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# Viscosity of Liquid *n*-Heptane by Dynamic Light Scattering

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The dynamic viscosity of liquid *n*-heptane was measured in the temperature range 293–353 K by dynamic light scattering employing a newly designed optical setup. Commercial stearyl-coated silica particles were used as a seed, where a calibration of particle sizes to obtain absolute viscosity values was performed in other alkanes. The measurements included experimental runs at various particle concentrations and scattering vectors and in both a heating and a cooling cycle with a total standard deviation of 0.8–0.9%. As established reference values exist for alkane viscosities, from which the deviations were below 1% over the whole range of relevant temperatures, the experiments may also be regarded as a successful test of the accuracy of the method.

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**KEY WORDS:** alkanes; dynamic light scattering; *n*-heptane; photon correlation spectroscopy; viscosity.

## 1. INTRODUCTION

Dynamic light scattering (DLS) has been widely and successfully used for the determination of thermophysical properties of fluids [1, 2]. Besides for thermal diffusivity [3] and the mutual diffusion coefficient [4], the technique has been employed for the measurement of the dynamic viscosity [5, 6], a transport property of great importance for both theoretical concepts and practical engineering purposes. This application of DLS is based on the determination of the diffusion coefficient $D_p$ of suspended seed particles, which is related via the Stokes–Einstein relationship

$$D_p = \frac{kT}{3\pi\eta d} \tag{1}$$

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with the desired viscosity $\eta$, particle diameter $d$, Boltzmann's constant $k$, and temperature $T$.

When laser light is scattered in a particle dispersion, a fluctuating interference pattern results. The diffusion coefficient can be obtained by observation of the intensity fluctuations on a suitable detector, which are analyzed by calculating the autocorrelation function (ACF) $\hat{g}^{(2)}(t)$, which in the idealized case of monodisperse, freely diffusing particles, is of the form

$$\hat{g}^{(2)}(t) = a + b_{\exp}(-t/\tau_c) \qquad (2)$$

with experimental constants $a$ and $b$ and the decay time $\tau_c$. In the case of a homodyne detection the decay time $\tau_c$ is related with the particle diffusion coefficient by

$$D_p = 1/(2q^2\tau_c) \qquad (3)$$

where the modulus $q$

$$q = \frac{4\pi n}{\lambda_0} \sin \frac{\Theta}{2} \qquad (4)$$

of the scattering vector is determined by the refractive index $n$ of the liquid investigated, the laser wavelength *in vacuo* $\lambda_0$, and the scattering angle $\Theta$.

In a previous paper [7] we have shown that, a careful experimental procedure and suitable seed particles provided, DLS may be employed successfully for viscosity measurements in a variety of liquids near ambient conditions. In this contribution we want to investigate for an exemplary liquid how DLS measurements may be performed reliably in an extended range of temperature. There have been a number of former investigations on DLS viscosimetry over an extended temperature range, e.g., the early and pioneering work by Saad and Gulari [5] and various measurements by Byers et al. [6, 8]. This earlier work, however, suffers from a number of drawbacks and restrictions. Saad and Gulari used very small micelles, of some 1.5-nm radius, where the size of this seed changes by nearly 10% over a temperature interval of 40 K. Thus, this early approach is hardly suitable to obtain data with a high accuracy. In the work of Byers et al. the refractive index of the liquid investigated was estimated on the basis of the Eykman equation, which results in an uncertainty of more than 1% in the refractive index and thus of more than 2% in the viscosity by this effect alone. Moreover, these authors found significant differences of several percent between the results before and those after a temperature cycle. Finally, these measurements were performed in a relative manner, i.e., reference values for the viscosity of the liquid under investigation itself at ambient temperature were used for calibration.

In this paper measurements of the viscosity of *n*-heptane with the aims of a good reproducibility and small absolute uncertainty are presented. This specific liquid was chosen not only because there have been investigations by DLS described in the literature, but mainly because reliable reference data from a recent compilation by Dymond and Øye [9] are available both for *n*-heptane itself and for *n*-hexane and *n*-octane, which were used for the calibration of particle size. The measurements performed rely on a newly developed light-scattering system which allows the determination of the viscosity without having to know the refractive index of the liquid investigated.

