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An Accurate Estimation Method of Kinematic Viscosity for Standard Viscosity Liquids

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Deming's method of least squares is introduced to make an accurate kinematic viscosity estimation for a series of 13 standard-viscosity liquids at any desired temperature. The empirical ASTM kinematic viscosity–temperature equation is represented in the form $\log\log(\nu + c) = a - b \log T$, where ν (in $\mathrm{mm^2 \cdot s^{-1}}$) is the kinematic viscosity at temperature T (in K), a and b are the constants for a given liquid, and c has a variable value. In the present application, however, c is assumed to have a constant value for each standard-viscosity liquid, as do a and b in the ASTM equation. This assumption has since been verified experimentally for all standard-viscosity liquids. The kinematic viscosities for the 13 standard-viscosity liquids have been measured with a high accuracy in the temperature range of 20–40°C using a series of the NRLM capillary master viscometers with an automatic flow time detection system. The deviations between measured and estimated kinematic viscosities were less than ±0.04% for the 10 standard-viscosity liquids JS2.5 to JS2000 and ±0.11% for the 3 standard-viscosity liquids JS15H to JS200H, respectively. From the above investigation, it was revealed that the uncertainty in the present estimation method is less than one-third that in the usual ASTM method.

KEY WORDS: Deming's method of least squares; kinematic viscosity; master viscometer; standard-viscosity liquid.

1. INTRODUCTION

In Japan, in accordance with the provision of Japanese Industrial Standard (JIS) Z8809, a series of 13 standard-viscosity liquids, called Standard

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Liquids for Calibrating Viscometers, is supplied to manufacturers and consumers associated with viscometers. Table I shows the nominal viscosity and kinematic viscosity values at stated temperatures for these standard-viscosity liquids. The 10 standard-viscosity liquids JS2.5 to JS2000 are highly refined mineral oils without additives and the 3 standard-viscosity liquids JS15H to JS200H are good-quality polybutenes. According to the provisions of JIS Z8809, the National Research Laboratory of Metrology (NRLM) is responsible for measuring the actual values of viscosity and kinematic viscosity for these standard-viscosity liquids. The stated uncertainties in certified actual values of both viscosity and kinematic viscosity are ±0.1% for JS2.5 to JS2000 and ±0.2% for JS15H to JS200H, respectively, which are provided by JIS Z8809.

Based on the above-mentioned information, it appears that the actual values of both viscosity and kinematic viscosity are certified only at the stated temperatures of 20, 25, 30, and 40°C. Therefore, users of the standard-viscosity liquids frequently need to estimate viscosities or kinematic viscosities accurately at other temperatures in order to calibrate their viscometers at any desired temperature. To meet such needs and to enhance the usefulness of the standard-viscosity liquids for practical usage, the American Society for Testing and Materials (ASTM) kinematic viscosity–temperature equation was used for an accurate estimation of the kinematic viscosity at any desired temperature in the stated temperature range of 20 to 40°C.

Table I. Nominal Values of Kinematic Viscosity ν and Viscosity η at the Stated Temperatures for the Standard Liquids for Calibrating Viscometers

	ν (mm^2·s^{-1})				η (mPa·s)			
Designation	20°C	25°C	30°C	40°C	20°C	25°C	30°C	40°C
JS2.5	2.5	—	2.1	1.7	2	—	1.6	1.3
JS5	5	—	4	3	4	—	3	2.5
JS10	10	—	7	6	8	—	6	5
JS20	20	—	14	10	17	—	11	8
JS50	50	—	30	21	42	—	25	18
JS100	100	—	60	35	85	—	50	30
JS200	200	—	110	60	170	—	90	52
JS500	500	—	250	140	450	—	220	120
JS1000	1,000	—	500	250	900	—	440	220
JS2000	2,000	—	900	440	1,800	—	800	390
JS15H	16,000	—	6,000	3,000	14,000	—	5,000	2,000
JS60H	56,000	—	21,000	9,000	50,000	—	19,000	8,000
JS200H	170,000	100,000	—	—	150,000	90,000	—	—

The kinematic viscosities for the 13 standard-viscosity liquids were measured with a high accuracy at nine temperature points between 20 to 40°C using NRLM normal-flow U-tube capillary master viscometers with an automatic flow time detection system. Then the constants in the ASTM kinematic viscosity–temperature equation for each standard-viscosity liquid were determined by introducing Deming's method of least squares using several measured kinematic viscosities. If the certified numerical values for an equation are precisely determined, it is possible to estimate kinematic viscosities accurately at temperatures other than the stated temperatures.

