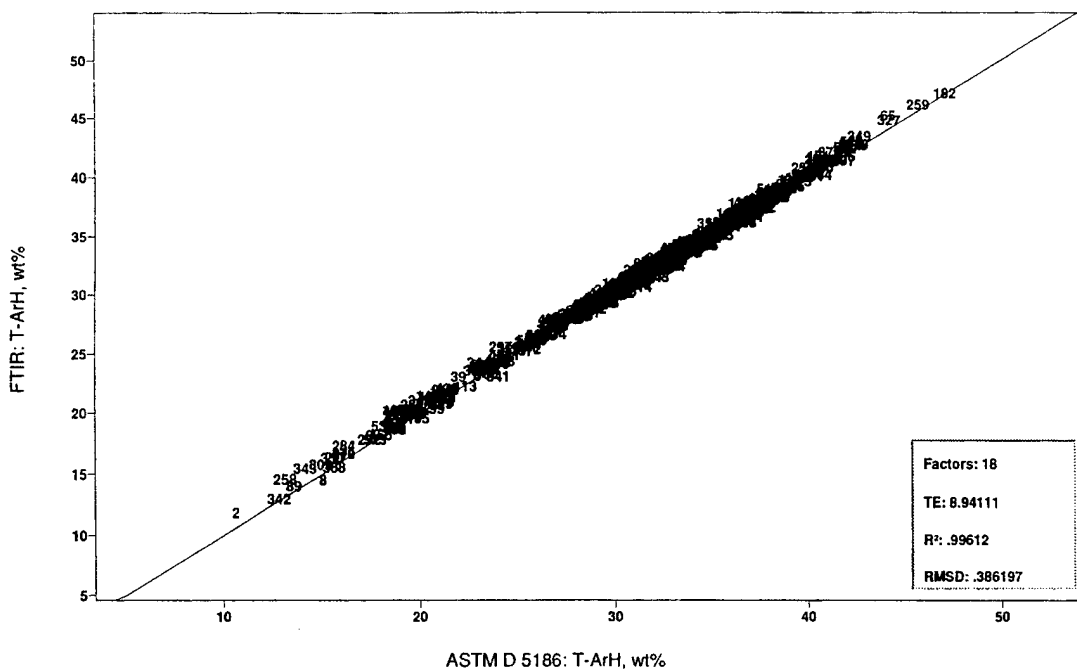


**Figure 59. Calibration for Total Aromatics: ASTM D 5186 vs FT-IR
Fuel Set D, Instrument B**

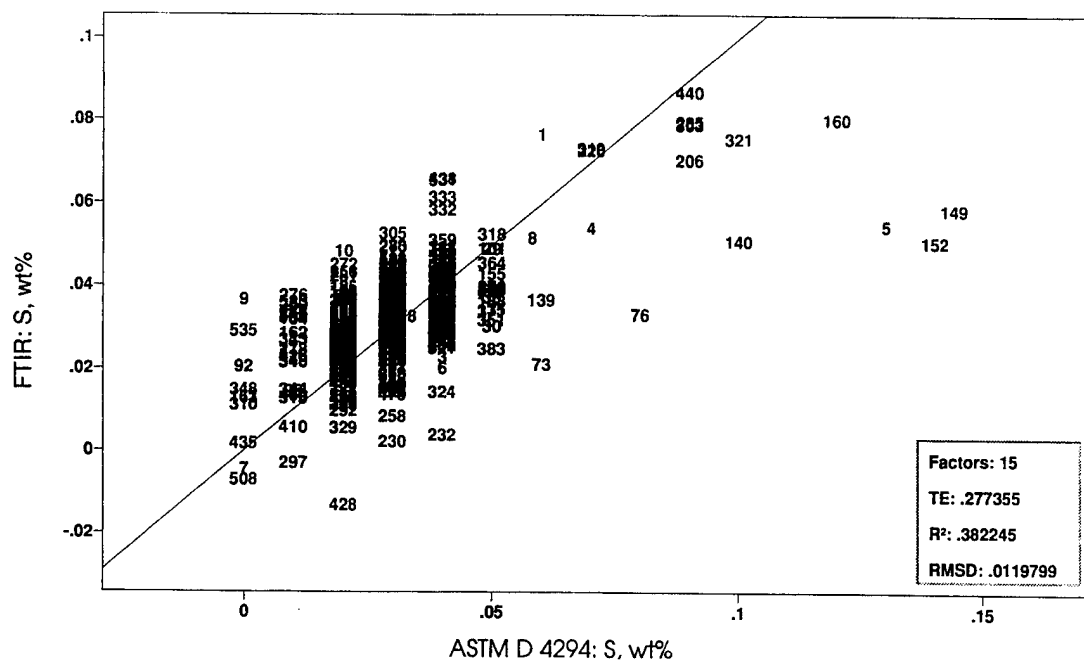


**Figure 60. Calibration for Sulfur Content: ASTM D 4294 vs FT-IR
Fuel Set D, Instrument B**

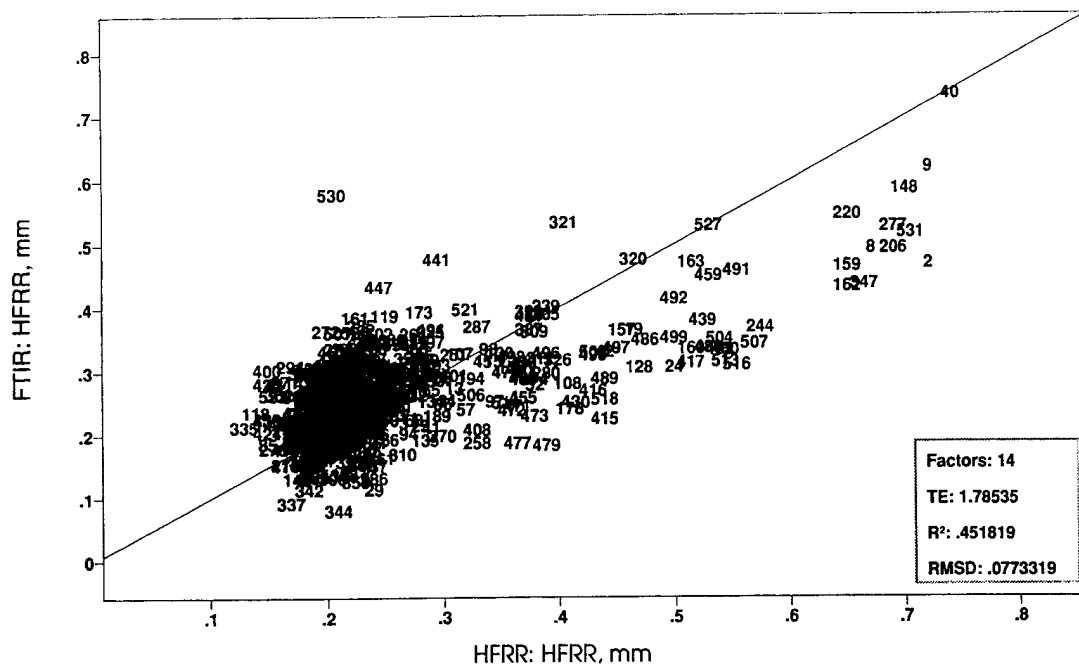


Figure 61. Calibration for Lubricity by HFRR: ISO/CD12156 vs FT-IR Fuel Set D, Instrument B

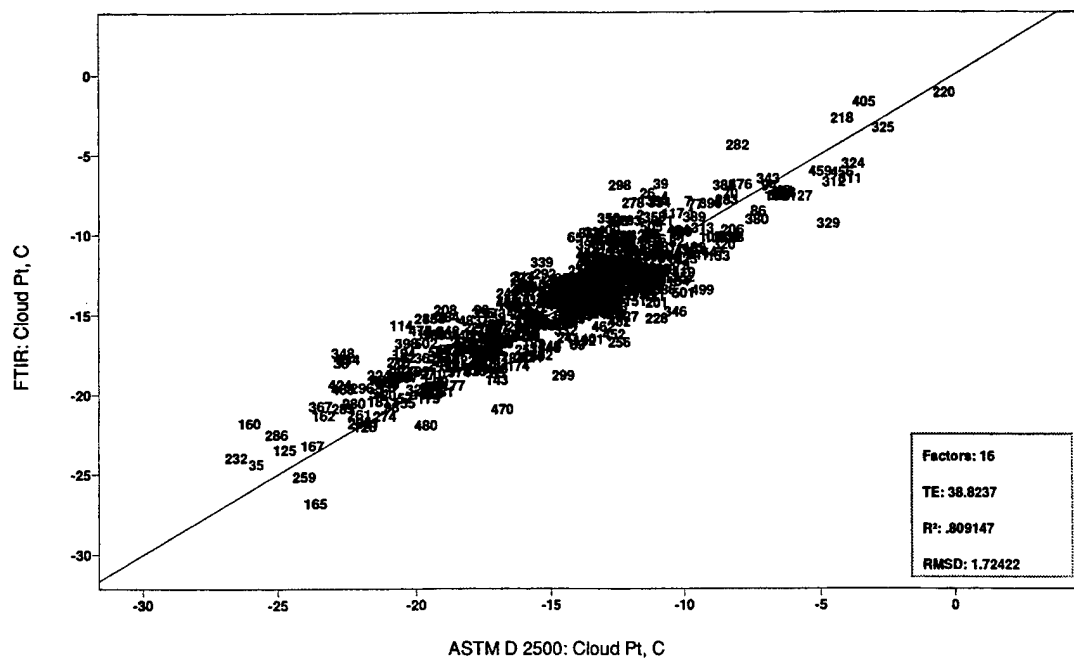


Figure 62. Calibration for Cloud Point for Diesel Fuels: ASTM D 2500 vs FT-IR Fuel Set D, Instrument B

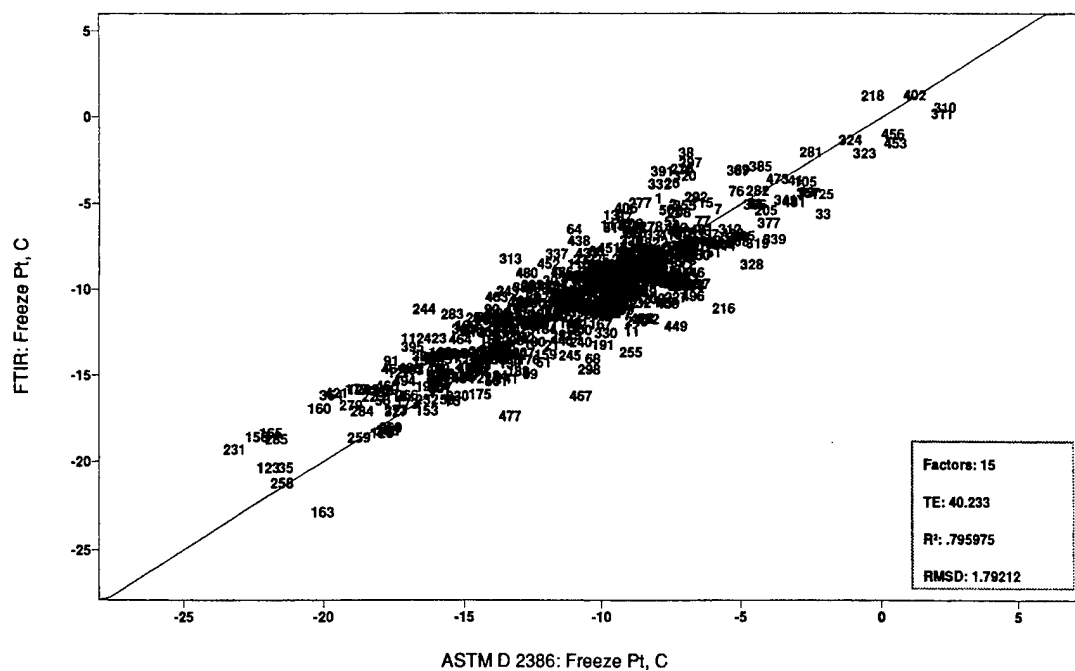


Figure 63. Calibration for Freeze Point for Diesel Fuels: ASTM D 2386 vs FT-IR Fuel Set D, Instrument B

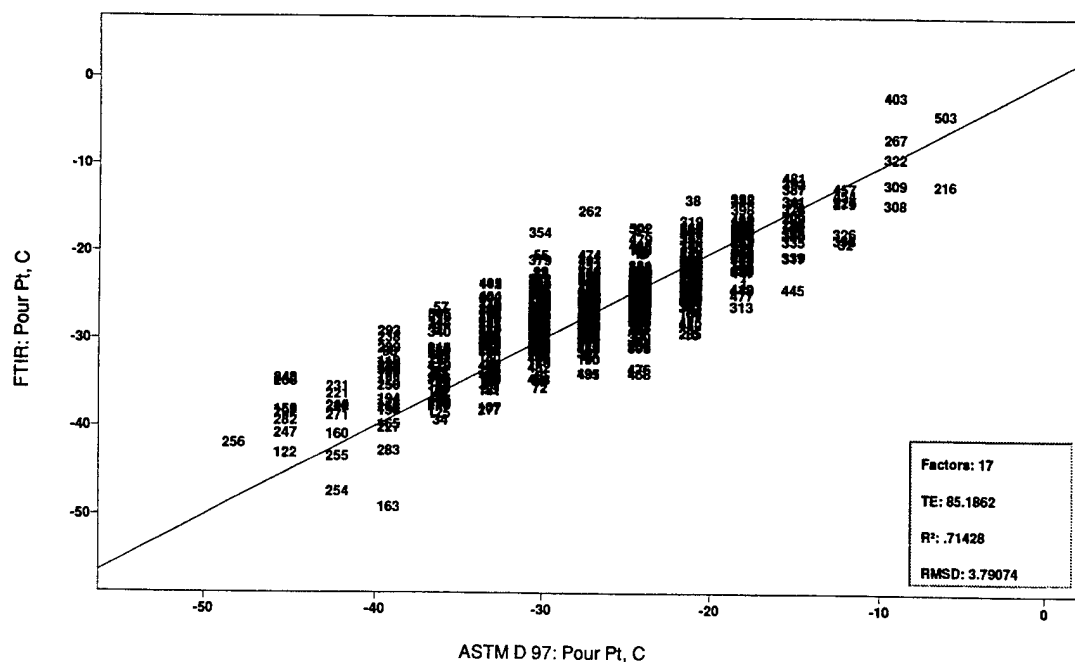


Figure 64. Calibration for Pour Point for Diesel Fuels: ASTM D 97 vs FT-IR Fuel Set D, Instrument B

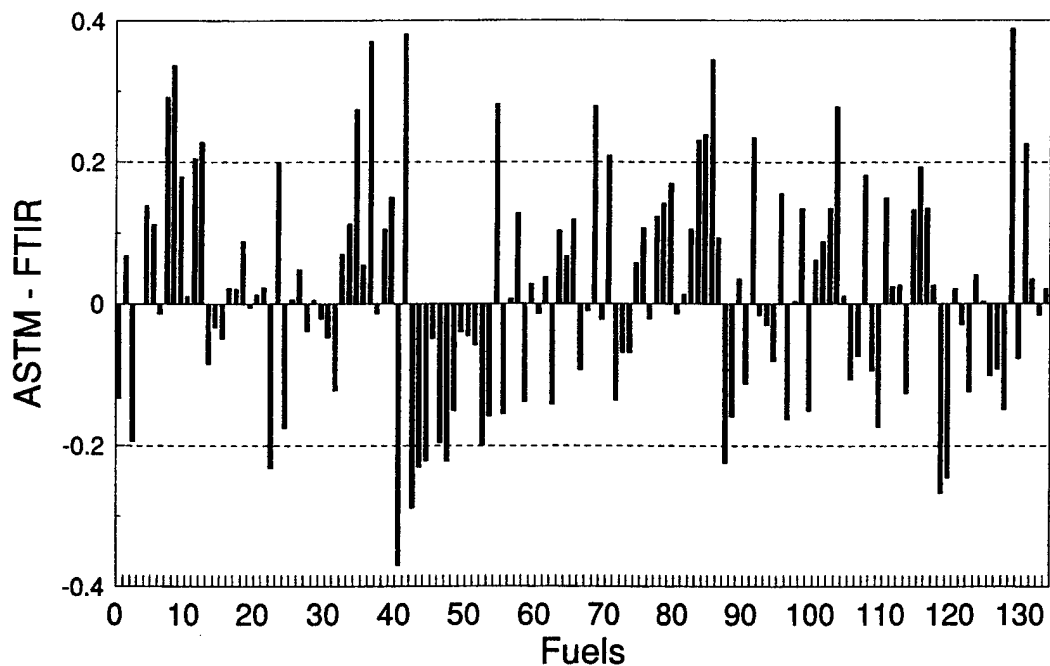


Figure 65. Residual Error in API Gravity Validation: ASTM D 1298
Fuel Set D, Instrument B

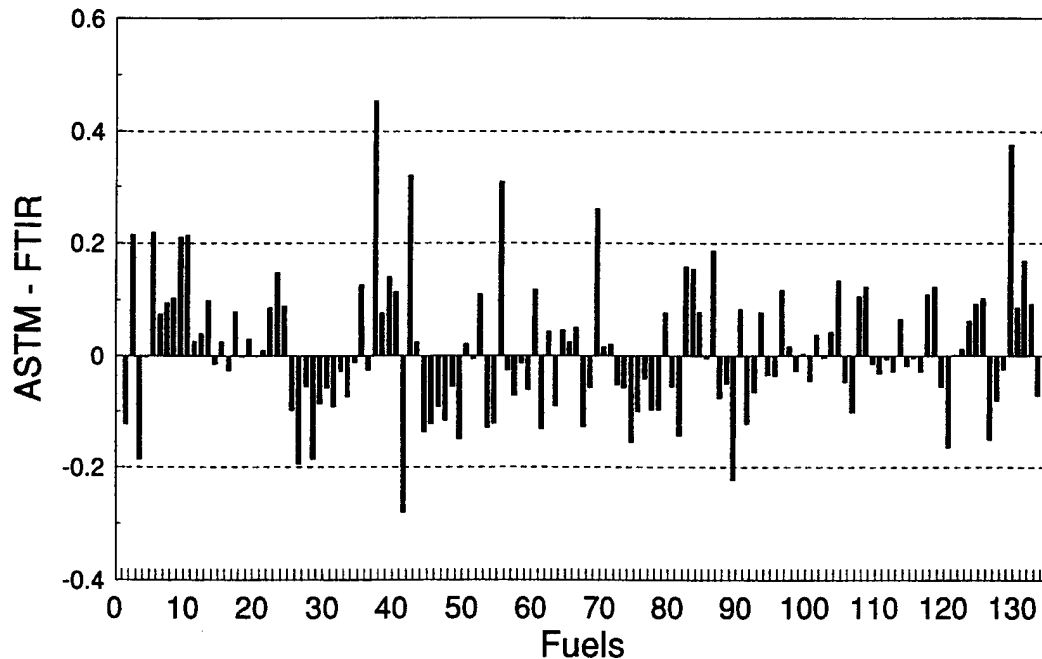


Figure 66. Residual Error in API Gravity Validation: ASTM D 4052
Fuel Set D, Instrument B

