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Determination of particle density by sedimentation methods —

Part 1: Isopycnic interpolation approach

Détermination de la densité de particules par méthodes de sédimentation —

Partie 1: Approche par interpolation isopycnique

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CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
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Foreword

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This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

A list of all parts in the ISO 18747 series can be found on the ISO website.

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Introduction

Dispersions are widely used in industry and everyday life. There is a need to understand the density of dispersed particles or droplets, e.g. for physico-chemical calculations like kinematic viscosity of dispersions (ISO 3105), determination of particle size distribution by separation techniques[4][5][7], characterization of core/shell or capsule-like particles, determination of particle compressibility[10] or optimization of dispersion stability by density matching[11].

The density of a body is its mass divided by its volume. This is straightforward for the mass of a larger body or particle. However, experimental determination of the volume of a macroscopic body is difficult. The geometrical volume (length, width and thickness) and the volume relevant for the determination of density can differ due to surface irregularities, fractures, fissures and open and closed pores or the measuring techniques employed.

Density determination of micro-particles, in particular nanoparticles dispersed in a liquid, raises issues, not only for the determination of mass and volume due to the small size but also, and mainly, because of the boundary between the liquid and the particle, which is fuzzy. Molecules in the continuous phase are partially immobilized at the surface. Physico-chemical properties (e.g. viscosity, ion concentration) in the fuzzy coat differ from bulk. This is especially important for small microparticles and nanoparticles which are dispersed in a polymer or biological fluid[12]. The so-called corona can be interpreted as an integral part of the particle and increases the effective/apparent volume compared to the space occupied by the dry material. The thickness of this layer ranges between a few to tens of nanometres and the effective/apparent volume deviates increasingly from the "geometrical" volume, if the particles become smaller. As a consequence, density determination by traditional methods is affected.

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Determination of particle density by sedimentation methods —

Part 1: Isopycnic interpolation approach

1 Scope

This document specifies a method for the determination of the density of solid particles or liquid droplets (below referred to generically as "particles") dispersed in a liquid. The method is based on the fact that a particle wholly immersed in fluid experiences buoyancy equal to the weight of the fluid displaced by this particle (Archimedean principle), and if its mass force matches the buoyant force, it stops gravitational or centrifugal settling/creaming and the particle remains suspended. This implies that the density of the particle equals the density of the liquid. In this document, particle density determination is conducted by analysing the direction of the migration movement of particles dispersed in liquids with densities that are lower and higher than particle density. All particles are of the same material composition.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14887:2000, *Sample preparation — Dispersing procedures for powders in liquids*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
dynamic viscosity
measure of the resistance of a fluid which is being deformed by shear stress

Note 1 to entry: Dynamic viscosity is calculated by shear stress divided by shear rate and determines the dynamics of an incompressible Newtonian fluid.

3.2
migration
directed particle movement (sedimentation or creaming/flotation) due to acting gravitational or centrifugal fields

Note 1 to entry: Sedimentation occurs when density of particles is larger than that of liquid density. Creaming/flotation occurs when density of particles is smaller than that of liquid density. In these two processes, particles move in opposite directions.

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3.3
migration velocity
absolute value of sedimentation or creaming/flotation terminal velocity

Note 1 to entry: Velocity of creaming/flotation is indicated by a negative sign.

3.4
true particle density
ratio of particle mass to particle volume excluding all pores, closed or open, and surface fissures

3.5
skeletal density
ratio of the mass of discrete pieces of solid material to the sum of the volumes of the solid material in the pieces and closed (or blind) pores within the pieces

[SOURCE: ASTM D3766]

3.6
buoyant density
ratio of particle mass to particle volume including filled or closed pores as well as adjacent layers of liquid or other coating materials

4 Symbols

For the purposes of this document, the following symbols apply.

Quantity	Symbol	Unit	Derivative unit
Acceleration	a	m/s^2	
Angular velocity	ω	rad/s	
Dynamic viscosity	η	Pa·s	mPa·s
Force due to buoyancy	F_B	N	
Force due to gravity	F_G	N	
Liquid density	ρ_L	kg/m^3	
Particle density	ρ_P	kg/m^3	
Radius	r	m	mm
Rotational frequency	n	s^{-1}	min^{-1}
Standard acceleration due to gravity	g	m/s^2	
Temperature	ϑ	°C	
Time	t	s	
Velocity	v	m/s	
Velocity measurand	y		
Volume	V	m^3	
Wavelength	λ	m	nm

5 Basic principle of method

The Archimedean principle states that a particle wholly immersed in liquid experiences buoyancy equal to the weight of the fluid displaced by this particle. The balance of the weight forces of a particle due to gravity, F_G, and of buoyancy force, F_B, determines whether its net gravitational motion is upward, downward, or neither [Formula (1)].

$$F_G = \rho_P \cdot V \cdot g = F_B = \rho_L \cdot V \cdot g \quad (1)$$

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where

ρ_P is the density of the particle;

V is the particle volume;

g is the gravitational acceleration;

ρ_L is the density of the liquid.

A particle stops migration if forces F_G and F_B are equal, and therefore the density of the particle shall equal the density of the liquid ($\rho_P - \rho_L = 0$). These considerations are applicable, independent of size or shape of the particle, as well as independent on dynamic viscosity of continuous phase. In the case of multicomponent dispersions, all particle species stay suspended only when the density of all particles is the same as the liquid density.

Formula (1) and the above considerations also hold for dispersed particles in a centrifugal field, where g shall be replaced by a centrifugal acceleration a [Formula (2)].

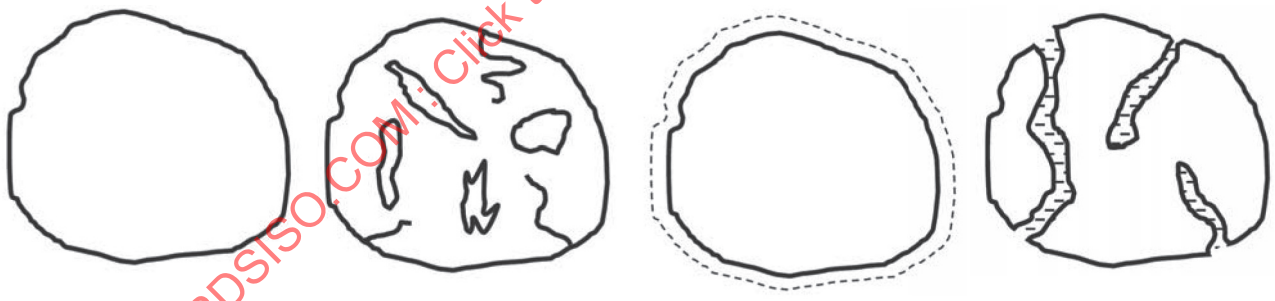
$$a = \omega^2 \cdot r = (2 \cdot \pi \cdot n)^2 \cdot r \quad (2)$$

where

ω is the angular velocity of the rotor;

r is the distance of the particle under consideration from the centre of revolution;

n is the number of rotations per seconds of the rotor.



a) Macroscopic solid particle **b) Particle with closed pores** **c) Particle with an adjacent/immobilised layer to its surface** **d) Particle with open pores**

Figure 1 — General structures of particles regarding density determination [21]

According to Formula (1), density corresponds to the volume of displaced liquid. This equals the geometrical volume of a macroscopic solid particle [Figure 1 a)]. It follows that determined density equals true solid particle density, which corresponds to the true material density. If the particle is made of different materials, true density reflects the (material) density and volume/mass fraction of each material.

