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Viscosities of Pure Polyaromatic Hydrocarbons

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Experimental measurements of the viscosities of eight aromatic and polyaromatic compounds are reported for the temperature range 298–498 K. These viscosity data, with an experimental uncertainty of 0.5%, represent a substantial expansion of the data base for some materials (such as toluene) and establish a data base for such compounds as picoline and quinoline. A comparison of these data with the results of the Morris correlation and the equation of van Velzen et al. gave average errors in the 15% range.

Introduction

In the design and subsequent operation of coal conversion plants, methods for accurately predicting transport coefficients are important. For instance, it is necessary to estimate the viscosity of complex mixtures over wide ranges of concentration and temperature in order to accurately size the pumps and piping systems of these plants as well as their reaction vessels and separation units. The viscosities of polyaromatic compounds and their mixtures offer important insights into the energy relationships in organic liquid mixtures. Such information is particularly useful for compounds in the critical region, where intermolecular interactions change rapidly with temperature and pressure.

It would be logical to use generalized viscosity correlations for engineering work, when such correlations are available. However, a review of the literature reveals that predictive relationships for pure components in the temperature range well removed from the critical point are unreliable (*1*). Furthermore, the predictions generally do not apply at temperatures above the normal boiling point. This temperature range is particularly important for coal processing and near-critical extraction processes. Therefore, a program was begun to collect the data needed to develop a method for predicting viscosities for mixtures of organic pure components, particularly at temperatures above the normal boiling point. As part of this study, a laser-light-scattering technique was developed to investigate high-temperature liquid transport properties (*2*). Another facet of the program was the investigation of viscosities at temperatures below the boiling point, to provide the data required for confirmation of the laser-light-scattering method. The data base and correlations developed as a result of this work are the subjects of this report.

Solvent-based coal conversion processes produce thousands of compounds and isomers (*3*). From a list compiled by Weinstein (*4*), a series of stable representative compounds was selected for in-depth study. Only limited data have been reported for most of the pure compounds on the list, and only toluene was considered to have a satisfactory data base. Some compounds were not documented prior to this study.

Experimental Methods and Materials

The apparatus used in this study is shown schematically in Figure 1. The major component is a glass Ubbelohde viscometer manufactured by Schott Geräte (Model Series 24500). Normal viscometer operation involves drawing the sample fluid into a reservoir bulb and measuring the time required for the gravity efflux of a fixed fluid volume. This process is automated in the Schott viscometer, and fiber-optic detectors replace the visual reading. When the fluid interface passes the plane of a light beam from the transmitter to a detector unit, the voltage signal changes, starting a timer. As the interface passes a second detector, denoting the efflux of a fixed volume, the signal change stops the electronic timer. Efflux time can be determined accurately to within 0.01 s, essentially removing one of the persistent sources of experimental error in older devices. More details on the operation of capillary viscometers may be found in the literature (*5–7*).

Our experimental equipment also included a constant-temperature bath and a piping system for drawing samples into the capillary tube. Because of the temperature range involved in the experiments and the possibility of oxidation, argon (at a pressure slightly greater than ambient) was selected as a blanket gas. The temperature bath is a Haake, Model N2, recirculating unit placed in a well-insulated, cylindrical, glass container. Measurements were made at temperatures from 298 to 498 K. Over this range, it was possible to maintain a constant temperature to within 0.02 K. For convenience, water was used as the bath medium at temperatures <368 K; Dow Corning's Silicone Fluid No. 550 (phenylmethylpolysiloxane) was used at higher temperatures. A calibrated, platinum, resistance thermometer connected to a digital Fluke RTD sensor (Model 2180A) made it possible to measure temperatures accurately to within 0.1 K. The entire experimental unit (except for readout devices) was covered and heavily insulated to minimize temperature gradients. Details of equipment operation are given elsewhere (*8*).

The viscometer stand, on which the fiber-optic detectors are precisely affixed, allows the rapid interchange of viscometers. During the course of the study, several glass units were used. In all instances, the manufacturer (Schott Geräte) provided calibration constants that relate the efflux time to the kinematic viscosity.

