Predicting Cetane Numbers of n-Alcohols and Methyl Esters from their Physical Properties

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Cetane numbers (C#) for the homologous series of straight-chain, saturated n-alcohols, C_5-C_{12} and C_{14}, were determined according to ASTM D 613. Measured C# ranged from 18.2–80.8 and increased linearly with carbon number (CN). Regression analyses developed equations that related various physical properties or molecular characteristics of these alcohols to calculated C#. The degree of relationship between measured and calculated C# was expressed as R^2. The decreasing order of the precision with which these properties correlated with C# was: boiling point (bp) > melting point (mp) > CN > heat of combustion (HG) > refractive index (n^{20}_D) > density (d). This ranking was based upon R^2 (0.99–0.96) and the Average % Error (2.8–7.2%). C# were also determined for straight-chain homologs of saturated methyl esters with CN of 6, 10, 12, 14, 16 and 18. C# ranged from 18.0–75.6 and increased curvilinearly with CN. Equations were also developed that related physical properties of these esters to C#. The precision with which these properties correlated with C# was: bp > viscosity (V) > heat of vaporization (HV) > HG > CN > surface tension (ST) > mp > n^{20}_D> d. R^2 ranged from 0.99 for bp to 0.98 for d. Equations for the alcohols were linear or quadratic, while equations for the esters were linear, quadratic or cubic based upon statistical considerations that included a Student's t-test. With related physical properties and these equations, accurate predictions of C# can be made for saturated n-alcohols and methyl esters.

KEY WORDS: Alternative fuels, prediction, regression analysis, saturated alcohols, saturated esters, statistics.

Alcohols and esters are being evaluated as fuel alternatives to #2 diesel oil for farm tractors. Shorter chain-length alcohols (C_4-C_{14}) are effective dispersing agents in various hybrid fuels, including aqueous alcohol-in-oil microemulsions (1-4). An important indicator of fuel quality for diesel fuel is its cetane number(s) (C#). C# is a measure of fuel performance determined in a special engine according to ASTM D 613. It is the percentage of cetane (hexadecane, C# = 100) which must be mixed with heptamethylnonane (C# = 15) to give the same ignition performance, under standard conditions, as the fuel in question. The C# of C_1-C_4 alcohols have been reported (5). However, except for our preliminary publication (6), no one has systematically determined C# for C_5-C_{12} and C_{14} alcohols.

Fatty esters have also been examined as an alternative fuel because their viscosities and boiling point ranges are much closer to those of #2 diesel oil than are vegetable oils (7-12). The use of methyl esters as alternative diesel fuel has been reviewed (13). C# of a number of vegetable oil esters have been reported. These include the esters of soy (9), sunflower (8), rape (14), peanut and palm oils (11). C# of esters ranging in carbon number (CN) from 8-18 have been measured (15) and will be discussed later. In the present work, C# for a series of straight-chain, saturated methyl esters with CN of 6, 10, 12, 14, 16 and 18 were determined.

C# are measured at considerable expense in a specially designed single-cylinder diesel engine that requires from 1 pint to 1 quart of liquid per run. Equations that predict C# might thus avoid this expensive and time-consuming test. Such equations relating measured C# and physical properties or molecular characteristics of the alcohols and esters were developed through regression analysis. For simplicity, the term physical properties will be used throughout this paper to discuss both physical properties and molecular characteristics. In this paper, the ASTM C# and calculated values for these alcohols and esters, as well as the corresponding equations, are reported.

EXPERIMENTAL PROCEDURES

Materials and C# determinations. C_5-C_{11} alcohols were purchased in purities of 97-99% from Aldrich Chemical Co. (Milwaukee, WI). Dodecanol and tetradecanol, with purities of 99% and 96%, were purchased from Sigma Chemical Co. (St. Louis, MO). The C# of longer-chain alcohols, such as hexadecanol and octadecanol, could not be measured because their high melting points prevented them from being treated as liquid fuels. C# were determined according to ASTM D 613 by Southwest Research Institute, San Antonio, TX.

Methyl caproate (Me 6:0, 99%), was purchased from Aldrich. Quantum, Emory Division (Los Angeles, CA) donated methyl decanoate (99%), laurate (96%), myristate, palmitate and stearate, all 95% pure. The composition of esters with purities of 95-96% was known. The impurities were generally higher and lower homologs of the ester being tested. Calculations that allowed for the weighted percentage of these impurities showed no significant change in measured C#. It is believed that the same would apply to the alcohols above. Southwest Research Institute determined the C# of these esters by ASTM D 613.