In case of a particle with closed pores [Figure 1 b)] or pores not filled with liquid of continuous phase, as well as with a surface layer [Figure 1 c)], Formula (1) determines the buoyant density, ρ_B (also called

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effective, average, or apparent density), of the particle of total volume, V_{tot}, which can be calculated according to Formula (3):

$$\rho_B = \frac{\rho_1 \cdot V_1 + \rho_2 \cdot V_2}{V_{tot}} \quad (3)$$

where

ρ_1 is the skeletal density;

V_1 is the volume of the skeleton;

ρ_2 is the density of the material of closed and open not filled pores;

V_2 is the volume of the pores;

V_{tot} is the total volume.

Figure 1 b) shows particles with closed pores. The same applies to particles coated with a layer of a different material of a volume of V_2 [Figure 1 c)]. The term "coated" is used in a generic way. It can refer to a shell of different materials (e.g. silica coated magnetic nanoparticles), a particle brushed by polymers or macromolecules or even an immobilised (unstirred) layer of liquid molecules. The layer itself can be porous or exhibit material gradients. Formula (3) is based on the assumption that the composition of volume V_2 does not change when dispersing the particles into different test liquids (see Clause 6). Volume V_2 is not mixed or exchanged with the continuous phase or at least only with a time constant larger than the experimental measuring time. The term "unstirred" also reflects the fact that, in case the particle exhibits any movement (e.g. due to electrical or gravity fields), then volume V_2 shall be treated as an "integral part" of the particle, which moves together with the main particle. It is obvious that the density determined according to Formula (3) deviates more and more from the true particle density if particles become smaller (e.g. nanoparticles).

Figure 1d) shows a particle that has open pores whose content can be freely exchanged with the continuous phase. In other words, density determination applies to particles whose pores are filled with the continuous phase. In this case, skeletal density is determined.

This document focuses on buoyant density, which coincides with the true (material) density [Figure 1 a)], with apparent density [Figure 1 b) and Figure 1 c)] and with skeletal density [Figure 1 d)]. The above four cases are separately discussed for clarity, but particles which belong to several cases exist, e.g. particles with closed pores and open pores filled with the liquid of continuous phase.

6 Measuring techniques to determine the direction of gravitational and centrifugal migration of dispersed particles

The determination of particle density, according to Formula (1) or (2), is based on the detection (measurement) of the migration direction of particles dispersed in continuous liquid phases with different densities, which are lower or higher than the expected particle density.

Any method which allows detection of the direction of particle migration (sedimentation or creaming/flotation) is appropriate. Particle migration may be driven by earth gravity or centrifugal field. Basic principles available include:

a) monitoring the concentration change near an appropriate interface, e.g. below the meniscus (dispersion/air interphase), or above the bottom of the measuring cell, either directly or by a concentration related signal, e.g. voltage, current, light intensity, X-ray absorption, conductivity, or electro-acoustic;

b) measuring the value of migration velocity or a directly correlating measurand of dispersed particles in the bulk of the dispersion;

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c) sedimentation or creaming/flotation in a density gradient[1)].

Attention should be paid to temperature changes. Instruments which allow for temperature setting and control are preferable. Alternatively, measurements can be conducted within a short time. Multichannel instruments are advantageous as they increase the sample throughput, and samples are measured under similar experimental conditions. Analytical cuvette centrifugation is especially appropriate for nanoparticles and continuous phases of high viscosity.

The volume fraction of dispersed phase does not enter into the calculations in Formulae (1) and (2). Choose the final volume concentration in accordance with the specification of the analytical technique employed. High volume fraction of particles (greater than 5 %) should be avoided in that, because migration velocity decreases with increasing volume concentration due to hydrodynamic interaction (hindrance)[13][14][20], experimental determination of the migration direction can be more difficult and time-consuming.

7 Preparation of samples

7.1 Solutions

Solutions which differ in density but cover the expected particle density range should be prepared. It is convenient to start with a concentrated solution and dilute with a solvent until the liquid density ρ_L, is smaller than the buoyant particle density, ρ_P. Prepare two solutions as a minimum, but for more precise results, a series of five to eight test liquids where the median density corresponds to the approximate particle density is recommended. If possible, density spacing should be about equidistant. A number of suitable solutions are tabulated in the literature (e.g. References [15] and [16]). Typical examples are shown in Figure 2.

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1) The buoyant density centrifugation method is not part of this document. Nevertheless, a short description is given in Annex D.

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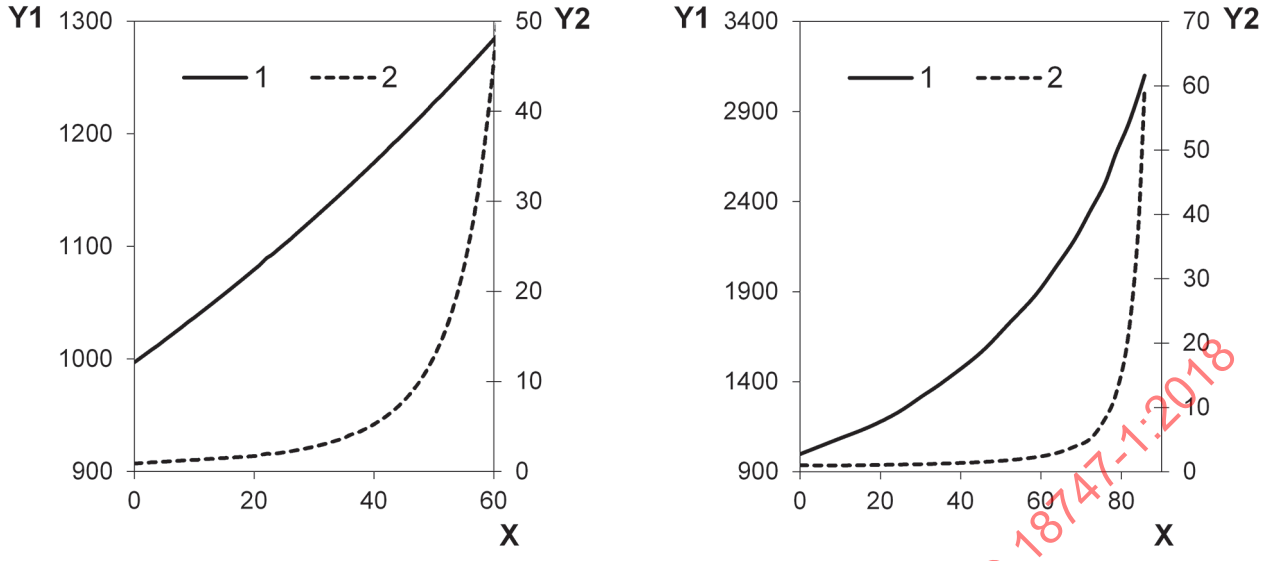

a) Sucrose **b) Sodium polytungstate**

Key

X mass fraction in %

Y1 density ρ in kg/m^3

Y2 dynamic viscosity η in mPa·s

1 density dependence

2 viscosity dependence

NOTE See References [15] and [16] for more information.