## 2. EXPERIMENTAL SETUP AND PROCEDURE

### 2.1. Light-Scattering System

The light-scattering system is based on a newly developed scattering cell which utilizes the principle of a symmetrical setup and is described in detail elsewhere [10]. The idea of the symmetrical geometry is to replace the term $n \sin(\Theta/2)$, which is required for the evaluation of the scattering vector through Eq. (4), by quantities which are easily accessible experimentally. With the use of a square cuvette this can be accomplished if one tilts the cuvette from the usual position, where the exit window is perpendicular to the axis of observation, by an angle $\Theta_i/2$, where $\Theta_i$ is the angle between the incident laser beam and the axis of observation. From Snell's law and simple geometrical considerations, the relation

$$n \sin\frac{\Theta}{2} = n \sin\frac{\pi-\alpha}{2} = n \sin\frac{\pi-(\pi-2\beta)}{2} = n \sin\beta = n_e \sin\frac{\Theta_i}{2} \qquad (6)$$

follows (see Fig. 1). The angle of incidence can be easily measured; if the surrounding medium is air, the external refractive index may be set equal to unity, otherwise a single determination of $n_e$ suffices, independently of the liquid to be investigated. It should be noted that the scattering vector $\mathbf{q}$ for a fixed angle of incidence is thus independent of the individual liquid under investigation, whereas the scattering angle $\Theta$ is not.

On the basis of the symmetrical setup described we have designed a thermostated scattering cell for a temperature range of approximately 280–400 K, which is made of an anodized aluminium alloy. A commercial square cuvette contains the liquid under investigation. The sample temperature is measured directly by a calibrated Pt-100-resistance probe

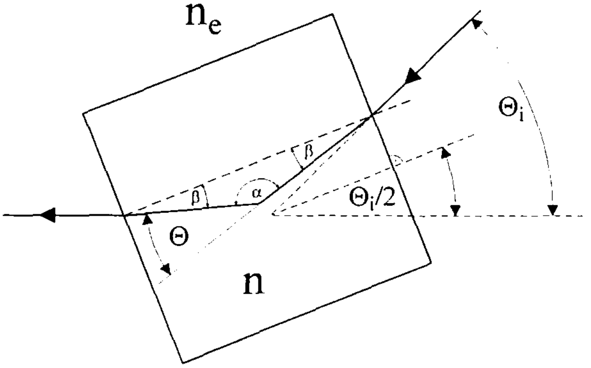

**Fig. 1.** Principle of the symmetrical setup, where the cuvette is tilted from the perpendicular position by $\Theta_i/2$; here the case $n > n_e$ is illustrated.

immersed with an estimated uncertainty of better than 0.05 K. The cuvette is fixed to an adjustable support, which can be aligned to the angle required by a rotating table. The cell contains an oil, which serves as a thermostating medium and the refractive index of which ($n_{20\ \mathrm{C},\ 633\ \mathrm{nm}} = 1.47$) closely matches that of the glass material of the cuvette ($n = 1.47$) and of the outer cell windows ($n = 1.52$), respectively. The oil is temperated via an external computer-controlled water or oil circuit. The temperature stability of the sample during an experimental run is better than $\pm 0.01$ K near room temperature and better than $\pm 0.02$ K at the higher temperatures employed. Optical access is realized by several windows, which allow perpendicular incidence for angles of 30, 60, and 90°. As a specific example, these angles of incidence result in scattering angles of 32, 64, and 97° for *n*-heptane at 298 K when the symmetrical alignment is used.

The symmetrical setup sets the user free from measuring the refractive index of the liquid under investigation. Only that of the index-matching fluid must be determined once. This may be accomplished be the use of commercial refractometers or by measuring the refraction at a prism immersed [10].

