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A Correlation of United States Tar Sand Bitumen Viscosities With NMR Spectroscopic Parameters -- Progress Report

Topical Report

D.A. Netzel
T.F. Turner

June 1989

Work Performed Under Contract No.: DE-AC21-86MC11076

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

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SUMMARY

Many physicochemical properties of bitumens must be known in order to mathematically model and simulate the processes used for the recovery of bitumen from tar sands. One fundamental property is the temperature dependence of viscosity. This property determines the heat transfer and flow characteristics of a bitumen through a porous media.

A method has been developed whereby the viscosity of a tar sand bitumen at any temperature can be calculated from nuclear magnetic resonance parameters. The method is semiempirical but is based upon some fundamental theoretical concepts for molecular mobility and intermolecular interactions. Using this method, the viscosities of three United States tar sand bitumens have been correlated to the weighted average spin-spin relaxation rates for the semiliquid, solidlike mobile, and solidlike rigid phases of the bitumens. The results indicate that bitumens with a high viscosity have a greater amount of solidlike rigid phase and lesser amounts of solidlike mobile and semiliquid phases than do the bitumens with low viscosity. It is also shown that the viscosity of a tar sand bitumen over a 100 degree temperature range can be determined from a single NMR experiment conducted near room temperature.

INTRODUCTION

Tar sand bitumens are complex mixtures of high molecular weight aliphatic and hetero- and polycondensed-aromatic compounds. Because of the asymmetric structure of the molecules and the strong intermolecular interactions between molecules, tar sand bitumens are highly viscous materials resembling heavy residues or "bottoms" produced by the distillation of crude oils. The bitumens and heavy oils from various geographic locations differ widely in their chemical composition and physical properties.

A knowledge of the chemical, thermodynamic, and physical properties of the bitumen in tar sands is required to mathematically model and simulate the processes used in the recovery of bitumen from tar sands. A major and common hindrance to the development of a mathematical model, and in turn, the economic recovery and processing of all bitumens and also that of heavy oils, is the lack of a fundamental description of the viscosity as a function of temperature, pressure, and dissolved gases. It is viscosity that determines the heat transfer and flow characteristics of a bitumen or heavy oil through porous media such as encountered during in situ extraction processes.

Because of the time and expense involved in obtaining the necessary experimental viscosity data used in the mathematical models of reservoir and extraction simulations, a method to accurately predict the viscosity of bitumens and heavy oils over a wide temperature range is required. Although the viscosity of liquids is one of the most commonly studied physical properties, it is still a difficult property to predict from molecular structural data for highly associated materials.

Viscosity may be thought of as a measure of the force needed to overcome the mutual attraction of the molecules so that they can be displaced relative to each other. The stronger the molecular interaction, the higher is the viscosity of a liquid. With increasing temperature, the increase in the kinetic energy of the molecules overcomes the intermolecular forces, increases molecular mobility, and reduces the viscosity. Liquid viscosities are very sensitive to the structure and association effects of the constituent molecules at temperatures below the reduced temperature (T/T_c, where T_c is the critical temperature) of about 0.7 (Reid et al. 1987). The molecular theory of viscosity is discussed by Reid et al. (1987). A satisfactory theory of viscosity has yet to be developed. Because of the lack of a well-defined theoretical description of the liquid state, in particular the viscosity of a liquid, many empirical relationships between viscosity and the molecular structure and physical properties of the liquid have been developed (Reid et al. 1987; Lymann et al. 1982).

The temperature dependence of viscosity can be obtained using property estimation methods when few or no experimental data are available. Most methods are limited to pure fluids. Recently, Twu (1985) described a simple, reliable, accurate, and internally consistent method for the calculation of liquid viscosities of petroleum fractions. For light distillate fuel products, the method is quite successful.

However, for tar sand bitumens, heavy petroleum residues, and coal liquids, all of which contain high concentrations of hetero- and polyaromatic molecules and, therefore, have strong intermolecular interactions, this method may be inadequate. Few viscosity correlations have been published for United States tar sand bitumens (Christensen et al. 1984). The viscosity as a function of temperature for several Alberta tar sand bitumens have been reported by Svrcek and Mehrotra (1988) and the references cited therein.