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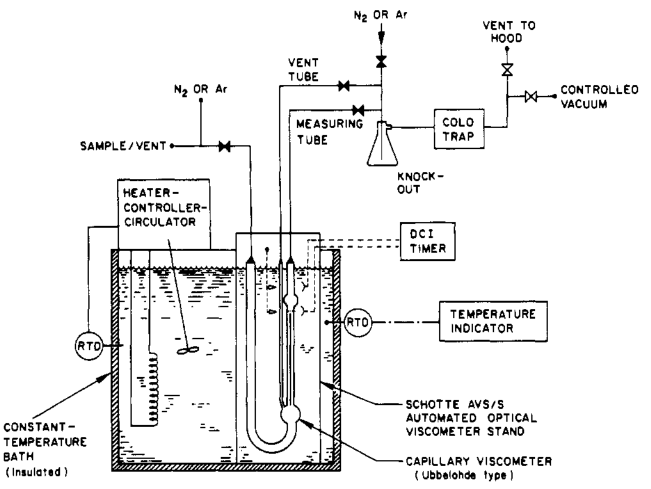

Figure 1. Experimental apparatus for measurement of viscosities of model fluids at ambient pressure.

Table I. Viscosity Data for Pure Toluene

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
298.2	0.646	0.557	0.264
308.2	0.574	0.489	0.013
318.2	0.522	0.440	0.035
328.2	0.476	0.397	0.008
338.2	0.439	0.362	0.020
348.2	0.406	0.331	0.009
358.2	0.377	0.304	0.010
368.2	0.352	0.280	0.014

To obtain the dynamic viscosity, it is necessary to determine the fluid's density at the temperature of the experiment. The densities of all of the pure components used in this study were measured by standard gravimetric methods (pycnometer) at several temperatures throughout the temperature range in which viscosities were measured. The temperature variation of density is linear throughout the range of temperatures examined for these liquids, but to assure accuracy of interpolation, a polynomial fit of the data was made (*9*). The experimental uncertainty of the method is 0.3%. The densities of pure components were also predicted by using the method of Gunn and Yamada (*10*). This method requires critical data, an acentric factor, and density data at the critical point and one additional temperature. Using our density data at 25 °C and the other factors tabulated in Reid, Sherwood, and Prausnitz (*1*), the results of the Gunn and Yamada correlation were calculated for all components. A comparison of our experimental data and the predictions of the Gunn and Yamada correlation indicated that the agreement was better than the uncertainty of the measurements for all components (*9*). The experimental data were used to compute the dynamic viscosities from the kinematic viscosity. Because of the experimental uncertainty of the density measurements, the uncertainty in the dynamic viscosity is ~0.8%, while the kinematic viscosity is known to be 0.5%.

Viscosities and densities for water are accurately known and tabulated for the range of interest (*11*). Therefore, distilled water was used to confirm the manufacturer's calibration constants for all units used in this study. Where necessary, the constants were corrected in accordance with our measurements. The most severe case of recalibration required a correction of ~1% to the calibration constant. There appears to be no temperature trend in the corrections made to the calibration.

All organic chemicals used in this study were reagent grade (99+ wt % pure). There were problems associated with impurities found in two compounds. Tetralin contained a peroxide that promoted coking at high temperatures. This caused yellowing of the solutions at higher temperatures, so it was necessary to purify the tetralin before use by extraction with aqueous ferrous ammonium sulfate. A slight discoloration caused by impurities in the 2-methylnaphthalene was overcome by use of an argon blanket.

Experimental Results

Toluene Standard Verification and Comparison with Literature. Since the manufacturer's calibration constant for the viscometer tube used in this study (No. 68609) appeared to have a 1% error when compared with our data for water, a new constant (0.000 950 0, as opposed to 0.000 940 5 given by the manufacturer) was assigned, and a second fluid (toluene) was selected to check the constant. Reagent-grade toluene was used, and viscosity measurements were taken at several temperatures between 298 and 368 K (see Table I). A correlation approach was used for comparisons. Over the temperature range in question, the two-constant Andrade correlation (*6*) fit our data with a coefficient of correlation (r^2) of 0.999 85 and a standard error of estimate of 0.004 mPa·s. The Andrade constants for toluene are given in Table II. A number of statistical parameters, arising from the taking of replicate data and the performance of regression analyses, were used in this work. A summary of the definitions of these parameters (i.e., r^2) is given in Table III. Toluene data from a number of standard sources (*1*, *12*, *13*) were compared with the correlation data obtained in the current study (Figure 2). With the exception of two apparently questionable points, the literature values deviated from the correlation by an average of 0.15%. An experimental error analysis indicates that the anticipated error is 0.5%. Thus, these data fall well within experimental error. As shown in Figure 2, the literature data points are randomly distributed relative to the current experimental data, indicating that a reliable calibration was obtained and that reliable data are available for toluene, at least to its normal boiling point.