In this paper, Deming's method of least squares is introduced to make an accurate estimation of kinematic viscosity for all standard-viscosity liquids at temperatures other than the stated temperatures by applying the ASTM kinematic viscosity–temperature equation, except that here c is assumed to be constant for each standard-viscosity liquid.

In addition, this paper describes the deviations between measured and estimated kinematic viscosities and the results of a comparison between Deming's method of least squares and the linear regression analysis based on the ASTM kinematic viscosity–temperature equation.

2. ASTM KINEMATIC VISCOSITY–TEMPERATURE EQUATION

For the variation of petroleum oil's kinematic viscosity with temperature, the following purely empirical equation expressed in terms of the logarithm to base 10, has been developed and is available from the ASTM Standard D341-77.

$$\log\log(v + c) = a - b \log T \tag{1}$$

where v is the kinematic viscosity in $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$, c is the variable value expressed in terms of exponential as the function of kinematic viscosity, a and b are the constants for a given oil, an T is the temperature in K.

In this equation, the applicability of kinematic viscosities is in the range from 0.21 to $2 \times 10^7\ \mathrm{mm}^2 \cdot \mathrm{s}^{-1}$. If v is greater than $3\ \mathrm{mm}^2 \cdot \mathrm{s}^{-1}$, the value of c becomes constant, $0.7\ \mathrm{mm}^2 \cdot \mathrm{s}^{-1}$, as shown in Fig. 1. This relationship has been derived by Wright [1] with computer assistance on the basis of the most reliable of modern data on pure hydrocarbons and petroleum fractions and has been widely used in interpolation or extrapolation of kinematic viscosities at temperatures other than the given temperatures. The constants a and b are determined by the usual linear regression analysis using several measured values of kinematic viscosity. Then interpolating or extrapolating kinematic viscosities at any desired temperature for a given oil can be carried out from Eq. (1).

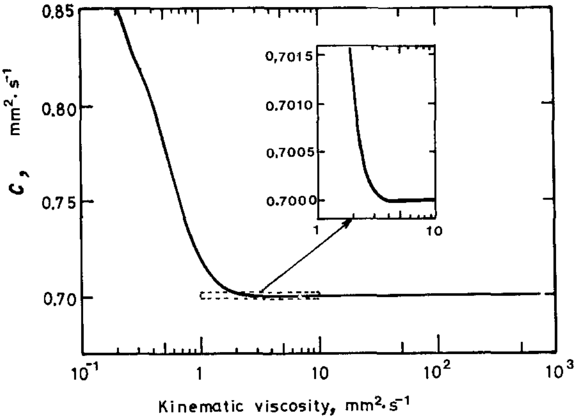

Fig. 1. Relation between the value of c and the kinematic viscosity derived by Wright [1].

3. FORMULATION OF DEMING'S METHOD OF LEAST SQUARES

In the ASTM kinematic viscosity–temperature equation, c is extremely complicated and is significant only at low-kinematic viscosity regions, as is evident from Fig. 1; therefore in the present analysis, we assume that c is also taken as a constant parameter to be determined for each standard-viscosity liquid. This means that there are three unknown parameters (a, b, and c) in Eq. (1). From this equation, the observation equation F is given as follows:

$$F(v, T; a, b, c) = a - b \log T - \log\log(v + c) = 0 \qquad (2)$$

According to the principle of Deming's method of least squares [2], the linear normal equation is obtained from the observation equation throughout the following analysis. Assuming approximate values of unknown parameters as a_0, b_0, and c_0 and denoting residuals of the parameters and n sets (more than four sets are required) of observed values v_i and T_i ($i = 1, 2,..., n$) by A, B, C, V_{v_i}, and V_{T_i} ($i = 1, 2,..., n$), respectively, the reduced conditions are given as follows:

$$\left.\begin{aligned} v_{ia} &= v_i - V_{v_i} \\ T_{ia} &= T_i - V_{T_i} \\ a &= a_0 - A \\ b &= b_0 - B \\ c &= c_0 - C \end{aligned}\right\} \qquad (3)$$

where v_{ia} and T_{ia} are the most probable values of observed values v_i and T_i, respectively. The relations given by Eq. (3) are substituted in the observation equation, Eq. (2). By expanding the equation by Taylor's series and retaining only the first powers of the residuals, Eq. (4) is obtained.

$$F_{0i} - F_{v_i} V_{v_i} - F_{T_i} V_{T_i} - F_{ai} A - F_{bi} B - F_{ci} C = 0 \tag{4}$$

where $F_{0i} = F(v_i, T_i;\, a_0, b_0, c_0)$, $F_{v_i} = (\partial F/\partial v_i)$, $F_{T_i} = (\partial F/\partial T_i)$, $F_{ai} = (\partial F/\partial a)$, $F_{bi} = (\partial F/\partial b)$, and $F_{ci} = (\partial F/\partial c)$. According to the principle of the well-known method of least squares, the weighted sum of squares of residuals $S = \sum (W_{v_i} V_{v_i}^2 + W_{T_i} V_{T_i}^2)$ must be minimized under the restriction of Eq. (4), where W_{v_i} and W_{T_i} are the weights for the observed values v_i and T_i, respectively. The problem is solved by the method of Lagrangian multiplier, leading to the following normal equation:

$$\begin{pmatrix} \sum (F_a F_a/L) & \sum (F_a F_b/L) & \sum (F_a F_c/L) \\ \sum (F_b F_a/L) & \sum (F_b F_b/L) & \sum (F_b F_c/L) \\ \sum (F_c F_a/L) & \sum (F_c F_b/L) & \sum (F_c F_c/L) \end{pmatrix} \times \begin{pmatrix} A \\ B \\ C \end{pmatrix} = \begin{pmatrix} \sum (F_a F_0/L) \\ \sum (F_b F_0/L) \\ \sum (F_c F_0/L) \end{pmatrix} \tag{5}$$

where $F_a = 1$, $F_b = -\log T$, $F_c = [-1/(v+c)]$, $F_v = F_c$, $F_T = -b/T$, $F_0 = a_0 - b_0 \log T_i - \operatorname{loglog}(v_i + c_0)$, and $L = F_{v_i}^2/W_{v_i} + F_{T_i}^2/W_{T_i}$.

After the residuals of the unknown parameters A, B, and C are determined by solving the normal equation, the adjusted parameters a, b, and c are determined immediately by Eq. (3). The weights W_{v_i} and W_{T_i} are regarded as constant and equal each other.

When the precision of the parameters a, b, and c obtained by the first adjustment is not sufficient, the iteration of the calculation is carried out by denoting $a_0 = a$, $b_0 = b$, and $c_0 = c$. In the present calculation, a sufficient precision is achieved by the second or third iteration. The computations were carried out by a personal computer.

4. KINEMATIC VISCOSITY MEASUREMENT

4.1. The Master Viscometers

In order to obtain high-accuracy, relative measurements of the kinematic viscosity for the 13 standard-viscosity liquids, we used a series of the new NRLM normal-flow U-tube capillary master viscometers of a type similar to that of the previous model, designed and introduced by Kawata et al. [3]. Figure 2 shows a cross-sectional view of the master viscometer

