BRUKER

TD-NMR FOOD ANALYSIS

minispec Droplet Size Analyzer 2.0 for food emulsions application note

New user interface, GoScan for minispec, and improved data analysis including the calculation of Multimodal droplet size distributions

Innovation with Integrity

Introduction

Droplet size distributions (DSDs) play a major role in the development of new products, influencing mouth feel, stability of emulsions, shelf life, microbial keeping properties, etc.

The droplet size distribution characteristics need to be regularly checked during production quality control, ensuring that the end-product will present the desired characteristics.

Examples of daily products where droplet size distributions are verified in both, Research & Development (R&D) and production are: mayonnaise, salad dressing and soft cheese containing oil droplets dispersed in a water phase; margarine, low fat spread and butter, containing water droplets dispersed in an oil phase.

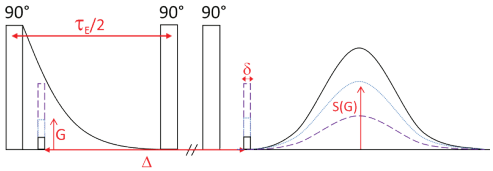
Unlike conventional techniques as light microscopy, laser diffraction, light scattering or electric sensing, Time Domain (TD) NMR requires minimum sample preparation and no dilution. Samples remain unaltered and opacity or presence of solid particles, do not disturb the measurements.

Food Market:

- Margarine
- Low fat spread
- Butter
- Other W/O emulsions
- Mayonnaise
- Salad dressing
- Soft cheese
- Other O/W emulsions

Application Method

The TD-NMR method is based on the physical laws of restricted diffusion of oil or water molecules in droplet particles. The pulse sequence is based on the stimulated echo with pulsed field gradients (PFG-STE), where the measurement is performed as function of the gradient (G) amplitude.



This pulse sequence can be used, with small modifications, for droplet size measurements, in either oil in water (O/W) and water in oil (W/O) emulsions. As this method relies on the measurement of the ^{1}H NMR signal as a function of gradients' amplitude, the application requires a relation between the signal attenuation S(G) and the molecular diffusivity of the liquid that is confined in the droplet.

The size of the droplets can be calculated based on the geometrically restricted diffusion of the dispersed phase's molecules inside the droplets, as first described by Murday and Cotts [3].

The resulting datasets include the information of the DSDs. Parameters of distribution functions are automatically determined to the user choice between Lognormal and Multimodal analysis mode.

Advantages of the new Droplet Size Analyzer 2.0 in comparison with previous versions

- New GoScan for minispec GUI.
- Two different analysis modes: Lognormal and Multimodal.
- Calculation of the $d_{3,2}$ parameter for the Lognormal analysis mode.
- While an acquisition is running, it is possible to check the display of the results and re-analyze other data without disturbing the measurement in progress.
- The graphical display of the results is always available together with new interactive tools.
- Calculation of Number DSDs in the Lognormal analysis mode.
- PDF reports available for single results or batch measurements.