Heat of combustion (HG) of alcohols and esters. HG values for C_5, C_7 and C_8 alcohols were those of Kharasch (16). HG values for C_{10}, C_{12} and C_{14} alcohols were previously reported (17). Based on the linear relationship between HG and CN for the six alcohols above, HG for the C_6, C_9 and C_{11} alcohols were determined by regression analysis. Procedures for determining HG of the esters as well as their HG values have been reported (18).

Physical properties. For the alcohols, boiling points (bp), melting points (mp), density (d) and refractive

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index (n^{20}_D) were taken from the Aldrich catalog (19), Lang (20) and Bailey (21). For the esters, bp and mp were compiled from Aldrich (19) and Lange (20). Data for viscosity (V), heat of vaporization (HV), n^{20}_D, d and surface tension (ST) were obtained from Bailey (21).

Statistical procedures. Lotus 1-2-3 (Lotus 1-2-3 is a registered trademark of Lotus Development Corp., Cambridge, MA) was used to determine data regression and other statistical data. The output for regression analysis included calculated Y, Error, (the difference between measured and calculated Y), % Error, R^2 (correlation coefficient squared), the X coefficient(s), the Y intercept, the Standard Error of Y Estimate and the degrees of freedom. From these statistical data, equations were developed that linked C# (Y) to the physical constants (X).

These equations were found to be either linear, quadratic or cubic. As the polynomial degree increased, so did the R^2. To decide which polynomial degree was justified, a statistical significance test was employed from Lotus 1-2-3. Briefly, a linear regression analysis was first performed and the R^2 was determined. Next, a quadratic regression analysis was performed that resulted in a higher R^2. A Student's t-value was calculated with a formula that used the increase in R^2, the degrees of freedom and the R^2 (22). This formula is:

$$t \text{ value} = [(\text{increase in } R^2)*(\text{degrees of freedom})/(1-R^2)]^{1/2}$$

This calculated t-value was then compared to a tabular t-value at the 95% confidence level in a Student's t-distribution table. If the calculated t-value exceeded the tabular t-value, the goodness of fit was considered significant. The process was then repeated at the next higher polynomial degree until the calculated value no longer exceeded the tabular value. The equation corresponding to the last polynomial degree having significance was the one used. This procedure objectively allows selection of the proper polynomial degree rather than guessing at a polynomial degree that may be incorrect.

RESULTS AND DISCUSSION

C# and physical properties of the alcohols. In Table 1 are listed the measured C# for the C_5-C_{12} and C_{14} alcohols and their physical properties. Obviously, a very close relationship exists between CN, electron number (EN) and molecular weight (MW) for a given alcohol. EN was determined as described by Kharasch (16). For example, the EN of an alcohol was defined as the number of valence electrons related to the residual carbon and hydrogen atoms after subtracting H_2O from the empirical formula. Thus, CH_3OH has an EN of 6. We have shown that these three characteristics for a given saturated compound may be used interchangeably to produce the same calculated Y value, % Error and R^2, etc. (17,18). Therefore, only CN is discussed further in this paper as representative of these variables.

The source for most of the physical properties in Table 1 was the Aldrich Catalog (19). A check of several handbooks as additional sources for these properties showed some small differences from the values listed, but these differences did not significantly change the conclusions derived from using the data in Table 1.

Prediction of C# from physical properties of the alcohols. By using physical properties from Table 1 as X and measured C# as Y variables, regression analyses produced equations for determining calculated C#. These values were then compared to the measured C# as summarized in Table 2. R^2 shows the correlation between measured and calculated C#. The physical properties were ranked according to descending order of precision. Based on these criteria, the equation based on bp predicted C# with the highest R^2 (0.9931), with mp a close second. A ranking of the physical properties of the n-alcohols in descending order according to their ability to predict C# is: bp > mp > CN > HG > n^{20}_D> d.