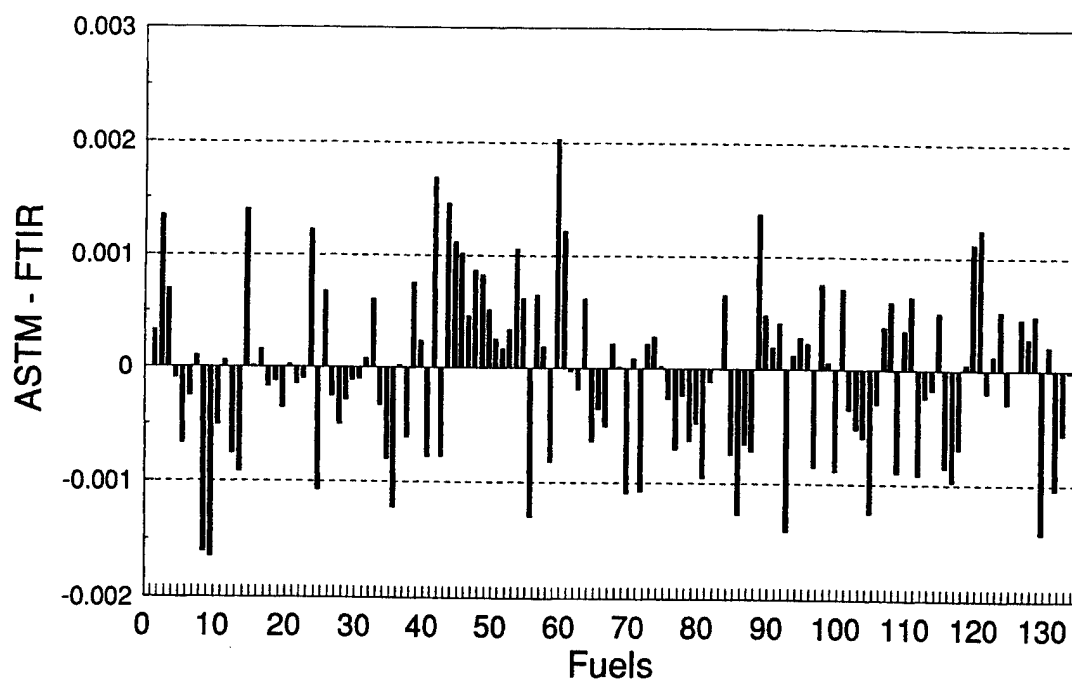


Figure 67. Residual Error in Density Validation: ASTM D 1298
Fuel Set D, Instrument B

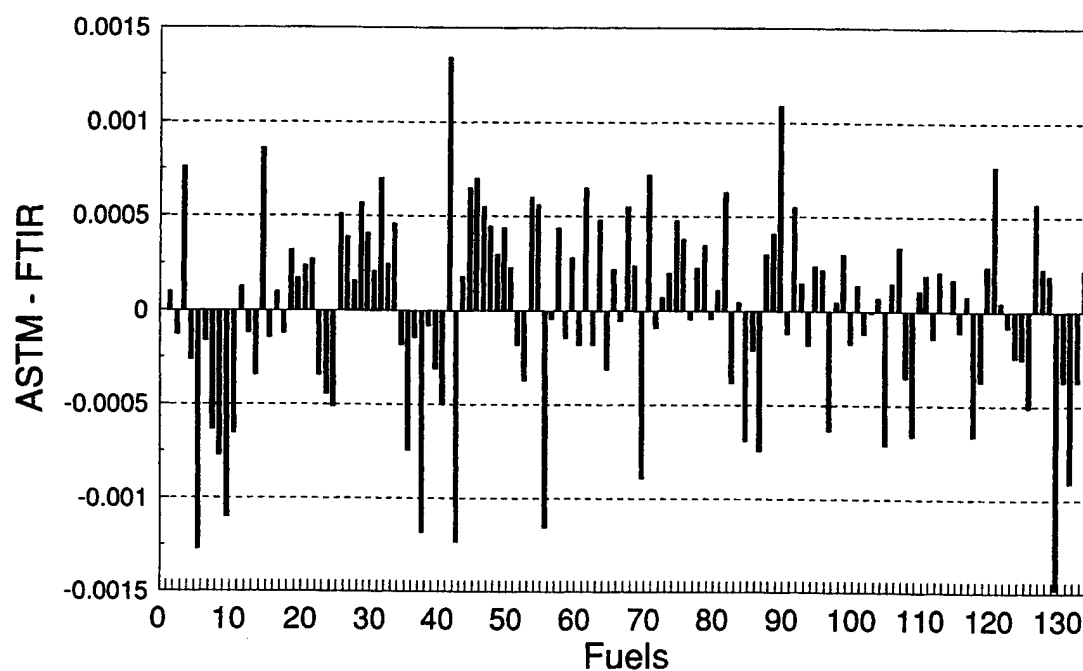


Figure 68. Residual Error in Density Validation: ASTM D 4052
Fuel Set D, Instrument B

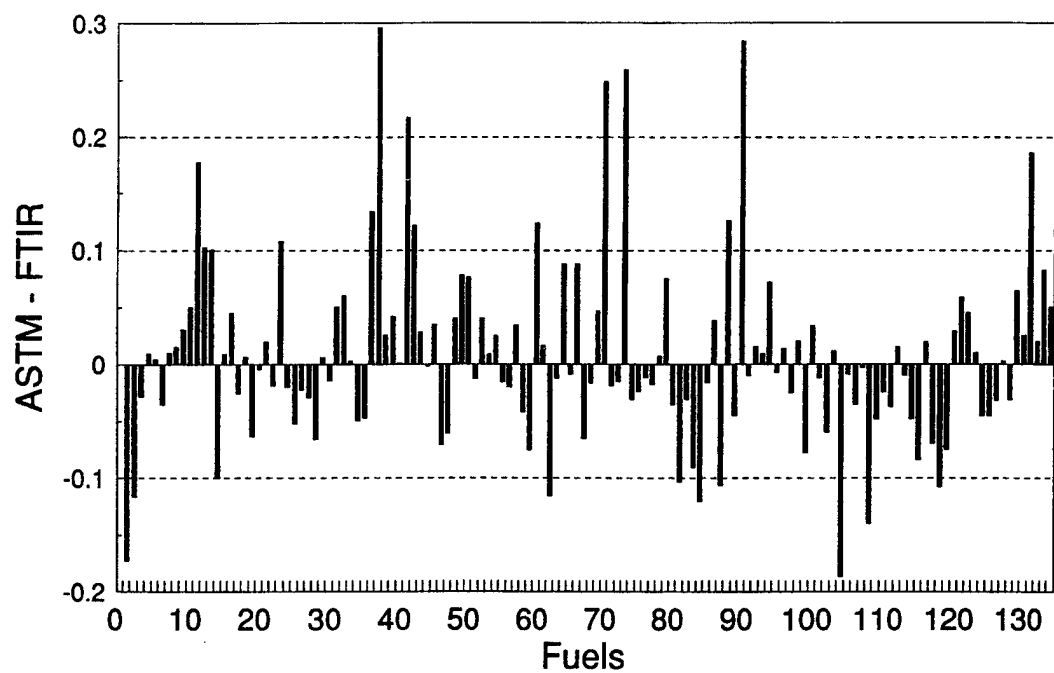


Figure 69. Residual Error in Kinematic Viscosity Validation, cSt, °C: ASTM D 445
Fuel Set D, Instrument B

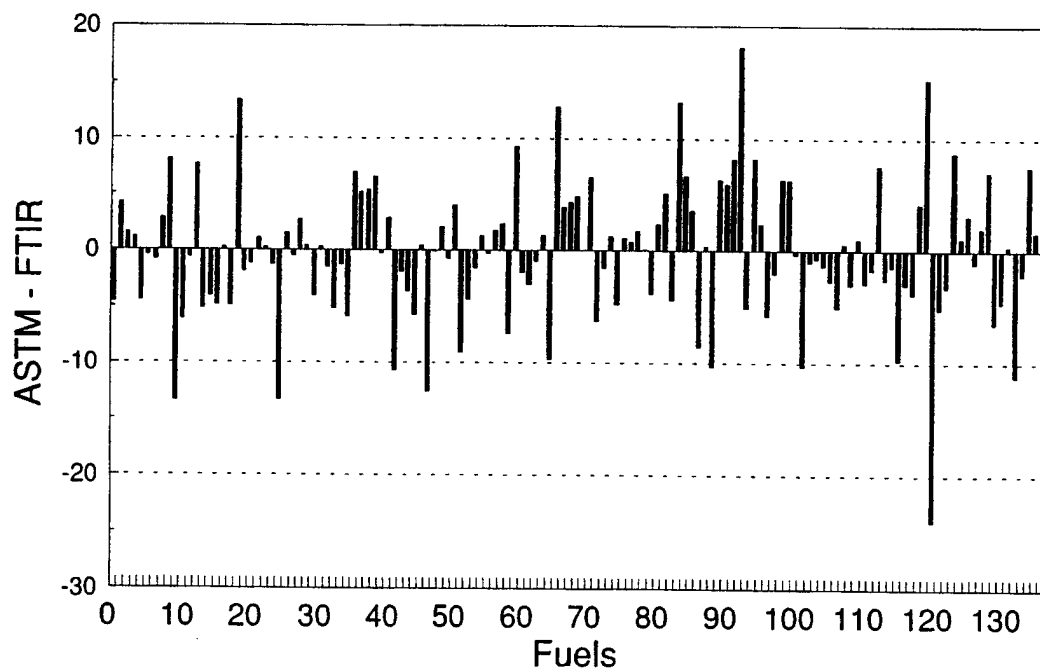


Figure 70. Residual Error in Flash Point Validation: ASTM D 93
Fuel Set D, Instrument B

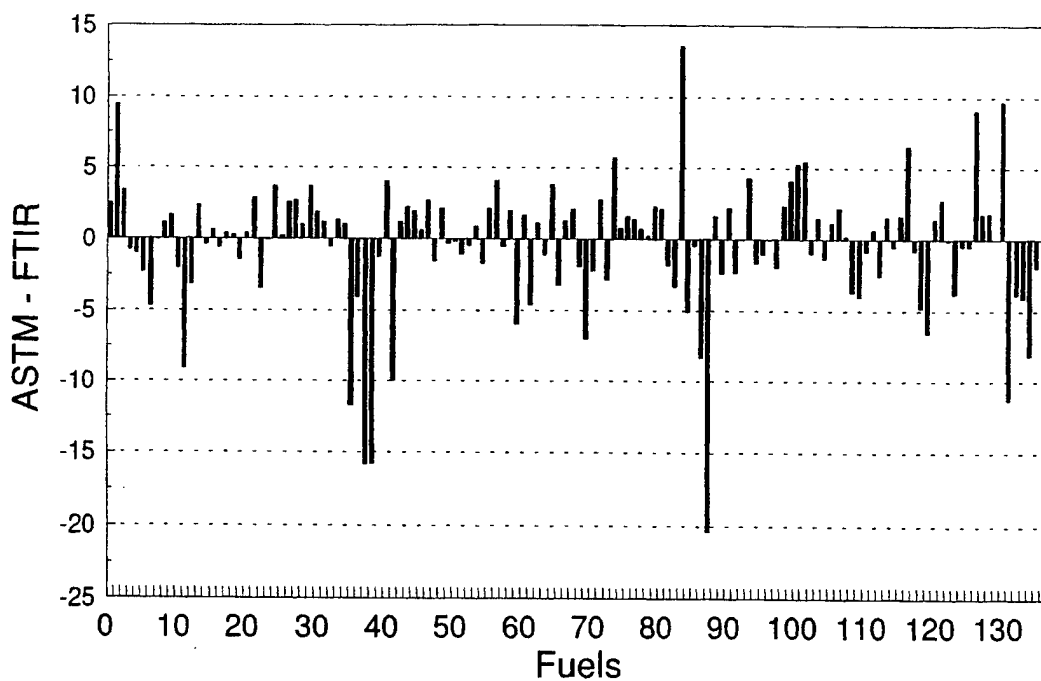


Figure 71. Residual Error in Cloud Point Validation: ASTM D 2500
Fuel Set D, Instrument B

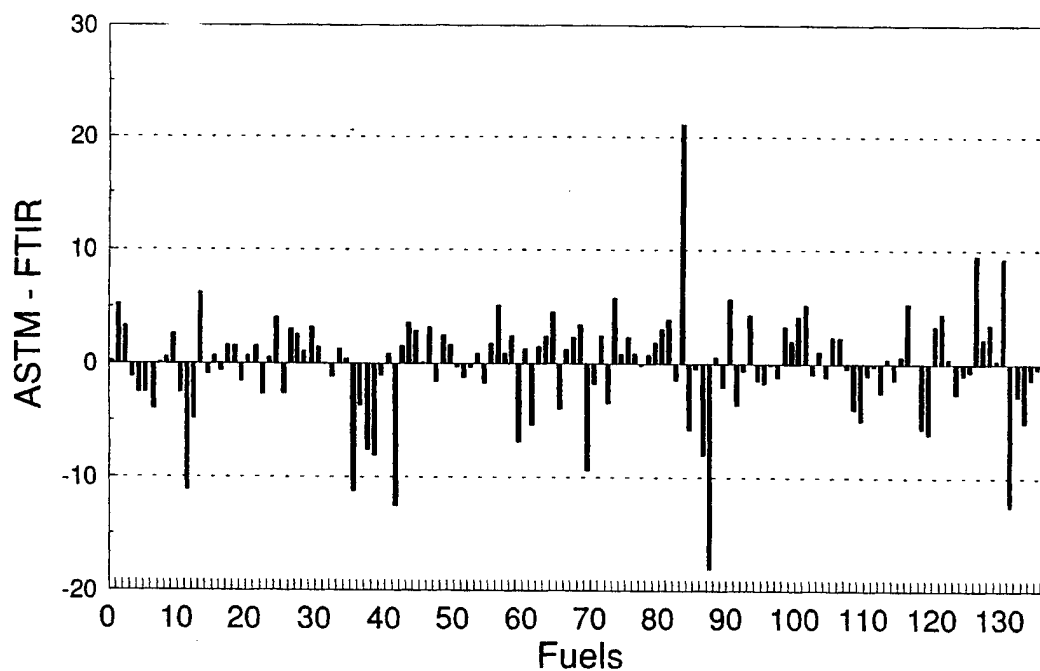


Figure 72. Residual Error in Freeze Point Validation: ASTM D 2386
Fuel Set D, Instrument B

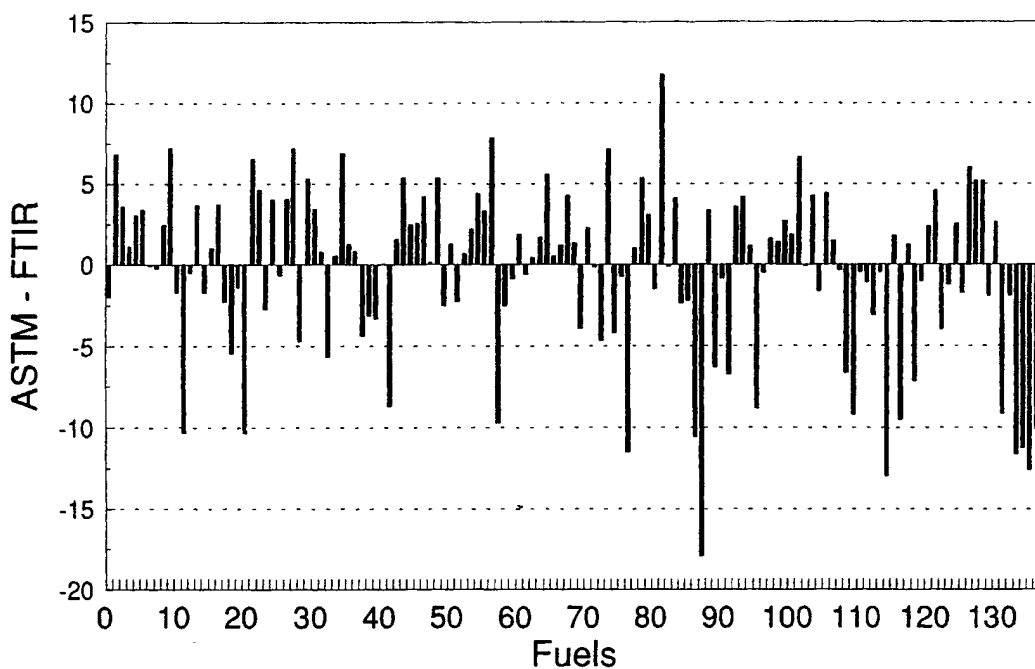


Figure 73. Residual Error in Pour Point Validation: ASTM D 97
Fuel Set D, Instrument B

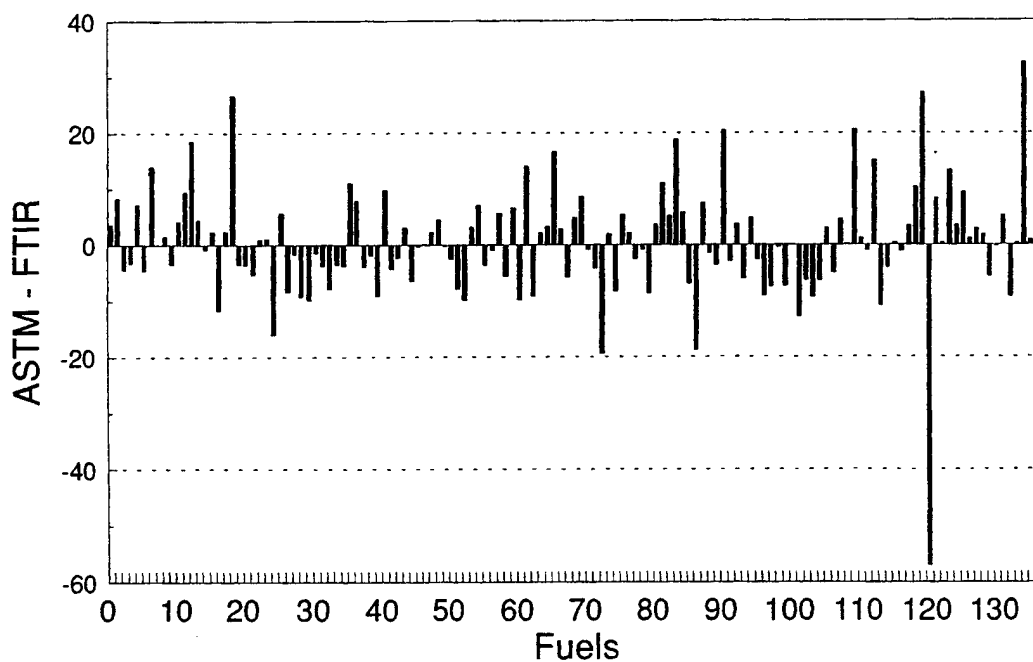


Figure 74. Residual Error in Initial Boiling Point Validation: ASTM D 86
Fuel Set D, Instrument B

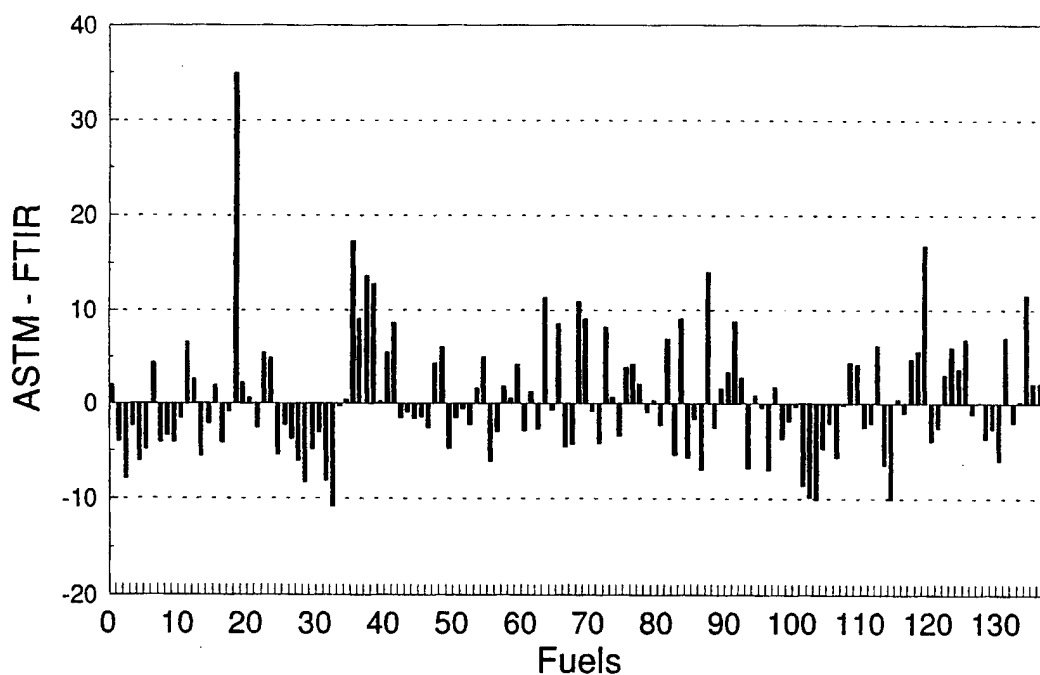


Figure 75. Residual Error in Boiling Point at 10% Validation: ASTM D 86
Fuel Set D, Instrument B