Figure 2 — Density and dynamic viscosity dependence on sucrose and sodium polytungstate mass fraction at ϑ = 25 °C

Another approach consists of mixing two liquids of different density. Typical examples include water-ethanol-mixtures or water-glycerol mixtures. Both of them are well characterized[18][19]. Densities of these mixtures range from 789,7 kg/m^3 to 998,2 kg/m^3 and from 998,2 kg/m^3 to 1 263,9 kg/m^3 at ϑ = 20 °C, respectively.

NOTE Numerical values of density as well as dynamic viscosity are functions of temperature. If the density and viscosity values are not known for a specific temperature, they can be experimentally determined according to International Standards (viscosity: ISO 3105, density: ISO 2811-3).

CAUTION — Particles, especially of organic or hydrocolloid matter, should not swell or shrink in chosen liquids due to the effects of solvation or osmotic pressure. In case of particles with open pores, liquids shall wet the pore material (contact angle > 90°) and the preparation time of dispersion shall allow fully filled pores. Furthermore, the liquid selected should not allow gelation or particle network formation.

7.2 Dispersing procedure

Disperse powders in the test liquids in accordance with the procedures specified in ISO 14887. A mild dispersing procedure is sufficient because, in contrast to other methods, e.g. particle sizing, any aggregates, agglomerates or flocks do not disturb the density measurement. Wet all particles thoroughly to avoid density underestimation due to adhering gas bubbles remaining in the test liquid.

The volume concentration of all samples should be the same and in accordance with the requirements of the measuring technique. In general, high concentrations should be avoided, as the sensitivity of migration detection will be reduced due to particle hindrance[20].

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If the original samples are provided as dispersion, a high volume concentration is desirable. These samples should be centrifuged and the supernatant should be discharged and replaced by the corresponding test liquid. This procedure should be repeated until the continuous phase of the original dispersion is exchanged.

With particles of a fractured nature or dense flocks, care should be taken to ensure the removal of all the included original liquid.

If the density and mass of the original continuous phase of the test sample are known, the liquid density can also be adjusted by adding defined amounts of density changing fluids or soluble substances.

Avoid bubbles attached to particles, which can give false low-density values, or bubbles in test liquids, which can interfere with particle migration.

Finally, the particle density measured is valid only for a batch of material if the test sample taken is representative of this batch.

8 Measurements

After dispersing particles into five to eight test liquids, fill and incubate corresponding measurement cells, e.g. in a water bath, to obtain the temperature specified for density values of test liquids. Then gently shake the cells (homogenization of dispersed phase; avoid air bubbles) and insert them into the measuring instrument (preconditioned to the same temperature). Measurement is terminated if migration direction is identified by monitoring concentration changes or if velocity is determined. Measurement time will depend on the measurement principle and necessary test liquids. Repeat this procedure for all five to eight dispersed samples.

In case the change of migration direction or change in velocity direction cannot be detected or takes place only in the sample of the lowest or highest liquid density, further determinations in adequate test fluids with broader density range shall be performed.

Gravitational migration velocity can be very slow in the case of submicron particles or even nanoparticles. In these cases, analytical centrifugation is advantageous to accelerate the evaluation of particle migration (ISO/TR 13097, ISO 13318-2). Multichannel instruments increase the throughput and allow for increased similarity in measurement conditions.

If instruments do not have temperature control, room temperature variations shall be prevented (< 1 K) and sample temperature should be measured precisely. Density data of test liquids employed shall correspond to measurement temperature. Nevertheless, such an approach without adjustable temperature control should be avoided, if possible, as the precision of density determination is decreased.

Annex A describes the determination of migration direction. Annex B provides more information on migration velocity determination for liquid and solid particles dispersed in solutions with different densities.

9 Data analysis

9.1 General

Measurements according to Clause 8 yield quantitative data describing the change of a particle migration velocity related measurand in arbitrary units v^* or particle migration velocity v depending on density ρ_L of a series of i different test liquids (continuous phases). As detailed in Annex C, experimental data v^* and v obtained for each test liquid shall be linearized by multiplying with the corresponding liquid viscosity η at measurement temperature to increase the precision of interpolation, especially for curves with low slopes.

NOTE In the following, the measurand in arbitrary units v^* or particle migration velocity v is named y.

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Examples of typical data are compiled in Table 1. The figures given have been obtained according to migration direction approach [Clause 6 a)], with backscattering intensity measured just above cell bottom for suspensions of polystyrene particles. The increase in backscattering intensity indicates an increase in particle concentration, i.e. sedimentation, and vice versa, creaming.

Table 1 — Exemplary data for density determination by detection of migration direction of particles dispersed in solutions of different sucrose mass fractions

Sucrose mass fraction	Liquid density	Liquid viscosity	Determined backscattering intensity	Backscattering intensity multiplied by viscosity
	ρ_L	η		
%	kg/m³	mPa·s	a.u.	a.u.·mPa·s
0	998,2	1,018	12,08	12,30
5	1 017,8	1,250	4,97	6,21
10	1 038,1	1,481	0,74	1,10
15	1 059,1	1,713	−0,99	−1,70
20	1 081,0	1,945	−1,82	−3,54
30	1 127,0	3,187	−2,72	−8,67
NOTE For further information, see Annex A.				

Particle density is determined by plotting changes in viscosity normalized backscattering (column 5 of Table 1) over the density of corresponding test liquids (column 2 of Table 1) as depicted in Figure 3 a). Particle density should be estimated based on this plot by interpolating data points nearest to the value "zero" of backscattering intensity multiplied by liquid viscosity (in this case for sucrose mass fractions of 10 % and 15 %). In general and mathematically speaking, interpolation can be done by drawing a straight line between these two measurement points or by any other fit-function including more measurement points.

9.2 Graphical interpolation

The density of dispersed particles, ρ_P, can be obtained graphically by drawing from the 0-value of the Y-axis a parallel line to the X-axis. The X-value of the crossing point equals the liquid density where particles do not move. By definition [see Formula (1)], this liquid density equals particle density ρ_P and, according to Figure 3 a), is approximately 1 047 kg/m³.

9.3 Two point interpolation

Particle density, ρ_P (corresponds to buoyant density, see 3.6), can also be calculated based on the coordinates of the two points (ρ_{L1}, y_1) and (ρ_{L2}, y_2) according to Formula (4), where index 1 corresponds to the lower test liquid density data and index 2 to the higher one.

$$\rho_P = \rho_{L1} - \frac{\rho_{L2} - \rho_{L1}}{y_2 - y_1} y_1 \tag{4}$$

Based on the data in Table 1, a density of 1 046,3 kg/m³ is calculated according to Formula (4), which is close to graphical determination but is the exact interpolation value.