Khan et al. (1984) reviewed several viscosity models for Athabasca tar sand bitumen. Their attempt to correlate the bitumen viscosity with modified Eyring or Hildebrand models resulted in limited success. Most successful were the two-parameter empirical viscosity correlations based on experimental data-fitting using regression techniques. Mehrotra and Svrcek (1987) and Johnson et al. (1987) used the corresponding state method for calculating the viscosity of bitumens obtained from four regions in Alberta: Athabasca, Peace River, Marguerite Lake, and Wabasca. The calculated bitumen viscosities compare satisfactorily with published viscosity data over a wide range of temperatures. However, the corresponding state method requires the critical properties, acentric factors and mole fraction of each of the bitumen pseudocomponents as input data. The number of parameters needed for calculating the viscosity make this method less attractive than others for use in mathematical models. Recently, Svrcek and Mehrotra (1988) investigated eight empirical correlations involving two to five empirical constants for the temperature dependence of the viscosity of Alberta bitumens. They developed a one-parameter generalized viscosity equation. To determine the parameter, only one viscosity measurement is required. However, the correlation has little, if any, theoretical basis. To overcome the theoretical inadequacy of the empirical estimation methods, these correlative methods must be more directly related to molecular motion which, in turn, is related to molecular structure and interactions.

In this paper, a method is described whereby the viscosities as a function of temperature for United States tar sand bitumens is estimated from nuclear magnetic resonance (NMR) parameters. The viscosity-NMR correlation is based upon theoretical consideration of molecular motion and intermolecular interactions.

NMR RELAXATION METHODOLOGY

Nuclear magnetic resonance relaxation spectroscopy (Netzel and Miknis 1977) is commonly used to investigate molecular mobility and association. With this technique, the nuclear dipolar spin-lattice (T_1) and spin-spin (T_2) relaxation times are measured. For a single-component system, the theoretical correlation between viscosity and the nuclear relaxation times has been developed (Abragam 1973). Because of the chemical complexity of tar sand bitumen, the following development of the correlation between the viscosity and NMR parameters is based on analogy and is, therefore, semiemperical.

If a nuclear spin system (^1H or ^{13}C) is perturbed by a short radiofrequency (rf) pulse, there is a redistribution of spins in the upper and lower energy states. After the perturbation, the spin system must revert to its original equilibrium condition. The recovery process by which the spin system returns to the equilibrium state is referred to as relaxation. The relaxation process occurs through the coupling interaction of the nuclear spin moments with resonance frequencies generated by fluctuating local magnetic and electric fields, which, in turn, are produced by thermal motions of the molecules. The extent of coupling depends upon the magnitude and rate of fluctuation of the local fields. The time dependence of the fluctuating local magnetic fields is related to the molecular dynamics of a molecule in any given physical state. The literature on the theoretical descriptions of the nuclear relaxation phenomena is extensive. Books by Abragam (1973), Noggle and Schirmer (1971), Becker (1980), Farrar and Becker (1971), and the references cited therein are suggested for the interested reader.

The length of time a molecule remains in any given position before a collision causes it to change its state of motion is the correlation time, τ_c. The correlation time describing molecular motion is related to the spin-lattice and spin-spin relaxation times:

$$1/T_1 \text{ or } 1/T_2 = n_i f(\tau_c) \qquad (1)$$

where n_i is the nuclear interaction constant. The relationship between the correlation time and the relaxation time is shown qualitatively in Figure 1. For nonviscous liquids, the average correlation time for the molecule to remain in one state of motion is about 10^{-12} seconds. Because T_2 involves the exchange of energy between nuclei, rather than between the nuclei and the environment (lattice), the dependence of T_2 on molecular motion differs from that of T_1 (Becker 1980). As shown in Figure 1, T_2 decreases monotonically with increasing τ_c throughout the liquid range and reaches a limiting value that is characteristic of a completely rigid solid lattice.

By applying some very stringent assumptions (Noggle and Schirmer 1971), the molecular correlation time of molecules in the liquid state can be related to viscosity using the microviscosity theory of Gierer and Wirtz (1953):

$$\tau_c = \eta V_m f_r / kT = f(\eta) \qquad (2)$$

where

η = the viscosity

f_r = microviscosity factor

k = Boltzman constant

T = temperature

V_m = volume of the molecule.