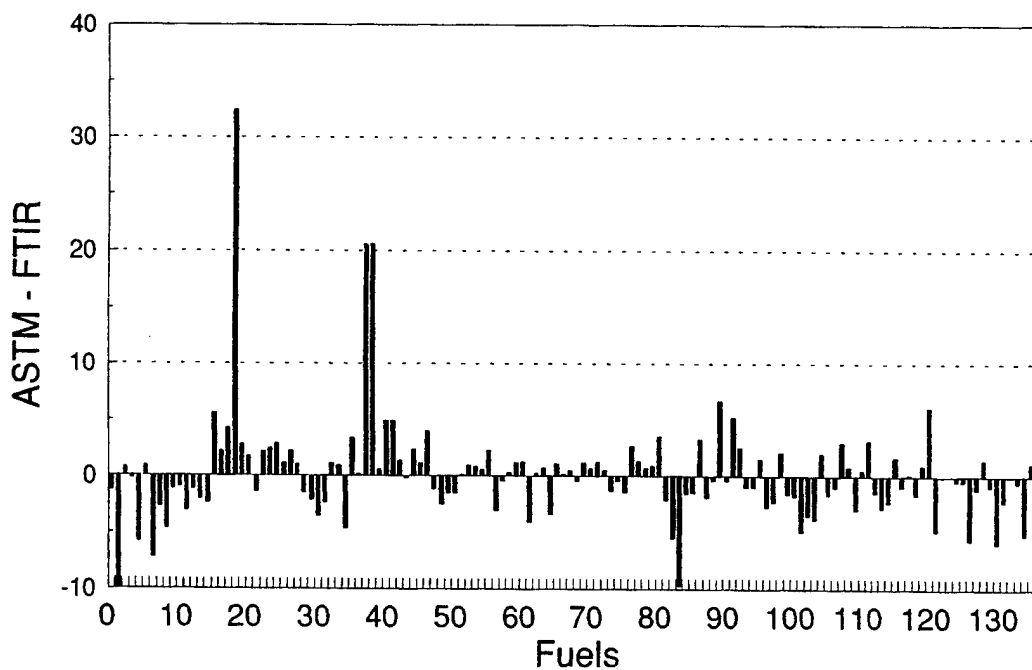


Figure 76. Residual Error in Boiling Point at 50% Validation: ASTM D 86
Fuel Set D, Instrument B

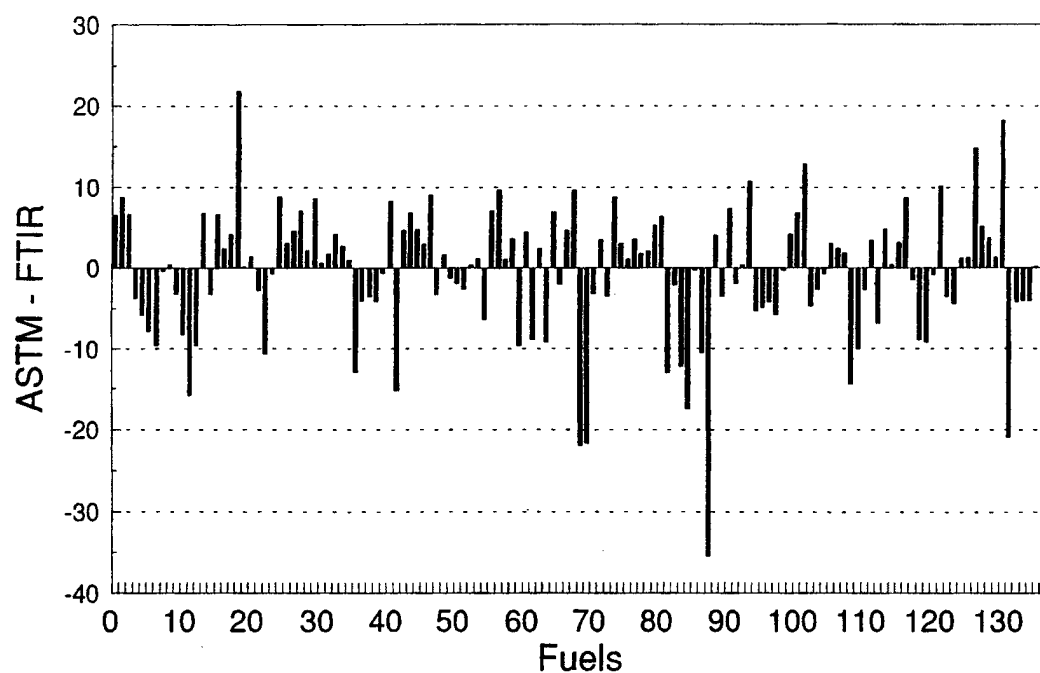


Figure 77. Residual Error in Boiling Point at 90% Validation: ASTM D 86
Fuel Set D, Instrument B

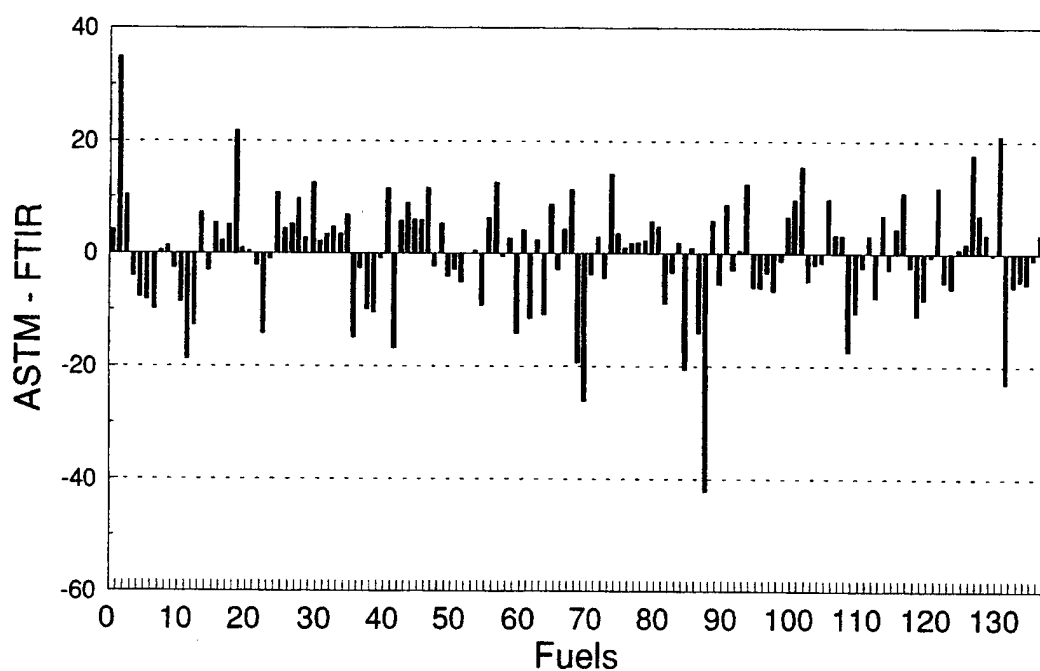


Figure 78. Residual Error in Boiling Point at 95% Validation: ASTM D 86
Fuel Set D, Instrument B

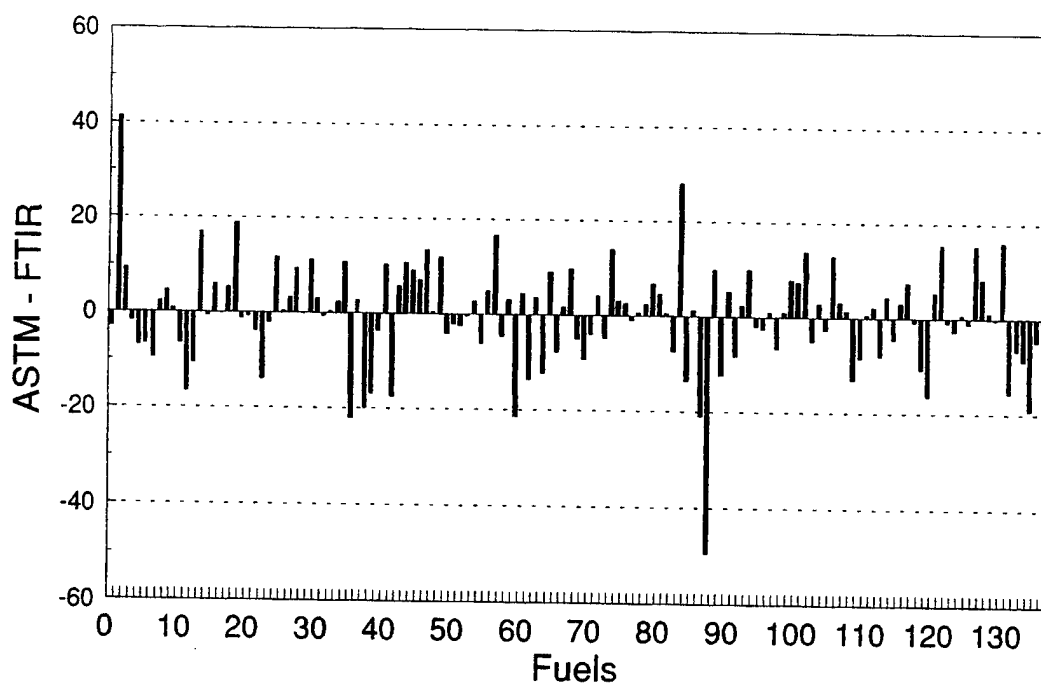


Figure 79. Residual Error in Final Boiling Point Validation: ASTM D 86
Fuel Set D, Instrument B

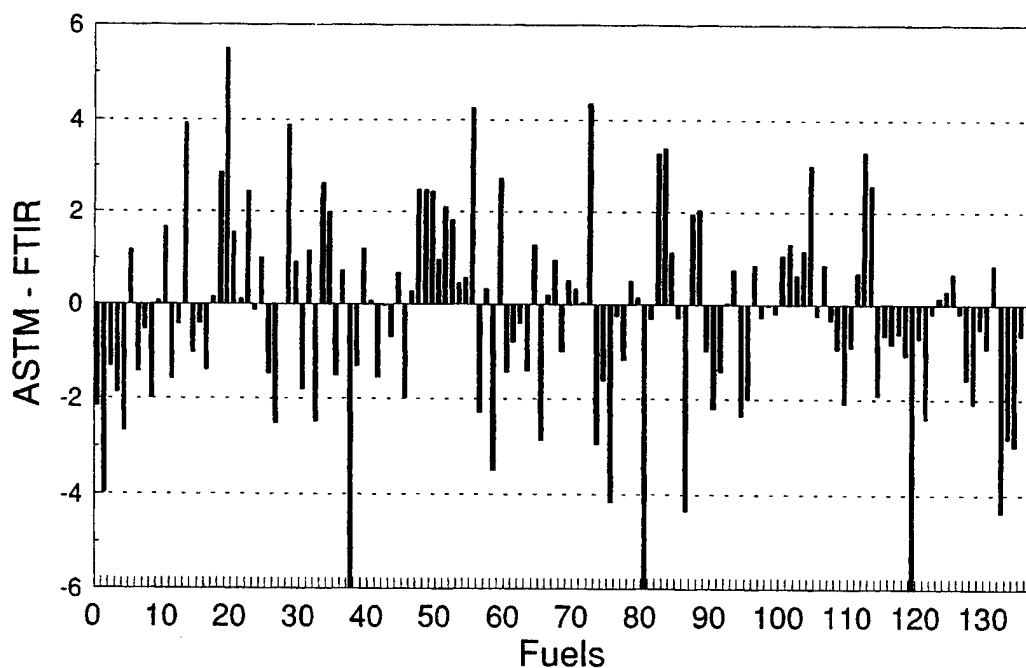


Figure 80. Residual Error in Cetane Number Validation: ASTM D 613
Fuel Set D, Instrument B

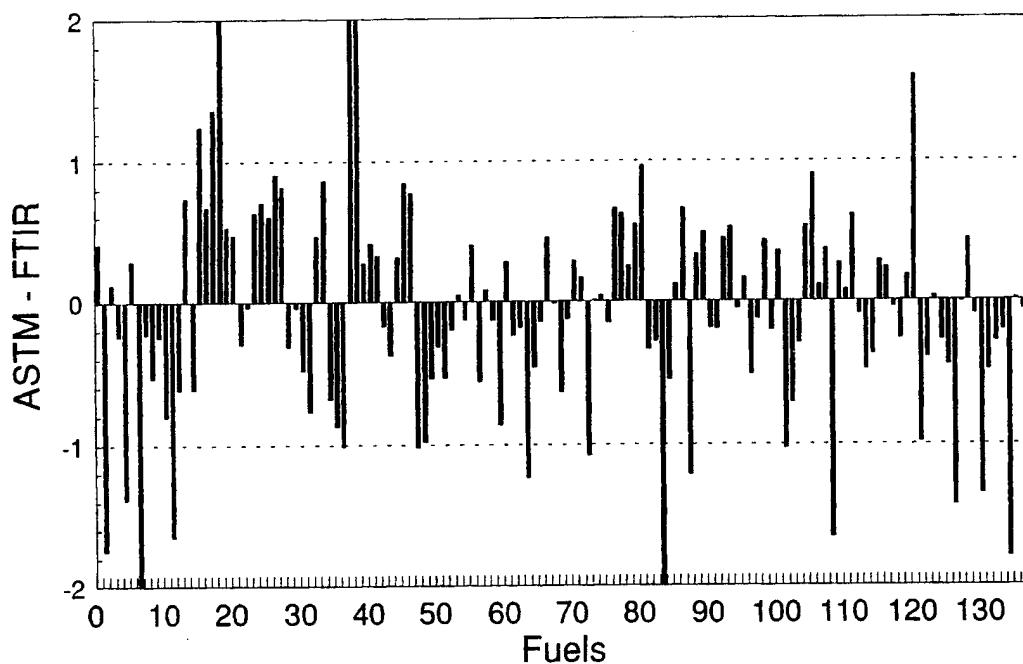


Figure 81. Residual Error in Calculated Cetane Index Validation: ASTM D 976
Fuel Set D, Instrument B

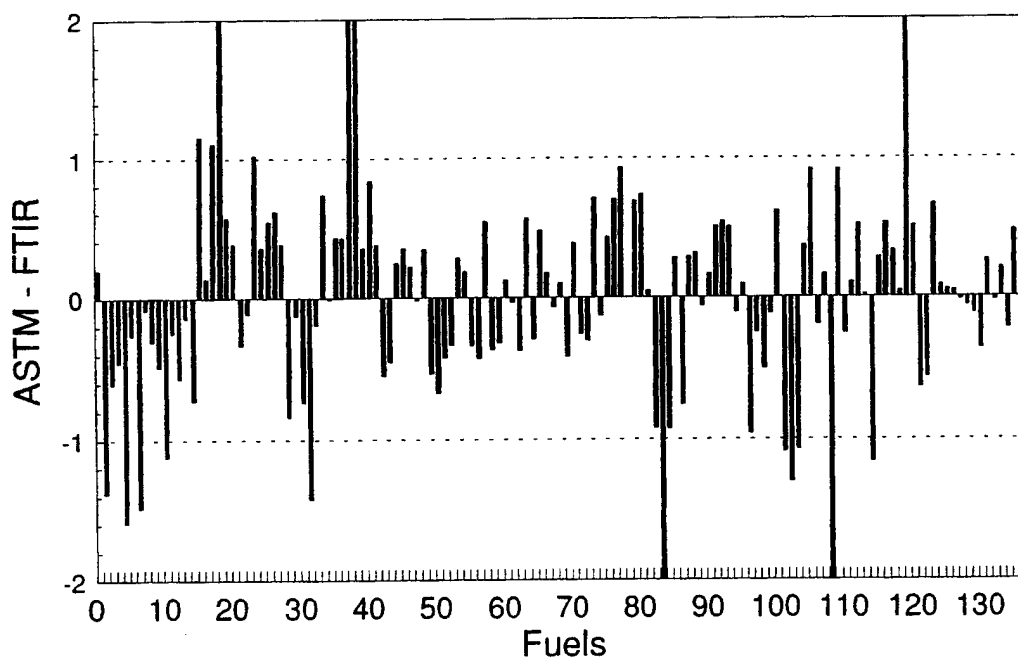


Figure 82. Residual Error in Calculated Cetane Index Validation: ASTM D 4737
Fuel Set D, Instrument B

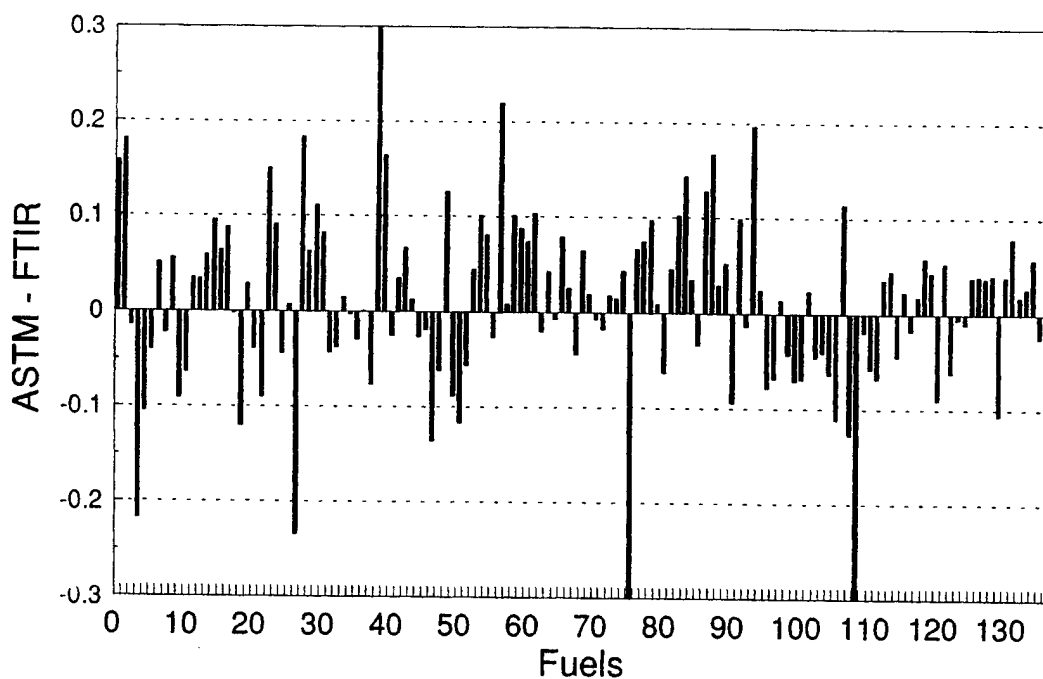


Figure 83. Residual Error in Hydrogen Content Validation: ASTM D 5291
Fuel Set D, Instrument B

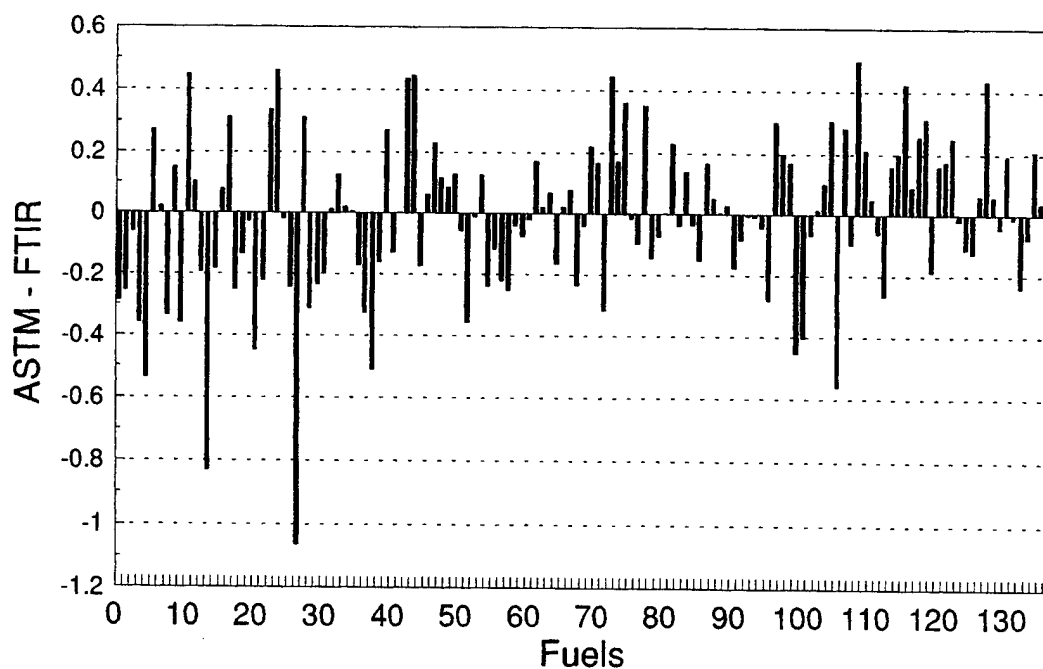


Figure 84. Residual Error in Carbon Content Validation: ASTM D 5291
Fuel Set D, Instrument B