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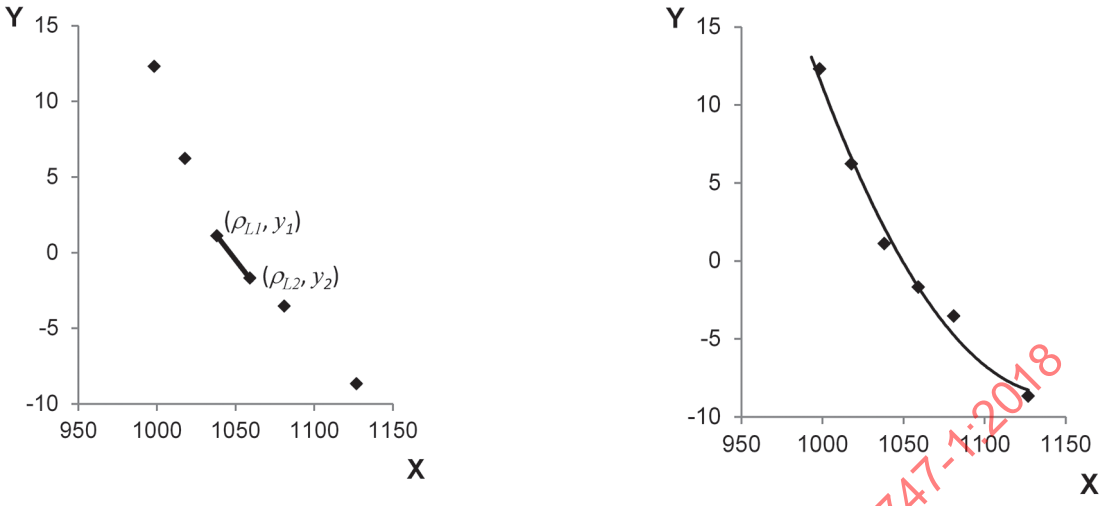

a) Linear interpolation[a] **b) Non-linear fit-function**[b]

Key

X liquid density ρ_L in kg/m^3

Y backscattering multiplied by viscosity in a.u. · mPa·s

a Data points of Table 1. Graphically linear interpolation between points (ρ_{L1}, y_1) and (ρ_{L2}, y_2).

b Data points of Table 1 fitted to non-linear fit-function.

Figure 3 — Linear interpolation and non-linear fit-function

9.4 Linear or non-linear fit

Linear or non-linear fit-function including more measurement points can be applied to analyse experimental values. In this case, we obtain a general equation such as Formula (5):

$$y = f(\rho_L) \tag{5}$$

Density obtained by regression line of an appropriate chosen quadratic non-linear fit-function corresponds to $y = 0{,}000\,9\rho_L^2 - 2{,}163\,1\rho_L + 1\,229{,}3$ [Figure 3 b)]. Particle density is obtained by setting $y = 0$ and calculating ρ_L, which equals particle density. It reads $\rho_P = 1049{,}0$ kg/m^3.

For practical reasons, it is advantageous to plot the data inversely, i.e. liquid density versus particle velocity measurand multiplied by liquid viscosity (Figure 4), and to perform regression according to Formula (6):

$$\rho_L = f(y \cdot \eta) \tag{6}$$

By this transformation, particle density equals the absolute term of Formula (6) for any chosen fit-function. Data from Table 1 [Figure 3 b)] result in $\rho_L = (0{,}238\,4y^2 - 6{,}999\,1y + 1\,049{,}0)$ kg/m^3 and a density of $\rho_P = 1\,049{,}0$ kg/m^3 is obtained setting $y = 0$.

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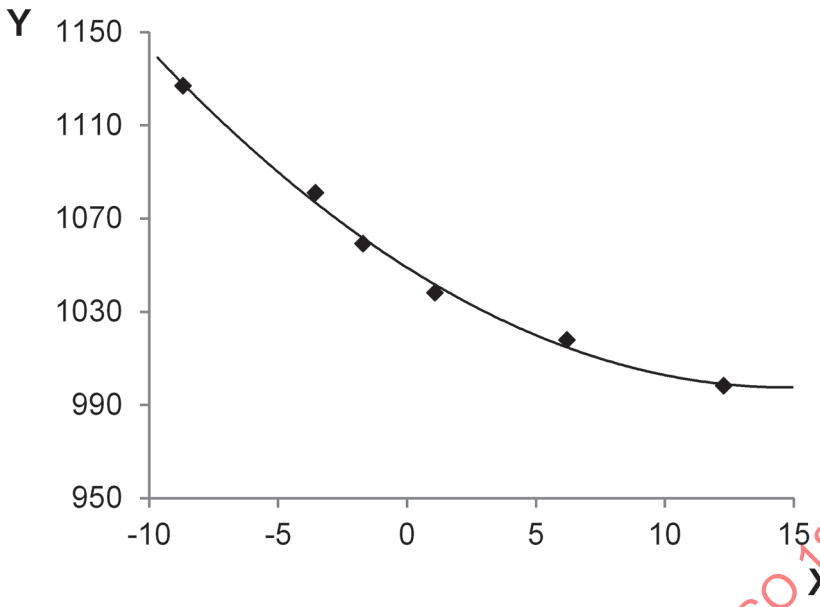

Key

X backscattering multiplied by viscosity in a.u. · mPa·s

Y liquid density ρ_L in kg/m^3

NOTE Data used is the same as in Figure 3 b).

Figure 4 — Plot of liquid density versus normalized velocity data fitted to an arbitrary quadratic polynomial

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10 Reference materials and measurement uncertainty

10.1 Reference materials

Particles of any shape may serve as reference material if density and its uncertainty are well known or shall be determined by an independent technique. Certified reference particles are advantageous. Density uncertainty shall be smaller than accuracy demands of particle density, which should be analysed. Any particle density distribution should be avoided. Reference particles shall be specified regarding useable test fluids and temperature of use. They should be easy to disperse into test fluids but shall be inert against those test liquids (no swelling, shrinking or dissolution). Strong flocculation, agglomeration and aggregation should be avoided (refer to ISO 14887). Reference particles without pores are also advantageous in this regard.

10.2 Measurement uncertainty

Calculate the precision and accuracy of analytical instruments in accordance with operational and performance validation of used measurement techniques recommended by the manufacturer.

The uncertainty of ρ_P depends on the uncertainty of velocity determination, the distance of interpolation points and the accuracy of liquid density and viscosity data.

Particle density is estimated according to Formula (4). Based on uncertainty propagation rules, the uncertainty of particle density can be calculated based on absolute, respective relative uncertainties of experimental quantities on the right side of the equation.

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The propagation of uncertainty according to Formula (4) is given by Formula (7):

$$\Delta\rho_P = \sqrt{\left(\frac{y_2}{y_2 - y_1}\right)^2 \cdot \Delta\rho_{L1}{}^2 + \left(\frac{y_1}{y_1 - y_2}\right)^2 \cdot \Delta\rho_{L2}{}^2 + \left(\frac{y_2 \cdot (\rho_{L1} - \rho_{L2})}{(y_2 - y_1)^2}\right)^2 \cdot \Delta y_1{}^2 + \left(\frac{y_1 \cdot (\rho_{L2} - \rho_{L1})}{(y_2 - y_1)^2}\right)^2 \cdot \Delta y_2{}^2} \quad (7)$$

where

$\Delta\rho_P$ is the absolute uncertainty of the particle density;

y_1 is the value of the migration for in solution 1;

y_2 is the value of the migration for in solution 2;

$\Delta\rho_{L1}$ is the absolute uncertainty of the liquid density of solution 1;

$\Delta\rho_{L2}$ the absolute uncertainty of the liquid density of solution 2;

ρ_{L1} is the liquid density of solution 1;

ρ_{L2} is the liquid density of solution 2.

The derivation of uncertainty propagation and some exemplary data are given in Annex E.

The uncertainty of particle density in the case of linear or non-linear regression [Formulae (5) and (6)] to interpolate the "zero velocity" can also be calculated based on slope and absolute constant by more advanced regression models[26].