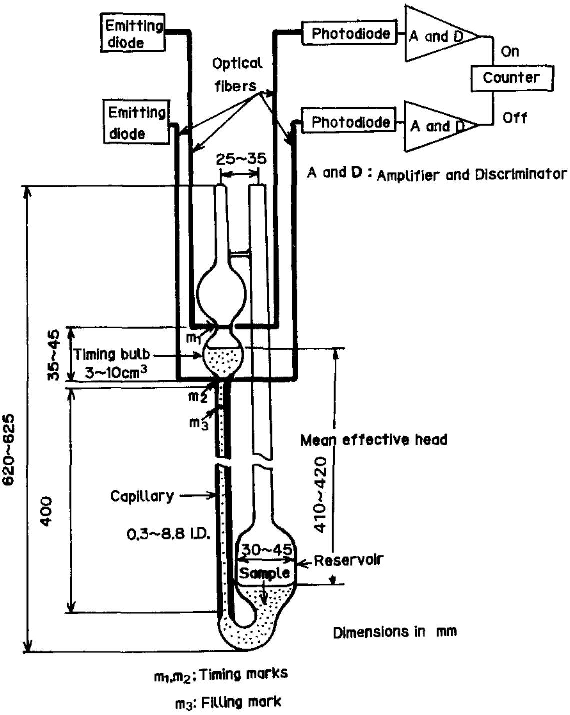

Fig. 2. The NRLM normal-flow U-tube capillary master viscometer and a block diagram of the automatic flow time detection system.

and a block diagram of the automatic flow time detection system. Fully annealed, low-expansion ($3.2 \times 10^{-6} \cdot {}^{\circ}\mathrm{C}^{-1}$) borosilicate glass was used for the construction of all master viscometers, which consisted of a reservoir, timing bulb, and capillary in a U-tube arrangement. Table II shows the essential dimensions of the master viscometers. They consist of 18 viscometers divided into nine groups with nine different capillaries inside the diameter, as shown in Table II. In order to eliminate the operator reaction time of hand-to-eye response and to improve the accuracy of kinematic viscosity measurement, the new master viscometers have an automatic flow time detection system, as shown in Fig. 2. The flow time is automatically obtained by detecting the leading edge of the liquid meniscus passing between the upper (m_1) and the lower (m_2) timing marks of the timing bulb with the aid of an optoelectronic technique using optical fibers. We replaced the two timing marks with optical fibers. When the leading edge of the meniscus passes through an infrared light path from an emitting diode, the intensity of light is changed. The change in light intensity was

Table II. Essential Dimensions of the NRLM Normal-Flow U-Tube Capillary Master Viscometers

Viscometer No.	Instrumental constant K ($mm^2 \cdot s^{-2}$)	Capillary radius (cm)	Capillary length (cm)	Mean effective head (cm)	Timing bulb volume (cm^3)	Filling volume (cm^3)	Reservoir radius (cm)
1-11	0.00048	0.017	40	42	7	15	1.5
1-21	0.00061	0.018	40	42	7	14	1.6
2-1	0.0034	0.023	40	41	3	11	1.5
2-31	0.0046	0.024	40	42	3	11	1.5
3-1	0.019	0.035	40	41	3	11	1.5
3-31	0.017	0.034	40	41	3	11	1.5
4-1	0.092	0.051	40	42	3	11	1.5
4-31	0.097	0.052	40	42	3	11	1.5
5-1	0.49	0.078	40	41	3	11	1.5
5-31	0.50	0.078	40	41	3	11	1.4
6-1	2.5	0.13	40	42	5	16	1.5
6-31	2.7	0.13	40	42	5	15	1.5
7-1	11	0.23	40	42	10	35	2.2
7-31	11	0.23	40	42	10	34	2.2
8-1	50	0.33	41	42	10	41	2.3
8-31	45	0.33	40	42	10	42	2.3
9-1	140	0.43	41	42	10	52	2.2
9-31	151	0.44	40	42	10	53	2.3

converted to a working voltage for an electronic time counter by the photodiode, amplifier, and discriminator. The upper and lower parts of the timing bulb correspond to the beginning and ending of the flow time, respectively. The measured flow time was indicated with a resolution of milliseconds.