The cell, placed on two translation stages, is illuminated by a vertically polarized 10-mW He–Ne laser, which is mounted on a rotatable arm for a variation of the angle of incidence (cf. Fig. 2). The laser bundle is focused by two cylindrical lenses with different focal lengths, where a short focal length is used to focus the laser bundle in the direction perpendicular to the scattering plane. This approach ensures a high signal-to-noise-ratio of the resulting correlation function and prevents a significant lack of definition in the scattering angle. The light scattered is collected by two apertures at a

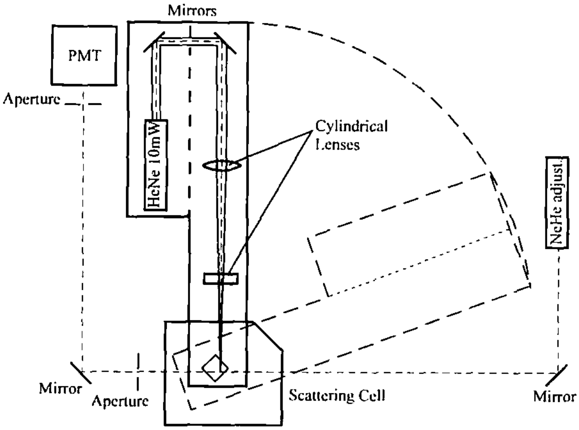

Fig. 2. Light-scattering system.

distance of 0.5 m, where the first one is a slit, variable both in height and in width for an optimal adjustment, and the second one is circular with a 1-mm diameter. The signal from the photomultiplier tube is amplified, discriminated, and fed to an ALV 5000/E correlator with a pseudologarithmic channel spacing.

## 2.2. Sample Preparation and Experimental Conditions

A key feature for a successful determination of the dynamic viscosity is the choice of suitable seed particles. As silica particles exhibit excellent chemical stability and may be used for a large variety of liquids, commercial silica particles have been chosen for this study. The particles used are rendered organophilic by grafting stearyl ($C_{18}$) groups to the surface and exhibit a standard deviation in particle size of less than 10%. The refractive index was measured to be $n_{633\ \mathrm{nm}} = 1.433$.

The liquids employed had certified purities of 99% (*n*-hexane), 99.5% (*n*-heptane), and 99.6% (*n*-octane), respectively, and were used without further purification. Particles were dispersed in the liquid of investigation and submitted to ultrasonic treatment for several hours to improve dispersion and to break up aggregates. Samples were then directly prepared by filtering the dispersions through syringe filters with a 0.45-$\mu$m pore size in order to remove dust and residual aggregates. As the filter efficiency cannot be determined easily and may vary depending on solid contents, the particle concentration in the samples could not be measured directly. An estimate was obtained by comparing the scattered intensity of filtered samples with those of unfiltered samples with defined concentration, where a certain amount of dry particles was weighed into the liquid. The resulting particle volume fractions $\Phi$ were in the range of $1\text{–}3 \times 10^{-4}$ for all samples investigated. Such low concentrations were chosen in order to rule out completely possible influences of multiple scattering or particle interactions. This small solid content resulted in net transmissions of well above 90% in all cases.

For both *n*-heptane and the liquids used for calibration, measurements were performed at four scattering angles. Three of these measurement series were performed with the use of the symmetrical geometry (denoted "s") at angles of incidence of 30, 60, and 90°, respectively. Additionally, one measurement series was performed with a conventional perpendicular geometry ("p") and an angle of incidence of 90°, which of course is also the scattering angle in this case. In this instance, the refractive index of the liquid under investigation must be known. This was determined with the help of an Abbe refractometer, where the refractive index for the laser wavelength used was derived from that measured at the sodium *D* line (589 nm) and a small correction employing the mean dispersion measured by the refractometer. For measurements at these four angles a medium ("m") particle concentration of $\Phi \approx 2 \times 10^{-4}$ was used. Additionally, measurement series at an angle of incidence of 60° were performed, employing both a higher ($\Phi \approx 3 \times 10^{-4}$) and a lower ($\Phi \approx 1 \times 10^{-4}$) particle concentration, denoted "h" and "l," respectively. Hence, in total, six experimental series were carried out.