Regression equations for the alcohols. The regression equations that were used to calculate C# as well as related statistical data are shown in Table 3. These equations show a curvilinear relationship between C# and bp, mp and d, but a linear relationship between

TABLE 1

Cetane Numbers and Physical Properties of C_5–C_{12} and C_{14} n-Alcohols

Saturated n-alcohol	Measured cetane number	Carbon number	Electron number	Molecular weight	Heat of combustion kg-cal/mole[a]	Boiling point, °C 760 mm[b]	Melting point, °C[b]	Density[b]	Refractive index[b]
1-pentanol	18.2	5	30	88.15	794	138	−78	0.811	1.4093
1-hexanol	23.3	6	36	102.18	950	156.5	−52	0.814	1.4179
1-heptanol	29.5	7	42	116.21	1105	176	−36	0.822	1.4232
1-octanol	39.1	8	48	130.23	1262	196	−15	0.827	1.4290
1-nonanol	46.2	9	54	144.26	1422	215	−6	0.827	1.4334
1-decanol	50.3	10	60	158.29	1582	231	7	0.829	1.4370
1-undecanol	53.2	11	66	172.31	1736	243[c]	11	0.830	1.4400
1-dodecanol	63.6	12	72	186.34	1899	260	24	0.831[d]	—
1-tetradecanol	80.8	14	84	214.40	2202	289	38	—[e]	—

[a] See Experimental Procedures.
[b] Reference 19.
[c] Reference 21.
[d] Reference 20.
[e] The density of 1-tetradecanol was not listed here because it was determined at 38°C or higher, and thus could not be compared to the other densities that were determined at room temperature.

TABLE 2

Prediction of Cetane Numbers from Physical Properties of n-Alcohols

		Boiling point		Melting point		Carbon number		Heat of combust.		Refractive ind.		Density	
Alcohol chain length	Measured cetane #	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.
5:0	18.2	18.6	−2.2	18.8	−3.3	17.1	6.0	17.2	5.5	15.2	16.5	20.6	−13.2
6:0	23.3	23.7	−1.7	23.3	0.0	23.9	−2.6	23.9	−2.6	25.8	−10.7	19.9	14.6
7:0	29.5	29.8	−1.0	28.5	3.4	30.6	−3.7	30.5	−3.4	32.3	−9.5	29.3	0.7
8:0	39.1	36.8	5.9	38.4	1.8	37.4	4.3	37.3	4.6	39.4	−0.8	43.6	−11.5
9:0	46.2	44.2	4.3	43.7	5.4	44.2	4.3	44.2	4.3	44.9	2.8	43.6	5.6
10:0	50.3	50.9	−1.2	52.4	−4.2	50.9	−1.2	51.1	−1.6	49.3	2.0	51.2	−1.8
11:0	53.2	56.3	−5.8	55.3	−3.9	57.7	−8.5	57.7	−8.5	53.0	0.4	55.4	−4.1
12:0	63.6	64.4	−1.3	65.6	−3.1	64.4	−1.3	64.7	−1.7	—	—	59.8	6.0
14:0	80.8	79.4	1.7	78.2	3.2	78.0	3.5	77.7	3.8	—	—	—	—
Average			—		—		—		—		—		—
% Error			2.8		3.1		3.9		4.0		6.1		7.2
R^{2a}		0.9931		0.9914		0.9877		0.9871		0.9769		0.9627	

[a]Correlation coefficient squared for measured C# vs calculated C#.

TABLE 3

Regression Equations for Cetane Numbers (Y) vs Physical Properties (X) for n-Alcohols

Physical property	Equation	R^2	Std. error of Y estimate
Boiling point	$Y = 0.85 + (-0.0021X) + 0.000948X^2$	0.9931	1.9
Melting point	$Y = 47.52 + 0.663X + 0.0038X^2$	0.9914	2.1
Carbon number	$Y = (-16.69) + 6.761X$	0.9877	2.4
Heat of combustion	$Y = (-17.00) + 0.0430X$	0.9871	2.4
Refractive index	$Y = (-1721.18) + 1232.06X$	0.9769	2.3
Density	$Y = 86366.66 + (-212286.18X) + 130477.92X^2$	0.9627	3.6

C# and CN, HG and n^{20}_D. The curvilinear relationship between C# and bp, for example, is shown in Figure 1. The close juxtaposition of data points and calculated line attest to the excellent correlation (R^2, 0.9931) between measured and calculated C#. The linear relationship between C# and CN is illustrated in Figure 2. The goodness of fit is nearly equal to that shown in Figure 1 (R^2, 0.9877).

The standard Error of Y Estimate indicates the amount of error in the calculated C#. Except for d, 3.6, the error values in Table 3 are less than the reproducibility range of 2.5–3.3 specified by ASTM D 613. Thus, R^2 and Standard Error data give assurance that the equations in Table 3 (except the one for d) can be used to predict C# satisfactorily from the corresponding physical properties of saturated n-alcohols.