4.2. Calibration

The instrumental constants for the master viscometers were calibrated by the stepping-up method [3] on the basis of an internationally accepted value of 1.0038 $mm^2 \cdot s^{-1}$ for the kinematic viscosity of distilled water at 20.00°C and atmospheric pressure [4] as the basis for the NRLM viscosity scale. Figure 3 shows the general procedure of the stepping-up method for the calibration of the master viscometers. In this method, the first group of master viscometers, Nos. 1-11 and 1-21, with the smallest-radius capillaries was calibrated with distilled water at 20.00°C. The first group of master viscometers, calibrated with distilled water, was used to determine the kinematic viscosity of standard-viscosity liquid JS2.5 at 20.00°C, which was then used to calibrate the second group of master viscometers, Nos. 2-1 and 2-31. In this process, a correction due to the difference in surface tension between water and JS2.5 for the master viscometers Nos. 1-11 and 1-22 was experimentally considered by the same procedure described in our previous publications [3, 5]. Similarly, the kinematic viscosity of the standard-viscosity liquid JS10 at 20.00°C was determined with the

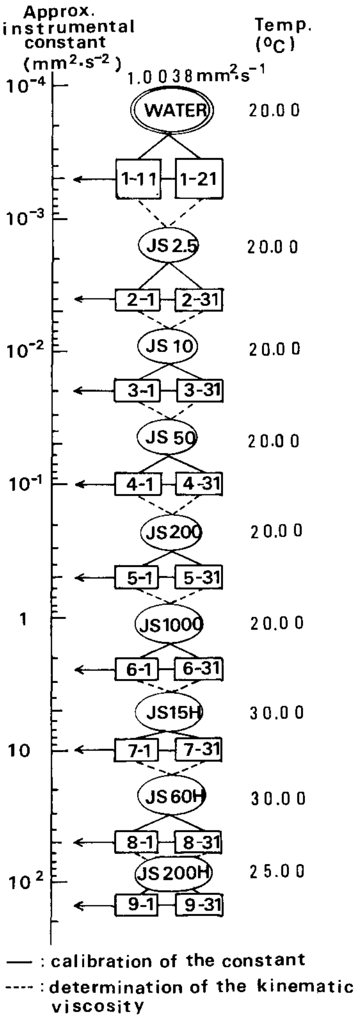

Fig. 3. General procedure of the stepping-up method.

calibrated second group of master viscometers, and the third group of master viscometers, Nos. 3-1 and 3-31, was calibrated with JS10 at 20.00°C. In this measurement of the kinematic viscosity of JS10, the surface tension correction can be neglected. Subsequently, the master viscometers Nos. 4-1, 4-31, 5-1, 5-31, 6-1, 6-31, 7-1, 7-31, 8-1, 8-31, 9-1, and 9-31 were likewise calibrated with a series of standard-viscosity liquids, as shown in Fig. 3.

In this stepping-up method, calibrations for the 1st series of master viscometers (Nos. 1-11, 2-1, 3-1, 4-1, 5-1, 6-1, 7-1, 8-1, 9-1) and the 31st series of master viscometers (Nos. 1-21, 2-31, 3-31, 4-31, 5-31, 6-31, 7-31, 8-31, 9-31) were made independently of each other. Table III shows the results of the calibration. The uncertainty given in Table III was taken as the square root of the sum of the squares of the variation of the ratio of flow times between the calibrated viscometer and the viscometer to be calibrated [5]. The uncertainties may be accumulated due to the errors of flow time measurements in the process of the stepping-up method. All of the flow time measurements for calibration were made by the automatic detection system; the variations of the ratio of flow times were very small, especially for the viscometers used in the low-kinematic viscosity regions. Therefore some of the uncertainty values obtained for these viscometers are thought to be of the same order, as is evident from Table III.

Table III. Results of Calibration of the NRLM Normal-Flow U-Tube Capillary Master Viscometers

Viscometer No.	Instrumental constant K (mm$^2\cdot$s^{-2})	Uncertainty of K (%)	Viscometer No.	Instrumental constant K (mm$^2\cdot$s^{-2})	Uncertainty of K (%)
1-11	0.00047786[a]	0.006	1-21	0.00060732[a]	0.001
1-11	0.00047845[b]	0.007	1-21	0.00060812[b]	0.004
2-1	0.0034257	0.007	2-31	0.0045657	0.005
3-1	0.019384	0.007	3-31	0.017229	0.006
4-1	0.092007	0.009	4-31	0.097248	0.007
5-1	0.48974	0.010	5-31	0.49718	0.007
6-1	2.4575	0.012	6-31	2.6865	0.008
7-1	11.463	0.012	7-31	10.747	0.011
8-1	49.654	0.022	8-31	44.879	0.014
9-1	139.99	0.030	9-31	151.28	0.023

[a] Calibrated with distilled water.