All measurement were first performed at a temperature of 298.15 K, the reference temperature used in the work of Dymond and Øye [9]. In the case of *n*-heptane itself, a temperature cycle was then initiated, starting at 303.15 K and going up in steps of 10 to 353.15 K, the highest temperature used in this investigation, as it is already somewhat outside the range of the reference regression with a maximum temperature of 346 K. Measurements were also performed for the same temperatures when lowering the temperature stepwise. The lowest temperature employed in this study was 293.15 K, as at lower temperatures air humidity started to condense on the sample cell windows. Lower temperatures can be realized if desired, e.g., by flushing the cell windows with a stream of dry air or working in an environment with low humidity.

### 2.3. Experimental Procedure and Data Evaluation

During the measurement series seven single experimental runs were performed under each condition in the particle calibration procedure and at each point in the temperature cycle for *n*-heptane described above. Measurement times of as long as typically 20 min were chosen for a single experimental run to achieve a low statistical uncertainty of the correlation functions obtained. The ACFs were analyzed for the decay time $\tau_c$ by the multifit procedure described in more detail previously [11] to ensure agreement with the underlying model. With this approach a nonlinear fit on the basis of Eq. (2) is performed to a variety of lag-time intervals (cf. Fig. 3). The decay time is taken from the mean value of these 99 fits; the standard deviation in $\tau_c$ may be regarded as a measure of quality reflecting both possible systematical deviations and noise in the ACF. An upper limit of 0.3% in this value was established as a criterion for acceptance of a single run. In most instances the standard deviations in the decay time were clearly smaller. Hence, for all conditions at least five single runs were accepted; in many cases this could be done with all seven of them. As a consequence of the high quality of the individual experiments, the standard deviation in the diffusion constants obtained for consecutive runs was typically 0.1–0.3%.

As the laboratory thermostat could be given only a certain nominal temperature value for an internal control, but did not allow a real feedback

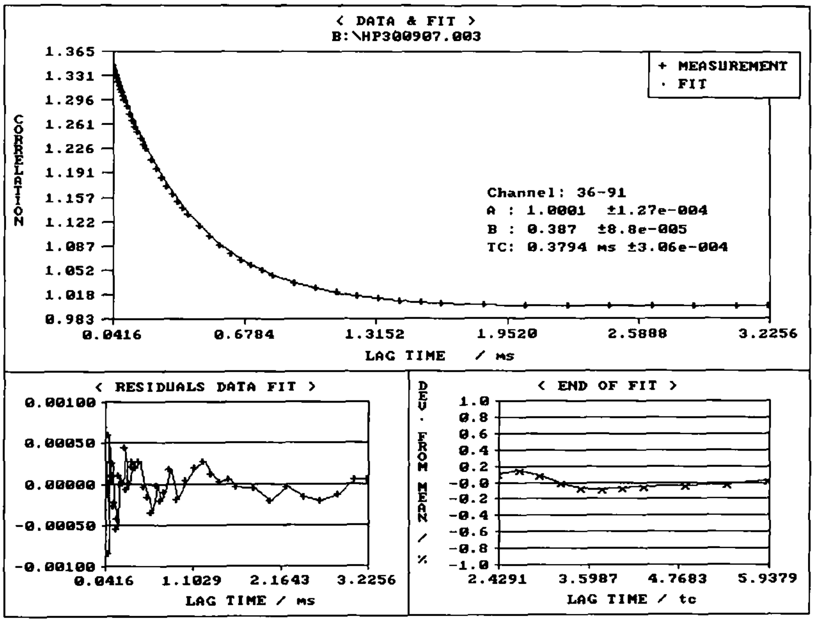

Fig. 3. Multifit procedure, where a fit is performed to various time intervals. The insets show the residuals between data and fit (left) and the deviations of fits up to various lag times from the mean value of all fits (right).

loop from the temperature probe, measurements could be performed not exactly at the nominal temperatures, but at values deviating up to ±0.3 K from them. For reasonable data evaluation and representation the obtained viscosity values thus had to be corrected to the nominal temperatures. Therefore, on the basis of the measured viscosities the relative deviation from the reference value was calculated, and this deviation was then used to express the viscosities at the relevant nominal temperatures. Combining the various concentrations and scattering angles, measurements in the heating and the cooling phase, and various individual runs, more than 60 single measurements form the basis for the viscosity value obtained at a specific temperature.