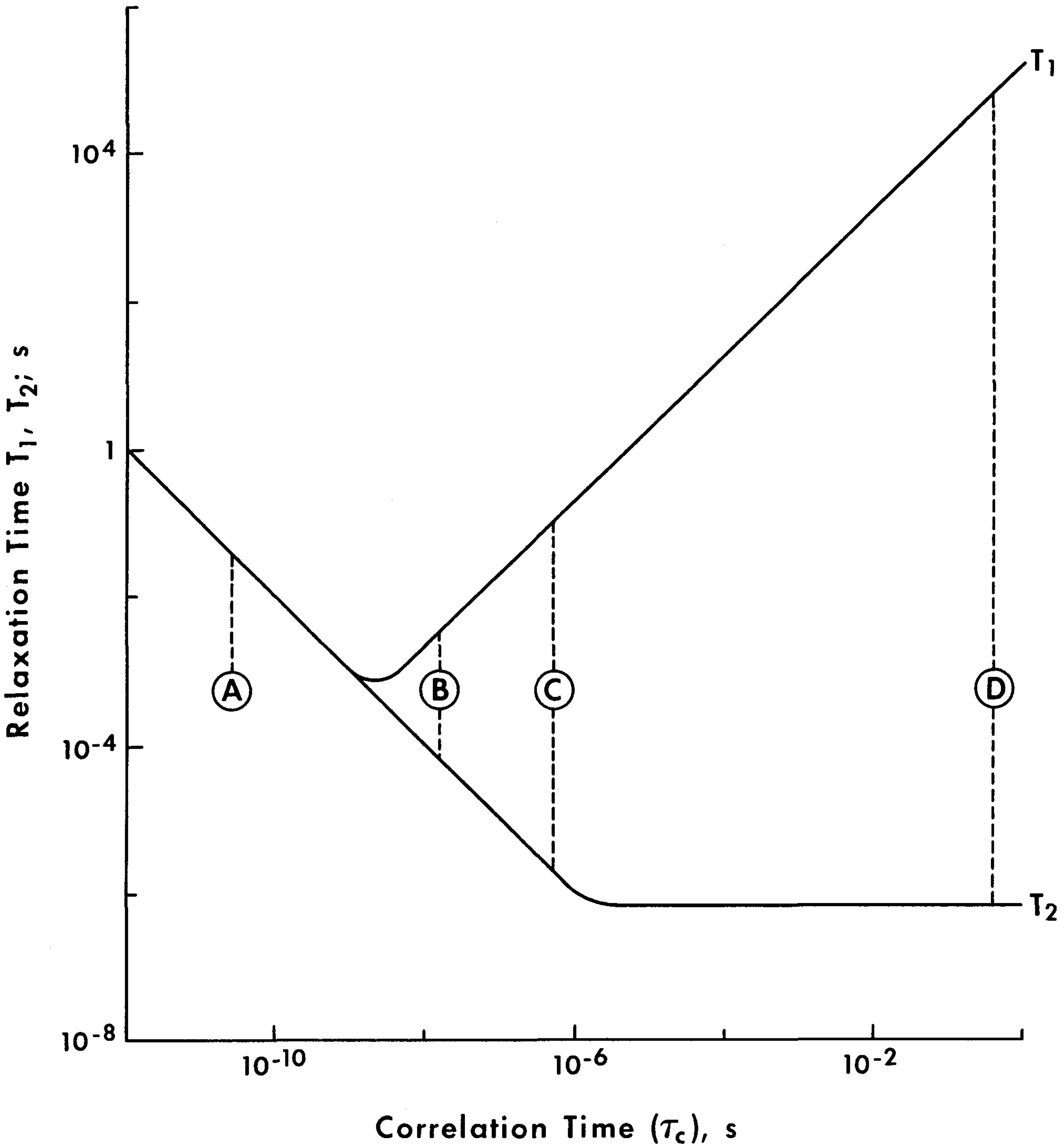

Figure 1. A Schematic Representation of the Dependence of Relaxation Times on Molecular Correlation Time for (A) Nonviscous Liquid, (B) Viscous Liquid, (C) Nonrigid Solid, and (D) Rigid Lattice.