[b] Corrected constant for surface tension effect between water and JS2.5.

4.3. Viscometer Bath

A thermostated viscometer bath with water has the capacity to set two master viscometers so that the upper part of the timing bulb is about 80 mm below the water surface. Over the time required for the kinematic viscosity measurements, it was confirmed that the temperature control of the viscometer bath was maintained within ±0.002°C in the present temperature range of 20 to 40°C over the length of the viscometers and between two viscometers. The temperature of the bath was measured with a calibrated high-precision platinum resistance thermometer (Leeds & Northrup Company, Model 8163QB) together with a Mueller temperature bridge (Leeds & Northrup Company, Model 8069-B) having an accuracy of ±0.001°C, and the temperature control of the viscometer bath using an SCR proportional controller was monitored with a quartz thermometer (Hewlett Packard Company, Model 2804A) with a resolution of ±0.0001°C.

4.4. Measurement of Kinematic Viscosity

Each standard-viscosity liquid was measured by two master viscometers to cover the desired kinematic viscosity range, which belong in the same group, and three measurements of the flow time were carried out with each master viscometer at nine temperatures: 20, 22, 25, 28, 30, 32, 35, 38, and 40°C. The kinematic viscosity v (in $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$) is as follows:

$$v = Kt(1 + \varepsilon) \tag{6}$$

where K is the instrumental constant for a given master viscometer (in $\mathrm{mm}^2 \cdot \mathrm{s}^{-2}$), t is the average flow time in seconds, and ε is the correction for the effects of the air column in the viscometer and the thermal expansion of the liquid charge for the measured t. The measured value of the kinematic viscosity was obtained from the average of the values measured in two master viscometers.

5. RESULTS AND DISCUSSION

The uncertainty of the kinematic viscosity determined by the master viscometers is estimated to be within ±0.06% in two standard deviations in the range from 1.7 to 170,000 $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$. This value assumes to have no uncertainty in the value for the kinematic viscosity of distilled water adopted as the primary viscosity standard. The individual flow time measurement by the automatic detection system produces an error of less

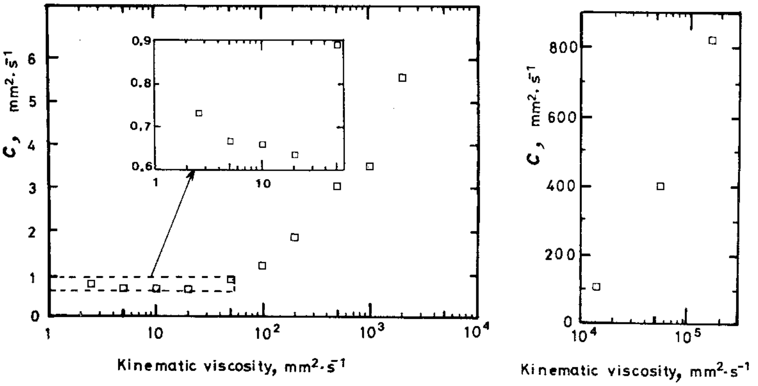

Fig. 4. Relation between the experimental value of c and a kinematic viscosity at 20°C for each standard-viscosity liquid.