## 3. RESULTS AND DISCUSSION

For the measurement of absolute viscosity values it is essential to calibrate particle size. This is best performed by using the DLS technique itself, where liquids with well-documented viscosities and with chemical and physical properties similar to those to be investigated are to be chosen. In this investigation we calibrated particle sizes by measurement series as described above for *n*-hexane and *n*-octane at 298.15 K. With the reference values of Dymond and Øye at these temperatures, 0.2949 mPa · s ± 0.4% for hexane and 0.5092 mPa · s ± 0.3% for octane, particle sizes of 222.8 nm ± 0.6% and 222.4 nm ± 1.1% resulted, where the uncertainties given reflect solely the standard deviation of the calibration measurements. For the following measurements we used the mean value of 222.6 nm, with a resulting total standard deviation of 0.9%. Taking into account also the uncertainty of the reference values themselves would result in a slightly larger uncertainty.

On the basis of this calibration the viscosity of *n*-heptane was measured as described above, where the resulting mean values for the specific temperatures are composed of a great multitude of individual measurements. These results are documented in Table I together with the individual standard deviations and plotted in Fig. 4 in comparison with the reference regression and light-scattering results of Brunson and Byers [6], where measurements were performed in a relative manner based on the viscosity of heptane at ambient temperature. The deviation of our results from the reference regression (cf. Fig. 5) is −0.5% at low temperatures and −1.0% at the highest temperature covered by the regression, and it is thus well within the combined uncertainties. The deviation would accordingly be smaller if all values were referred to the value at 298 K. It is also worth mentioning that the standard deviation of our measurements is remarkably constant over the whole temperature range.

**Table I.** Viscosities and Experimental Standard Deviations Obtained for *n*-Heptane

| $T$ (K) | $\eta$ (mPa·s) | $\sigma$ (%) |
|---|---|---|
| 293.15 | 0.409 | 0.9 |
| 298.15 | 0.387 | 0.9 |
| 303.15 | 0.367 | 0.9 |
| 313.15 | 0.332 | 0.9 |
| 323.15 | 0.301 | 0.8 |
| 333.15 | 0.275 | 0.8 |
| 343.15 | 0.251 | 0.8 |
| 353.15 | 0.230 | 0.9 |

An essential point in performing DLS viscosity measurements is the stability of particle dispersions. Williams and Byers [8] in some instances reported marked differences of several percent in the diffusion coefficients measured before and after high-temperature operation. In our experiments deviations between the final and the initial viscosities measured, which might also be expressed as effective particle diameters, were comparatively small, with one extreme value of −0.9% in one case, but also with one positive deviation of +0.2% in another one. The average values, again plotted as deviations from the reference regression, for the temperature

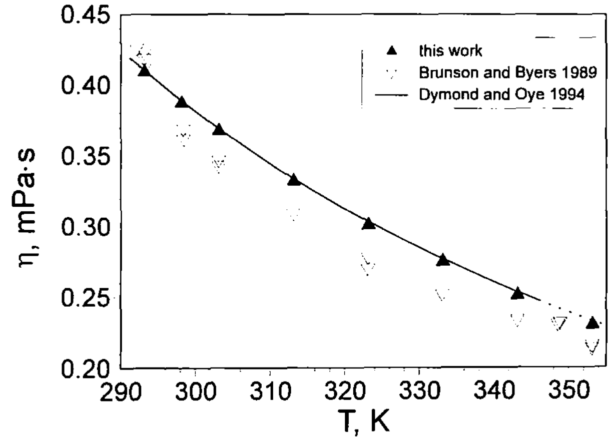

Fig. 4. Measured viscosities in comparison with the light-scattering results of Brunson and Byers [6] and the reference of Dymond and Øye [9].

