Cloud Point of Non Ionic Surfactants
Automatically Determined

Cloud point is an easy and accessible quality control parameter for non-ionic surfactants present in many body care products and detergents. Since the turbid/clear transition in cloud point analysis happens so suddenly, manual measurements lack accuracy and repeatability. Melting Point Excellence instruments from METTLER TOLEDO efficiently, reliably and automatically determine cloud point.

Non-ionic surfactants (NIO) are active ingredients in products, such as industrial cleaners (for fabric cleaning and returnable bottles), home care (cleaning agents) and body care (e.g. cosmetics and moisturizers). They are also used in special, industrial applications for dispersion purposes, including the dispersion of dyes in ink or print colors.

What is special about NIO?
The hydrophilic parts of NIO comprise Ethoxy- and OH groups. The relation between chain length and ethoxylation degree (ED) is an important parameter for the emulsifying properties (solubility of fat in water) of an NIO making it very important in terms of quality.

The solubility of NIO in water is dependent upon the non-polar part, the number of ethoxy groups (ethoxylation degree) and temperature. Solutions of NIO in water or alcohols become turbid (cloudy) above a certain temperature and this is a reversible process. The ED determines the cloud point temperature, which can therefore be used for determination. NIO with low ED are always cloudy in aqueous solutions whereas highly ethoxylated NIO do not become cloudy below the boiling point of water. Cloud point is a quickly accessible indicator of the degree of ethoxylation of an NIO as raw material and its quality.

Superior automatic determination
Manual methods are based on the process of heating up the sample solution.
with a Bunsen burner until it becomes turbid and then recording the temperature when it becomes clear again upon cooling. This turbid-clear transition occurs quite suddenly and is, therefore, strongly dependent on the observing operator, which significantly affects result repeatability (and provides no means of recording the event).

Our MP Excellence instruments offer fully automated determination and recording of the cloud point based on the well-established measurement of the transmittance change used for automatic melting point tests.

**Simple measurement**

During sample preparation, the MP furnace is heated to a predefined start temperature that is slightly below the expected cloud point. The previously prepared NIO sample solution is transferred into a melting point capillary with an injection needle and then heated with a constant heating ramp until the cloud point is automatically detected.

At the cloud point temperature, the transmittance shows a distinct change from high to low values due to the suddenly occurring turbidity (Figure 1). Such characteristic curve shapes can easily be calculated to obtain the corresponding temperature. A PC-based software tool evaluates this data automatically as soon as the data is transferred from the MP instrument. Depending on the model, a maximum of either four or six samples can be analyzed simultaneously. Video recording stores the test run and enables inspection upon request.

The MP Excellence instruments provide fully automated and highly efficient sample analysis. Unbiased, objective cloud point temperatures of high accuracy and repeatability guaranteed.

Figure 1: A turbidity curve of ethoxylated Nonylphenol, an NIO with an average ethoxylation degree of 10.

Text: Dr. Hans-Joachim Muhr
Manager SPG Melting/Dropping Point

www.mt.com/one-click-melting
To Melt or Not to Melt
Thermal Analysis of Lipsticks

At a given temperature, one lipstick melts while another remains solid. DSC analysis can be used as a tool to obtain melting profiles in order to characterize the quality of different lipsticks.

DSC (Differential Scanning Calorimetry) is used to study the thermal behavior of cosmetic materials and to investigate events and processes that characterize them, such as melting, crystallization, and evaporation. DSC methods also provide information about the enthalpy of transitions and the influence of impurities upon melting behavior. Most of the effects are related to enthalpy changes initiated by increasing or decreasing temperature. DSC analysis can be used as a quality control tool to obtain melting profiles and to characterize and distinguish between different lipsticks.

Figure 1 shows the heating curves of five different lipsticks, labeled Lipstick A, B, C, D, and E, measured in the range -50 °C to 140 °C. Measurements like these are typically performed at heating rates of 5 or 10 Kelvin per minute.

Waxes and oils are initially solid but melt on heating, giving rise to endothermic peaks on the DSC curves. The results also provide information about the practical performance of lipsticks. For example, we expect a lower-melting lipstick like Lipstick D to spread well and a higher-melting lipstick like Lipstick C to wear well.

The measurements were performed with a METTLER TOLEDO DSC 1 instrument which measures from -150 °C to 700 °C at heating rates of up to 300 K/min. The DSC utilizes an innovative patented DSC sensor with 120 thermocouples which guarantees unmatched sensitivity.

Dr. Matthias Wagner
Product Manager Materials Characterization

www.mt.com/dsc
Optimize Industrial Crystallization
Track Particle Size Inline

Crystallization and precipitation are two of the most important separation and purification processes in the chemical industry. Crystallization throughput and product quality are improved by optimizing the particle size distribution. Today, established technologies measure particle size, shape and count inline in order to make real-time, informed decisions and, therefore, ensuring quality and replicability.

