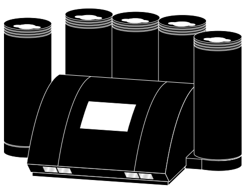
Application Note

Predicting shelf-life with MultiScan MS 20 based on ISO/TR 13097 standard

The shelf-life of a product is a critical factor in ensuring that the product performs as intended, maintains the required quality and is safe for the consumer over its entire lifetime. One of the primary challenges in the development of new dispersion-based products is accurately predicting their shelf-life based on early data acquisition or by accelerating the destabilisation process. Predicting the shelf-life of a product is a complex process that requires a combination of accelerated stability testing, including exposure to temperature and mechanical stress, with data extrapolation using mathematical models. To address this challenge, the MultiScan MS 20 from Dataphysics Instruments has been developed as an analytical instrument (Fig. 1), providing comprehensive insight into the stability of dispersion-based products.[1] The following note provides detailed explanations on how shelf-life can be predicted using Multiscan technology in accordance with the ISO/TR 13097 standard.

Measurement device
MultiScan dispersion stability analysis system



Measurement method
Optical dispersion stability analysis

Measured quantities
Transmission intensity
Backscattering intensity

Environmental conditions
40 °C

Samples
Dispersion formulations

Industries
All dispersion formulations

dataphysics

Fig. 1: DataPhysics Instruments stability analysis system MultiScan MS 20 with six independent ScanTowers

What is Shelf-life?

The ISO/TR 13097 defines the term "shelf-life" as "the recommended time period during which a product can be stored, throughout which the defined quality of a specified property of the product remains acceptable under expected (or specified) conditions of distribution, storage, display and use."[2] It is based on how stability metrics change. Faster alteration means a shorter shelf-life. The standard focuses on thermodynamic instability, as illustrated in Fig. 2. It excludes phenomena related to radiation, chemical or enzymatic reactions, and biological processes like bacterial growth and metabolism.

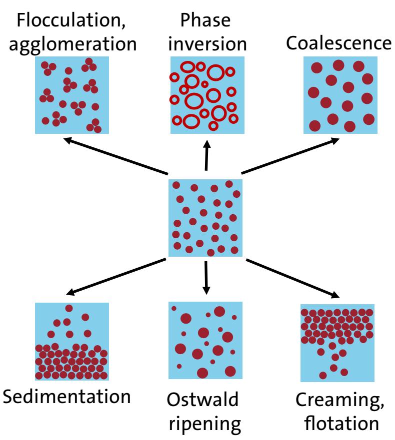


Fig. 2: Primary destabilization phenomena in liquid-solid or liquid-liquid dispersions

How to predict Shelf-life?

ISO/TR 13097 describes two methods for shelf-life prediction: comparative and predictive analysis.

- Comparing analysis compares the stability metric with a reference sample. This approach is reliable, does not require complex calculations, and can be used for all types of dispersions. It is the quickest and easiest way to predict how long a dispersion will be stable.

- In predictive analysis, data is modelled and extrapolated to check if the stability metric stays within the stability criteria for the desired amount of time. However, the models are not currently well adapted to predict the stability of complex systems. Using such methods to predict the stability of real dispersions can lead to false results.

To speed up the quantification of long-term stability, the following approaches are possible:

- Thermal acceleration: Test at higher temperatures (up to 50 °C) or use thermal cycles.

- Mechanical acceleration: Test the product using centrifugation or vibration.

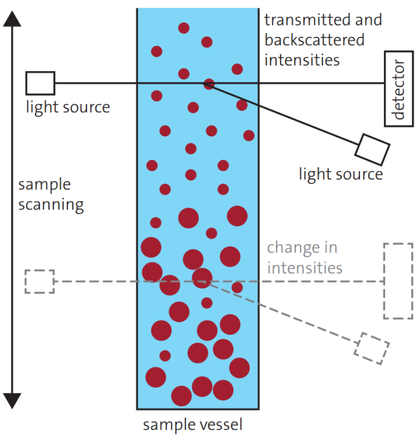


Fig. 3: MultiScan measuring principle

- Physico-chemical perturbation: Add substances (solvent, acid, etc.) to vary the sample composition and test the resistance to this change.