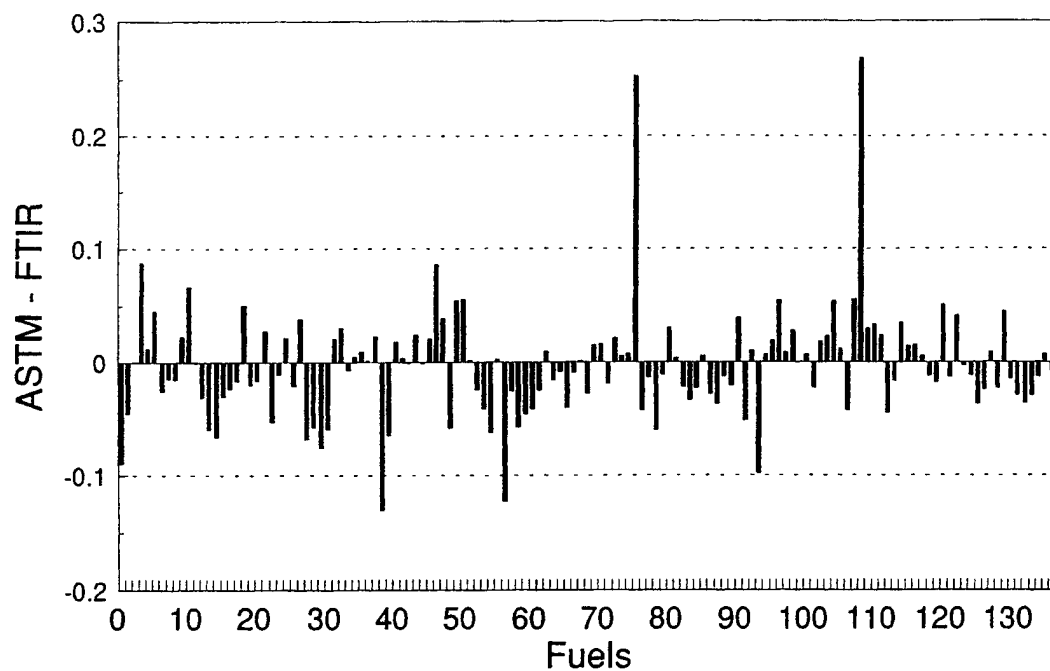


Figure 85. Residual Error in Carbon-to-Hydrogen Ratio Validation: ASTM D 5291
Fuel Set D, Instrument B

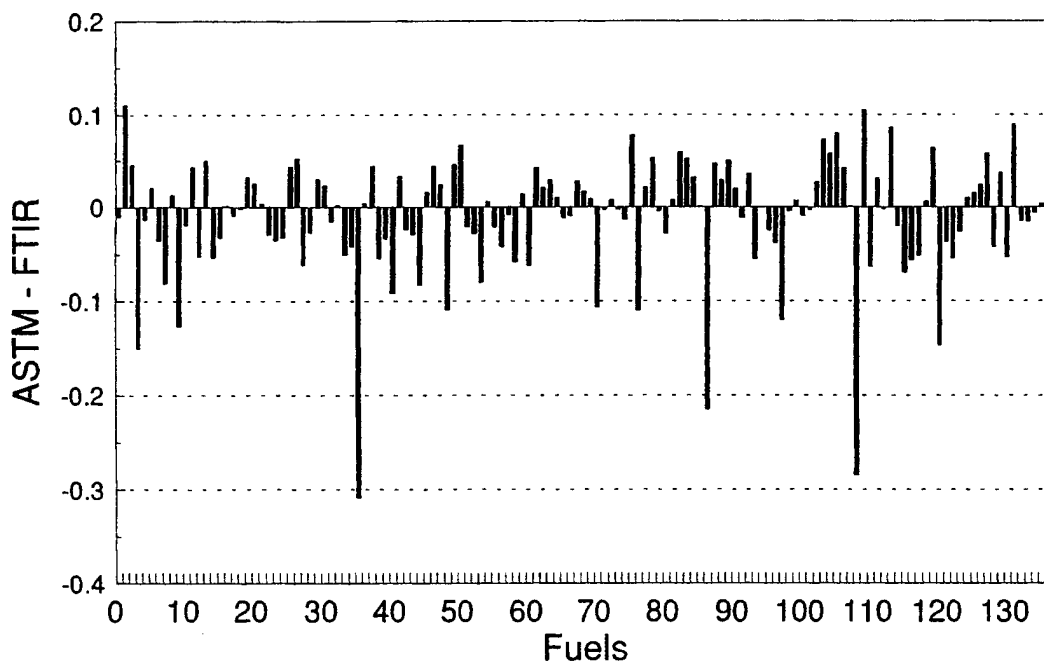


Figure 86. Residual Error in Net Heat of Combustion, MJ/kg, Validation: ASTM D 240
Fuel Set D, Instrument B

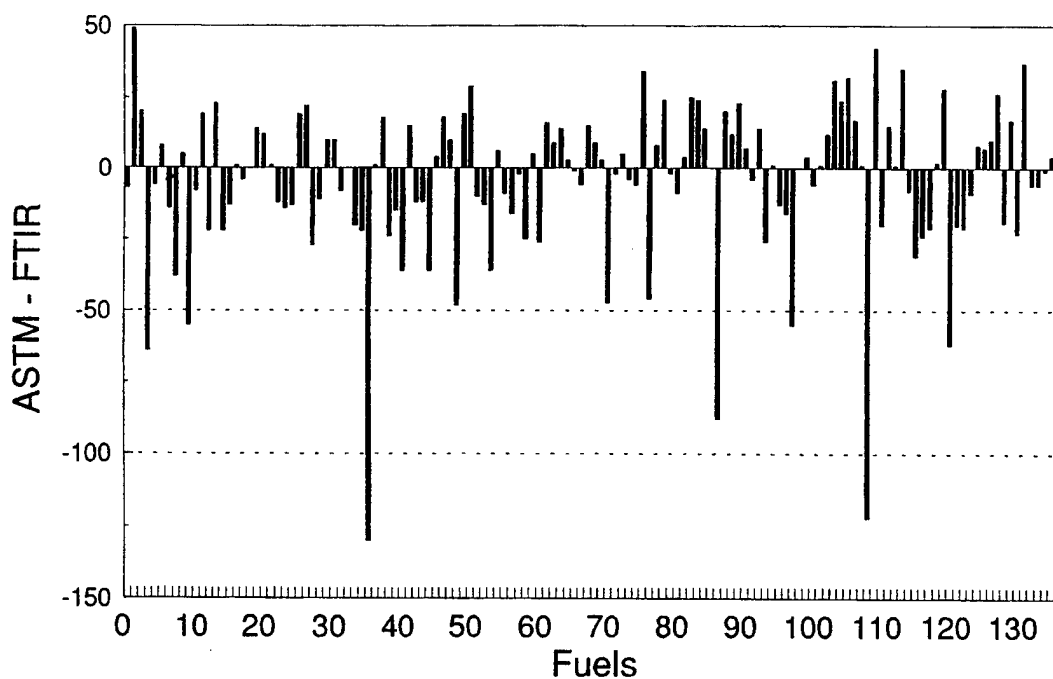


Figure 87. Residual Error in Net Heat of Combustion, Btu/lb, Validation: ASTM D 240
Fuel Set D, Instrument B

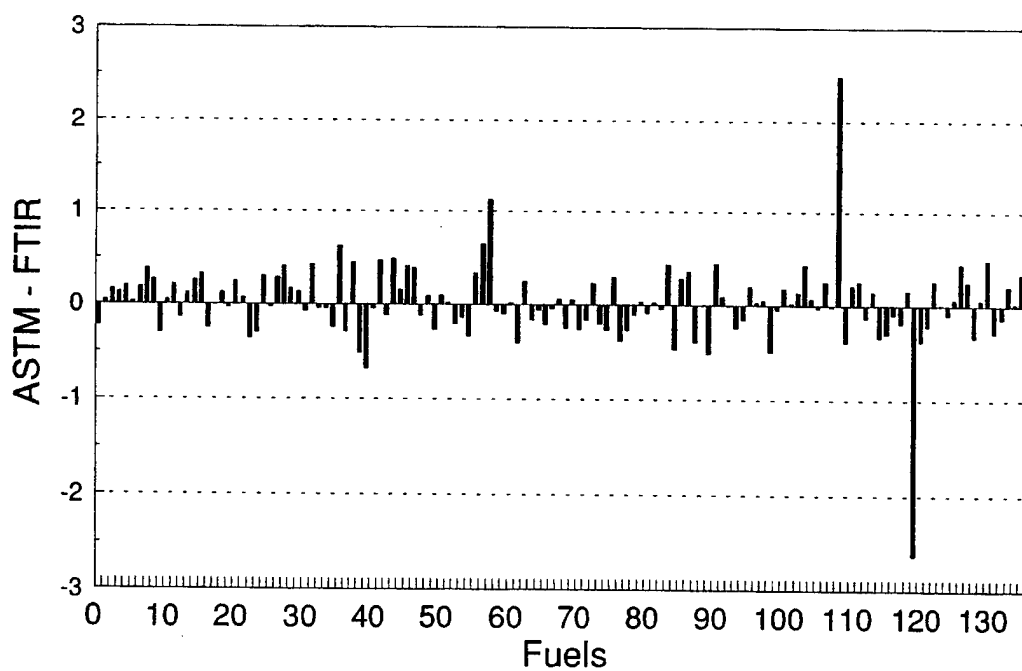


Figure 88. Residual Error in Monocyclic Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

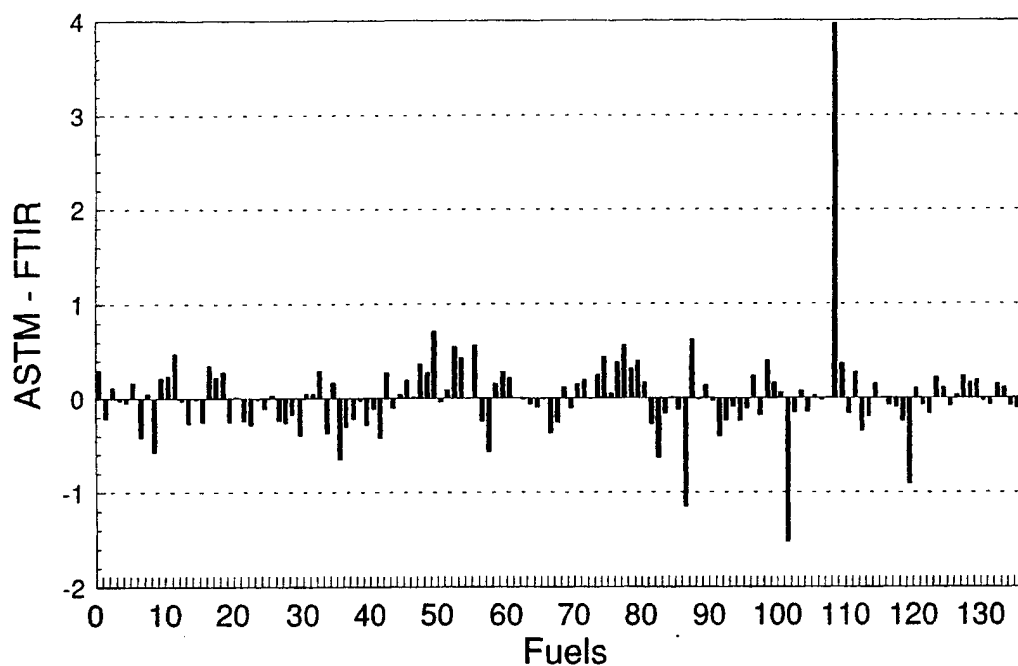


Figure 89. Residual Error in Dicyclic Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

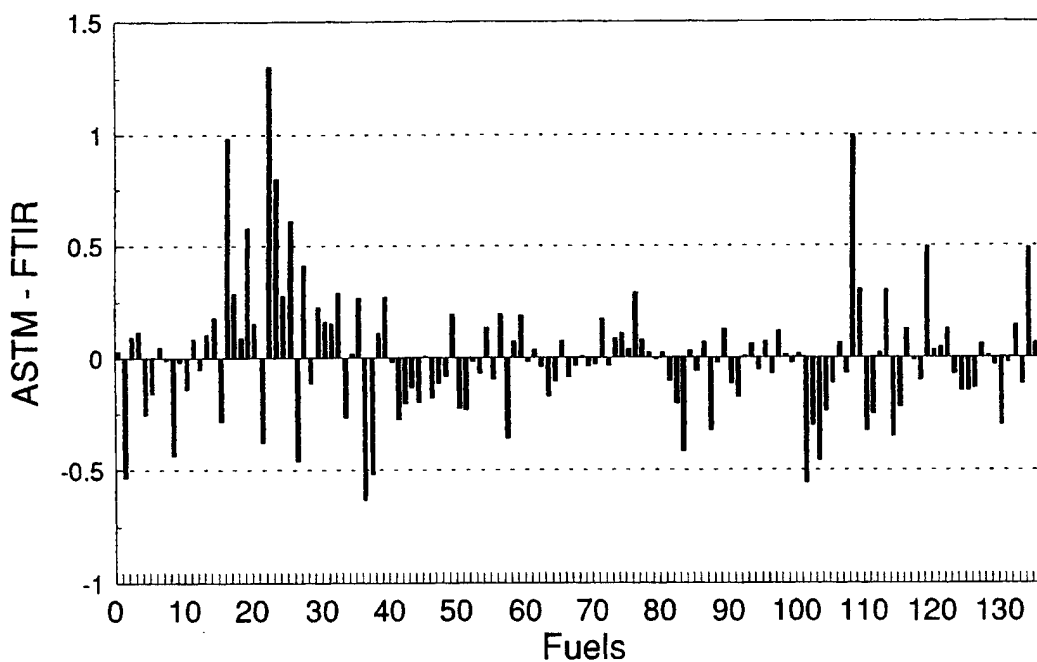


Figure 90. Residual Error in Polycyclic Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

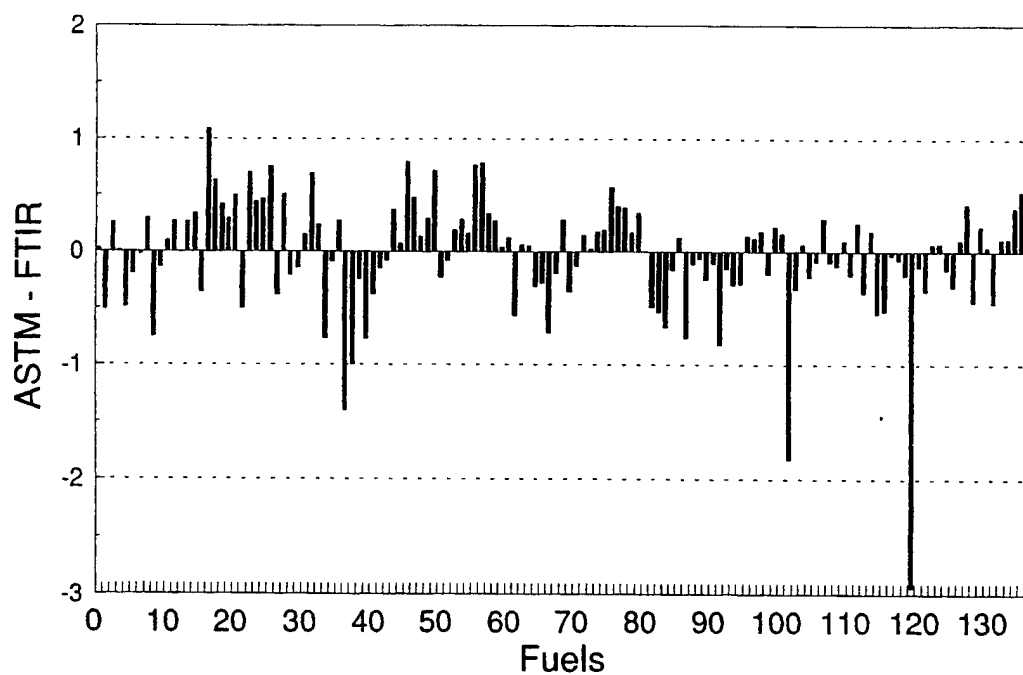


Figure 91. Residual Error in Total Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

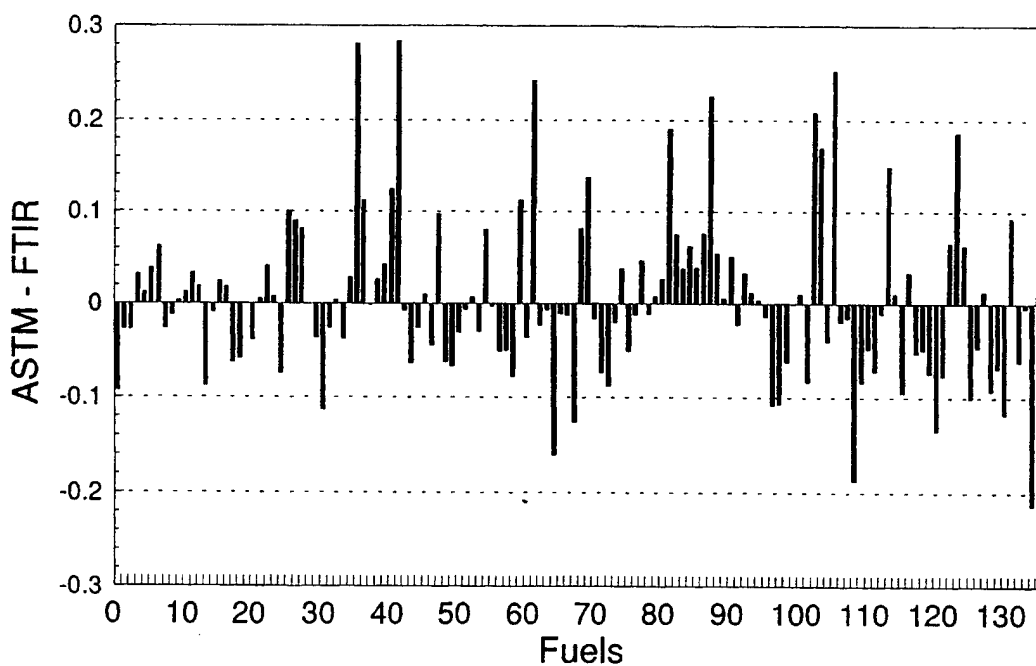


Figure 92. Residual Error in Lubricity by HFRR Validation: ISO/CD12156
Fuel Set D, Instrument B