Chemical companies are under increasing pressure to understand and develop scalable crystallization processes faster, at lower costs and with higher quality. Companies must control crystallization conditions if they want to improve cycle times and optimize product quality and yields. The need to produce products with a specific particle size distribution continues to drive crystallization research.

Challenges with crystallization
If misunderstood, the thermodynamics and kinetics of crystallization can sometimes result in unexpected behavior, such as rapid precipitation or agglomeration, which leads to variability in yield, centrifugation times and other quality attributes. It has been established that the root cause of this variability is often inconsistency in the particle size distribution. Fine particles or agglomerates can form in the crystallizer. These cause slow centrifuge throughput caking, dust, or bulk density problems in transport and consequently particle size leading to quality variability in the final product. These problems can be controlled in the crystallizer by measuring crystal growth rates while optimizing solvent addition rates, mixing conditions, seeding and cooling rates.

Understanding crystallization with in situ analysis
A well-designed crystallization or precipitation process can be scaled-up to manufacturing giving the desired crystal size distribution, yield, form and purity. Inline particle size and shape analysis allow scientists and engineers to measure and observe crystal behavior in situ without sampling or dilution. Crystal nucleation and growth are tracked at full concentration and operating temperature and this information is then used to troubleshoot the root cause of variability and optimize processes. By applying real-time measurements, scientists and
engineers understand when and why in-consistencies occur and can then make informed decisions on how to produce repeatable, high quality products.

Tracking real-time particle size distributions

ParticleTrack inline particle measurements ensure a clear understanding of how changes in fundamental process variables directly impact upon the final product. Inline technologies track critical parameters, such as the crystal size and shape, crystal form and even the level of supersaturation, all at full process concentration and in translucent or opaque slurries. ParticleTrack provides process knowledge that enables real-time improvements in yield, throughput and profitability of laboratory- or process-scale crystallization. It accelerates the characterization of critical operating parameters for increased productivity and identifies undesired events in real-time to fully ensure final product quality.

Read our white paper to better understand how in-line particle technology is used to comprehend, optimize and control crystallization in order to improve product quality and yield.

Text: Ben Smith
Market Manager Particle System Characterization

www.mt.com/particle_track

www.mt.com/wp-industrial-crystallization

ParticleTrack E25
Irritation-Free
Cosmetic Testing

In the cosmetics and body-care world, ensuring a product will not irritate the user’s skin is critical. Cosmetics manufactures outsource quality control related tests to independent laboratories. European test lab Farcos, where operators handle 300+ samples daily, has learned that the right equipment and data-handling solutions can also make its stringent quality control processes irritation-free.

European chemistry and microbiology test lab Farcos provides quality control test procedures for cosmetic manufacturers worldwide. Founded in 1994, its labs, including those in its sister company Farcoderm, ensure sought-after consumer products maintain expected attributes, such as look, feel, analytical and microbiological stability, wearability and safety.

Pressure to perform
The company has long prided itself on the surety of the results it provides to its clientele. However, while its operators offered a wealth of technical skills, Farcos still relied on the handwritten transcription and tracking processes that it developed nearly 20 years ago; therefore processing took time, which was detrimental to the expanding company’s productivity.

Additionally, the balances that were in use failed to perform reliably. Unscheduled maintenance put more pressure on the lab’s testing schedule meaning more stress for operators.

LabX software connects the balances, label printers and barcode readers for assured traceability at Farcos.
**Enhancing surety and speed**

Farcos sought a comprehensive weighing solution that would help them maintain the quality testing their clients expected while simultaneously saving time and eliminating the error potential inherent in handwritten tracking protocols. METTLER TOLEDO was chosen for its quality products and reputation for providing the continued service and technical expertise necessary to ensure reliable and consistent balance operation.

The final installation included four XS802 balances, one XS205DU balance, three label printers and a barcode reader, all connected by LabX software for assured traceability. The new, integrated configuration takes full advantage of LabX’s barcode functionality. Automatically generated barcodes uniquely identify each sample in Farcos’ sample-receiving department. These barcodes enable fast and error-free sample management that ensures the traceability of all test results and LabX then handles all data produced automatically.

**Effortless processing**

This new METTLER TOLEDO equipment setup has made information management virtually effortless at Farcos. All processing has become faster, fully traceable and compliant so that operators can now concentrate on helping additional clients rather than dealing with the irritation associated with handwritten transcriptions and unreliable balances.