C# and physical properties of the esters. The measured C# and physical properties of the saturated, straight-chain methyl esters used in our study are shown in Table 4. This Table includes the same properties employed with the alcohols, and in addition, V, HV and ST. Examination of the ester data for measured C# and CN suggests a curvilinear relationship between these two variables, in contrast to the linear relationship shown in Figure 2 for the alcohols. Indeed, regression analysis and the Student's t-test established that a quadratic equation was justified in describing the relationship between C# and CN for these esters. The small increase in C# noted when progressing from Me 14 to Me 16 to Me 18 are not significantly different.

The C# of straight-chain, saturated methyl esters with CN of 8, 10, 12, 14, 16 and 18 have also been reported by Klopfenstein (15). Our values in Table 4 are in good agreement with values reported for his esters with CN of 10, 12 and 16. The C# of his Me 14 and Me 18 esters, however, differed from those in the Table by 7 and 11, respectively. There are several reasons that might explain these differences: i) His esters were prepared from commercial grades of fatty acids and reagent grade alcohols that were not further purified. The esters were purified in the final step by vacuum distillation. Fatty acid composition was deter-

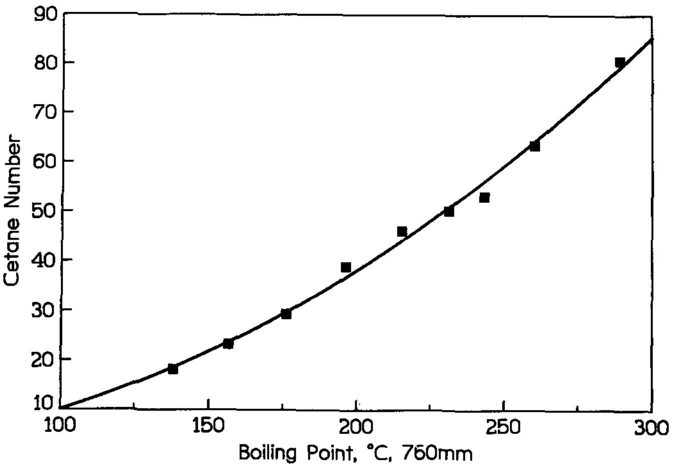

FIG. 1. Plot of cetane number vs boiling point for n- alcohols.

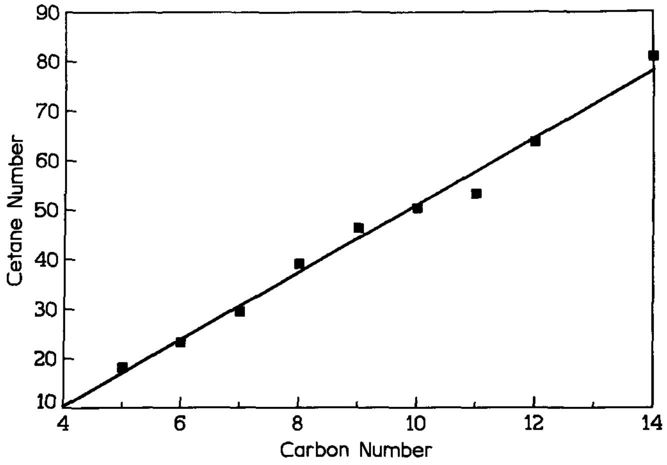

FIG. 2. Plot of cetane number vs carbon number for n-alcohols.

mined by gas-liquid chromatography. As this analysis was not described as quantitative, errors in the composition could have been introduced at this stage. ii) The purity of his Me 18 ester was 92.1%. The 7.9% unknown impurities may have affected the reported C#. iii) The C# for Klopfenstein's Me 18 ester was determined as an ester-petroleum ether mixture (70:30, v/v). Although the ASTM method permits the use of secondary reference fuels, such fuels must be calibrated to primary reference fuel blends. Instead, the C# of the petroleum ether used was determined by calculation from a 70:30 mixture of Me 16:0 and petroleum ether. iv) The viscosity and surface tension differences resulting from diluted vs neat ester might further explain the difference between his value (86.9) and the value in Table 4 (75.6).