Table II. Pure-Component Viscosity Correlations[a]

compound	melting point, K	boiling point, K	exptl temp range, K	viscometer tube no.	Andrade constants: A, mPa·s	Andrade constants: B, K	coeff of correln
toluene	178.0	383.8	298–368	68609	1.535×10^{-2}	1.069×10^{3}	0.9998
phenol	314.0	455.0	313–363	82456	3.033×10^{-4}	3.028×10^{3}	0.9979
tetralin	242.0	480.7	293–353	85564	1.292×10^{-2}	1.487×10^{3}	0.9990
			358–458	85564			
phenyl ether	300.0	531.7	303–363	85564	9.123×10^{-3}	1.757×10^{3}	0.9981
			363–483	85564			
1-methylnaphthalene	242.7	517.8	308–358	85564	1.109×10^{-2}	1.631×10^{3}	0.9982
			363–503	85564			
2-methylnaphthalene	307.7	514.2	308–363	85564	9.590×10^{-3}	1.611×10^{3}	0.9993
quinoline	257.6	510.3	373–463	70159	1.003×10^{-2}	1.745×10^{3}	0.9980
picoline	276.9	418.5	303–363	86713/2450100	1.552×10^{-2}	1.192×10^{3}	0.9998

[a] Andrade correlation constants: $\mu = A \exp(B/T)$.

Table III. Summary of Statistical Parameters[a]

1. Linear regression parameters ($y = \ln \mu$, $x = 1/T$)
   a. standard error of estimate (S_e)

$$S_e = \left[\frac{1}{N-2}\sum^{N}(Y_{\text{data}} - Y_{\text{fit}})^2\right]^2$$

   b. coefficient of correlation (r^2)

$$r^2 = (\sigma_{xy}{}^2)^2/\sigma_x{}^2\sigma_y{}^2 = \frac{(N\sum xy - \sum x \sum y)^2}{[N\sum x^2 - (\sum x)^2][N\sum y^2 - (\sum y)^2]}$$

2. Measures of replication
   a. coefficient of variation (CV) for replicated data

$$\%\ \text{CV} = (\text{standard deviation}/\text{mean}) \times 100 = (X_x/\bar{X}) \times 100$$

   b. av % deviation or error

$$\%\ \text{av error} = \sum^{N}\frac{|Y_{\text{ref}} - Y_{\text{expt}}|}{Y_{\text{expt}}} \times 100/N$$

[a] Experimental values are calculated from the Andrade equation in order to exactly match the literature (ref) temperatures (except as noted in Table XI).

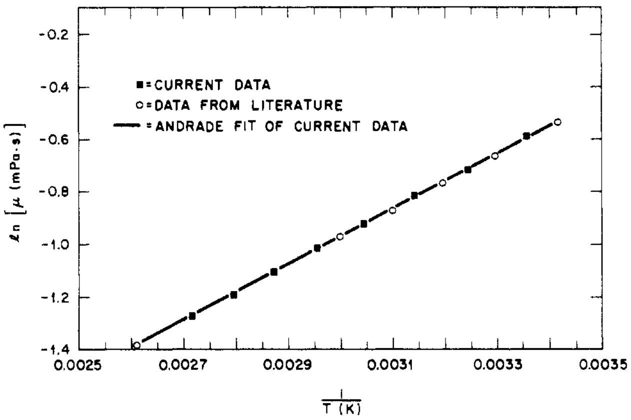

Figure 2. Viscosity of toluene (298–368 K): experimental results and literature values.