The spin-spin relaxation time as a function of viscosity can then be written:

$$1/T_2 = n_i f(\eta) \tag{3}$$

For simplicity, it is assumed that the magnetization of a single-component system, M_t, measured at time, t, is an exponential function of the spin-spin relaxation time:

$$M_t = M_o \; e^{-t/T_2} \tag{4}$$

where M_o is the magnetization at time zero. This equation represents the observed free induction decay (FID) time domain spectrum. For chemically complex systems, the FID is a summation of the decay for many individual components. The FID for a multicomponent system is:

$$M_t' = \Sigma M_{oi} e^{-t/T_{2i}} \tag{5}$$

In equation 5, the assumption is that all components exhibit an exponential decay.

A tar sand bitumen is composed of three phases: solidlike rigid, solidlike mobile, and semiliquid (Sobol et al. 1985). These phases can be regarded as different degrees of molecular dipolar, aromatic π-bonding, and hydrogen-bonding interactions resulting in rigid and semimobile molecules through association. Thus, assuming a three-component system, equation 5 becomes:

$$M_t' = M_{ol} e^{-t/T_{2l}} + M_{om} e^{-t/T_{2m}} + M_{os} e^{-t/T_{2s}} \tag{6}$$

where M_t' is the measured magnetization at time, t, and M_{ol}, M_{om}, M_{os} are the initial magnetization for the semiliquid, solidlike mobile, and solidlike rigid phases at t=0. Also at t=0, M_t' is equal to the sum of the initial magnetization of each phase:

$$M_{t(o)}' = \Sigma M_{oi} \tag{7}$$

The fractional amount of each phase can be obtained from the ratio of the initial magnetization to the total magnetization:

$$X_i = M_{oi}/\Sigma M_{oi} \tag{8}$$

Values of T_{2i} and M_{oi} in equation 6 are obtained using nonlinear regression analysis.

Woessner et al. (1969) have shown that the average relaxation rate, $1/T_{2av}$, for a multicomponent decay curve is related to the weighted average of the relaxation rates of the components:

$$1/T_{2av} = \Sigma X_i/T_{2i} \tag{9}$$

Because $1/T_2 \propto f(\eta)$, equation 9 suggests that the viscosity of a multicomponent system such as a tar sand bitumen can be obtained from the weighted average of the relaxation rates of the phases.

Equation 9 is of the same form used for calculating the viscosity from group contribution property estimation methods:

$$f(\eta_{av}) = \ln\eta = a(X_l/T_{2l}) + b(X_m/T_{2m}) + c(X_s/T_{2s}) \tag{10}$$

The coefficients a, b, and c can be obtained from a multiple linear regression analysis, if the viscosity and the weighted relaxation rates are known for each of the three tar sand bitumens. The $\ln\eta$ in equation 10 is an arbitrarily chosen function for viscosity and is based upon empirical relationships reported in the literature (Reid et al. 1987). Equation 10 is a generalized equation for relating the viscosity of any heavy residue or bitumen to the corresponding NMR parameters.

EXPERIMENTAL SECTION

The Asphalt Ridge, Sunnyside, and Arroyo Grande tar sand bitumens used in this investigation are from three United States tar sand deposits. The Asphalt Ridge tar sand sample was obtained from the Uinta County quarry, Vernal, Utah. This tar sand had a bitumen content of ~13.6% by weight. The Sunnyside tar sand sample was obtained from the GNC and Chevron mine site near Price, Utah. The bitumen content is 10.4 wt %. The Arroyo Grande tar sand sample was obtained from a site located in San Luis Obispo County, California, in Township 31 S., Range 13 E., Sec. 31, NW 1/4 (Kuuskraa and Hammershaimb 1984). The percentage of bitumen in this sample is 10.7 wt % on a dry basis. The bitumen from each tar sand was obtained by solvent extraction using a standard Soxhlet extraction technique for the recovery of organic materials from mineral matrices (Christensen et al. 1984; Netzel and Coover 1989).