Klopfenstein noted that, "It appears that for the methyl esters there is a nonlinear increase in cetane number with increasing chain length of the fatty acid." For his methyl esters he gave the equation:

$$Y = 24.48 + 8.431\ X + (-\ 0.1299\ X^2)$$

to show the nonlinear relationship between the calculated C#, Y, and the chain length of the fatty acid, X. His data were subjected to regression analysis and Student's t-test as discussed above, with the following results: i) The 24.48 in the equation above should be negative; and ii) only a linear equation is justified, that equation being:

$$Y = (-3.96) + 5.045\ X$$

With data reported by Ryan and Stapper (23) for hexane, octane, tetradecane and hexadecane, we obtained a curvilinear relationship by regression analysis of C# vs CN. For these hydrocarbons the equation was:

$$Y = (-30.90) + 15.446\ X + (-0.4562\ X^2)$$

where Y is C# and X is CN. Thus, the change in C# with CN can be described by quadratic equations for both esters and hydrocarbons within the reported ranges of CN. As noted earlier, the relationship between C# and CN for alcohols was linear over the C 5 to C 14 range studied. For the esters over the C 6 to C 14 range, the relationship between C# and CN was also linear (R^2, 0.9981). Only Me 16 and Me 18 caused the change in slope in the curve. This suggests that had it been possible to determine C# for C 16 and C 18 alcohols, the alcohol curve might have been similar in shape to that of the ester curve. By the same reasoning, had the C# for the C 16 and C 18 alcohols been available, the C# vs bp plot for the alcohols may have tended to level off at the higher C#, as did the esters.

Prediction of C# from physical properties of the esters. The measured C# and physical properties of the esters were analyzed to obtain regression equations and to rank the physical properties from R_2 values. The results are shown in Table 5. Based on R^2, the descending order of precision for predicting C# from physical properties is: bp > V > HV > HG > CN >ST > mp > n^{20}_d> d. As with the alcohols, bp predicted C# most precisely. The R^2 was essentially perfect (0.9999), and the Average % Error, 0.1, was at least an order of magnitude lower than the other values shown in the Table. Regression analysis of the C# - bp data and the Student's t-test showed that a cubic equation was justified for relating these two variables. Figure 3 is a plot of C# vs bp data. Other correlations having R^2 values of at least 0.99 were V, HV, HG and CN. These properties may also be used to predict C#.

Regression equations for the esters. Regression equations and associated statistical data that relate the C# of the esters to selected physical properties are shown in Table 6. C# are related to bp and V by cubic equations; to HV, HG, CN and ST by quadratic equations; and to mp, n^{20}_d and d by linear equations. The cubic equations have the highest R^2 and the lowest Standard Errors. Thus, the bp equation is preferred for use with saturated esters. The equation for V with its high R^2 (0.9985) and relatively low Standard Error

TABLE 4

Cetane Numbers and Physical Properties of Methyl Esters

Methyl esters	Measured cetane number	Boiling point, 760 mm^a	Viscosity, cSt, 40°C^c	Heat of vaporizat., cal/g, 1 mm^c	Heat of combustion, kg-cal /mol^d	Carbon number	Surface tension, dynes/cm, 40°C^c	Melting point, °C^a	Refrac. index, 40°C^c	Density, 75°C^c
Methyl caproate	18.0	151	0.785	—	1000	6	24.2	−71	1.3963	0.8313
Methyl decanoate	47.9	224	1.69	81	1625	10	26.1	−18	1.4174	0.8271
Methyl laurate	60.8	262	2.28	78	1940	12	27.0	5	1.4240	0.8259
Methyl myristate	73.5	323	3.23	74	2254	14	27.8	18.4	1.4291	0.8252
Methyl palmitate	74.3	330^b	4.32	71	2550	16	28.4	28	1.4332	0.8247
Methyl stearate	75.6	356^b	5.61	68	2859	18	29.1	39	1.4365	0.8244

[a] References 19 and 20.

[b] Extrapolated from reduced pressure.

[c] Reference 21.

[d] Reference 18.