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Annex A
(informative)

Discrimination between sedimentation or creaming/flotation by bottom focused backscattering intensity

Reference [22] illustrates the isopycnic velocity approach. Polystyrene (PS) particles were dispersed in accordance with Clause 7 in continuous aqueous phases of different sucrose mass fractions (0 % to 40 %). These suspensions were analysed by a bottom focused light backscattering (BFLB) technique (LUMiCheck®[2)]). The measuring principle is described in Reference [22]. Monochromatic light illuminates a small region of interest near the cell bottom (incident wavelength 870 nm) and backscattering intensity by dispersed particles is monitored over time. Readings are given in arbitrary units. Any onset of particle migration due to gravity increases (sedimentation) or decreases (creaming, flotation) the number of scattering particles and correspondingly results in an increase or decrease of backscattering intensity, respectively. Therefore, the direction of particle movement can be easily detected.

Figure A.1 shows the change of the backscattering intensity over time for the polystyrene (PS) particles dispersed in different sucrose solutions. The start value for each sample is set to zero (normalized backscattering intensity). PS particles suspended in sucrose solutions of 0 % to 8 % exhibit an increase in light intensity over time, indicating sedimentation (an increase in the number of scattering objects). For these solutions, particle density is larger than sucrose solution density ($\rho_P - \rho_L > 0$) and migration velocity is $v > 0$. For higher sucrose solutions (14 % to 40 % mass fraction), particle density is smaller than the density of the sucrose solution ($\rho_P - \rho_L < 0$) and particles float. The decrease in particle number above the bottom causes a decline in backscattering intensity. The density of PS particles can be determined by comparing backscattering intensity at a given separation time. Five arbitrarily selected times are indicated in Figure A.1 by vertical arrows. Intersection points between time arrow and change in backscattering intensity due to particle migration for each sucrose mass fraction are plotted versus the density of the corresponding sucrose mass fraction (Figure A.2). By linear interpolation, the density of the sucrose solution can be obtained. It corresponds to the zero migration velocity and equals the density of analysed particles. Particle density does not depend on the arbitrarily chosen time for evaluation.

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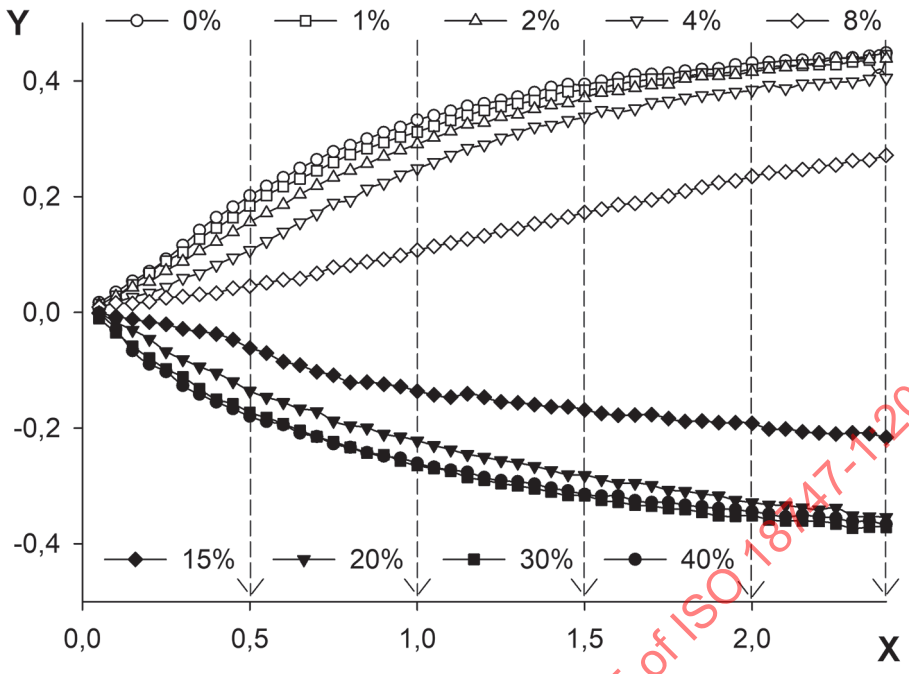

Key

X time t in min

Y normalized backscattering in a.u.

↓ selected time intervals for density calculation

Figure A.1 — Time course of the bottom focused backscattering intensity of polystyrene particles dispersed in different sucrose solutions (0 % to 40 % mass fraction)

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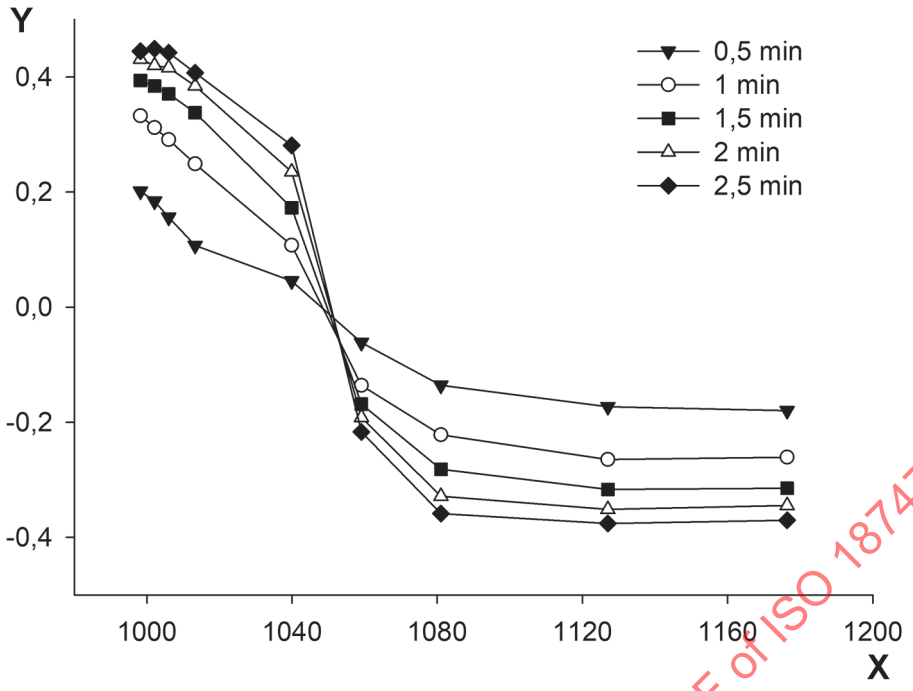

Key

X density ρ in kg/m^3

Y normalized backscattering in a.u.

NOTE Separation times correspond to the vertical arrows in Figure A.1.

Figure A.2 — Backscattering intensity monitoring PS-particle migration for selected separation times plotted against the density of corresponding continuous phase (sucrose solutions)

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Annex B
(informative)

Determination based on measurement of direct migration velocity

B.1 General

Annex B details some applications with respect to in-situ particle density determination as described in this document. In general, any measuring techniques allowing for, at least, the determination of the mean velocity of particles dispersed in appropriate liquids of the required density range may be employed. Chemical homogeneity regarding the density of particles to be probed can be investigated if the technique used allows analysing particle velocity distribution[17][25].

B.2 Density of poly(methyl methacrylate)-particles (PMMA particles)

Dry particle powder (SediTest™[3]) was supplied by Dr. Lerche KG, Germany.

PMMA particles were specified as follows:

— spherical monodisperse particles of sizes 15 µm, 19 µm, 29 µm and 38 µm;

— density: 1 200 kg/m^3;

— refractive index: 1,483.