Text: Roberto Tonani
Product Manager precision balances

> www.mt.com/LabX
Setting up a new Method in Your Instrument?
Let us Give You a Hand

Setting up a new measuring method in your instrument for the first time is not straightforward if lacking the right instructions. METTLER TOLEDO has attained over the years a wealth of know-how about instruments’ methods for a broad range of industry applications. This compendium of knowledge is just one click away from you.

+2000 methods
More than 2000 methods for our titrators, thermal analysis instruments and moisture analyzers are available to you. Find the right method in the shortest time thanks to the filtering options.

Always at hand
Search, download and store your methods in a convenient and easy way. The digital method library is always accessible from any digital device. No more leafing through endless book pages.

+60 years of knowledge
It is been long since in 1945 Dr. Erhard Mettler, a Swiss engineer, invented the substitution principle with a single-pan balance. Since then we have developed and tested many precision instruments. Benefit from this wealth of knowledge for free.
Four Steps to Your Desired Instrument Method.

1. Go to [www.mt.com/ta-applications](http://www.mt.com/ta-applications)
2. Define the search criteria either with filters or keywords
3. Get your search results
4. Download your application method

---

Thermal Analysis Application No. UC 293

Application published in METTLER TOLEDO Thermal Analysis UserCom 29

### Measurement of thin films in shear by DMA

**Introduction**

In the DMA, a sample undergoes periodic deformation. However, the force necessary to deform the sample acts not only on the sample but also on the sample holder. This means that the measured displacement amplitude is the sum of the deformation of the sample and the deformation of the sample holder. Ideally, the deformation of the sample holder should be negligible compared with the deformation of the sample.

When thin samples (thickness < 0.2 mm) are loaded in the shear sample holder, the danger is that the shear clamping plates tilt slightly and touch each other. The results from a DMA measurement performed under these conditions are then completely wrong. To make sure the plates do not touch each other, one intuitively tends to measure thin samples with large diameters. The stiffness of such samples might then be greater than the stiffness of the sample holder. In such cases, the deformation of the sample holder contributes more to the total deformation than the deformation of the actual sample. When the modulus is calculated, the measured total deformation or stiffness must be corrected. This is done using the so-called stiffness correction.

Thin films with a thickness of 50 to 200 µm are usually measured in tension in the DMA. They can, however, also be measured in shear if proper attention is paid to sample preparation and other factors. In this article, we present two examples to show how this is done.

![Figure 1. Determination of the sample holder stiffness from a “displacement scan” for the shear sample holder. The alignment insert for the small clamping assembly was used as a sample.](Image)
Put Your Lab InMotion™ for Flexible and Efficient Analyses

Automation in today’s laboratory has high demands for a variety of samples and workflows. Automation no longer means just analyzing one sample after another. Autosamplers assist with making workflows more flexible and efficient by eliminating process orders and sample data transcription errors.

Maximum throughput
Designed to maximize throughput in the minimum of space, InMotion autosamplers increase productivity without sacrificing laboratory bench space. The innovative robotic arms of the Pro and Max series reach into the sample tray to optimize space.

Flexible workflows
Intuitive and flexible instrument programming allows sample workflows to be tailored to specific requirements. Whether using PowerShower™ to clean components or more thorough sequences to clean and recharge electrodes, In-Motion is here to assist.

Modular and tailored
Every lab and sample has its own demands. Build an autosampler according to sample requirements with modular boards for extra pumps, CoverUp™ system for protecting samples and operators and water bath sample trays for temperature control.

▶ www.mt.com/InMotion
Worry-free Weighing
Follow the Green Light

Labs are facing increased pressure to comply with industry regulations, improve equipment effectiveness and increase profit margins. METTLER TOLEDO’s new XPE and XSE balances introduce a new dimension to analytical weighing with a combination of intelligent features designed to make daily weighing tasks easy and error-free. On top of outstanding weighing performance, full regulatory compliance and high process security are assured.

StatusLight™
StatusLight uses color intuitively to show at a glance if it is safe to start a weighing task. Green means the balance is level, functioning correctly and all calibration and routine tests are up to date: Safe!

StaticDetect™
The weighing error is measured when static charges are detected. If the user-defined limit is exceeded, a warning to employ anti-static measures is given in order to ensure the most accurate results.

RFID Solutions
RFID SmartSample tags enable secure transfer of titration sample data from balance to titrator. EasyScan™ checks test and calibration dates on tagged pipettes. The balance has a built-in pipette accuracy check application.
Every density and refractometry measurement contains some uncertainty. Put in practice a set of Good Density and Refractometry Practices in your QC lab and reduce the risk of error to the minimum.

Our Density and Refractive Index Measurement guides give you tips & hints along the measurement process: From instrument calibration to result verification.

www.mt.com/lab-density-tips

www.mt.com/lab-refractometry-tips