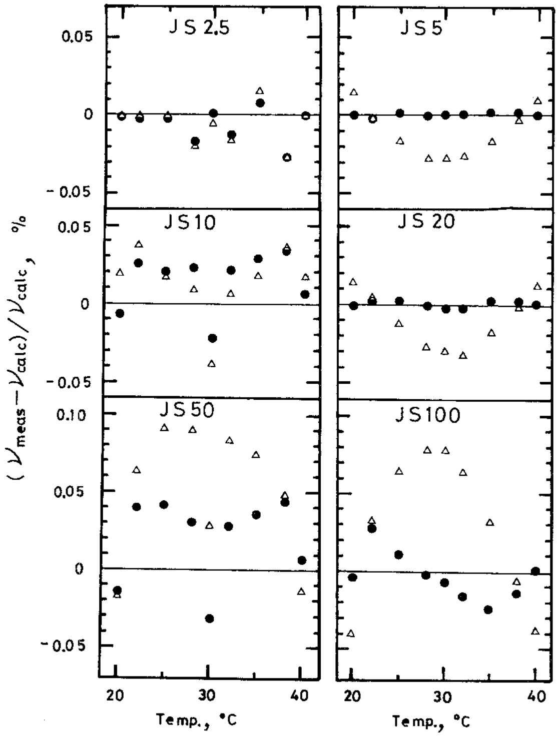

Fig. 5. Deviations between ν_{meas} and ν_{calc} for JS2.5 to JS100 obtained by the present Deming's method (●) and by the ASTM method (△).

than ±0.01 %, and the deviations of the kinematic viscosities determined by two master viscometers are less than ±0.03 %.

The results of some of the measured values of kinematic viscosity were applied to the ASTM kinematic viscosity–temperature equation with *a*, *b*, and *c* as the constant parameters for each standard-viscosity liquid, and adjusted parameters for an equation were determined by Deming's method of least squares. Figure 4 shows the results of the adjusted parameter *c* plotted against a kinematic viscosity at 20°C for each standard-viscosity liquid. Except for the low-kinematic viscosity regions, the value of *c* increased with increasing viscosity, and values ranged from about 0.63 to 822 $\mathrm{mm^2 \cdot s^{-1}}$, as is evident from Fig. 4. Figures 5–7 graphically show the deviations between measured values of the kinematic viscosity ν_{meas} and the estimated (calculated) values ν_{calc} obtained by the present Deming's method of least squares and results estimated by the usual linear regression

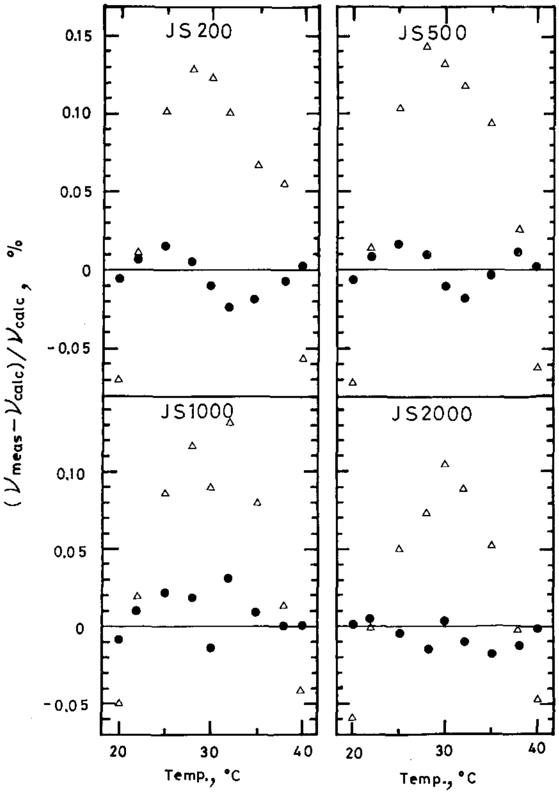

Fig. 6. Deviations between ν_{meas} and ν_{calc} for JS200 to JS2000 obtained by the present Deming's method (●) and by the ASTM method (△).

analysis based on the ASTM D341-77 (ASTM method). Figure 8 also graphically shows the results of a comparison between the present Deming's method and the usual ASTM method plotted against a kinematic viscosity at 20°C for each standard-viscosity liquid. In Figs. 5–8, the filled circles show the results estimated by Deming's method using the measured kinematic viscosities at 20, 25, 30, and 40°C. The open triangles show the results estimated by the usual ASTM method using the measured kinematic viscosities at 20, 30, and 40°C, respectively.