Dry particles were dispersed by vortexing (1 min) in a series of heavy liquid solutions (sodium polytungstate, TC-Tungsten Compounds GmbH, Germany) having different densities above and below the expected density of the particles. Densities in the range from about ρ = 1 100 kg/m^3 to ρ = 1 300 kg/m^3 were prepared by gravimetric dilution of sodium polytungstate stock solution (85,77 % mass fraction; ρ = 3 076 kg/m^3) with distilled water. The sedimentation/creaming velocities of the particles in the suspensions were analysed by analytical cuvette separation using LUMiReader®[4]. Samples were filled in 10 mm polycarbonate cuvettes, sedimentation/creaming was performed at normal gravity at ϑ = 30 °C, transmission profiles were recorded every 20 s at 870 nm wavelength (Figure B.1). Particle velocity distribution and its harmonic mean were calculated by SEPView®[4] 6.4 software using the following settings:

— analysis mode: constant position (all profiles);

— analysis positions: 3;

— range: 3 mm;

— data analysis regions/nodes: 38,0 mm, 41,5 mm, 45,0 mm (sedimentation), 34,0 mm, 37,0 mm, 40,0 mm (creaming) (Figure B.2).

Normalized particle velocities were plotted as a function of the density of the continuous phase (Figure B.3). The effective particle density was determined separately for each particle size class from the particle velocity data by interpolating the liquid density between the sedimentation and creaming

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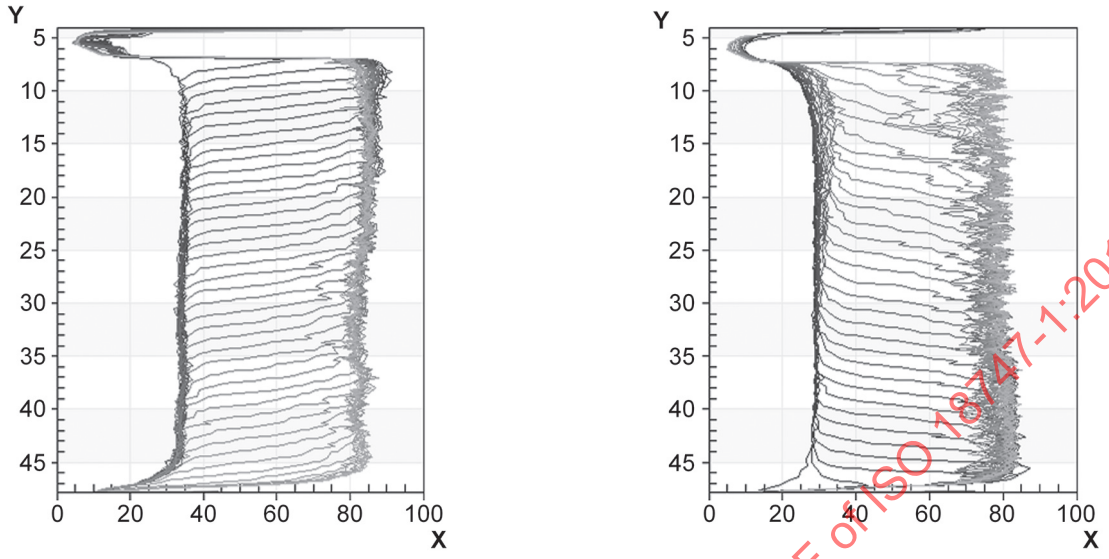
velocities closest to the floating point (zero velocity) of the particles. Densities are virtually not depending on size as mentioned in Clause 5. Mean value reads 1 201,5 kg/m^3, which corresponds to data from the manufacturer (ρ_P = 1 200 kg/m^3).



a) Settling **b) Rising**

Key

X transmission in %

Y position in mm

NOTE 1 Sodium polytungstate solutions with a particle mass fraction of 0,2 % were used.

NOTE 2 Results were recorded with LUMiReader® PSA at ϑ = 30 °C, λ = 870 nm, 10 mm PC cells.

NOTE 3 Figure B.1 a) shows particle sedimentation (ρ_L = 1 107 kg/m^3) and Figure B.1 b) shows particle creaming (ρ_L = 1 300 kg/m^3).

Figure B.1 — Space and time resolved transmission profiles of gravity migration of 15 µm PMMA particles in suspensions with different densities of the continuous liquid phase

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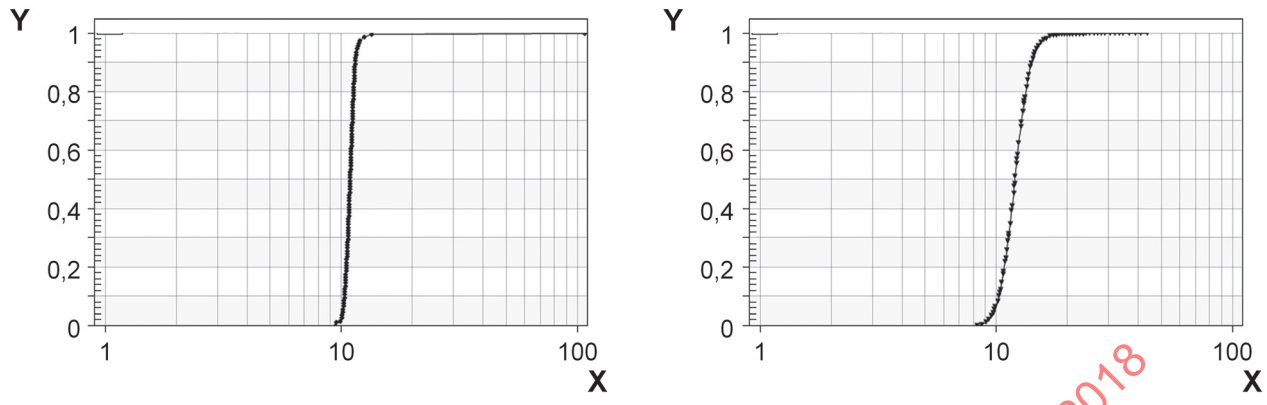

a) Settling **b) Rising**

Key

X sedimentation/creaming velocity v in µm/s

Y cumulative function (extinction-weighted) of the velocity distribution

NOTE 1 Sodium polytungstate solutions with a particle mass fraction of 0,2 % were used.

NOTE 2 Results were calculated from data shown in Figure B.1 using SepView® 6.4 software (settings as described above).

NOTE 3 In Figure B.2 a), liquid density ρ_L = 1 107 kg/m^3 and in Figure B.2 b), liquid density ρ_L = 1 300 kg/m^3.