An IBM PC-20 NMR spectrometer with a fixed magnetic field of 0.47 T and a frequency field of 20 MHz for hydrogen was used. The temperature of the magnet and probe assembly was held constant at 313 ± 0.01 K. A 7.5-mm variable-temperature solid/liquid probe modified with a 1-cm Teflon spacer in the bottom of the probe assembly was used. This spacer was added to ensure that the sample was within the 20-mm coil height when it was placed in a 7.5-mm flat-bottom NMR tube. Optimization of the instrumental parameters using a Tektronix 466 storage oscilloscope was achieved following manufacturer's recommended procedures prior to each NMR experiment. The following instrument settings were used for each tar sand bitumen: an attenuation factor of 25, pulse delay of 2 s, bandwidth of 1 MHz, and 64 time-averaged FIDs. The spectrometer was operated in the phase-sensitive detector mode.

The free induction decay signal following a 90° pulse was stored using a Nicolet 1070 signal-averaging system. This system consisted of a model SD-71B analog-to-digital converter and model SW-71B wide-range sweep control. The amplitude of the FID was stored in 2000 channels. Two dwell-time settings were used to obtain sufficient number of data points in the region of fast decay. Dwell times of 9.9 and 11.9 μs/channel were used to convert the channel numbers into time. The background FID signal due to instrumental artifacts and an empty NMR tube was subtracted from the measured FID signals for the bitumens.

Using a multiple-regression analysis program, the FID signal for each bitumen was resolved into a sum of three Lorentzian exponential decay functions from which the spin-spin relaxation times and initial magnetization values were obtained.

RESULTS AND DISCUSSION

Because of intermolecular interactions, a tar sand bitumen may be thought of as existing in various pseudo phases in which molecules exhibit different degrees of mobility. The various dynamic states of motion of the molecules give rise to different nuclear relaxation times. Due to the different relaxation times, the total nuclear magnetization after excitation, decays nonexponentially. The nonexponential decay of the magnetization can be resolved into a sum of exponential decays where each exponential decay represents molecules within a given mobility range. Sobol et al. (1985) measured the spin-spin relaxation times for extracted Athabasca tar sand bitumen and concluded that the bitumen can be represented by three phases: a semiliquid phase (T_2 = 550 μs), a solidlike mobile phase (T_2 = 74 μs), and a solidlike rigid phase (T_2 = 15 μs).

A plot of the logarithm of the magnetization decay versus time is shown for the Asphalt Ridge bitumen in Figure 2. Both Sunnyside and Arroyo Grande bitumens show a similar magnetization decay after a 90° excitation pulse. The nonexponential decay for each bitumen was resolved into the sum of three Lorentzian exponential decay functions. Lack of data in the region of fast decay precluded the use of a Gaussian exponential function. The term Gaussian or Lorentzian is used to indicate a time-domain component whose frequency-domain component corresponds to Gaussian or Lorentzian line shapes. The selection of three Lorentzian exponential functions was based on an evaluation of the goodness-of-fit statistics. For all bitumens, the three-phase model was better than a two-phase model. The square of the correlation coefficient, r^2, was better than 0.999, and the model selection criterion was better than 6.67 for each bitumen, assuming three phases. For each bitumen, Table 1 lists the spin-spin relaxation times for each phase type. The tar sand bitumens are arranged in order of increasing viscosity from Asphalt Ridge to Sunnyside to Arroyo Grande. Although the T_2 values within each phase differ for each bitumen, the magnitude of the T_2 value for each phase type is the same. The relaxation times for Asphalt Ridge bitumen are longer than those for Arroyo Grande bitumen, which is qualitatively in agreement with the NMR relaxation theory that more mobile systems have longer relaxation times. The Sunnyside bitumen relaxation times, in general, lie between the other two bitumens, as does its viscosity value.