TABLE 5

Prediction of Cetane Numbers from Physical Properties of Methyl Esters

		Boiling point		Viscosity		Heat of vapor.		Heat of combust.		Carbon number		Surface tens.		Melting point		Ref. ind.		Density	
Ester chain length	Measured cetane #	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.	Calc. C#	% Err.
6:0	18.0	18.0	0.0	17.7	1.7	—	—	17.2	4.4	17.2	4.4	17.2	4.4	18.8	−4.4	18.1	−0.6	16.3	9.4
10:0	47.9	48.0	−0.2	48.5	−1.3	47.9	0.0	50.1	−4.6	50.2	−4.8	50.4	−5.2	48.2	−0.6	50.2	−4.8	52.6	−9.8
12:0	60.8	60.7	0.2	61.1	−0.5	61.1	−0.5	61.7	−1.5	61.7	−1.5	61.8	−1.6	61.0	−0.3	60.3	0.8	63.0	−3.6
14:0	73.5	73.6	−0.1	72.0	2.0	72.2	1.8	70.0	4.8	69.9	4.9	69.5	5.4	68.4	6.9	68.0	7.5	69.0	6.1
16:0	74.3	74.3	0.0	75.3	−1.3	75.7	−1.9	74.7	−0.5	74.8	−0.7	73.9	0.5	73.8	0.7	74.3	0.0	73.3	1.3
18:0	75.6	75.6	0.0	75.4	0.3	75.1	0.7	76.5	−1.2	76.4	−1.1	77.4	−2.4	79.9	−5.7	79.3	−4.9	75.9	−0.4
Average			—		—		—		—		—		—		—		—		—
% error			0.1		1.2		1.0		2.8		2.9		3.2		3.1		3.1		5.1
R^{2a}		0.9999		0.9985		0.9930		0.9921		0.9919		0.9893		0.9822		0.9805		0.9799	

[a] Correlation coefficient squared for measured C# vs calculated C#.

TABLE 6

Regression Equations for Cetane Numbers (Y) vs Physical Properties (X) for Methyl Esters

Physical property	Equation	R^2	Std. err. of Y est.
Boiling point	$Y = (-41.30) + 0.2785\,X + 0.001209\,X^2 + 3\text{E-}06\,X^3$	0.9999	0.1
Viscosity	$Y = (-23.48) + 61.6828\,X + (-12.7738\,X^2) + 0.87697\,X^3$	0.9985	1.4
Heat of vapor.	$Y = (-1054.90) + 32.324\,X + (-0.23097\,X^2)$	0.9930	1.4
Heat of comb.	$Y = (-62.96) + 0.09700\,X + (-1.69\text{E-}05\,X^2)$	0.9921	2.6
Carbon number	$Y = (-57.26) + 14.892\,X + (-0.4149\,X^2)$	0.9919	2.6
Surface tension	$Y = (-1500.58) + 104.656\,X + (-1.7330\,X^2)$	0.9893	3.0
Melting point	$Y = 58.22 + 0.556\,X$	0.9822	3.4
Refrac. index	$Y = (-2107.38) + 1522.21\,X$	0.9805	3.5
Density	$Y = 7206.14 + (-8648.96\,X)$	0.9799	3.6

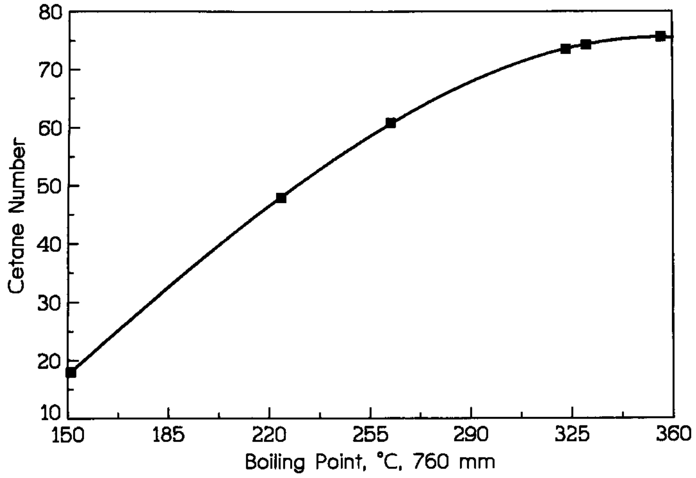

FIG. 3. Plot of cetane number vs boiling point for methyl esters.

(1.4) is a good second choice. Because the ASTM permits a reproducibility range of 2.5–3.3, equations for HV, HG, CN and ST could also be employed.

In conclusion, C# of n-alcohols and methyl esters can be precisely predicted from a variety of physical properties and molecular characteristics. In comparing the equations that relate C# to bp for both the alcohols and esters, the latter should give somewhat more accurate predictions of C# because of their higher R^2 and lower standard errors.

A useful extension of the present work is to have the ability to predict the C# of blends. Toward this end research has been initiated to determine the C# of unsaturated esters (Me 18:1, 18:2 and 18:3). These new data may permit the prediction of C# for blends of methyl esters of vegetable oils. In addition, C# for both saturated and unsaturated triglycerides are also being determined to see how well these values can be used to predict C# for vegetable oils. Preliminary results suggest that satisfactory predictions of C# for vegetable oils are feasible.

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