Figure B.2 — Velocity distribution of 15 µm PMMA particles in suspensions with different densities of the continuous liquid phase

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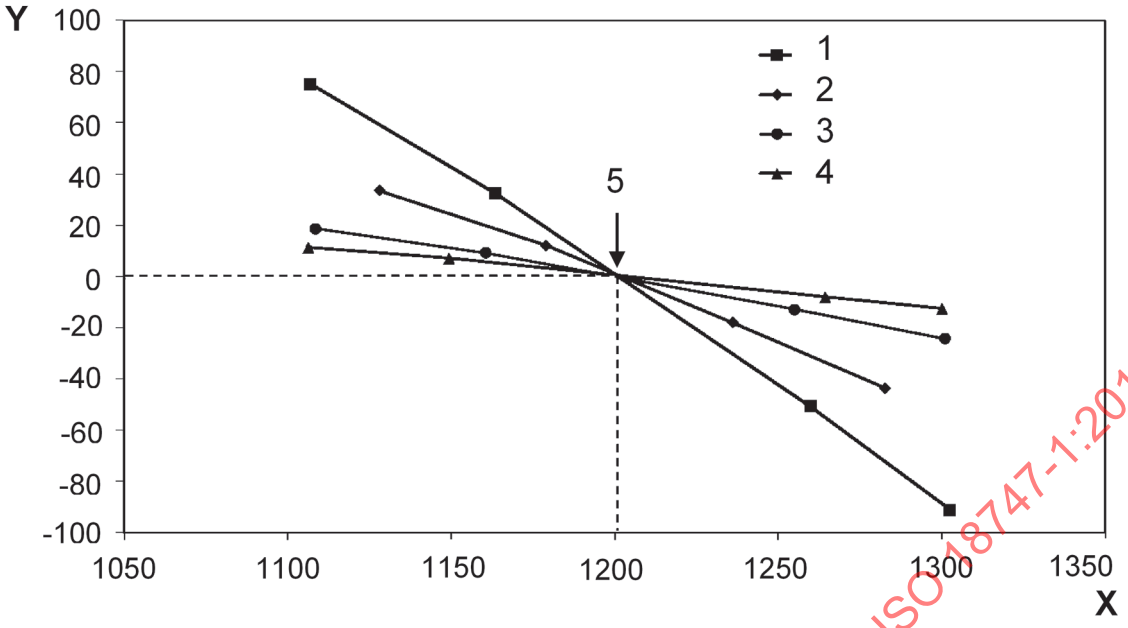

Key

X liquid density ρ_L in kg/m^3

Y particle velocity y multiplied by viscosity η in µm·mPa

1 PMMA particles 40 µm

2 PMMA particles 30 µm

3 PMMA particles 20 µm

4 PMMA particles 15 µm

5 ρ_P = 1 202 kg/m^3

NOTE 1 Solutions of different polytungstate mass fractions with a particle mass fraction of 0,2 % to 0,8 % were used at ϑ = 30 °C, λ = 870 nm, 10 mm PC cells.

NOTE 2 LUMiReader® PSA was used.

NOTE 3 For further information, see Reference [21].

Figure B.3 — Gravity sedimentation velocity of monodisperse PMMA particles of different sizes in dependence on different densities of continuous phase

Table B.1 — Data for calculation of effective densities of PMMA particles of different sizes by linear interpolation to the zero velocity of the particles according to Formula (4)

Particle size	Fluid density	Normalized particle velocity*	Fluid density	Normalized particle velocity*	Particle density
	ρ_{L1}	γ_1	ρ_{L2}	γ_2	ρ_P
µm	kg/m^3	µm·mPa	kg/m^3	µm·mPa	kg/m^3
15	1 150	7,025	1 265	−8,144	1 203
20	1 161	9,229	1 255	−12,928	1 200
30	1 179	11,852	1 236	−18,226	1 202
40	1 164	32,355	1 260	−50,767	1 201
				mean density of all particle size classes:	**1 201,5**

* Particle velocities were determined in duplicate; the mean was used for calculation of normalized particle velocity.

NOTE For interpolation, values shown in Figure B.3, which are the closest to the "zero velocity" point of the particles, were used.

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B.3 Density of birch oil particles (droplets)

Density determination of liquid particles (droplets) can be performed in an identical way as for solid PMMA particles. In this subclause, liquid particles will be referred to as "droplets", which the commonly used term.

A stabilized emulsion of birch oil droplets in water (o/w) was provided by Karlsruhe Institute of Technology (Germany). The emulsion consists of 2 % mass fraction of birch oil (Sigma-Aldrich GmbH, Germany) in water with 1 % mass fraction of nonionic surfactants (Lutensol®[5] TO 10) as emulsifier and was prepared using a conical colloid mill (IKA magic LAB®[6]) having a rotor diameter of 2,8 cm at speeds of n = 5 000 min^{-1} and n = 10 000 min^{-1} each for 5 min. The density of the continuous aqueous phase of the birch oil emulsion was varied by the addition of solid sucrose in defined amounts. The resulting density of the continuous phase was calculated based on the amount of sucrose added. The stability of the emulsion after adding sucrose was checked in advance and was found to be unaffected regarding droplet size. In this way, four different samples of the emulsion were prepared, varying in the density of the continuous aqueous phase in the range of 1 050 kg/m^3 to 1 220 kg/m^3 close to the expected density of the birch oil droplets. The migration velocities (sedimentation/creaming) of oil droplets in collected samples were analysed by analytical cuvette centrifugation using LUMiSizer®[7]. The samples were filled in 2 mm glass cuvettes, sedimentation/creaming was performed at n = 3 000 min^{-1} at ϑ = 25 °C, transmission profiles were recorded every 10 s at 865 nm wavelength (Figure B.4). Droplet velocity distribution and its harmonic mean were calculated by SEPView® 6.4 software using the following settings:

— analysis mode: constant position (all profiles);

— analysis positions: 3;

— range: 1 mm;

— data analysis regions/nodes: 118 mm, 119 mm, 120 mm (sedimentation) 120,5 mm, 121,5 mm, 122,5 mm (creaming) (Figure B.5).

Normalized droplet velocities were plotted as a function of the density of the continuous phase (Figure B.6). Calculation of the density of birch oil droplets was done by interpolation of the obtained linear regression function to the density of the continuous phase at the droplet zero velocity. The density of birch oil droplets amounts to 1 139,6 kg/m^3.

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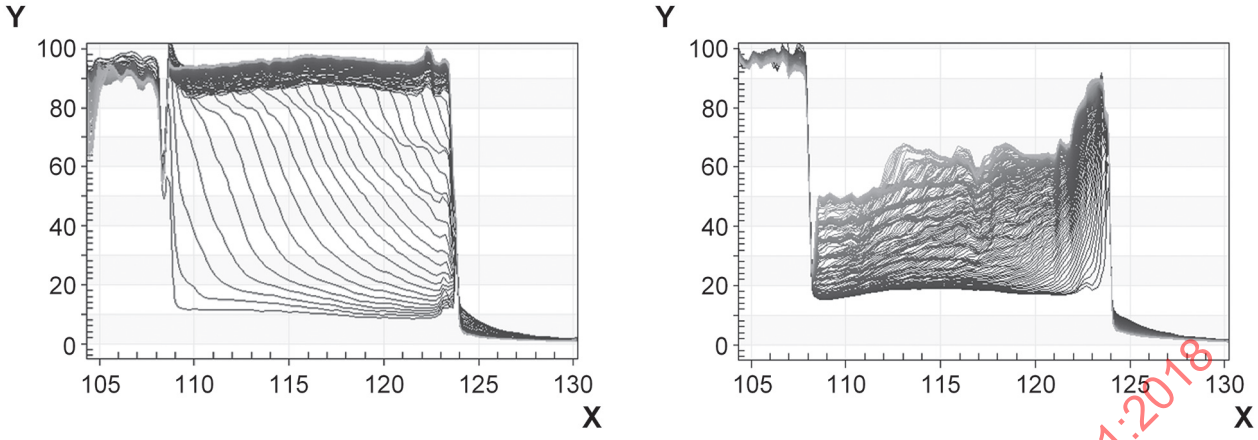