It is important to consider how long the product would normally last and how it would be used. The thermal acceleration method is the most used. It is useful for studying storage and shipping. Mechanical and physico-chemical acceleration are often leading to unnatural and unrealistic outcomes due to unexpected effects despite being efficient at speeding up certain destabilization processes.

ISO/TR 13097 specifically recommends instrumental methods since they are most objective and traceable. Additionally, they can detect destabilisation at an earlier stage much more reliably than conventional visual observation due to their high sensitivity and reproducibility. Moreover, it recommends that analysis should be conducted in the sample´s native state without dilution or perturbation.

MultiScan Technique

The MultiScan MS 20 technology is based on the static multiple light scattering (SMLS) principle (Fig. 3).[1] To conduct the measurement, the liquid dispersion is poured into a sample vial, which is then placed in one of the measuring chambers of the MultiScan MS 20. The MS 20 apparatus comprises a light source, a detector positioned opposite the light source for measuring the transmitted light, and a second light source, oriented at a 45-degree angle from the detector, for measuring the backscattered light. During each measurement, the light sources and detector move up and down along the sample vial, the transmission (Tr) signal and backscattering (BS) signal are obtained by repeated measurement at the full sample height h over a period of time. The composition of these scans enables the detection of physical instabilities in the dispersion process, including aggregation, sedimentation, and creaming, in both native status and storage conditions.

The MultiScan technology is in accordance with the recommendations in ISO/TR 13097 by employing optical scanning to quantify and study dispersions in their native state, as well as enabling thermal acceleration when required.

Moreover, the MSI (Multiscan Stability Index) can be employed to quantify the stability of samples in MultiScan technology. The MSI is an automated calculation embedded within the software that aggregates all destabilising factors into a single numerical value. A higher MSI value indicates a stronger tendency towards product instability (*for further details, please refer to the application note 'Multiscan Stability Index (MSI)-One-click stability evaluation'*).

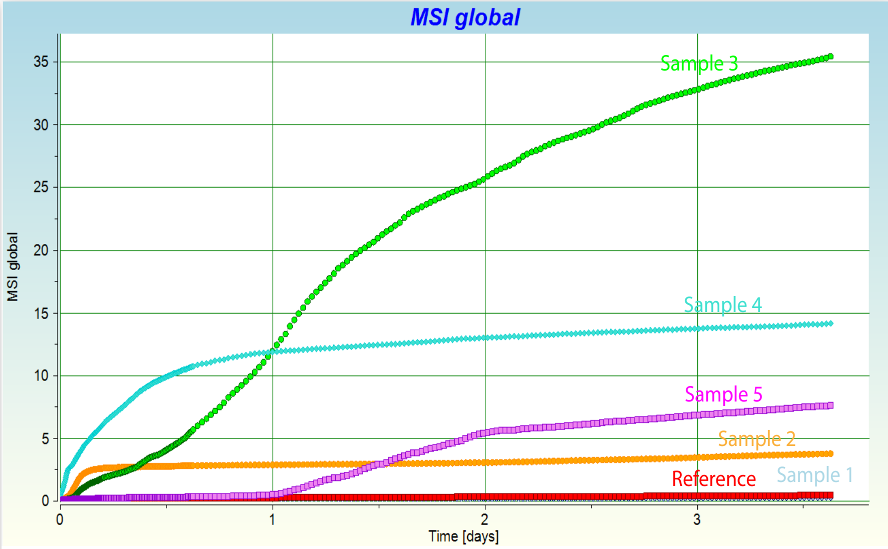


Fig. 4: Comparison of the MSI kinetic values of the samples to the reference sample

Application example

In the following study, five formulations with varying stabilisers were evaluated using the MS 20 to judge their shelf-life at a storage temperature of 40 °C after one month. Each sample was transferred into a vial and analysed over a duration of 3.5 days at a temperature of 40 °C. The global stability is assessed over time using the MSI. A higher value indicates a lower stability. The MSI kinetics were compared against a known, stable reference sample that had previously passed the visual test for stability conducted at the storage temperature for one month.

