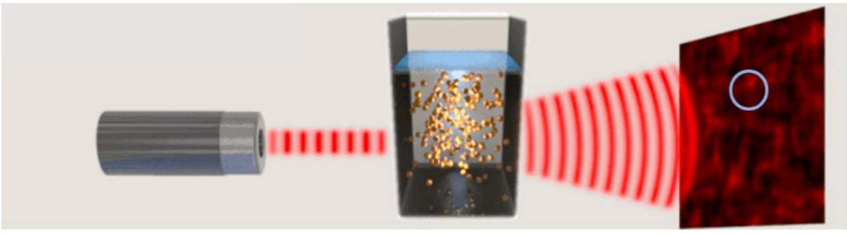
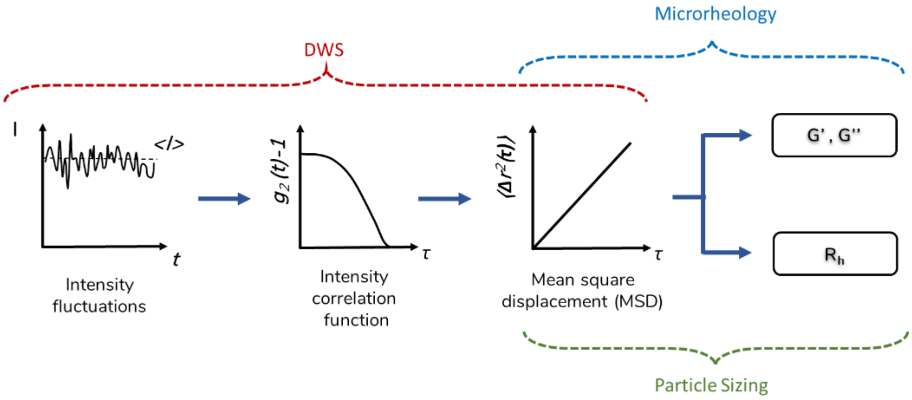
An Introduction to the DWS RheoLab

Webinar Q&A Transcript

1 Can you briefly summarize the principle of DWS Microrheology?

The DWS RheoLab measures the Brownian motion of particles in the sample and then computes the Mean-Squared Displacement. From there, one can already obtain information on the viscoelasticity and presence of a microstructure in the sample. Then, DWS Microrheology analysis can be performed to obtain the storage and loss moduli versus the frequency, as obtained via a mechanical rheometer. One can also perform particle sizing in purely viscous samples.
More detailed information can be found under the technology section on our website.





2 What is the frequency range of the RheoLab?

0.5 rad/s to 10^5 rad/s (with an option to extend it to 10^6 rad/s). The actual accessible range is sample dependent, although the sensitivity range can be tuned over a wide range by selecting an appropriate cell thickness L or tracer particle concentration.

3 What is the temperature range of the RheoLab?

4°C to 100°C, with stability better than 0.02°C. We offer an extended temperature range up to 180°C as an option.

4 What is the laser wavelength/power in the DWS RheoLab? Can it be changed?

The laser has a wavelength of 685nm and a power of 45mW.

The laser power and optical design is chosen such that it guarantees a high signal quality. It is therefore not necessary to adjust the laser power. For custom requests related to strong sample absorption at 685nm, please contact sales@instruments.ch

5 Is the laser beam safe for samples in it?

Yes, this is not an issue. The sample cell is in thermal contact with the metallic cell holder and the beam is expanded to about 8mm (diameter). The thermal heating induced by laser light absorption is not more than a fraction of a degree (°C).

6 Can the measurements be performed in reflection mode?

Yes, we have a backscattering mode available.

7 Are raw data, available for alternative data evaluation (open data access)?

All raw data can be exported in .csv files which can be directly be opened in Excel.

8 Is the software correcting for the refractive index of the solution?

The solvent or solvent refractive index can be entered manually. Several popular solvents are pre-defined, and the user can also define a custom solvent. We note that the influence of the solvent refractive index is relatively weak (a change from 1.33 to 1.4 would change the results by about 5%).

9 Can this technique be used for studying hydrogels?

DWS is well suited to study hydrogels.

See the below reference for more information:

Brushlike interactions between thermoresponsive microgel particles

10 Can this technique be used for emulsions?

Common emulsions are straightforward to be characterized with the RheoLab.
See the below references for more information:
Diffusing wave microrheology of highly scattering concentrated monodisperse emulsions.
Diffusing wave spectroscopy (DWS) methods applied to double emulsions.
Characterization of Mayonnaise

11 If my emulsion is stabilized by particles, can it be characterized by DWS?

Pickering emulsions are straightforward to be characterized with DWS as long as the droplets are below about 10 micrometer in diameter. The particles on the surface and the droplet itself will act as one particle.

12 Can we predict the morphology of the internal microscopic structure from the DWS data? Network of cross-linked polymers, for example.

The microstructure of the sample can be studied by DWS in a perfectly analogue way as in mechanical rheology – with the usual advantages of DWS in terms of speed, sample volume and frequency range.