a) Settling **b) Rising**

Key

X position in mm

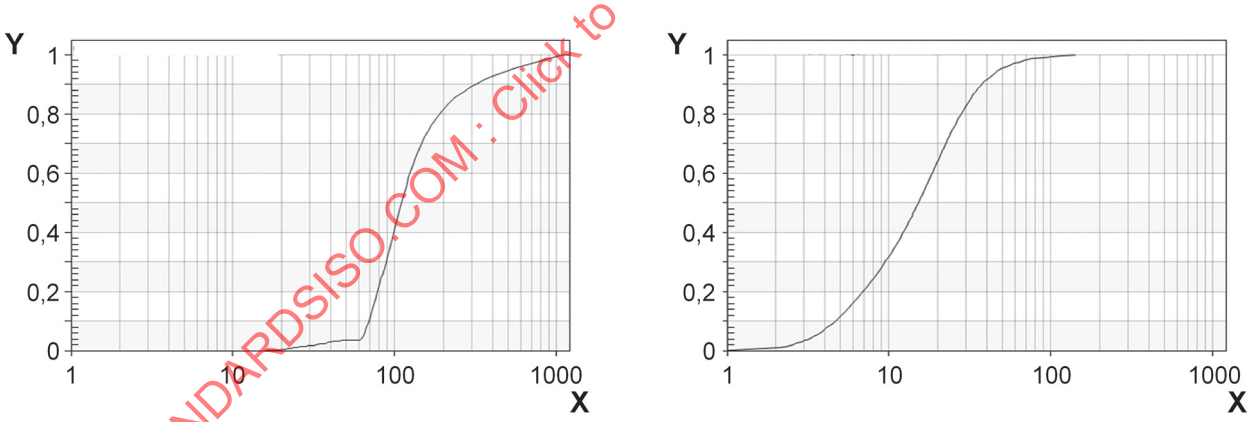
Y transmission in %

NOTE 1 Emulsions of birch oil droplets in water (o/w) with a mass fraction of 2 % were used.

NOTE 2 Results were recorded with LUMiSizer® at n = 3 000 min^{-1}, ϑ = 25 °C, λ = 865 nm, 2 mm glass cells.

NOTE 3 In Figure B.4 a), liquid density ρ_L = 1 050 kg/m^3 and in Figure B.4 b), liquid density ρ_L = 1 220 kg/m^3.

Figure B.4 — Space and time resolved transmission profiles of analytical cuvette centrifugation of birch oil droplets in emulsions with different densities of continuous liquid phase (sucrose solutions)



a) Settling **b) Rising**

Key

X sedimentation/creaming velocity v in µm/s

Y cumulative function (extinction-weighted) of the velocity distribution

NOTE 1 Emulsions of birch oil droplets in water (o/w) with a mass fraction of 2 % were used.

NOTE 2 Results were calculated from data of Figure B.4 using SepView® 6.4 software (settings as described above).

NOTE 3 In Figure B.5 a), liquid density ρ_L = 1 050 kg/m^3 and in Figure B.5 b), liquid density ρ_L = 1 220 kg/m^3.

Figure B.5 — Velocity distribution of birch oil droplets in emulsions (o/w) with different densities of the continuous liquid phase (sucrose solutions)

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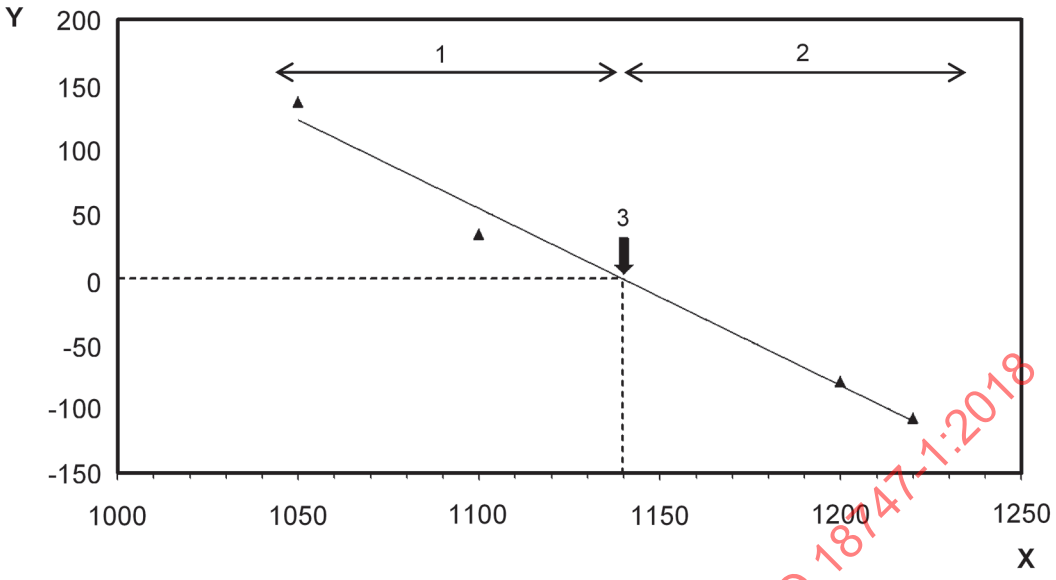

Key

X liquid density ρ_L in kg/m^3

Y particle velocity y multiplied by viscosity η in µm·mPa

1 sedimentation

2 creaming

3 droplet density, interpolation $y = 0$, $\rho_P = 1\ 139{,}6$ kg/m^3

NOTE Accelerated migration velocity was determined at $\vartheta = 25$ °C by cuvette analytical centrifugation (ISO 13318-2, LUMiSizer®, 2 mm optical glass-cells[21]).

Figure B.6 — Sedimentation and creaming of birch oil droplets

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Annex C
(informative)

Linearization of migration velocity versus density plots

If weight force and buoyancy force are not equal [Formula (1)], particles will sink or float in gravitational or in centrifugal fields with migration velocity v according to Stokes law [Formula (C.1)].

$$v = \frac{(\rho_P - \rho_L) \cdot x^2}{18 \cdot \eta} \cdot g \tag{C.1}$$

where

$\rho_P - \rho_L$ is the density difference;

x is the equivalent particle diameter for settling/creaming velocity;

g is the acceleration;

η is the viscosity of the test liquid.

Independent of limitations, it becomes evident that velocity depends linearly on the density difference between particles ρ_P and continuous phase ρ_L but inversely on the viscosity, η, of continuous phase. Due to different dependencies of the test liquid density (even more so of the density difference $\rho_P - \rho_L$) and liquid viscosity on solute concentration (Figure 2), migration velocity depends non-linearly on solute concentration. Based on this, velocity changes with increasing density of test liquid recorded by a linear sensor system are always more pronounced at concentrations where liquid density is lower than particle density and vice versa. Figure C.1 (left) demonstrates this for calculated velocities [Formula (C.1)] for spherical particles of different sizes and a density of 1200 kg/m^3 dispersed in sucrose solutions of mass fractions from 24 % ≙ 1 099 kg/m^3 to 62 % ≙ 1 298,3 kg/m^3 at ϑ = 20 °C. Data may be linearized by multiplying particle velocity at a given solute mass fraction with the corresponding solute viscosity. Correspondingly, the uncertainty of interpolated "zero" velocity can be reduced, especially if slopes are small as shown in Figure C.1 (right).

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