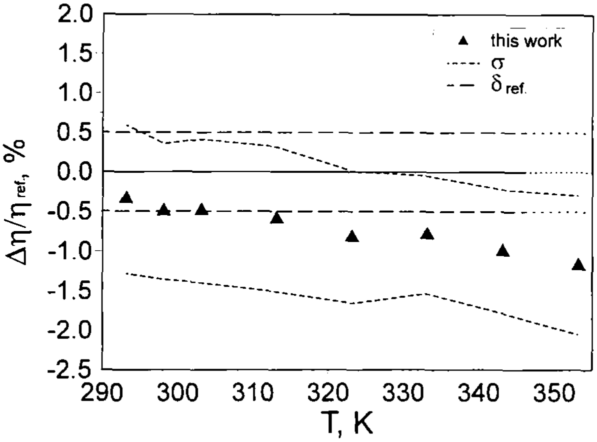

Fig. 5. Percentage deviation of measured viscosities from the reference regression [9]. Additionally, the experimental standard deviation $\sigma$ and the uncertainty $\delta_{\mathrm{rel}}$ of the reference are given.

cycle are shown in Fig. 6, from which also the average difference of $-0.4\%$ between the final and the initial value can be seen. Larger differences of typically $-2\%$ could be found when the samples where cooled more rapidly, i.e., in about 1 h, from the highest temperature down to 298 K. It is remarkable that the original values for this temperature could be reproduced when these samples were stirred. These findings indicate the possibility of a fractionation effect in the not exactly monodisperse sample,

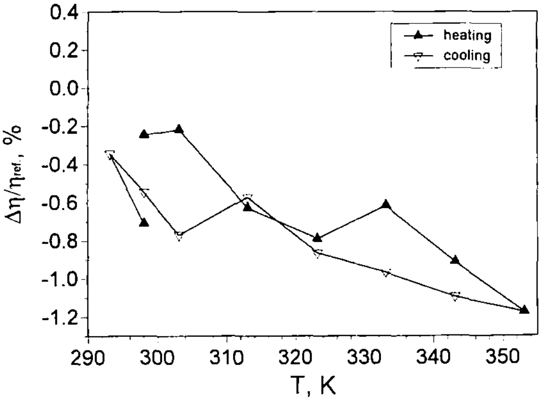

Fig. 6. Percentage deviation from reference, resolved for heating and cooling cycle.

where larger particles tend to accumulate in the bottom of the sample cell. Fractionation effects with similar particles have been described by Jansen et al. [12] in a variety of solvents. Although in their study significantly higher concentrations were investigated and clear phase separation effects were visible, it cannot be ruled out that also in our experiments similar effects due to different interaction potentials of particles of various sizes may be relevant. It should, however, be emphasized that in our experiments no signs of particle flocculation could be observed. Even if such fractionation effects should be given care in all viscosity measurements by DLS, the results obtained demonstrate that possible effects are small in our experiments.

Another point which should be given consideration in this type of experiment is the possible dependence of the diffusion coefficients measured on the experimental conditions, i.e., particle concentration and scattering vector employed, as undesired effects such as particle interactions and multiple scattering might manifest themselves. From the small overall standard deviation of our measurements it is obvious that such effects, if present, are small. There are some trends, however, which initiate further discussion and should also stimulate further work on this subject.