Analysis Examples

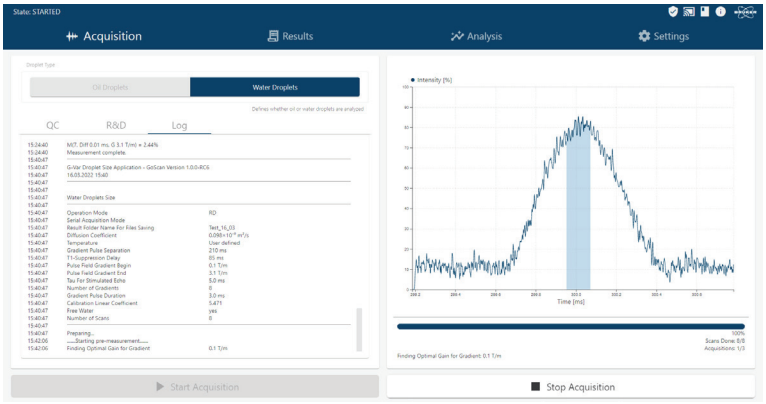


Figure 1 GoScan for minispec acquisition window

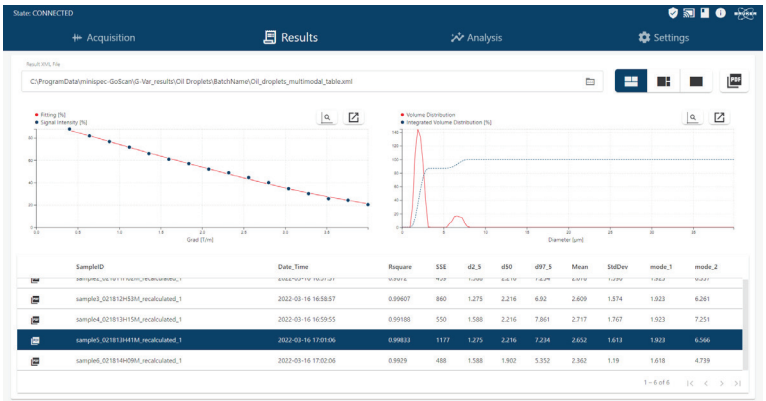


Figure 2 GoScan for minispec results window - Multimodal distribution

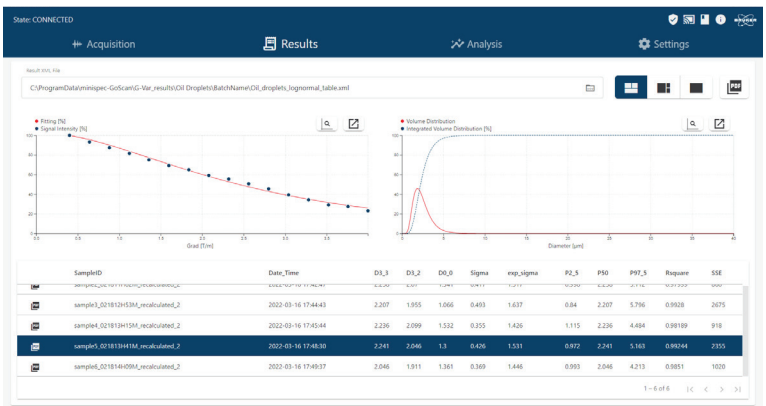
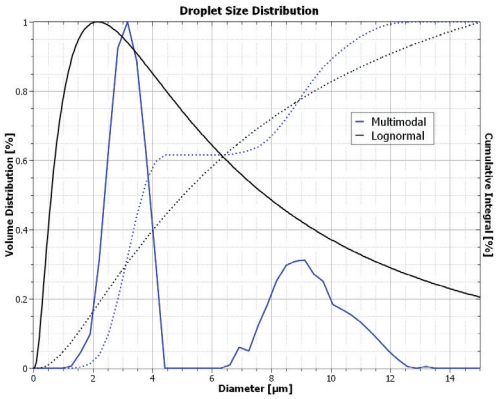


Figure 3 GoScan for minispec results window - Lognormal distribution

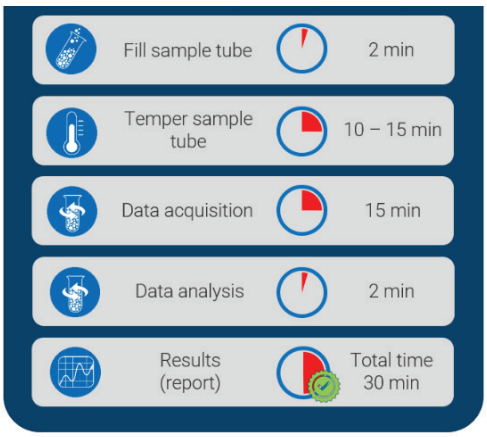
Data Analysis and Fittings

Usually, monomodal DSDs are assumed during data processing[4] and some of the experimentally observed signal decays can be described by this approach[1]. However, production processes and material instabilities lead to DSDs that cannot be described by a monomodal log-normal distribution. Also, the assumption of a log-normal distribution for the DSD of an emulsion is debatable as it is a statistical but not an experimental distribution.

To account for this, the minispec system is offering, on the GoScan for minispec GUI, a multimodal processing tool based on regularization methods, as proposed by Hollingsworth and Johns[2]. When choosing for the multimodal processing option the user is presented with the observed (measured) DSD without any assumption on its shape.
In both modes (lognormal and multimodal) the algorithm automatically calculates the volume distribution and the respective volume weighted mean droplet diameter $d_{50,3}$.



Workflow



Features and Benefits

- Minimum sample preparation with no sample alteration or dilution
- Able to measure both, W/O and O/W droplets
- Multimodal and Lognormal distribution results
- Similar or better precision compared to other analytical techniques such as microscopy, laser diffraction, and electric sensing
- NMR measures droplet size and not cluster size
- Fast method
- Has lowest cost-of-ownership per measurement of all methods

References:

1. J.P.M. van Duynhoven, G.J.W. Goudappel, G. van Dalen, P.C. van Bruggen, J.C.G. Blonk and A.P.A.M. Eijkelenboom. Magnetic Resonance in Chemistry 40, S51-S59 (2002). DOI: 10.1002/mrc.1115.
2. K.G. Hollingsworth, M.L. Johns. Journal of Colloid and Interface Science 258, 383-389 (2003). doi.org/10.1016/S0021-9797(02)00131-5.
3. J.S. Murday, J.M. Cotts, J. Chem. Phys. 48 (11) (1968) 4938.
4. K.J. Packer, C. Rees, J. Colloid Interface Sci. 10 (1972) 206.

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