The deviations between estimations using Deming's method and those measured kinematic viscosities were less than ±0.04% for the 10 standard viscosity liquids JS2.5 to JS2000 and ±0.11% for the 3 standard viscosity liquids JS15H to JS200H, respectively. On the other hand, the deviations between estimations using the usual ASTM methods and those measured kinematic viscosities were less than ±0.14% for JS2.5 to JS2000 and ±0.60% for JS15H to JS200H, respectively. From the results of a comparison between the present Deming's method and the usual ASTM method, it was revealed that the uncertainty in the present estimation method is less than one-third that in the usual ASTM method. This means that Deming's method gave better results for an accurate estimation of the kinematic viscosity than the usual ASTM method. The effectiveness of the present estimation method is especially recognized for the higher kinematic viscosity-standard liquids, as is evident from Figs. 5–8.

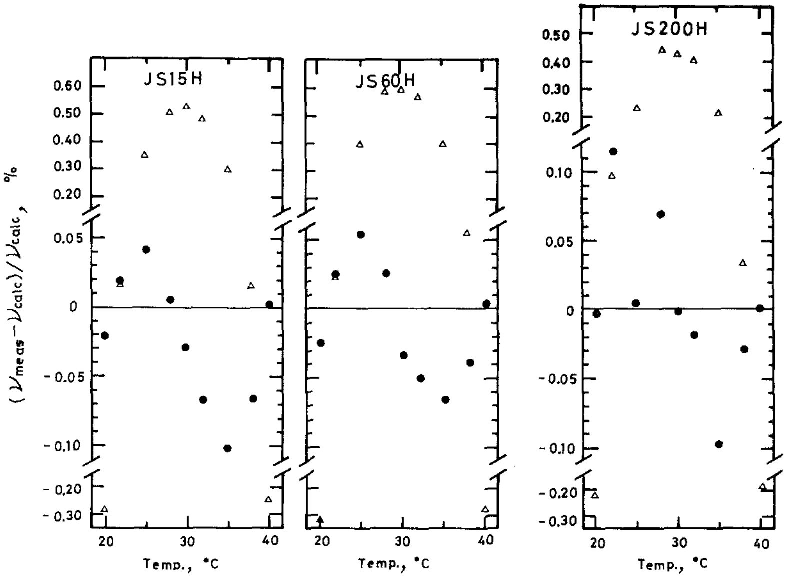

Fig. 7. Deviations between ν_{meas} and ν_{calc} for JS15H to JS200H obtained by the present Deminig's method (●) and by the ASTM method (△).

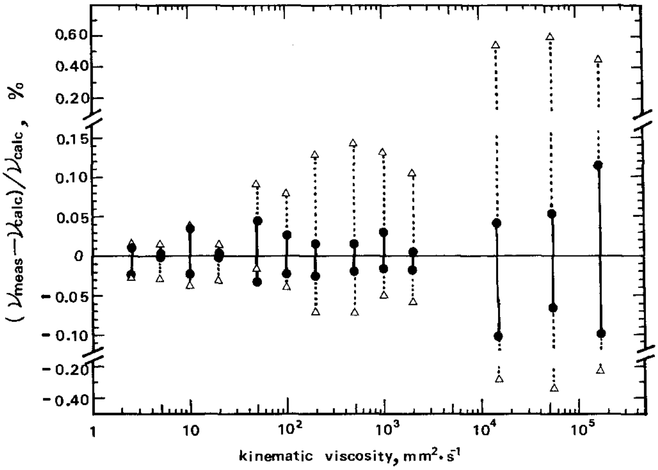

Fig. 8. Comparison between the present Deming's method (●) and the ASTM method (△) for each standard-viscosity liquid.

6. CONCLUSIONS

In the stated temperature range of 20 to 40°C, the present Deming's method of least squares can estimate the kinematic viscosity at any desired temperature within the stated uncertainties of ±0.1% for JS2.5 to JS2000 and ±0.2% JS15H to JS200H, respectively, which are provided by JIS Z8809.

Consequently, the estimation by the present Deming's method of least squares has been found to be available for a more accurate interpolation of kinematic viscosities for all of the standard-viscosity liquids. With the present method, the variable value *c* represented in the ASTM kinematic viscosity–temperature equation is assumed to have a constant value for each standard-viscosity liquid, as do *a* and *b* in the ASTM equation. This assumption has since been verified experimentally for all standard-viscosity liquids.

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