Giving the individual measurement series a closer look, as in Fig. 7, it can be recognized that the 60sh and the 30s measurements deviate from the other data over the whole temperature range. At first sight, some kind of experimental error may be assumed, e.g., in the alignment of the

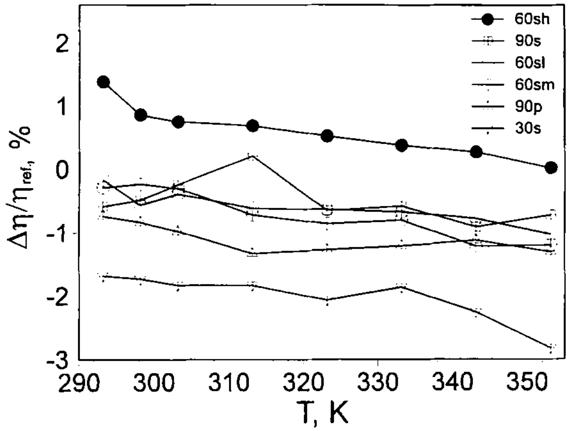

**Fig. 7.** Percentage deviation from reference, resolved for various measurement conditions. Numbers refer to angles of incidence; s, symmetrical; p, perpendicular; l, m, and h; low, medium, and high concentration, respectively.

setup, although this was done in a way so that associated errors should be significantly smaller. It is surprising, however, that a similar trend for these specific measurements may also be observed in the calibration measurements (cf. Table II), performed in hexane and octane, which indicates that there might be a more fundamental reason.

Therefore, the major reasons for possible systematical deviations are briefly discussed to judge if they might explain the observed trends in this case. This discussion also highlights the important considerations in performing DLS viscosity measurements in general.

### 3.1. Multiple Scattering

Although particle concentrations are low and the setup was designed in a way to suppress multiple scattering as far as possible, the probability of higher-order scattering events is naturally nonzero. As the decay time of double-scattered light is of the same order of magnitude as 180° single-scattered light [13], a significant amount of double-scattering should result in a shorter effective decay and thus in lower viscosities and particle diameters, respectively. Clearly, the trend is opposite for the 60° measurements, as at the highest concentration employed, where a possible effect of multiple scattering should be most evident, a slightly larger value for $\eta$ and $d$ is observed. From the large difference in decay times between multiple scattering and 30° single scattering, a possible effect would be consistent with small $\eta$–$d$ values for the 30 s measurements, although the high scattering amplitude for small scattering angles and the absence of systematic deviations in the ACF argue against this explanation.

**Table II.** Deviation of the Individual Measurement Series at 298 K from the Respective Mean Values

| Series | $n$-Hexane, $(d-\bar{d})/\bar{d}$ (%) | $n$-Heptane, $(\eta-\bar{\eta})/\bar{\eta}$ (%) | $n$-Octane $(d-\bar{d})/\bar{d}$ (%) |
|---|---|---|---|
| 30s | −0.7 | −1.2 | −1.7 |
| 60sn | −0.4 | −0.1 | +0.4 |
| 60sm | 0 | +0.2 | +1.3 |
| 60sh | +0.9 | +1.4 | +1.1 |
| 90p | −0.2 | −0.3 | −0.7 |
| 90s | +0.4 | +0.2 | −0.5 |

### 3.2. Reflections

DLS results may be influenced by reflections from cell windows resulting in secondary beams where the scattered light detected may form a contribution with a different scattering angle and thus a different decay time. This possibility may be ruled out, however, by the way the scattering cell is constructed and the alignment is performed. For an angle of incidence of 60° the lowest order which can be detected is after two reflections. These reflections are very weak, as the refractive indices of sample, cuvette, and oil are not too different. Moreover, the higher-order reflection in this special case would result in a contribution with a large scattering angle and thus with a too short decay time. For the 30° measurements the lowest reflection within the scattering volume is of at least fourth order and may thus be completely neglected.

### 3.3. Heterodyning

Heterodyning means that the detected light is not due purely to scattering from the particle dispersion itself, but also from direct laser reflections or flares. A heterodyne contribution would result in an additional term in the ACF with a decay time larger by a factor of 2. Thus, heterodyning cannot be the reason for the deviations with either the 30s or the 60sh series, as in the latter case a high particle concentration would reduce the relative contribution of a heterodyne term.