Pure-Component Viscosity Data. In addition to toluene, seven other compounds representative of the midboiling range of coal-conversion fluids were selected for low-temperature experiments. These fluids include 1-methylnaphthalene (Table IV), 2-methylnaphthalene (Table V), phenol (Table VI), phenyl ether (Table VII), picoline (Table VIII), quinoline (Table IX), and tetralin (Table X). Data for each of these liquids include at least one set of replicate measurements, often made with a different viscometer tube. Some data sets include several repetitions of measurements; the coefficient of variation reflects the reproducibility of the data. In Tables I–VIII, the kinematic viscosity, ν (in m^2/s), is the measured quantity. The fluid density is also required to calculate the viscosity, μ (in mPa·s). The density is an interpolation of experimental data for the component in question.

Correlation of Results. At temperatures substantially removed from the critical point, no reliable correlation exists to predict, from first principles, the viscosities of pure fluids. A number of empirical and semiempirical viscosity prediction methods exist; the four most often cited are the methods attributed to Orrick and Erbar, Morris, van Velzen et al., and Thomas, which are reviewed by Reid, Sherwood, and Prausnitz (*1*). All of these correlations take the general form

$$\ln \mu = \ln A + B/T \tag{1}$$

where the two constants, A and B, are generally estimated by some group-contribution method. For a wide range of compounds, Reid et al. (*1*) have shown that these correlations have an associated error of 15–20%; however, individual errors of as much as 100% were found. If the two constants in eq 1 are considered to be empirically fitted parameters, the relationship is called the Andrade equation.

Table IV. Viscosity Data for 1-Methylnaphthalene

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
	Nitrogen Atmosphere		
308.2	2.30	2.32	0.08
313.2	2.07	2.08	0.17
318.2	1.89	1.89	0.06
323.2	1.71	1.72	0.04
328.2	1.58	1.57	0.07
333.2	1.45	1.44	0.03
338.2	1.34	1.33	0.02
348.2	1.17	1.14	0.03
303.2	2.55	2.58	0.04
308.2	2.29	2.31	0.02
318.2	1.89	1.89	0.04
328.2	1.59	1.58	0.02
338.2	1.37	1.34	0.05
343.2	1.27	1.25	0.02
348.2	1.19	1.16	0.01
353.2	1.11	1.09	0.03
358.2	1.04	1.01	0.08
363.2	1.07	0.973	0.26
363.2	0.999	0.966	0.03
373.2	0.900	0.864	0.38
383.2	0.809	0.770	0.40
383.2	0.806	0.767	
393.2	0.732	0.691	0.10
403.2	0.671	0.628	0.20
403.2	0.670	0.628	0.09
413.2	0.617	0.572	0.25
423.2	0.566	0.521	0.19
433.2	0.526	0.479	0.10
443.2	0.490	0.443	0.10
443.2	0.495	0.448	0.07
453.2	0.459	0.411	0.14
463.2	0.430	0.382	0.06
463.2	0.440	0.390	0.10
473.2	0.405	0.355	0.02
483.2	0.381	0.331	0.03
493.2	0.360	0.310	0.06
503.2	0.342	0.291	0.01
	Argon Atmosphere		
363.2	1.00	0.967	[a]
373.2	0.900	0.863	
383.2	0.806	0.767	
413.2	0.618	0.573	
433.2	0.530	0.483	
443.2	0.494	0.446	
453.2	0.462	0.413	
463.2	0.434	0.384	
473.2	0.408	0.358	
483.2	0.386	0.336	

[a] Not computed for these runs.

Table V. Viscosity Data for 2-Methylnaphthalene

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
308.2	1.82	1.81	0.01
313.2	1.67	1.65	0.02
318.2	1.53	1.51	0.02
323.2	1.42	1.39	0.01
328.2	1.32	1.30	0.02
333.2	1.23	1.20	0.01
338.2	1.15	1.12	0.02
343.2	1.08	1.04	0.02
348.2	1.01	0.977	0.01
353.2	0.958	0.920	0.04
358.2	0.906	0.866	0.02
363.2	0.859	0.818	0.04

Table VI. Viscosity Data for Phenol

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
313.2	4.63	4.90	0.01
323.2	3.33	3.52	0.02
323.2	3.34	3.53	0.12
333.2	2.52	2.61	0.12
343.2	1.96	2.02	0.01
353.2	1.57	1.60	0.05
363.2	1.29	1.30	0.05