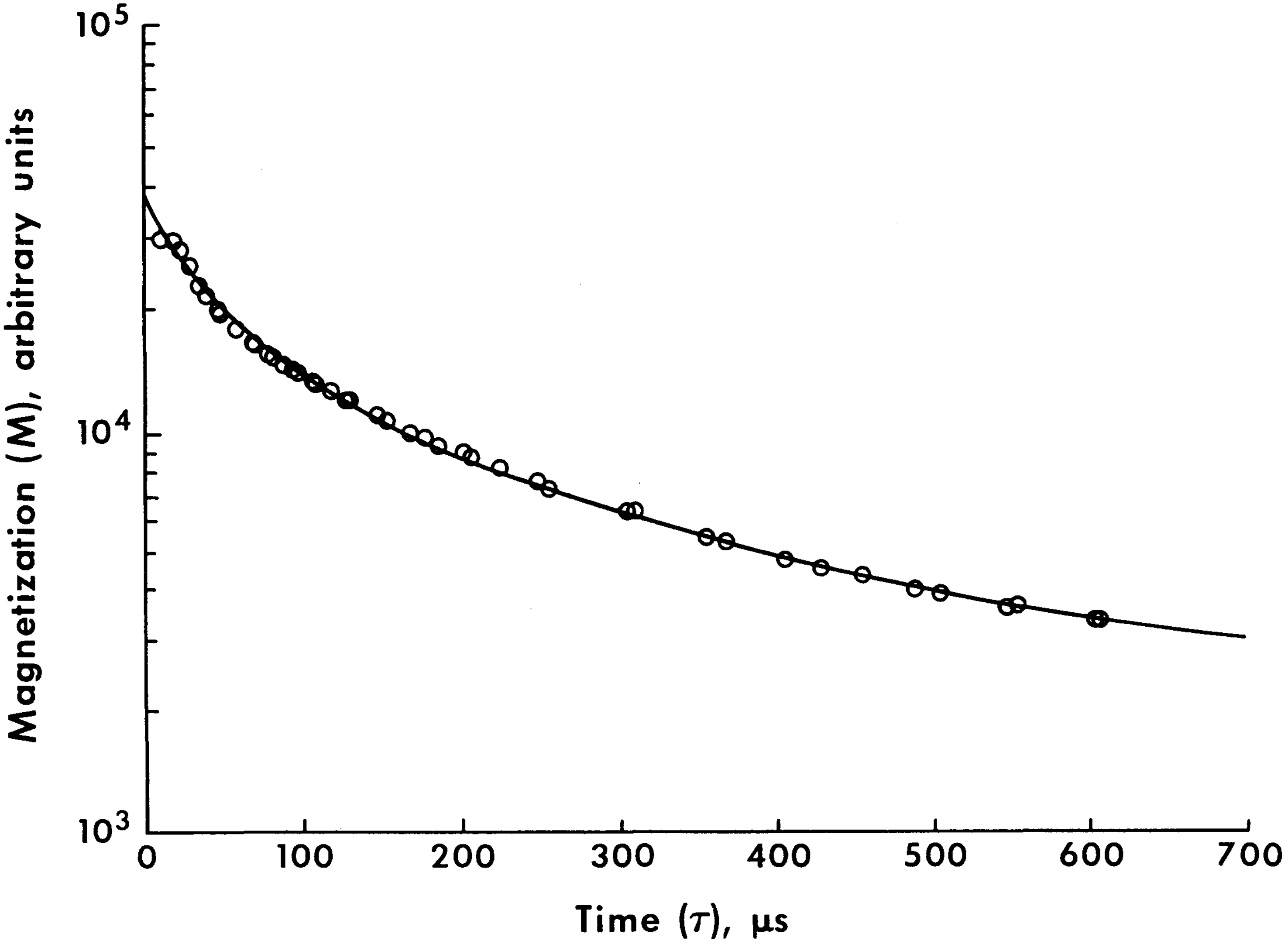

Figure 2. Free Induction Decay of Asphalt Ridge Tar Sand Bitumen

Table 1. Spin-Spin Relaxation Times for Tar Sand Bitumens at 313 K

	Spin-Spin Relaxation Time, μs		
Phase	Asphalt Ridge	Sunnyside	Arroyo Grande
Solidlike rigid	12	10	8
Solidlike mobile	70	49	39
Semiliquid	270	211	235

Beginning with the most mobile phase, successive extrapolation of the exponential decay for each phase back to zero time determines the magnetization content for that phase. The relative fraction of each phase can be determined by dividing the magnetization content of each phase by the total magnetization content. Table 2 lists the fraction of solidlike rigid, solidlike mobile, and semiliquid phases at 313 K in the Asphalt Ridge, Sunnyside, and Arroyo Grande bitumens.