Fig. 4 illustrates the comparison of the samples with the reference sample in terms of their respective MSI kinetic values. The MSI evolution of Sample 1 is slower than that of the reference sample. This indicates that the stability of Sample 1 is changing at a slower rate and will therefore have a higher stability than the reference sample. Furthermore, the MSI values of Samples 2, 3, 4 and 5 increase at a faster rate than the reference. This indicates that the stability of these samples is changing at a faster rate than the reference sample, suggesting that they will have a stability that is lower as that of the reference sample. Moreover, based on the MSI kinetic of the samples, it can be concluded that the stability order is as follows: Sample 1 (most stable) > reference > Sample 2 > Sample 5 > Sample 4 > Sample 3 (most unstable).

The results presented here were obtained based on a measurement period of 3.5 days. A similar conclusion may have been reached after only two days, rather than one month based on visual observation. The MSI represents an optimal alternative to the existing stability metric. It depicts the comprehensive stability evolution of the sample over time and is an objective, reproducible parameter that can be readily quantified with just a single click.

In addition, the MSI histogram based on the MSI scale allows the assessment of the degree of destabilisation at a given time. Fig. 5 depicts the MSI values for the samples at the measurement time of 3.5 days, presented in the form of a histogram. It is evident that there is no notable divergence in the reference sample and Sample 1. A

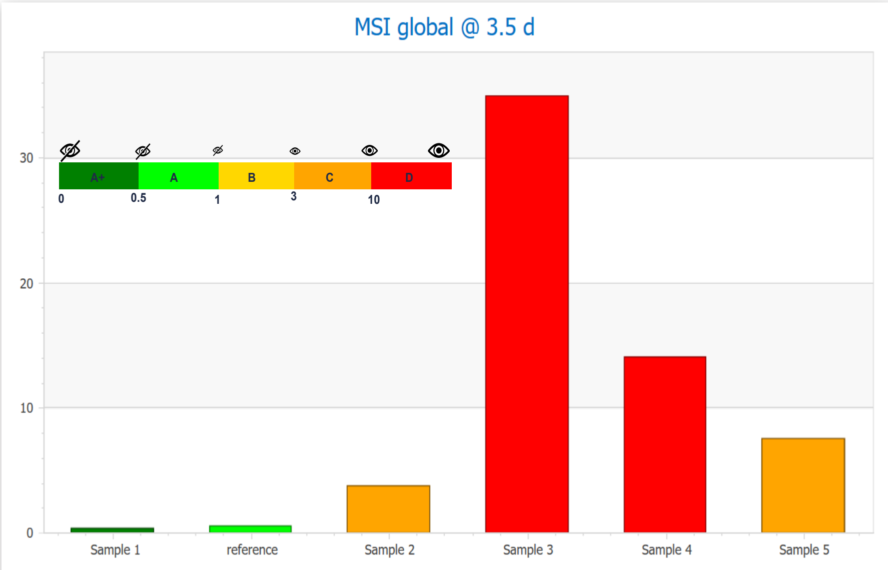


Fig. 5: The MSI values for the samples after the measurement time of 3.5 days in histogram form

noticeable alteration can be observed in Samples 2 and 5, while Sample 3 and Sample 4 exhibit even stronger changes.

Note that the MSI scale is based on an empirical analysis of a database comprising hundreds of measurements.

Summary

The highly sensitive SMLS technology allows the MS 20 to facilitate a straightforward and rapid investigation of formulation stability and shelf-life. The instrument permits the direct analysis of the native sample without any dilution. Furthermore, thermal stress can be employed to accelerate the stability measurement and/or simulate the conditions during shipping and storage. This allows for the prediction of shelf life, based on a comparison with a reference product, in accordance with ISO/TR 13097.

Reference

[1] https://www.dataphysics-instruments.com/products/ms/
[2] ISO/TR 13097:2013(en). Guidelines for the characterization of dispersion stability

**We will find a tailor-made solution for your surface science use case and will be pleased to provide you with an obligation-free quotation for the system that fits your needs.
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