Table VII. Viscosity Data for Phenyl Ether

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
303.2	3.03	3.23	0.04
308.2	2.70	2.87	0.02
313.2	2.42	2.56	0.02
318.2	2.19	2.31	0.03
323.2	2.00	2.09	0.01
333.2	1.68	1.75	0.02
338.2	1.55	1.60	0.02
343.2	1.44	1.48	0.01
353.2	1.25	1.28	0.02
363.2	1.10	1.11	0.02
363.2	1.10	1.11	0.05
373.2	0.977	0.981	0.04
383.2	0.875	0.870	0.04
393.2	0.791	0.780	0.09
403.2	0.718	0.702	0.02
413.2	0.657	0.636	0.00
423.2	0.603	0.579	0.10
433.2	0.558	0.530	0.04
443.2	0.517	0.486	0.03
453.2	0.481	0.449	0.10
463.2	0.450	0.416	0.01
473.2	0.423	0.387	0.07
483.2	0.397	0.360	0.37

Table VIII. Viscosity Data for Picoline

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
303.2	0.842	0.796	0.01
313.2	0.744	0.696	0.13
313.2	0.747	0.699	0.04
323.2	0.668	0.620	0.05
333.2	0.605	0.555	0.39
343.2	0.550	0.500	0.00
353.2	0.505	0.454	0.00
363.2	0.466	0.415	0.01

Table IX. Viscosity Data for Quinoline

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
373.2	1.03	1.06	1.50
373.2	1.05	1.08	1.07
373.2	1.04	1.07	1.19
413.2	0.711	0.710	0.96
433.2	0.560	0.550	0.04
443.2	0.521	0.507	0.02
453.2	0.487	0.470	0.06
453.2	0.487	0.470	0.02
463.2	0.457	0.438	0.02

The two-constant Andrade correlation was used to correlate all the data. As shown in Table II, the correlation is excellent; the standard error of estimate is <0.007 mPa·s or about 0.7%. This number is comparable to the estimated experimental accuracy of the method ~0.5%). The two constants, *A* and *B*, are similar for all of the compounds studied except for toluene, picoline, and phenol (with phenol exhibiting the most unique behavior).

Phenol is the only compound for which measurements were made in the subcooled liquid region. At these temperatures, intermolecular forces are substantially different from those at temperatures well removed from the freezing point. This is consistent with results of studies with other materials (*14*, *15*).

Table X. Viscosity Data for Tetralin

av temp, K	ν, m^2/s	μ, mPa·s	coeff of variation, %
	As Received		
293.2	2.25	2.18	0.08
303.2	1.87	1.80	0.14
313.2	1.58	1.50	0.07
323.2	1.36	1.29	0.20
333.2	1.19	1.11	0.15
343.2	1.05	0.979	0.17
353.2	0.929	0.859	0.04
358.2	0.872	0.803	0.07
363.2	0.830	0.760	0.04
373.2	0.752	0.684	0.03
383.2	0.687	0.614	0.05
393.2	0.632	0.565	0.03
403.2	0.582	0.516	0.05
413.2	0.539	0.474	0.07
423.2	0.505	0.440	0.07
433.2	0.470	0.406	0.05
443.2	0.442	0.379	0.01
453.2	0.416	0.353	0.02
458.2	0.402	0.340	0.14
	Purified		
383.1	0.688	0.620	0.04
393.1	0.630	0.563	0.03
403.5	0.581	0.575	0.02
423.4	0.500	0.436	0.17
443.6	0.437	0.374	0.09

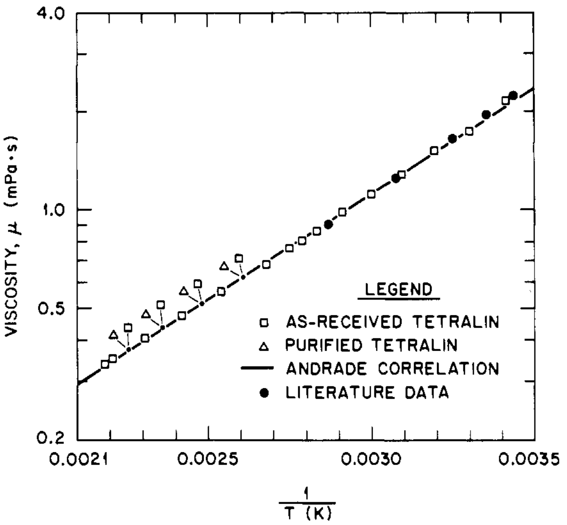

Figure 3. Viscosity of tetralin. Values are given for "as-received" and purified liquid tetralin as well as for data taken from the literature.