Table 2. Fraction of Solidlike Rigid, Solidlike Mobile, and SemiLiquid Phases in Tar Sand Bitumens

	Fraction of Phase at 313 K		
Phase	Asphalt Ridge	Sunnyside	Arroyo Grande
Solidlike rigid	0.480	0.569	0.633
Solidlike mobile	0.233	0.179	0.150
Semiliquid	0.287	0.252	0.228

The data in Table 2 show that as viscosity increases from Asphalt Ridge to Arroyo Grande, the fractional amount of solidlike rigid phase also increases, whereas the solidlike mobile and semiliquid phases decrease. These changes in the fraction of mobile phases with viscosity are what one would expect from a physicochemical argument.

The logarithms of the measured viscosity values at different temperatures for the three bitumens are listed in Table 3. The intercept, A, and the slope, B, for the viscosity as a linear function of temperature (equation 11) are listed in Table 4. The viscosity, n, as a linear function of temperature, T, is:

$$\ln\eta = A + BT \tag{11}$$

Using equation 11, the viscosity at 313 K (the temperature at which the NMR measurements were made) was calculated for each bitumen. These values are listed in Table 3.

Table 3. The Logarithm of the Measured Viscosity as a Function of Temperature for Asphalt Ridge, Sunnyside, and Arroyo Grande Tar Sand Bitumens[a]

Temperature K	ln[Viscosity, cp]		
	Asphalt Ridge	Sunnyside	Arroyo Grande
311	14.603968	18.890684	
313[b]	14.319409	18.794871	22.602042
333	11.472103	16.454568	
364		11.982929	
378			13.997832
398			11.350407
423			8.699515

[a] Measured viscosity (ASTM D-4402)
[b] From equation 11

Table 4. Linear Regression Coefficients of Viscosity for Tar Sand Bitumens[a]

Coefficients	Asphalt Ridge	Sunnyside	Arroyo Grande[b]
A (Intercept)	58.905621	59.901431	64.034165
B (Slope)	-0.142357	-0.131247	-0.132371

[a] $\ln\eta = A + BT$
[b] Based on the first two measured data points

The coefficients a, b, and c in equation 10 were evaluated from the spin-spin relaxation times, the fractional amounts of mobile and rigid phases listed in Tables 1 and 2, respectively, and the viscosities at 313 K listed in Table 3. The values of the coefficients are:

$$\ln\eta_{313} = 4979.591(X_l/T_{2l}) - 370.946(X_m/T_{2m}) + 248.6583(X_s/T_{2s}) \qquad (12)$$

The square of the correlation coefficient, r^2, for the correlation of the measured viscosities and the NMR parameters is 0.923. This reasonably good value is based on only five data points and should be enhanced with additional data.

Equation 12 is used to calculate the viscosity of a bitumen at 313 K. An equation for calculating the viscosity of a bitumen as a function of temperature is:

$$\ln\eta = a(X_l/T_{2l}) + b(X_m/T_{2m}) + c(X_s/T_{2s}) + B'(T-T_{313}) \qquad (13)$$

where $B' = -0.135325$ is the average of the viscosity temperature coefficients listed in Table 3 for the three bitumens.

Figure 3 shows a plot of the natural logarithm of the viscosity as a function of the temperature for the Asphalt Ridge, Sunnyside, and Arroyo Grande bitumens. The symbols are the measured viscosities using the ASTM D-4402 procedure. The lines represent the viscosity of the bitumen at any temperature calculated from the NMR data using equation 13. For the Sunnyside and the Arroyo Grande tar sand bitumen, the NMR viscosity values are the average of two experiments.

CONCLUSIONS

It has been demonstrated that a reasonably good correlation exists between the viscosity of a tar sand bitumen and the weighted average of the three spin-spin relaxation rates for the semiliquid, solidlike mobile, and solidlike rigid phases. Additional data are needed to enhance the correlations and to demonstrate the versatility of the method.