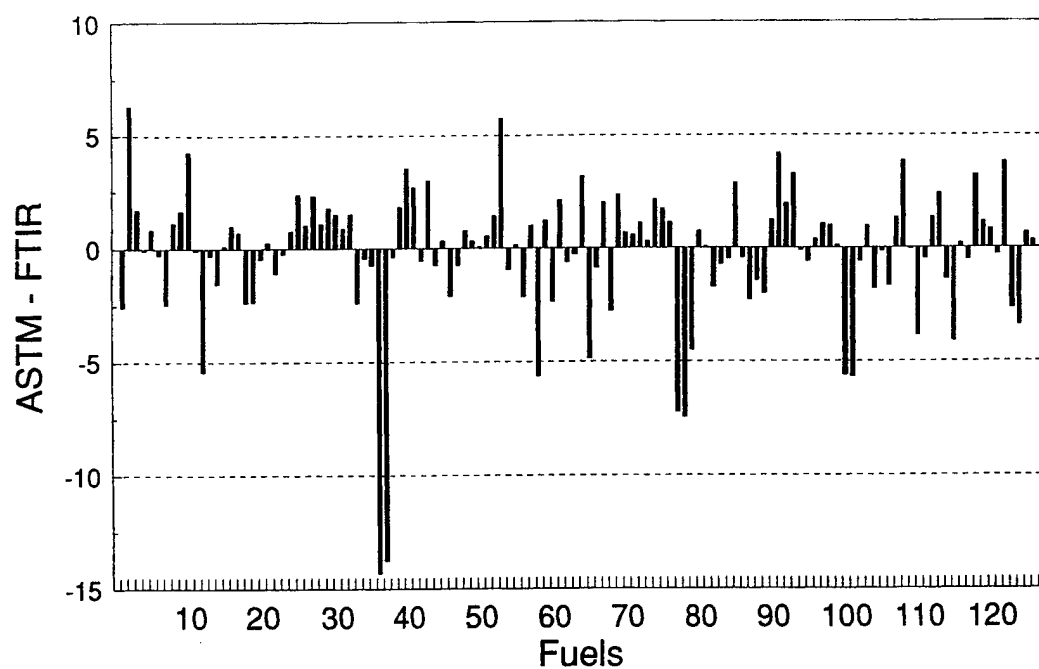


Figure 93. Residual Error in Cloud Point for Diesel Fuels Validation: ASTM D 2500
Fuel Set D, Instrument B

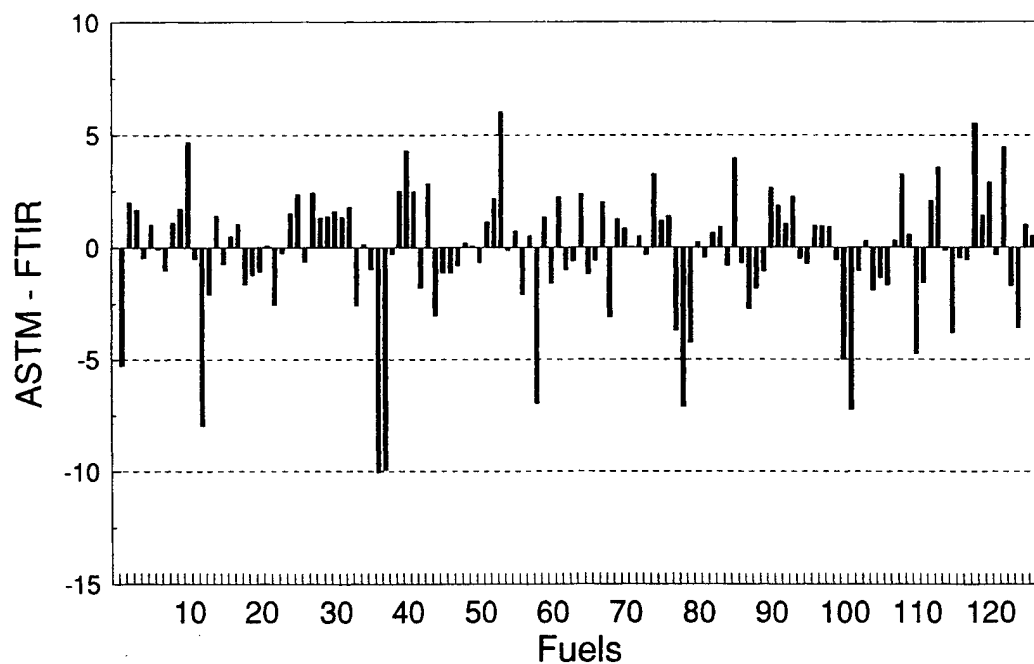


Figure 94. Residual Error in Freeze Point for Diesel Fuels Validation: ASTM D 2386
Fuel Set D, Instrument B

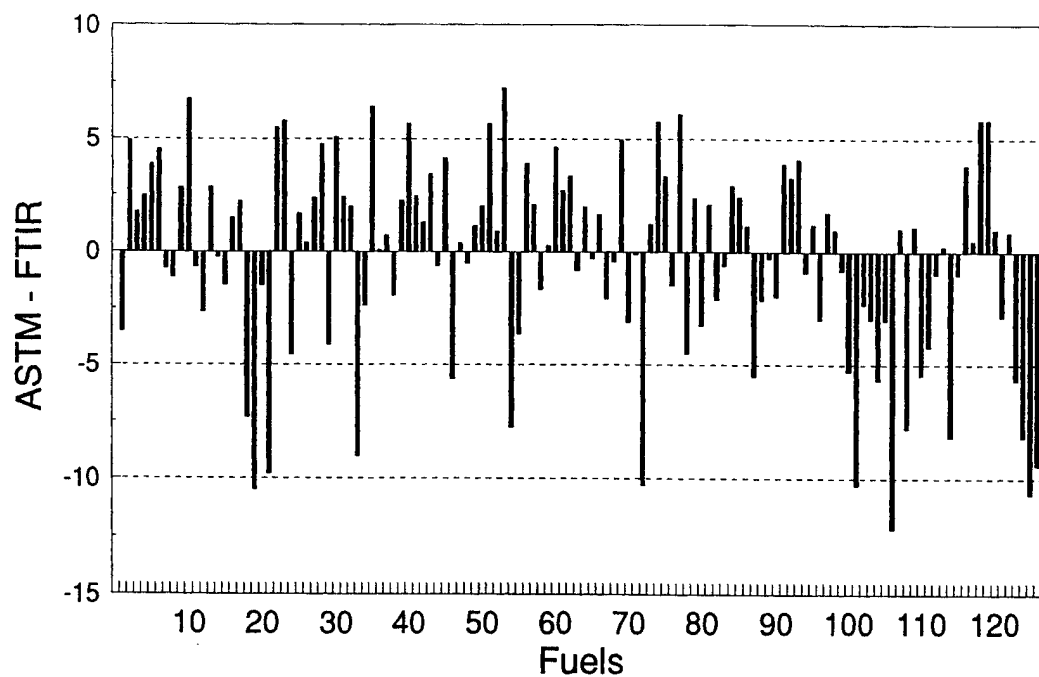


Figure 95. Residual Error in Pour Point for Diesel Fuels Validation: ASTM D 97 Fuel Set D, Instrument B

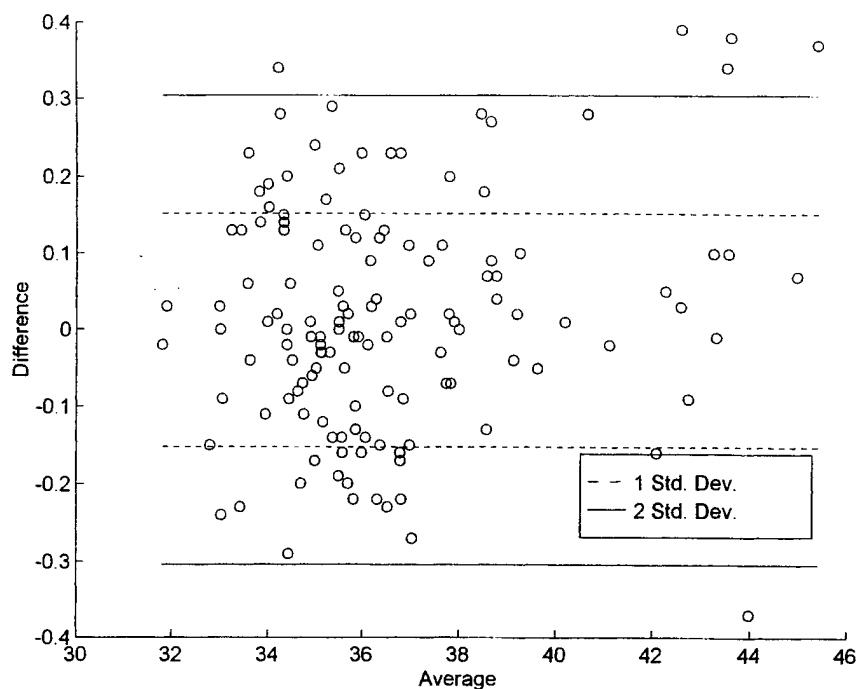


Figure 96. Limits of Agreement in API Gravity Validation: ASTM D 1298
Fuel Set D, Instrument B

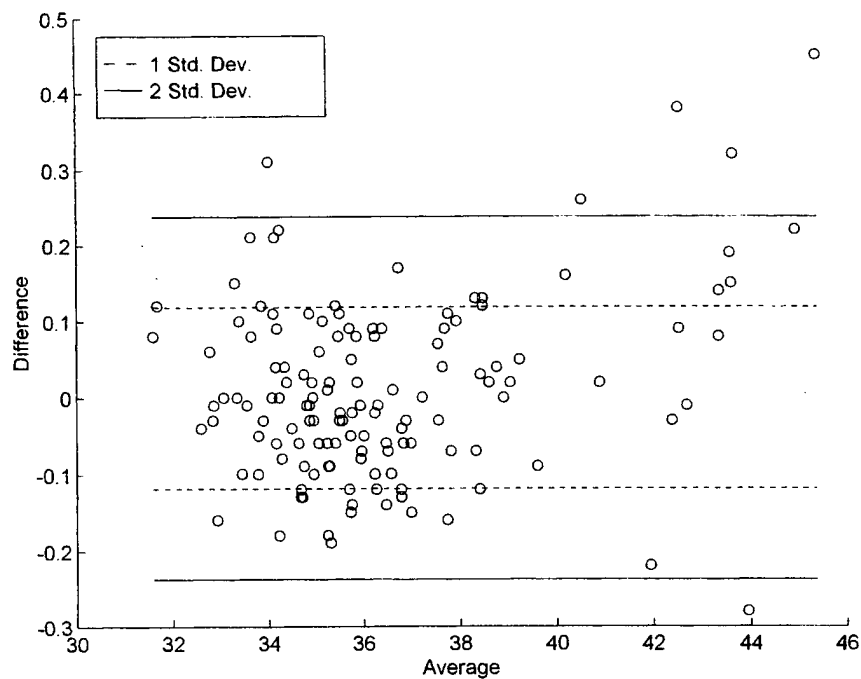


Figure 97. Limits of Agreement in API Gravity Validation: ASTM D 4052
Fuel Set D, Instrument B

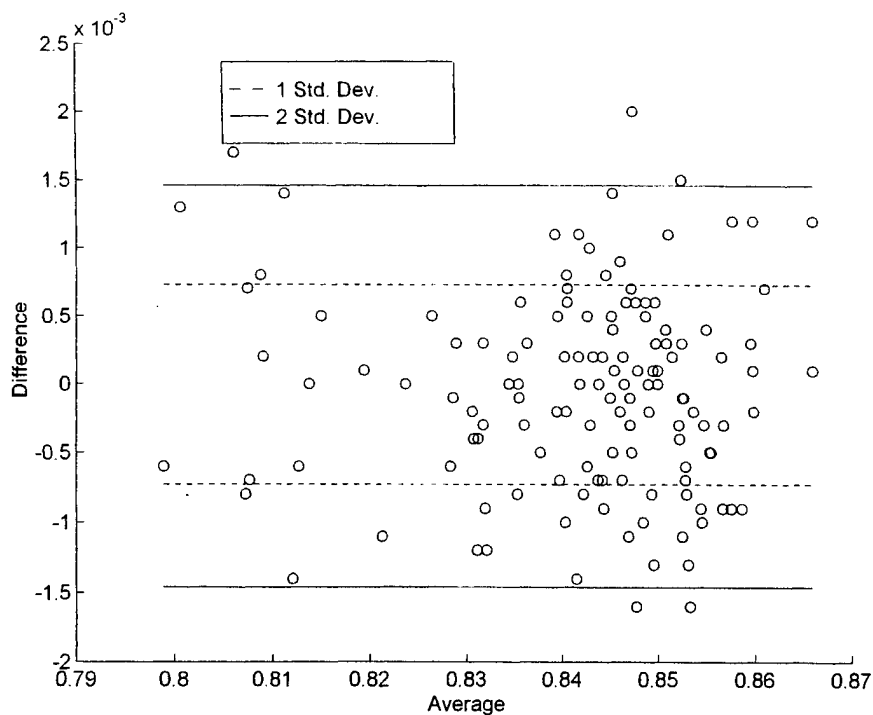


Figure 98. Limits of Agreement in Density Validation: ASTM D 1298
Fuel Set D, Instrument B

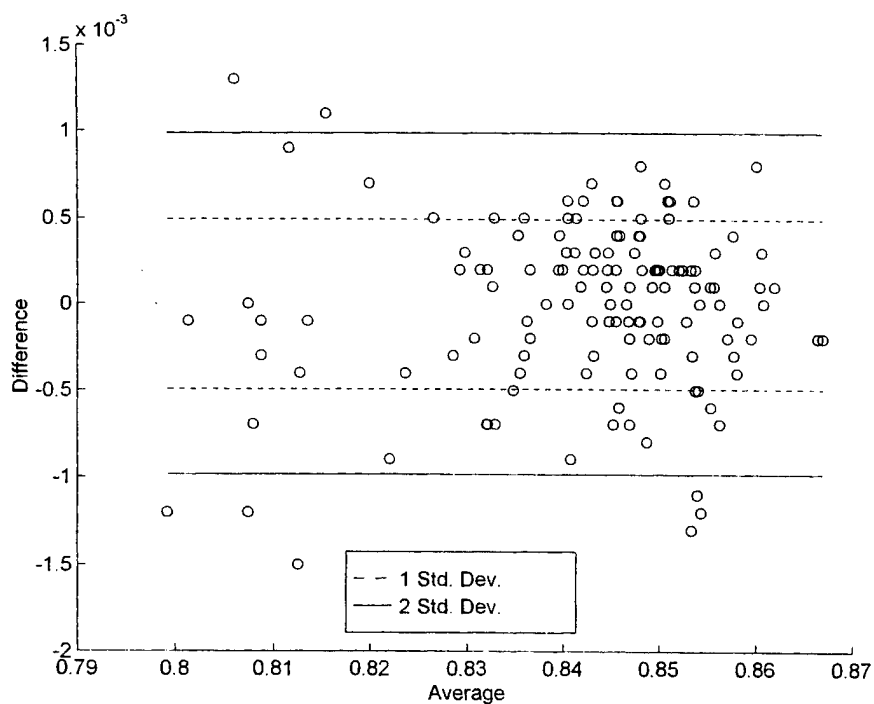


Figure 99. Limits of Agreement in Density Validation: ASTM D 4052
Fuel Set D, Instrument B

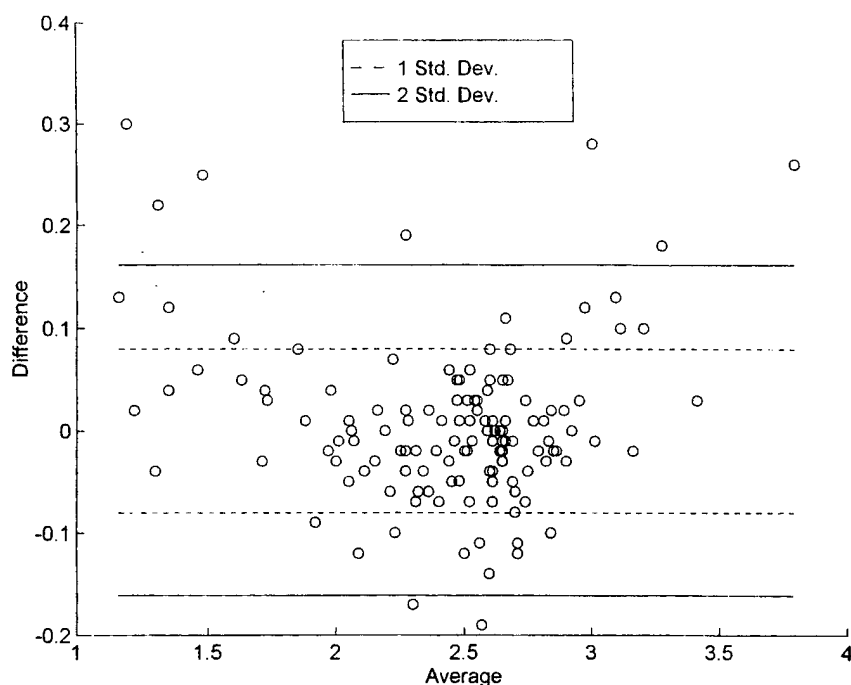


Figure 100. Limits of Agreement in Kinematic Viscosity Validation, cSt, 0°C:
ASTM D 445 Fuel Set D, Instrument B

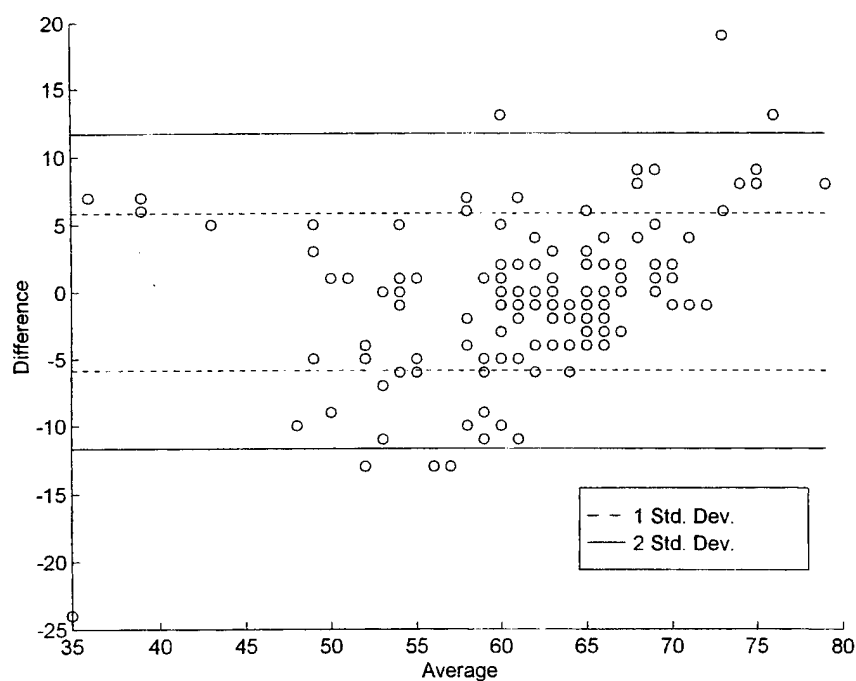


Figure 101. Limits of Agreement in Flash Point Validation: ASTM D 93
Fuel Set D, Instrument B

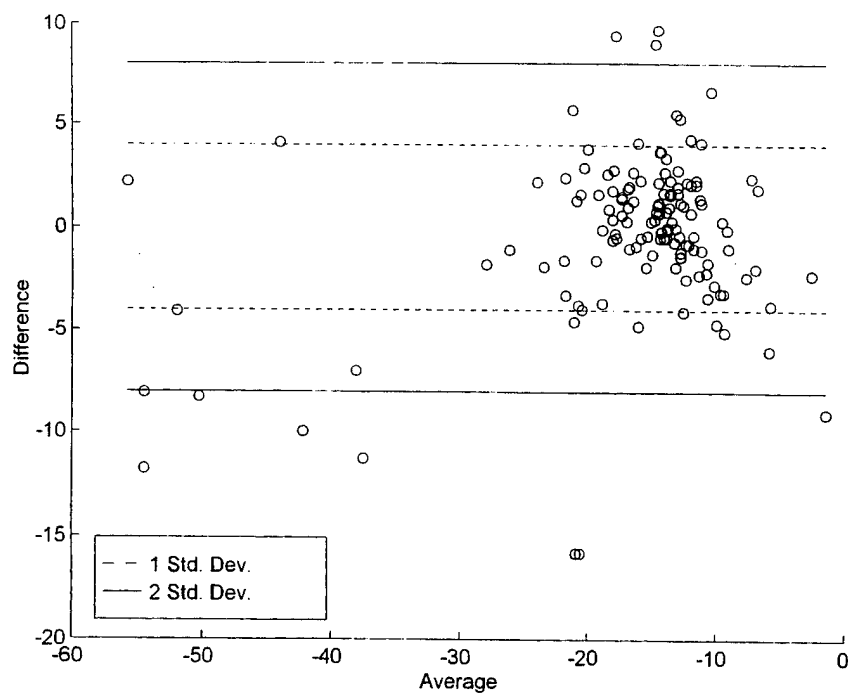


Figure 102. Limits of Agreement in Cloud Point Validation: ASTM D 2500
Fuel Set D, Instrument B