13 Can the DWS RheoLab be used to characterise dark opaque colloidal solutions in organic solvents?

Dark and colored turbid samples can be measured provided the scattering strength ($\mu^*=1/l^*$) is still much stronger than absorption, expressed by the absorption coefficient $\mu_a=1/l_a$. A discussion about the application of the DWS RheoLab on colored colloidal solutions can be found in the article cited below.

Improved diffusing wave spectroscopy based on the automatized determination of the optical transport and absorption mean free path

14 Is it possible study acid-induced gelation e.g. while changing pH?

In general, the DWS RheoLab will be perfectly suited for this task as is. Acidified milk has been measured using DWS in the past (see the references below), and even better measurements are possible these days with the latest technology developments implemented in the DWS RheoLab.
Titrating the pH with a dosing system is not possible with the DWS RheoLab. However, given the fact that measurements are carried out in sealed cuvettes one might prepare different samples at different pHs and measure them alternatively as the time elapses.

Aggregation and Gel Formation in Biopolymer Solutions
Characterization of the Gelling Process in Acidifying Milk

15 Are there samples which are challenging to characterize with DWS Microrheology? Can you shed some light on it?

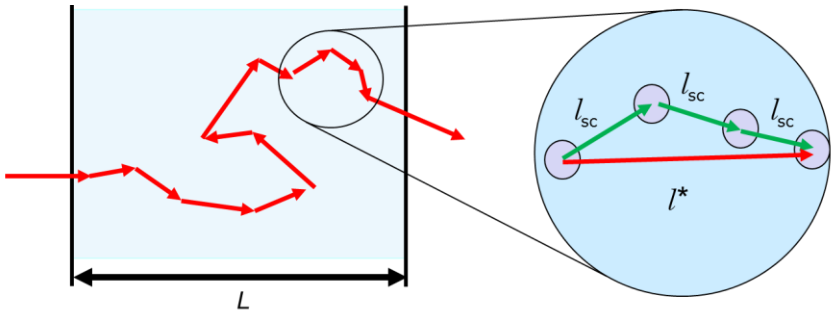
The DWS Microrheology approach is based on the assumption that the light reaching the detector has been scattered multiple times, and that samples under study are homogeneous on length scales smaller than the (tracer) particle size. Samples with large spatial heterogeneities or samples of low turbidity that are difficult to mix with tracer particles may be difficult to measure.

16 Do I need to add tracer particles?

Tracer particles can either be naturally present in a sample (e.g. droplets in an emulsion, particles in a suspension) or are added during the sample preparation for samples that are transparent or only slightly turbid.

17 How do I know if my sample is turbid enough to be characterized in its natural state?

In DWS we refer to the transport mean free path l^*, which is the mean distance the photon travels in the sample before its propagation direction is randomized.
The figure depicts a diffuse transmission through a turbid sample and illustrates the photon transport as a random walk with step size equal to l^*. The mean free scattering path l_sc is the average distance travelled by a photon before being scattered by a particle



Small values of l^* correspond to samples with high turbidity and, conversely, large values of l^* correspond to samples with low turbidity. In order to fulfill the assumptions of DWS, the ratio of the optical cuvette thickness L to l^*, must fulfill the condition:

$$5 < \frac{L}{l^*} < 40.$$

18 Do I need to determine the L/l* of my sample?

No! The LsLab software does this automatically once a measurement is started. The value of L/l* will be displayed and if outside of the working range, the user will be notified.

19 The LsLab software determines parameters automatically but our samples are dilute. How can we skip the L/l* check, and measure our sample anyway?

The LsLab software allows you to override the automatic settings and enter values for L/l* manually. Please bear in mind that the accuracy of your measurement depends on the values entered.

20 Is there a limit on sample absorbance or turbidity?

With our backscattering option, one can characterize samples that are too opaque to work in transmission mode. Furthermore, in transmission mode, very thin cuvettes can be used with optical path as low as 1 mm. It is very uncommon that samples are too turbid to be measured with the provided cuvettes.
If required, detachable cells can be ordered with path lengths down to L=0.01mm and thus there is no practical limit on the turbidity.

21 How do you cope with size polydispersity in the sample, such as would be the case for drops in an emulsions? Would you advise adding tracers in that case?

You do not need to add tracers unless the natural turbidity is very low ($L/l^* \ll 1$). The DWS RheoLab will be sensitive to the average size. For a monomodal size distribution, up to a polydispersity of 30-50%, DWS Microrheology is only weakly affected by polydispersity. The main consequence will be that frequency dependent features in $G^*(\omega)$ will be a little bit smeared out.

22 Would we face any issue with trying to characterize the viscoelasticity of an opaque dairy like solution with a very broad particle sizing?

Absolutely not, this is a typical system where a wealth of information can be deducted from the MSD without the need of knowing the precise particle size distribution.

23 What are the minimum requirements for the scattering particles?

Typically, the minimum size is 100-200 nm (diameter) and the minimum concentration 0.1-0.5 % (in volume). The refractive index contrast to the sample should be at least 0.1.

24 Do the particles need to be evenly distributed?

Yes, for accurate probing of your sample properties, the tracers (natural or added) should be well dispersed.