### 3.4. Polydispersity

It was shown by Pusey and van Megen [14] that even small polydispersities may result in a dependence of the diffusion coefficient measured on the scattering vector, if the angular distribution of scattered light is not homogeneous. From calculations based on the Rayleigh–Gans–Debey (RGD) theory, which can be regarded as a good approximation in this case due to moderate differences in the refractive indices of particle and solvent, a decrease in the effective $\eta$ or $d$ is predicted up to a first minimum at $qd = 9(1-\sigma)$, where $\sigma$ is the standard deviation in particle size. It is clear that this trend is contradictory to the deviations in the 30° measurements. As the minimum is not reached by the range of scattering vectors employed here, the effect of particle polydispersity is not pronounced in our measurements. With an assumed polydispersity of 8%, calculations yield deviations from the mean value over the four scattering vectors of +0.8% (30 s), +0.2% (60 s), −0.4% (90 p), and −0.6% (90 s).

In addition, it may be noted that due to weighting with the scattering amplitude, the mean diameter determined in the experiments does not coincide with the arithmetic mean of the particle distribution. This effect is irrelevant in the RGD limit as both the size calibration and the viscosity measurements rely on practically the same value. The reason is that almost-identical scattering vectors are employed in either case, with the use of the symmetrical geometry these are truly identical at a given temperature.

### 3.5. Particle Interactions

The effects of particle interactions on the effective diffusion coefficient measured have been extensively discussed in the literature, see, e.g., Refs. 15 and 16. In the case of the experiments described, particle volume fractions are low so that the average particle spacing $R$ of a few microns is larger than the inverse scattering vector $q^{-1}$ in all cases, where the maximum value of $q^{-1}$ is about 130 nm for a 30° angle of incidence, so that the motion of individual particles is probed. The particle interactions of this type of silica spheres in alkanes are determined by a comparatively weak van der Waals attraction, as the small difference in refractive indices between particles and solvents results in a small Hamaker constant, a hard-sphere repulsion, and a short-range interaction between the octadecyl chains on the particle surface [17]. With these types of interactions and the low volume fractions employed, the possible effect on the diffusion coefficients measured should be negligible.

Summarizing this discussion, no clear picture evolves to explain the experimental trends observed. It should be noted, however, that particularly the two deviating measurement series result in a comparatively high value for the total standard deviation of 0.8–0.9%. As both the actual measurement and the calibration series show similar trends, the calculation of an effective particle diameter for each measurement condition may practically account for this despite the underlying reasons remaining obscure. With the use of the respective diameter in the viscosity measurements, the experimental standard deviation is reduced to a value of 0.4–0.5%. This approach of course does not affect the actual mean viscosity value obtained.

## 4. CONCLUSION

Dynamic light scattering has been used to measure the viscosity of *n*-heptane in the temperature range 293–353 K with a deviation of the results from established reference values below 1%. Although the uncertainty of the DLS method cannot be exactly specified, the experimental execution with a variation of measurement conditions is in a way that possible errors may be regarded to be mainly of a statistical nature. Thus, the total standard deviation may be used as a guideline for experimental uncertainty. Combining the standard deviation of the measurements themselves of 0.8–0.9% with that of the calibration measurements and the given uncertainty of the reference values used for calibration, a value of about 1.4% results from a propagation of errors. As the results are not completely independent of the experimental conditions and a correlation has been observed between results in the measurements and the calibration, this combined error would reduce to about 0.9%, if an individual calibration under the respective condition were chosen for the evaluation of the viscosities. Such an approach, however, might be regarded as over-sophisticated, and so a fair estimate for the absolute uncertainty of the method should be in the range 1–2%. This value is not much worse than that of other more commonly used methods. With the necessary use of particle dispersions and effects such as particle polydispersity and surface effects given for this technique, it would be difficult to achieve absolute uncertainties clearly below 1%.

Dynamic light scattering, however, offers a number of inherent advantages, especially as it is may be used in thermal equilibrium and for the simultaneous acquisition of several transport properties. Thus, future work should be devoted not only to the mere improvement of the technique itself and acquisition of data for simple liquids, but also, especially, to the application of the technique to fluid mixtures with simultaneous determination of the mutual diffusion coefficient and to measurements in the vicinity of critical points.

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