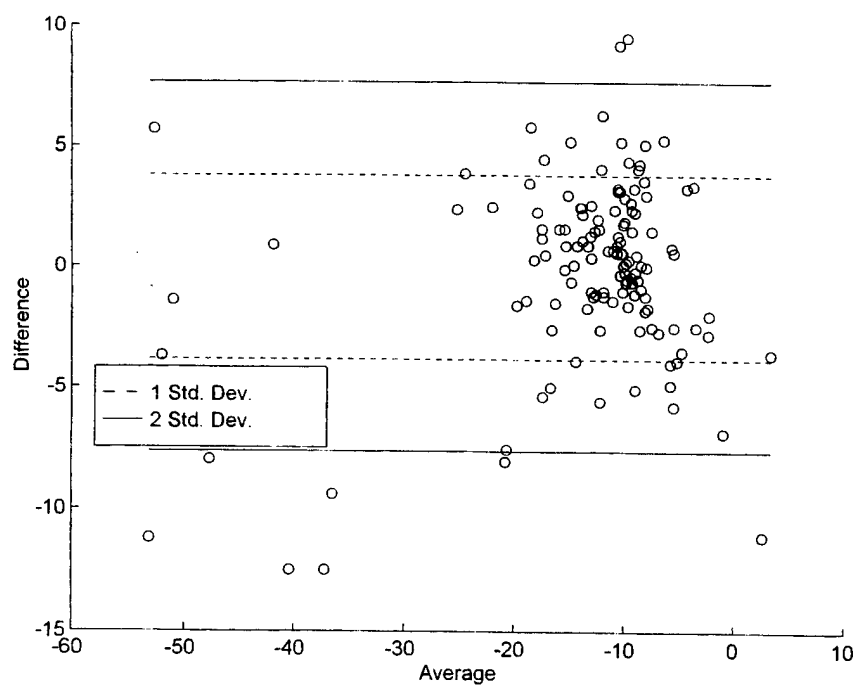


Figure 103. Limits of Agreement in Freeze Point Validation: ASTM D 2386
Fuel Set D, Instrument B

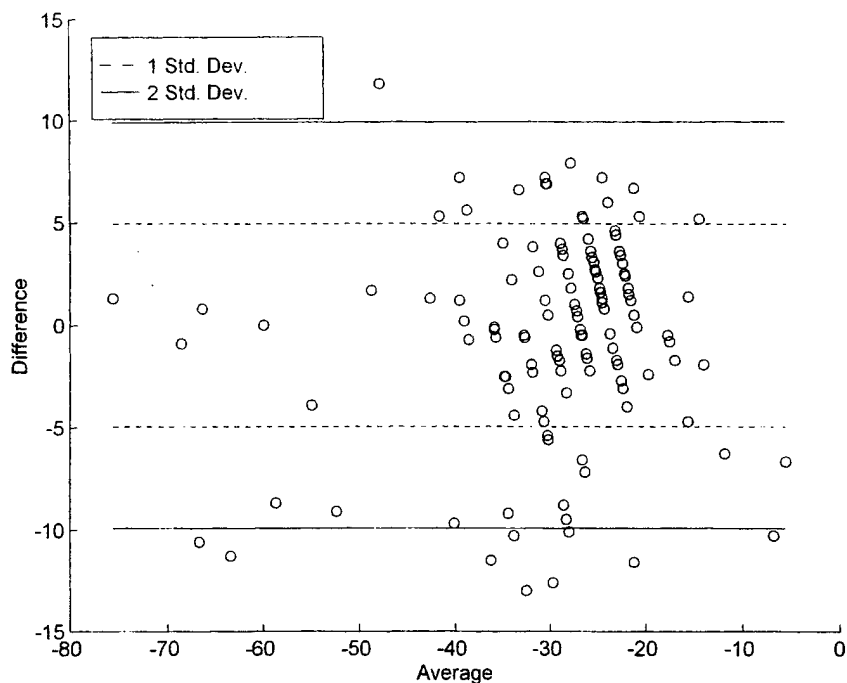


Figure 104. Limits of Agreement in Pour Point Validation: ASTM D 97
Fuel Set D, Instrument B

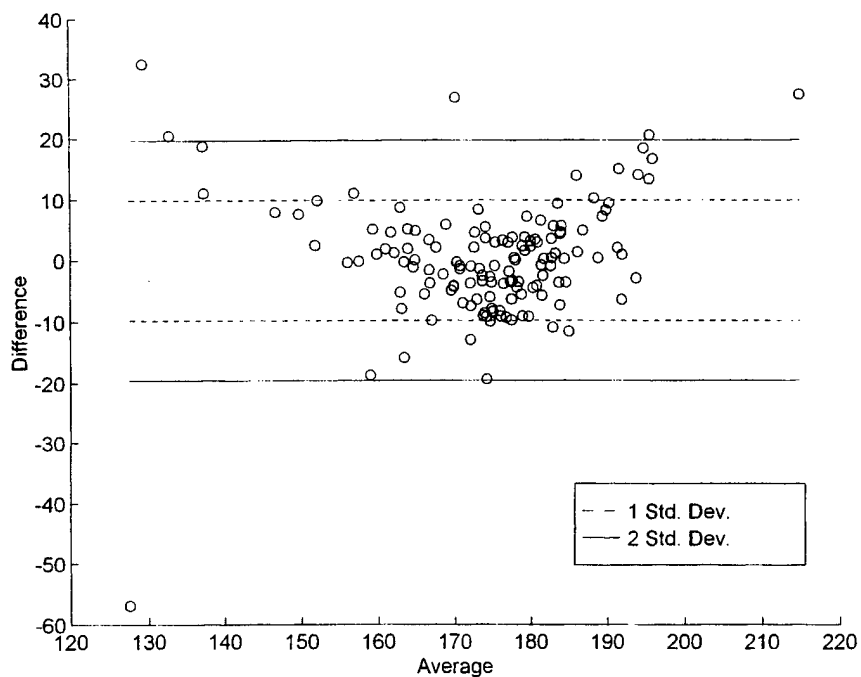


Figure 105. Limits of Agreement in Initial Boiling Point Validation: ASTM D 86
Fuel Set D, Instrument B

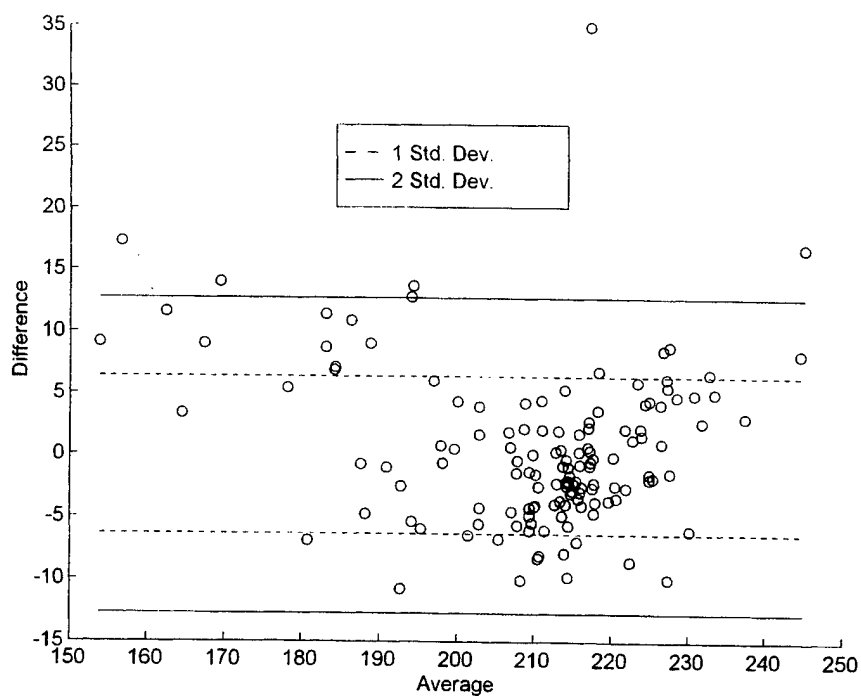


Figure 106. Limits of Agreement in Boiling Point at 10% Validation: ASTM D 86
Fuel Set D, Instrument B

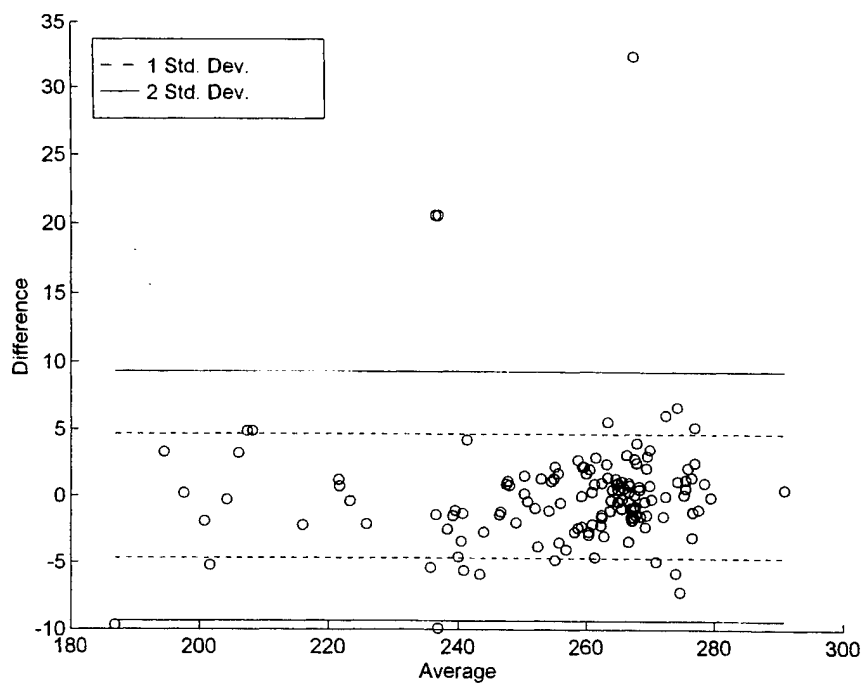


Figure 107. Limits of Agreement in Boiling Point at 50% Validation: ASTM D 86
Fuel Set D, Instrument B

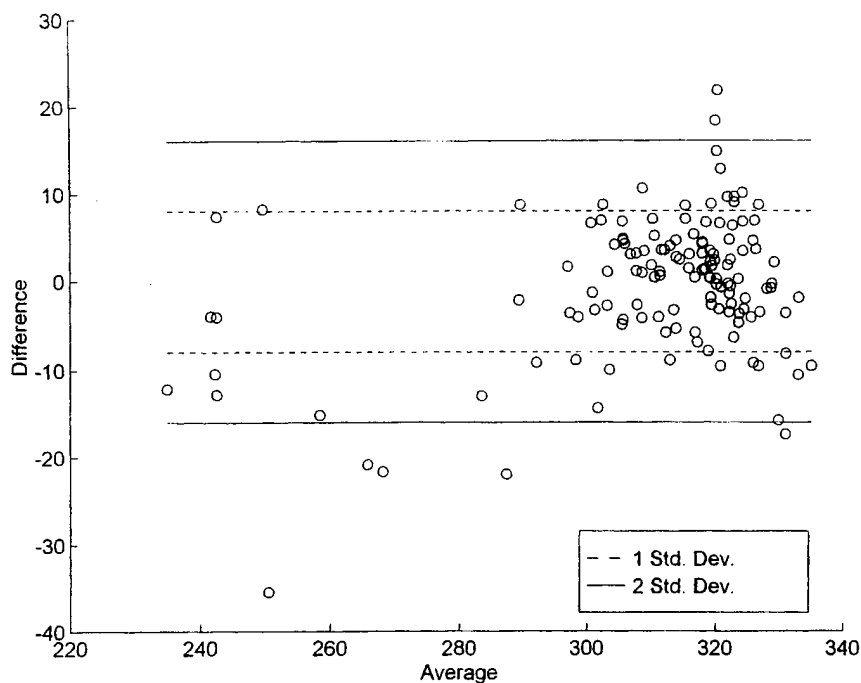


Figure 108. Limits of Agreement in Boiling Point at 90% Validation: ASTM D 86
Fuel Set D, Instrument B

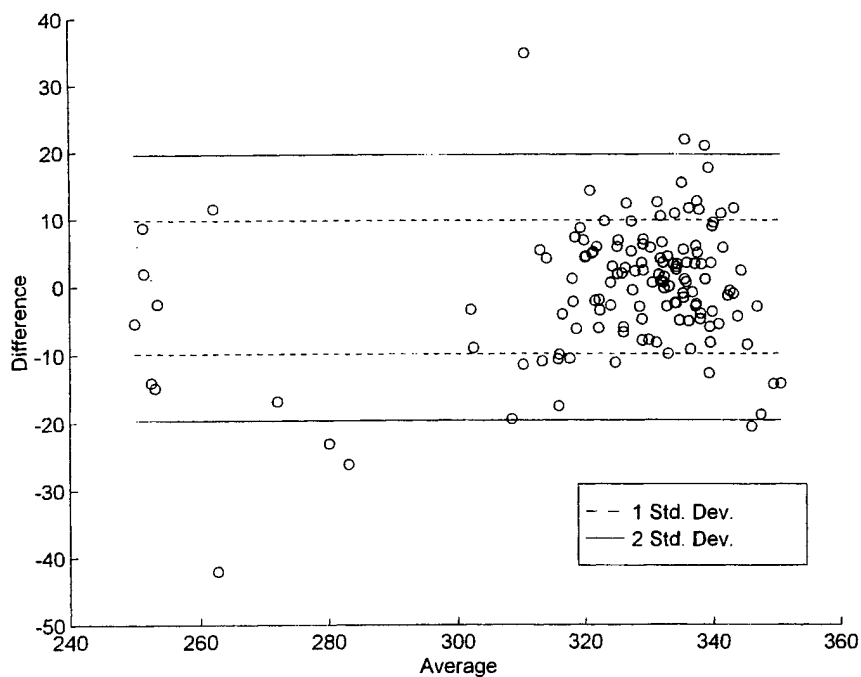


Figure 109. Limits of Agreement in Boiling Point at 95% Validation: ASTM D 86
Fuel Set D, Instrument B

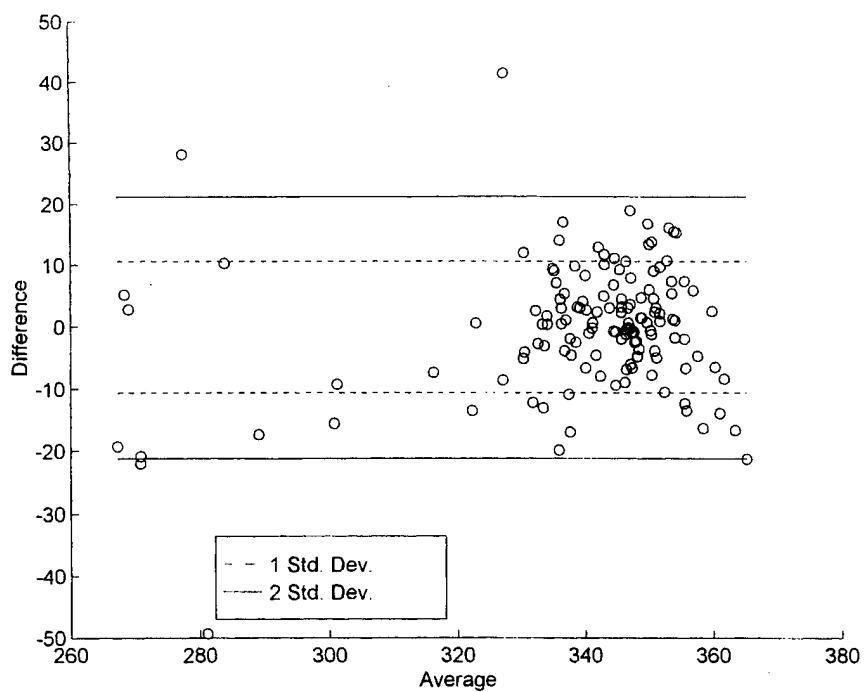


Figure 110. Limits of Agreement in Final Boiling Point Validation: ASTM D 86
Fuel Set D, Instrument B

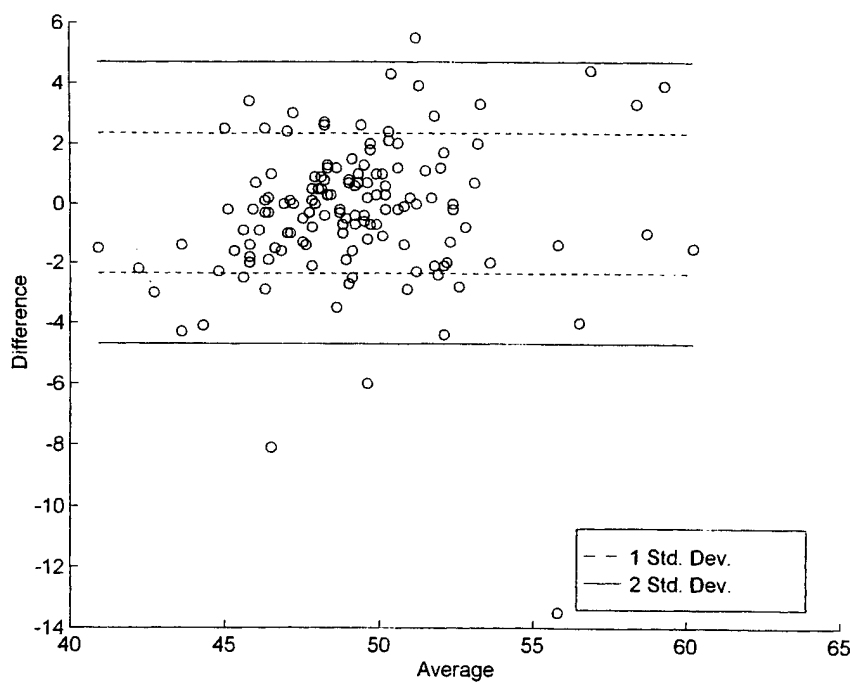


Figure 111. Limits of Agreement in Cetane Number Validation: ASTM D 613
Fuel Set D, Instrument B

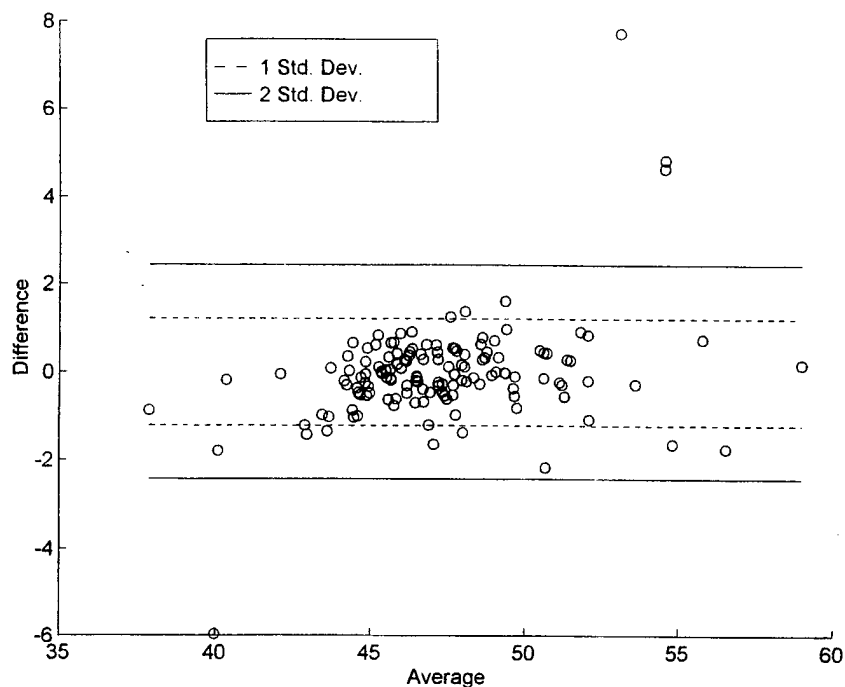


Figure 112. Limits of Agreement in Calculated Cetane Index Validation: ASTM D 976
Fuel Set D, Instrument B