25 How can I determine the particle size in a naturally white sample?

We note that the particle mean-square-displacement measured by the DWS RheoLab does NOT depend on whether we know the particle size. If the particle size is unknown, this data can be used directly.

Providing that the sample is purely viscous, one can perform a DWS sizing experiment or a DLS measurement using the NanoLab 3D or the LS Spectrometer.

26 Is the particle sizing still effective in a partially arrested system like a gel?

The DWS RheoLab performs particle sizing in purely viscous samples. If the particle size must be used for microrheology analysis purposes, we recommend a measurement on a dilute sample to obtain this information from DWS sizing.

In a viscoelastic system, it is still possible to manually analyze the mean free path l*, provided automatically by the instrument, and use this information to determine the mean particle size or particle size evolution.

27 If two kinds of particles with different size were put in the system, can the MSD data display the movement of each one?

For this specific measurement we recommend using our LS Spectrometer or NanoLab. Due to the multiple light scattering principle, the DWS RheoLab does not allow to disentangle the MSD data for multi-modal size distributions of particles.

28 What is the rule of thumb for the tracer size relative to the droplet size in the sample? As close as possible?

Note that droplets can act as tracer particles when studying emulsions.

If tracer particles must be added, the rule of thumb for the DWS microrheology analysis would be that the tracer particles are comparable or larger in size than the droplets.

Moreover, when working with tracer particles in fairly turbid systems, we recommend that the tracer turbidity (or optical density) 1/l* is at least an order of magnitude higher than the sample turbidity. If this cannot be reached, it will be better not to add tracers.

29 Which of your examples could be analysed through a DLS measurement with the Nanolab 3D?

The NanoLab 3D can measure very turbid samples, but not as turbid as the DWS RheoLab. Furthermore, the NanoLab can only measure liquid samples, hence not gels. DLS microrheology allows to obtain information on viscoelastic liquids, but not viscoelastic solids.

30 If the droplet size is not within the range of 0.1 um to 1 um, are DWS measurements possible?

The accuracy of a size measurement within the specified range is +/- 5%. Outside of the specified range, the accuracy of the measurement will be lower.

The upper size range is 5-10um as long as strong sedimentation is not an issue. The lower size limit is between 0.05.2mu (diameter) depending on the particle refractive index (the higher the smaller).

31 How does the size of tracer particles influence the measurement?

For microrheology, smaller particles are subject to comparatively larger thermal agitation and thus it is possible to measure larger elastic moduli. Larger particles provide a better separation of length scales between the sample and the tracer. We recommend a particle size around 0.3-0.8 micrometer in diameter.

32 Is it possible to study a sample under deformation?

The DWS RheoLab measures the Brownian motion of particles in the sample and thus, no deformation is applied.

As an alternative, one can submit the sample to various conditions outside of the RheoLab and characterize the resulting changes once the cuvette is re-inserted.

33 Is it possible to study a sample under variable atmospheric pressure?

In principle this is possible but, at the moment, we do not offer a pressure cell for the DWS RheoLab. A custom development adapting an existing pressure cell could be considered upon request. Please contact sales@instruments.ch

As an alternative, one can submit the sample to various conditions outside of the RheoLab and characterize the resulting changes once the cuvette is re-inserted. Custom made cells can be inserted (at your own risk) into the RheoLab and we'll be happy to provide information about the beam position on the cell surface.

34 What happens when you work with a sample above its boiling temperature? Do the condensation drops that occur inside the cuvette influence the measurement?

If condensation droplets develop, they will not occupy the scattering volume, hence they will not influence the measurement. It is however not recommended to measure boiling samples due to possible damage to the instrument due to vapor condensation. The gas bubble motion would also strongly perturb the DWS measurement and therefore we advise against such an experiment.

35 Can I open the cuvette to make a measurement under solvent evaporation?

This is not recommended due to possible damage to the instrument due to vapor condensation but is feasible under some precautions. Please contact LS Instruments for more details.

36 What is the most common material for tracer particles?

We recommend TiO_2 and Polystyrene colloidal particles, available from LS Instruments. Please contact sales@instruments.ch. Sizes are around 0.3-0.5 micrometer. Other commercially available particles are suitable as well, for example Melamine. The requirements are that the refractive index is larger compared to the sample (at least 0.1 larger) and the size should be between 0.2-2 micrometer.

37 Are there alternative cuvette materials in case a sample interacts with the cuvette surface?

Yes, one can use plastic, glass or quartz cuvettes depending on the interactions of the sample with the surface.

38 Are the square capped glass cuvettes suited for temperatures higher than 100 degrees

Yes, as long as the pressure build-up within the cell doesn't create problem to the cell itself.

39 What is the beam size? Can narrow cuvettes be used?

The beam is intentionally expanded for best results and the diameter is about 8 mm. For custom solutions, please contact LS Instruments.

40 We would like to buy disposable cuvettes. Do you recommend a certain polymer? Polystyrene? PMMA?

Provided the cells fit the RheoLab there are little restrictions on the choice of the material, or the optical quality of the cell walls as long as they are 10 mm in with and not more than 10 mm in depth. Due to poorer heat conduction, it may take longer for the sample to reach thermal equilibrium.