The viscosity data for pure tetralin are shown in Figure 3. Two separate sets of data are shown; the first and more extensive group of data points was from "as-received" tetralin, which contained a small quantity of peroxide. The second group of measurements was taken after purification of the tetralin. The differences are within the experimental error of the method. Andrade correlations (given in Table II) and data from the literature (*1*, *12*, *13*) are also incorporated in the graph. Our Andrade equation predictions are in good agreement with the literature data (*12*, *13*) (<1% average deviation) and there is greater internal consistency among our experimental measurements than among the literature values. Our experimental results have doubled the range of available data, providing a good basis for future comparisons.

The pure-component data for the other liquids studied show a similar trend, although the literature references in these cases are less plentiful than for toluene and tetralin.

Table XI. Prediction of Pure-Component Viscosities and Andrade Coefficients

	experimental		Morris correlation (*16*)			correln method of van Velzen et al (*17*)		
compound	*A*, mPa·s	*B*, K	*A*, mPa·s	*B*, K	av % error[a]	*A*, mPa·s	*B*, K	av % error
toluene	0.015 35	1069	0.014 85	1062.9	5.03	0.018 93	968.0	9.6
phenol	0.000 303	3028	0.000 13	3197.5	32.20	0.002 46	2210.0	22.5
tetralin	0.012 92	1487	0.009 65	1593	3.05	0.008 60	1516.0	27.8
phenyl ether	0.009 123	1757	0.007 44	1906.1	27.10	0.014 78	1561.2	5.8
1-methylnaphthalene	0.011 09	1631	0.011 13	1609.9	3.51	0.013 19	1521.9	9.8
2-methylnaphthalene	0.009 59	1611	0.011 3	1586.9	15.70	0.013 19	1521.9	8.1

[a] [(exptl viscosity − predicted viscosity) × 100]/exptl viscosity = % error.

Prediction methods for pure fluids generally involve variations of the Andrade equation, using a group-contribution method to predict the parameters. Methods by Morris and van Velzen et al. follow this precisely, while the methods of Orrick and Erbar and Thomas allow for slight curvature of the ln μ vs. $1/T$ curve (*1*). To test the effectiveness of these methods, Andrade coefficients were predicted for several of the components in the study by using both the Morris and the van Velzen techniques. The results are shown in Table XI. Predicted viscosities were compared with experimental data over the entire temperature range for each compound. The average absolute error of the predictions is given for each method. Errors of as much 32.2% were obtained, and both methods generally gave unacceptably erratic results. Since these group-contribution methods are based on an extensive collection of data points, the poor prediction results bring the usefulness of these approaches into question.

For heterocyclic compounds such as quinoline and picoline, no means of prediction is now available. A major problem associated with all of the prediction methods examined is their unreliability unless data are already available for compounds closely related to the compound for which one desires a prediction.

Conclusions

1. A viscosity data base has been compiled for selected, pure, model coal-processing fluid components. For some liquids (such as toluene), the data confirm and extend the range of earlier results. For other compounds (such as 4-picoline and quinoline), new data bases have been established.

2. The ability to predict viscosities of pure components is limited. Most predictive methods are based on group contributions, and some prior data must be available for homologues of the compound in question. Even so, errors of 15% or more are common.

Glossary

A	Andrade coefficient, mPa·s (cP)
B	Andrade coefficient, K
N	number of data points
S_x	standard deviation
r^2	coefficient of correlation
t	time, s
T	temperature, K
X	independent variable > $1/t$
X_1	mean of the independent variable
y	dependent variable ln μ

Greek Letters

μ	viscosity, mPa·s
ν	kinematic viscosity, m^2/s
ρ	fluid density, kg/m^3
ω	acentric factor
σ_{xy}	standard deviation (defined in Table III)

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