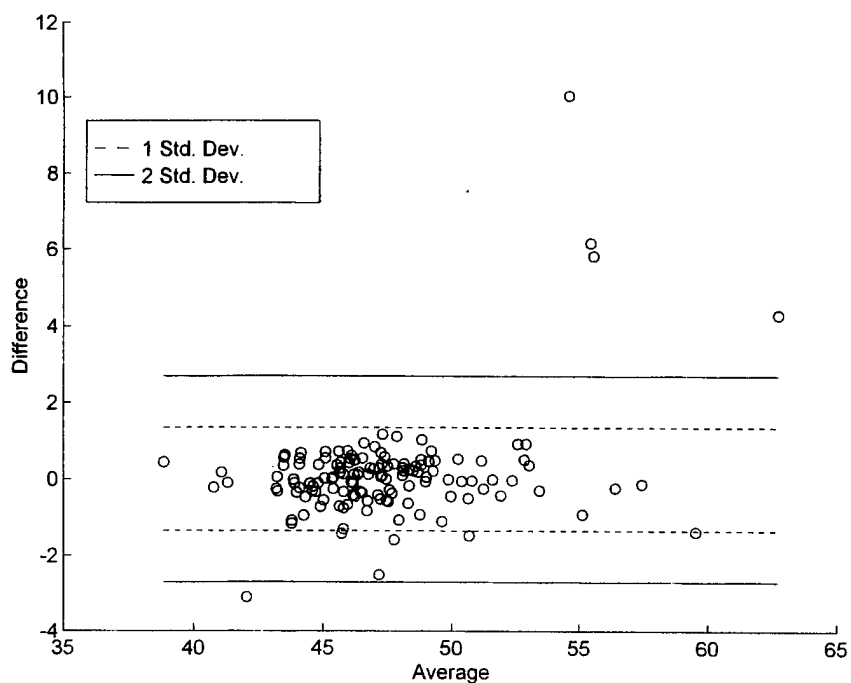


Figure 113. Limits of Agreement in Calculated Cetane Index Validation: ASTM D 4737
Fuel Set D, Instrument B

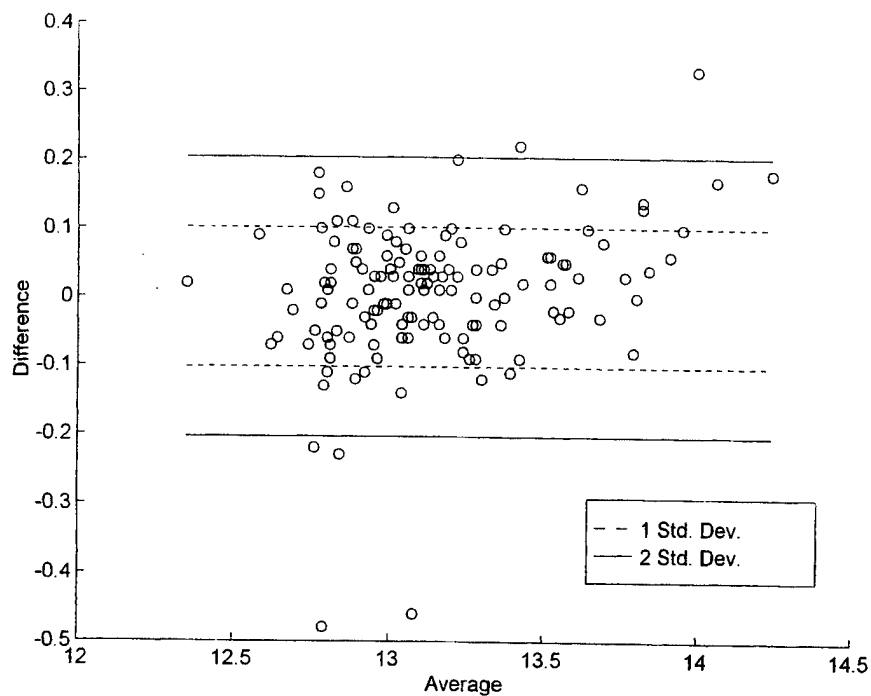


Figure 114. Limits of Agreement in Hydrogen Content Validation: ASTM D 5291
Fuel Set D, Instrument B

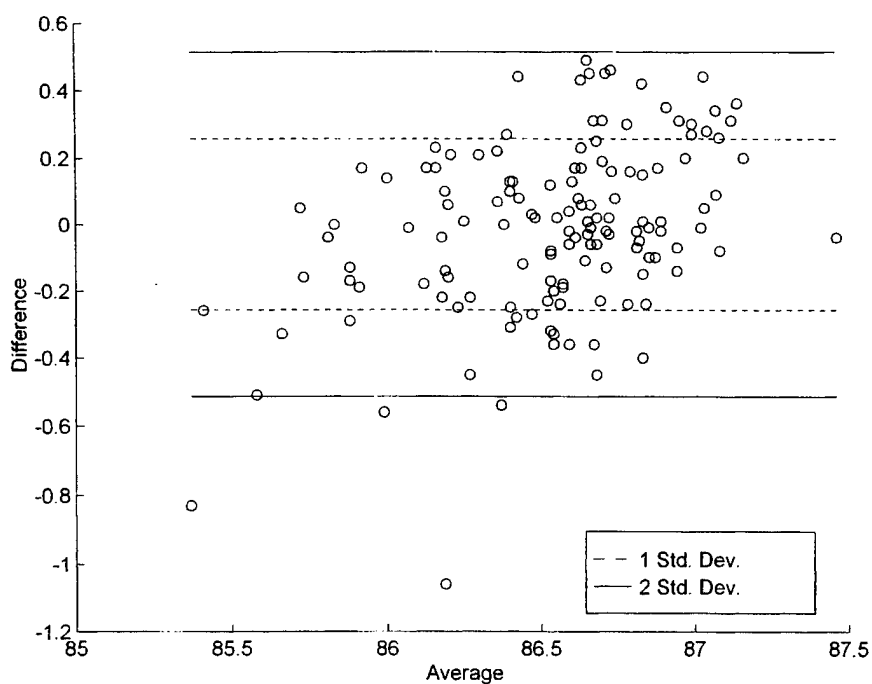


Figure 115. Limits of Agreement in Carbon Content Validation: ASTM D 5291
Fuel Set D, Instrument B

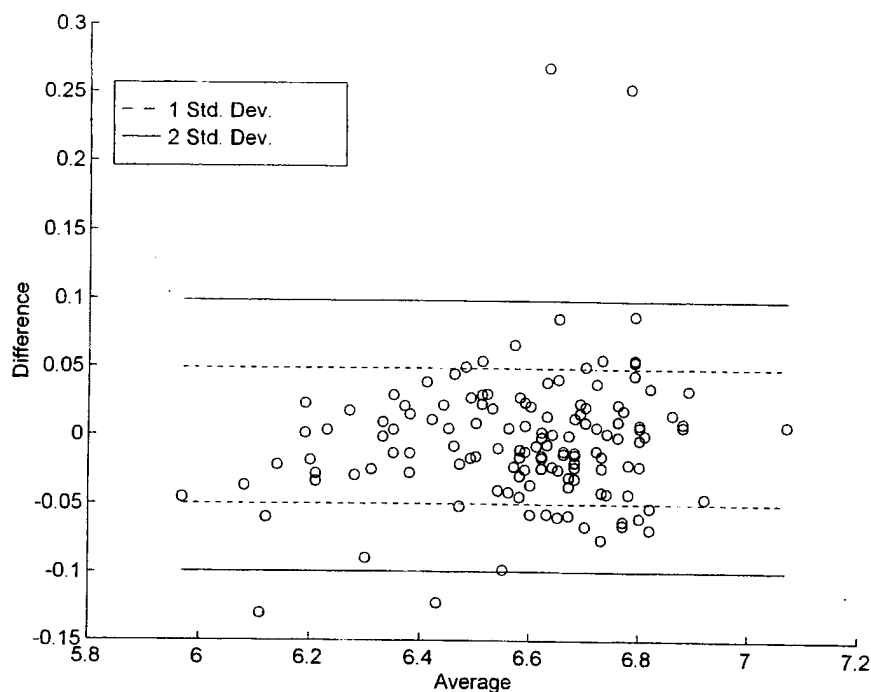


**Figure 116. Limits of Agreement in Carbon-to-Hydrogen Ratio Validation: ASTM D 5291
Fuel Set D, Instrument B**

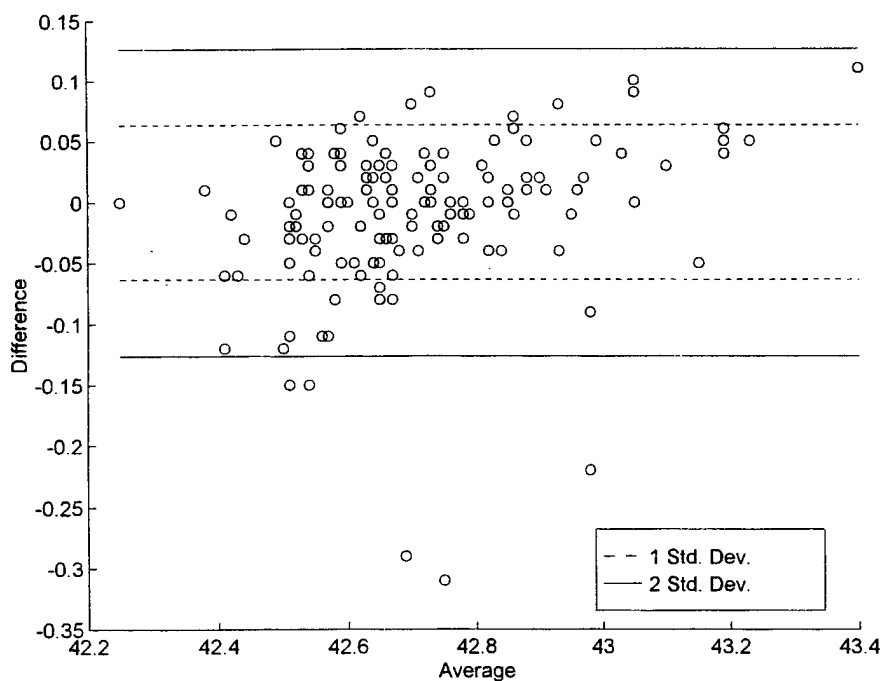


**Figure 117. Limits of Agreement in Net Heat of Combustion, MJ/kg, Validation:
ASTM D 240, Fuel Set D, Instrument B**

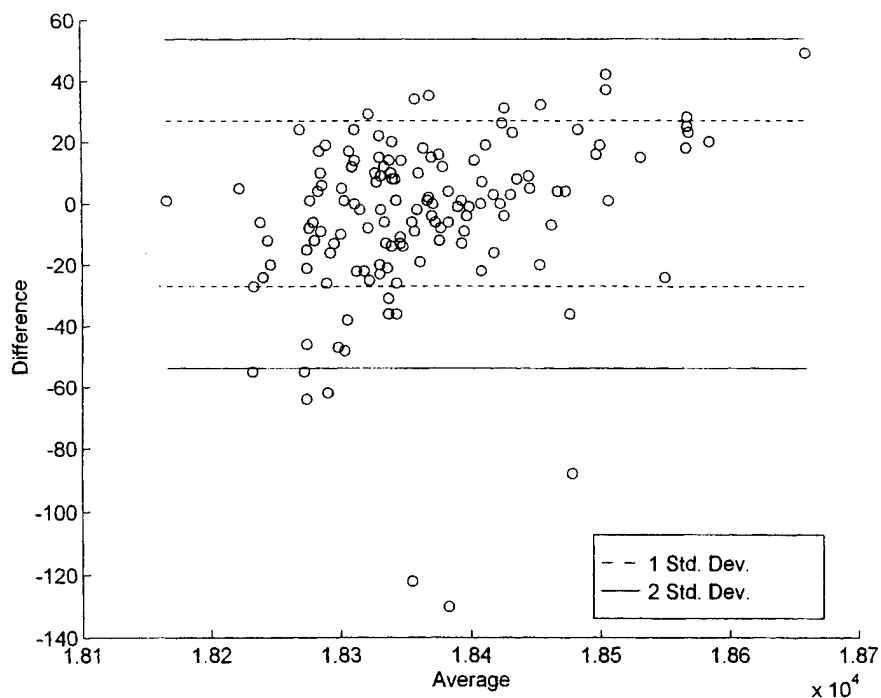


Figure 118. Limits of Agreement in Net Heat of Combustion, Btu/lb, Validation: ASTM D 240, Fuel Set D, Instrument B

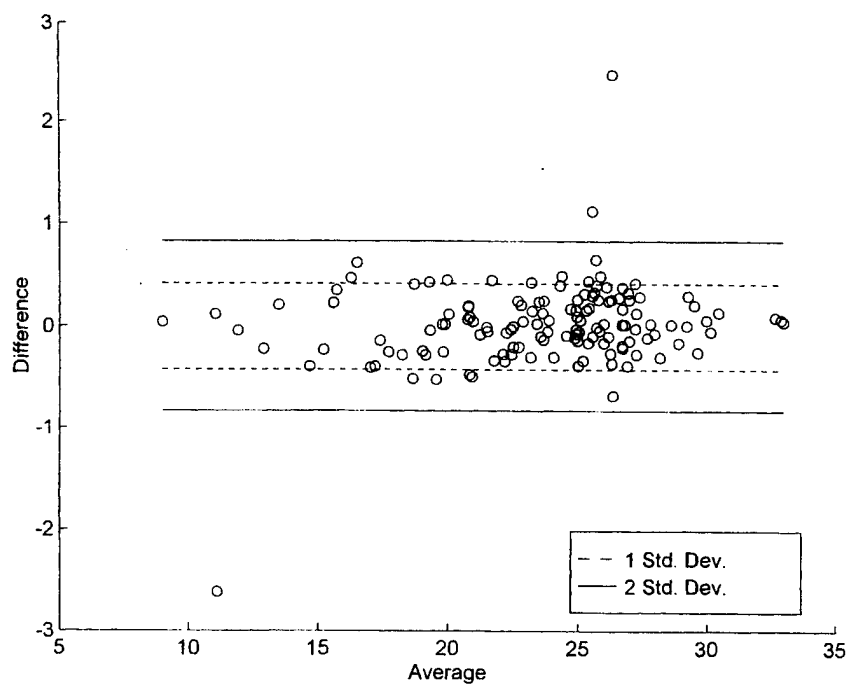


Figure 119. Limits of Agreement in Monocyclic Aromatics Validation: ASTM D 5186 Fuel Set D, Instrument B

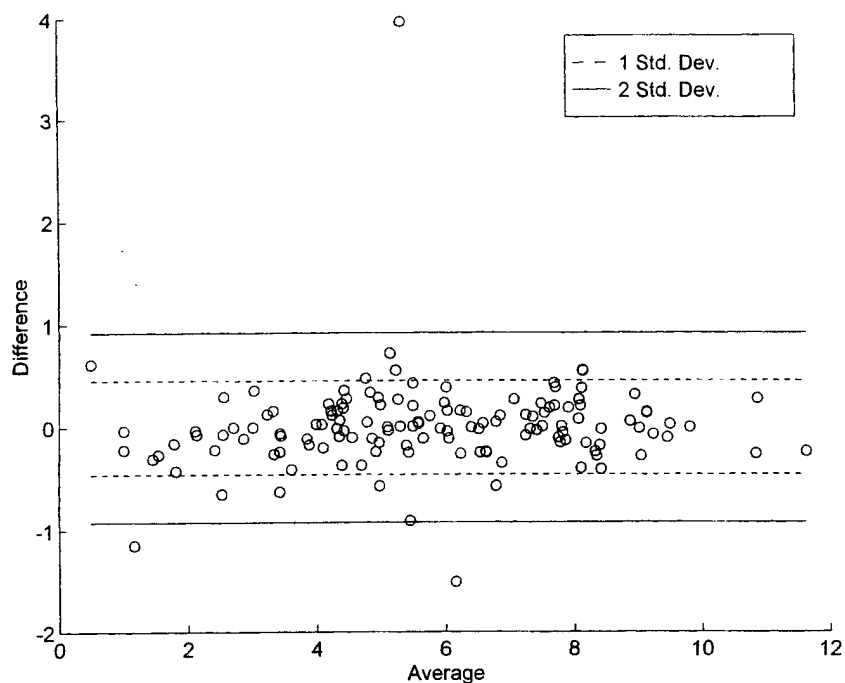


Figure 120. Limits of Agreement in Dicyclic Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

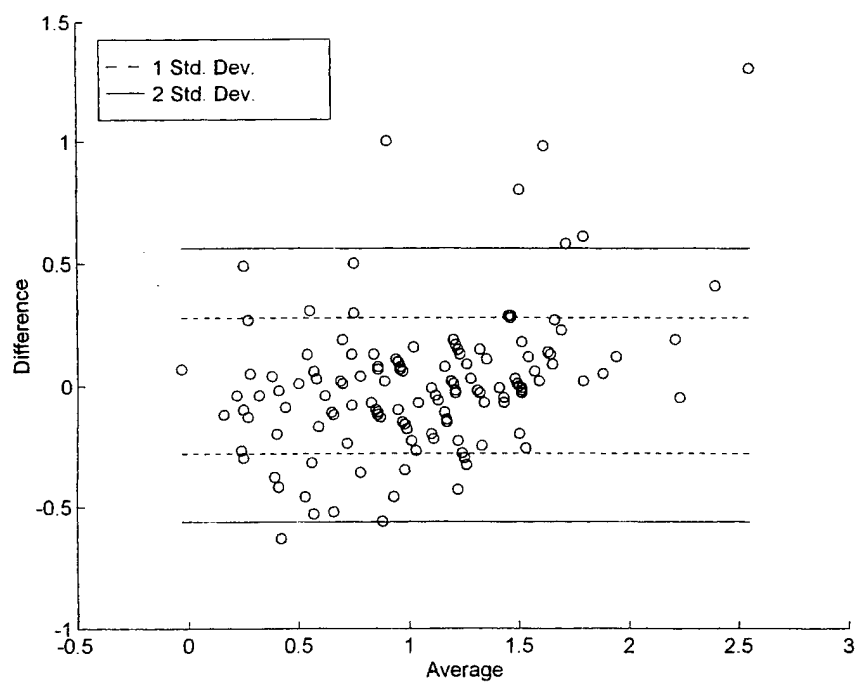


Figure 121. Limits of Agreement in Polycyclic Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

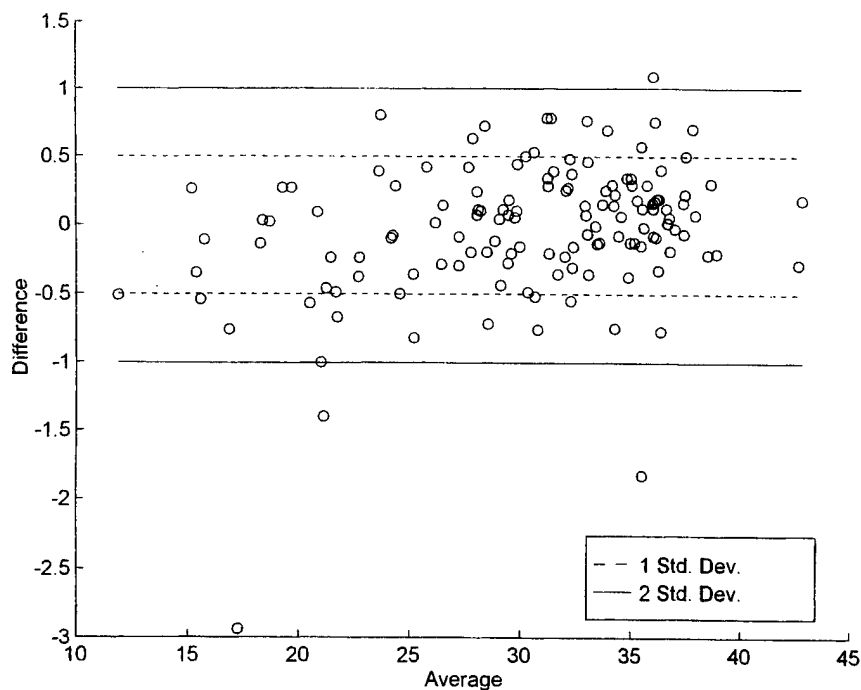


Figure 122. Limits of Agreement in Total Aromatics Validation: ASTM D 5186
Fuel Set D, Instrument B