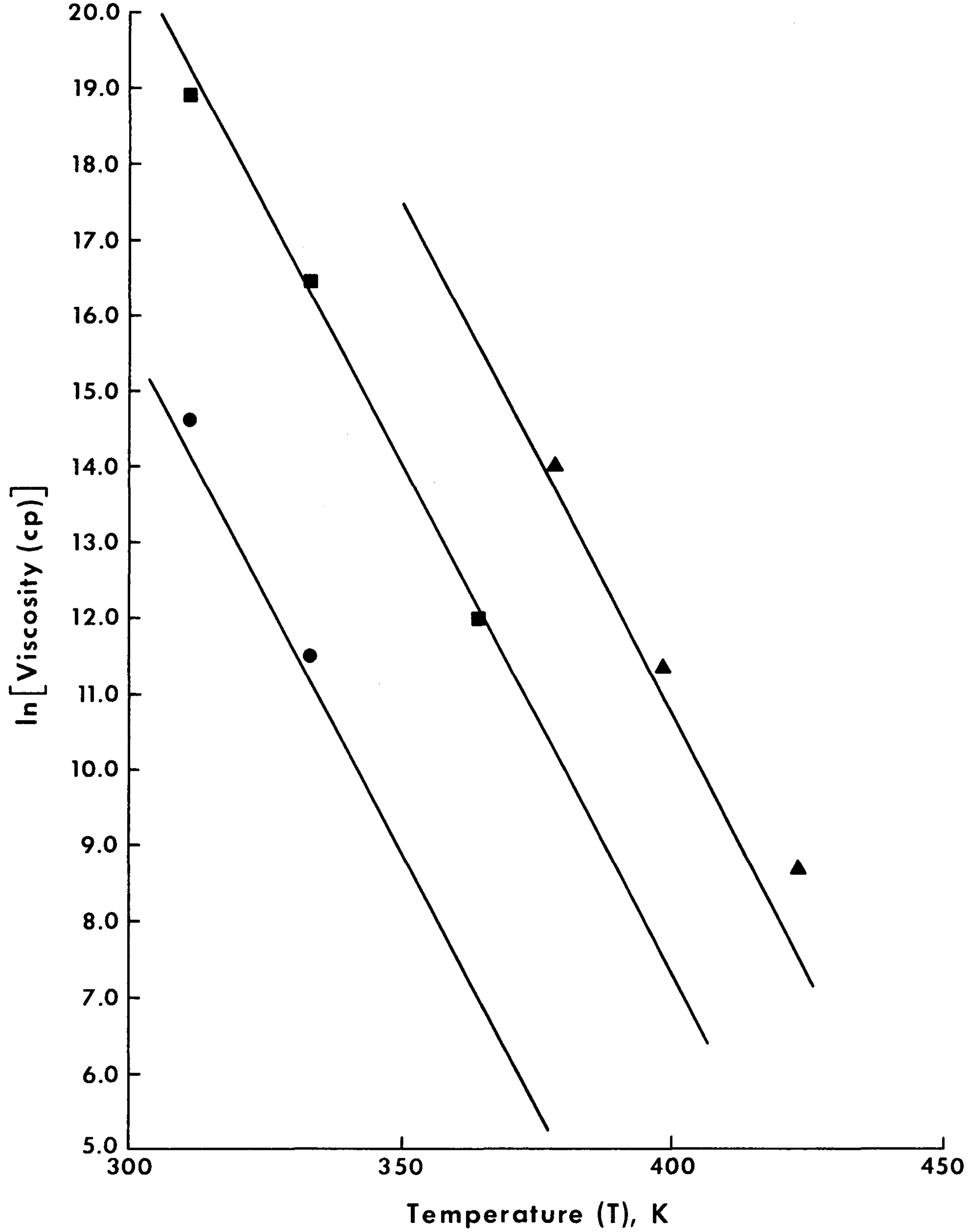

Figure 3. Correlation of Measured Viscosities with Viscosities Determined from NMR Parameters for ● Asphalt Ridge, ■ Sunnyside, and ▲ Arroyo Grande Tar Sand Bitumens

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