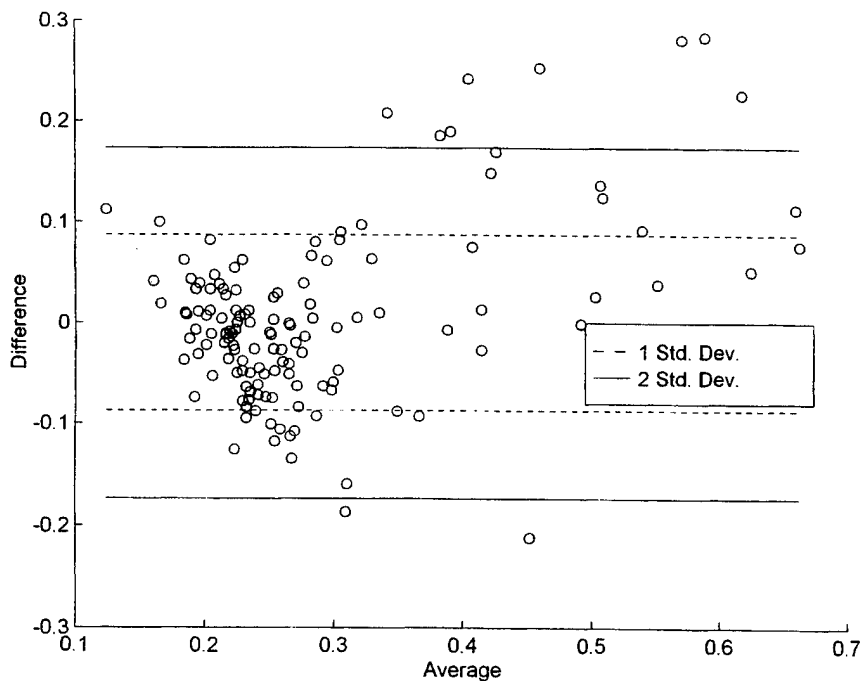


Figure 123. Limits of Agreement in Lubricity by HFRR Validation: ISO/CD 12156
Fuel Set D, Instrument B

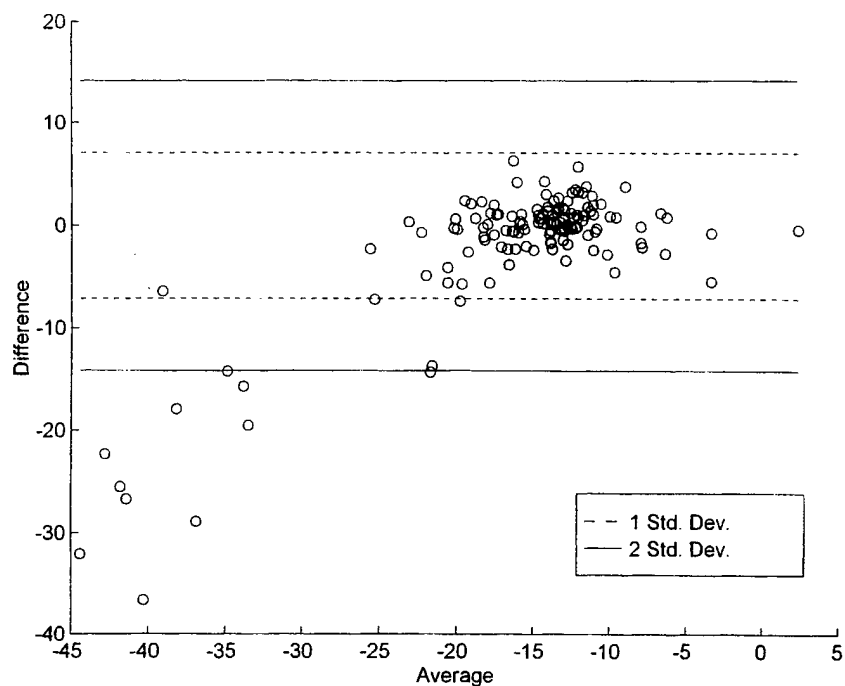


Figure 124. Limits of Agreement in Cloud Point in Diesel Fuels Validation: ASTM D 2500
Fuel Set D, Instrument B

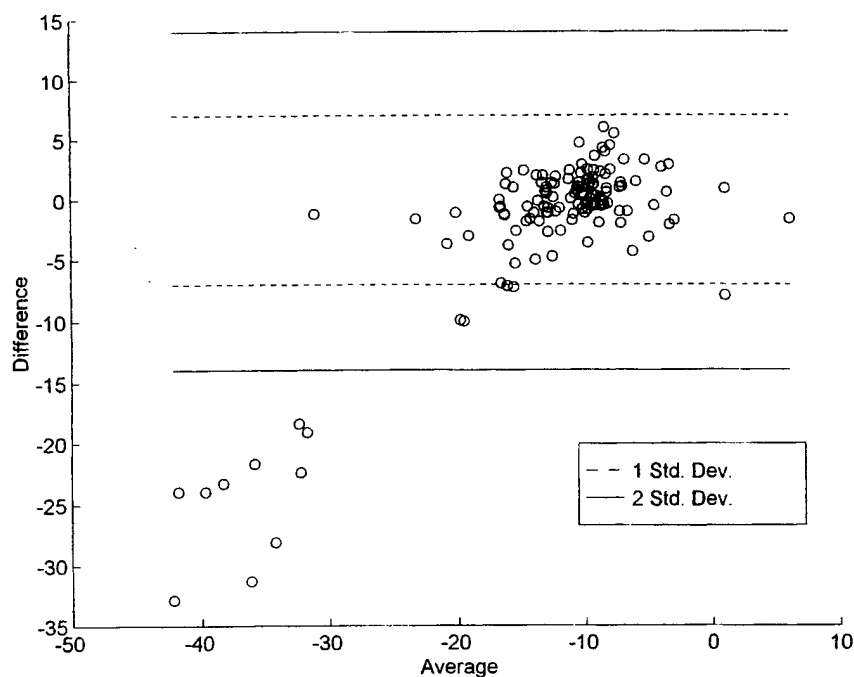


Figure 125. Limits of Agreement in Freeze Point in Diesel Fuels Validation: ASTM D 2386
Fuel Set D, Instrument B

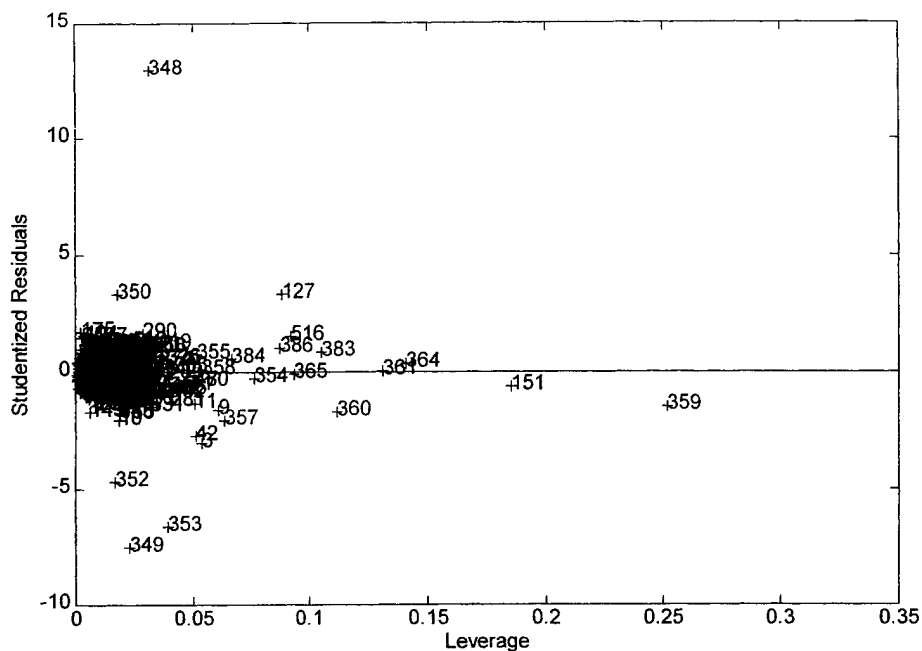


Figure 128. Density ASTM D 4052: Leverage vs. Studentized Residuals by Sample Number

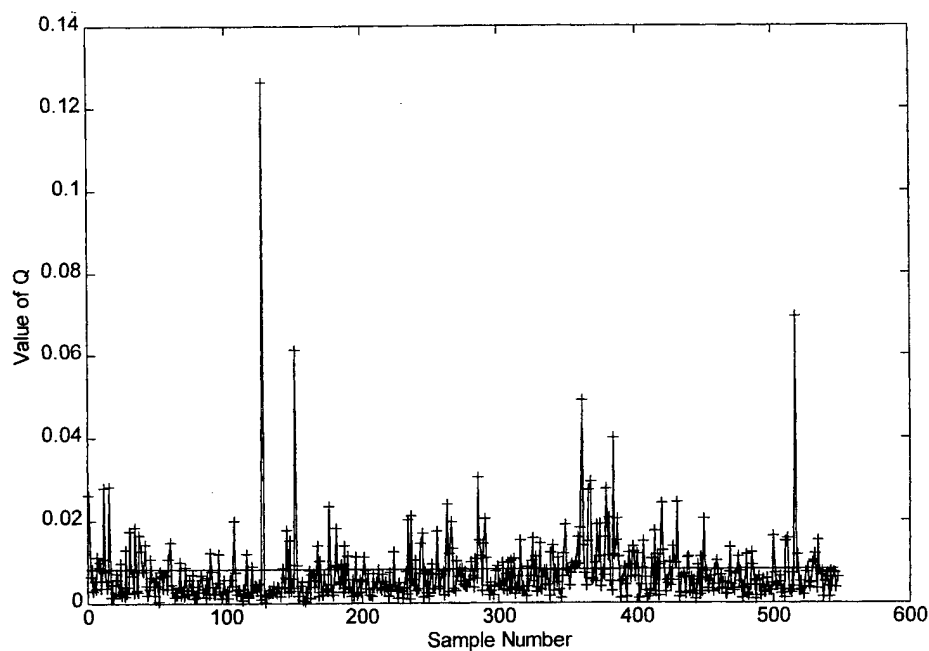


Figure 129. Density ASTM D 4052: Q vs Sample Number

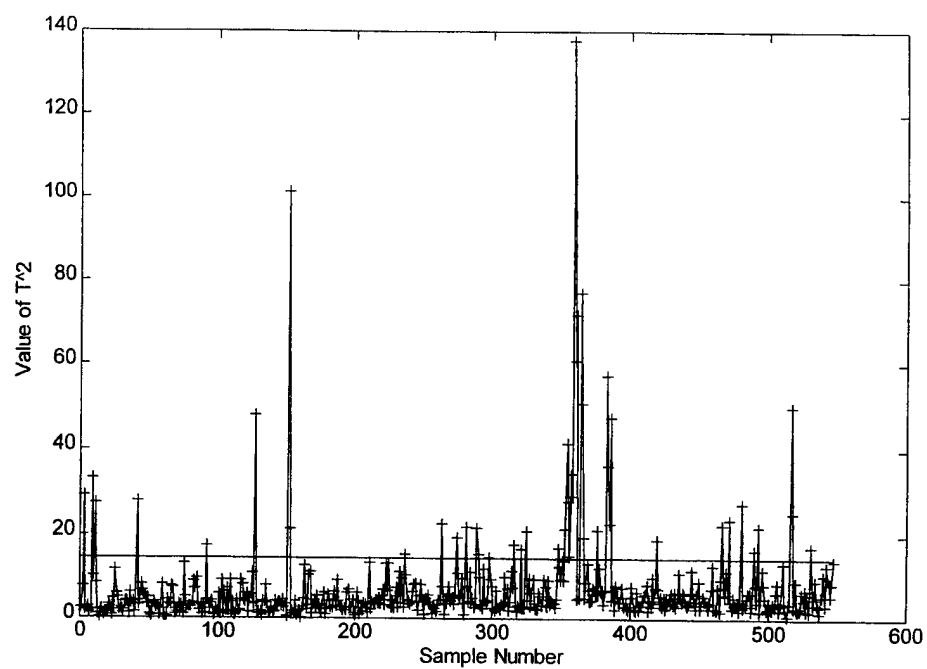


Figure 130. Density ASTM D 4052: T² vs Sample Number

Department of Defense

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US ARMY TACOM		CDR AMSAA	
ATTN: AMSTA TR-R/210 (L. VILLAHERMOSA)	10	ATTN: AMXSY CM	1
AMSTA TR-R/210 (T. BAGWELL)	1	AMXSY L	1
10115 GRIDLEY RD STE 128		APG MD 21005-5071	
FT BELVOIR VA 22060-5843			
PROG EXEC OFFICER		CDR ARO	
ARMORED SYS MODERNIZATION		ATTN: AMXRO EN (D MANN)	1
ATTN: SFAE ASM S	1	RSCH TRIANGLE PK	
SFAE ASM H	1	NC 27709-2211	
SFAE ASM AB	1	CDR AEC	
SFAE ASM BV	1	ATTN: SFIM AEC ECC (T ECCLES)	1
SFAE ASM CV	1	APG MD 21010-5401	
SFAE ASM AG	1		
CDR TACOM		CDR ARMY ATCOM	
WARREN MI 48397-5000		ATTN: AMSAT I WM	1
PROG EXEC OFFICER		AMSAT I ME (L HEPLER)	1
ARMORED SYS MODERNIZATION		AMSAT I LA (V SALISBURY)	1
ATTN: SFAE FAS AL	1	AMSAT R EP (V EDWARD)	1
SFAE FAS PAL	1	4300 GOODFELLOW BLVD	
PICATINNY ARSENAL		ST LOUIS MO 63120-1798	
NJ 07806-5000			
PROG EXEC OFFICER		CDR ARMY SOLDIER SPT CMD	
TACTICAL WHEELED VEHICLES		ATTN: SATNC US (J SIEGEL)	1
ATTN: SFAE TWV TVSP	1	SATNC UE	1
SFAE TWV FMTV	1	NATICK MA 01760-5018	
SFAE TWV PLS	1		
CDR TACOM		CDR ARMY ARDEC	
WARREN MI 48397-5000		ATTN: AMSTA AR EDE S	1
PROG EXEC OFFICER		PICATINNY ARSENAL	
ARMAMENTS		NJ 07808-5000	
ATTN: SFAE AR HIP	1	CDR ARMY WATERVLIET ARSN	
SFAE AR TMA	1	ATTN: SARWY RDD	1
PICATINNY ARSENAL		WATERVLIET NY 12189	
NJ 07806-5000			
PROG MGR		CDR APC	
UNMANNED GROUND VEH		ATTN: SATPC L	1
ATTN: AMCPM UG	1	SATPC Q	1
REDSTONE ARSENAL		NEW CUMBERLAND PA 17070-5005	
AL 35898-8060			
DIR		PETROL TEST FAC WEST	1
ARMY RSCH LAB		BLDG 247 TRACEY LOC	
ATTN: AMSRL PB P	1	DDRW	
2800 POWDER MILL RD		P O BOX 96001	
ADELPHIA MD 20783-1145		STOCKTON CA 95296-0960	
VEHICLE PROPULSION DIR		CDR ARMY LEA	
ATTN: AMSRL VP (MS 77 12)	1	ATTN: LOEA PL	1
NASA LEWIS RSCH CTR		NEW CUMBERLAND PA 17070-5007	
21000 BROOKPARK RD			
CLEVELAND OH 44135		CDR ARMY TECOM	
		ATTN: AMSTE TA R	1
		AMSTE TC D	1
		AMSTE EQ	1
		APG MD 21005-5006	

PROJ MGR PETROL WATER LOG		CDR ARMY INF SCHOOL	
ATTN: AMCPM PWL	1	ATTN: ATSH CD	1
4300 GOODFELLOW BLVD		ATSH AT	1
ST LOUIS MO 63120-1798		FT BENNING GA 31905-5000	
PROJ MGR MOBILE ELEC PWR		CDR ARMY AVIA CTR	
ATTN: AMCPM MEP T	1	ATTN: ATZQ DOL M	1
AMCPM MEP L	1	ATZQ DI	1
7798 CISSNA RD STE 200		FT RUCKER AL 36362-5115	
SPRINGFIELD VA 22150-3199			
CDR		CDR ARMY ENGR SCHOOL	
ARMY COLD REGION TEST CTR		ATTN: ATSE CD	1
ATTN: STECR TM	1	FT LEONARD WOOD	
STECR LG	1	MO 65473-5000	
APO AP 96508-7850			
CDR		CDR 49TH QM GROUP	
ARMY BIOMED RSCH DEV LAB		ATTN: AFFL GC	1
ATTN: SGRD UBZ A	1	FT LEE VA 23801-5119	
FT DETRICK MD 21702-5010			
CDR FORSCOM		CDR ARMY ORDN CTR	
ATTN: AFLG TRS	1	ATTN: ATSL CD CS	1
FT MCPHERSON GA 30330-6000		APG MD 21005	
CDR TRADOC			
ATTN: ATCD SL 5	1	CDR ARMY SAFETY CTR	
INGALLS RD BLDG 163		ATTN: CSSC PMG	1
FT MONROE VA 23651-5194		CSSC SPS	1
		FT RUCKER AL 36362-5363	
CDR ARMY ARMOR CTR			
ATTN: ATSB CD ML	1	CDR ARMY ABERDEEN TEST CTR	
ATSB TSM T	1	ATTN: STEAC EN	1
FT KNOX KY 40121-5000		STEAC LI	1
		STEAC AE	1
		STEAC AA	1
		APG MD 21005-5059	
CDR ARMY QM SCHOOL		CDR ARMY YPG	
ATTN: ATSM PWD	1	ATTN: STEYP MT TL M	1
FT LEE VA 23001-5000		YUMA AZ 85365-9130	
ARMY COMBINED ARMS SPT CMD		CDR ARMY CERL	
ATTN: ATCL CD	1	ATTN: CECER EN	1
ATCL MS	1	P O BOX 9005	
ATCL MES (C PARENT)	1	CHAMPAIGN IL 61826-9005	
FT LEE VA 23801-6000			
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CDR ARMY FIELD ARTY SCH		AMC FAST PROGRAM	
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FT EUSTIS VA 23604-5000		FT LEWIS WA 98433-5000	
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		RED RIVER ARMY DEPOT	
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WASHINGTON DC 20350-2000

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P O BOX 7176
TRENTON NJ 08628-0176

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ATTN: SEA 03M3
2531 JEFFERSON DAVIS HWY
ARLINGTON VA 22242-5160

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STE 3719
FT BELVOIR VA 22060-6224

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NAVAL SURFACE WARFARE CTR
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ANNAPOLIS MD 21402-5067

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NAVAL AIR SYSTEMS CMD
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1421 JEFFERSON DAVIS HWY
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MARINE CORPS SYS CMD
2033 BARNETT AVE
QUANTICO VA 22134-5080

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PROG MGR COMBAT SER SPT
MARINE CORPS SYS CMD
2033 BARNETT AVE STE 315
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2030 BARNETT AVE STE 315
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5880 CHANNEL VIEW BLVD
JACKSONVILLE FL 32226-3404

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CDR
MARINE CORPS LOGISTICS BA
ATTN: CODE 837
814 RADFORD BLVD
ALBANY GA 31704-1128

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PSC BOX 20090		CAMP PENDLETON	
CAMP LEJEUNE		CA 92055-5702	
NC 28542-0090			

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HI 96861-4118	

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WASHINGTON DC 20330-1030			

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WASHINGTON DC 20330-1030		KELLY AFB TX 78241-6439	

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WL/POSF	1	ROBINS AFB	
1790 LOOP RD N		GA 31098-1647	
WRIGHT PATTERSON AFB			
OH 45433-7103			

AIR FORCE MEEP MGMT OFC	1
OL ZC AFMC LSO/LOT PM	
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BLDG 613 STE 2	
ENGLIN AFB FL 32542-5303	

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		WASHINGTON DC 20585	

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220 N. VIRGINIA		ANN ARBOR MI 48105	
BARTLESVILLE OK 74003			

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800 INDEPENDENCE AVE SW	
WASHINGTON DC 20590	