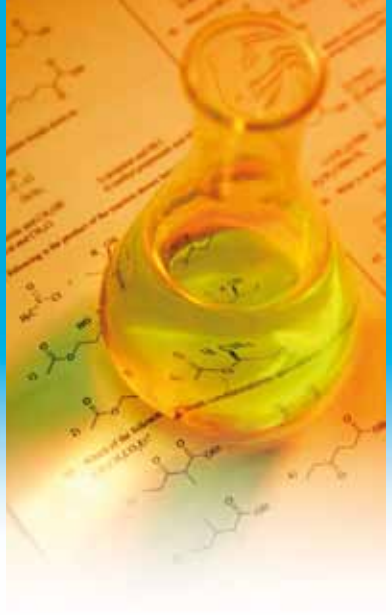
Chemicals
Analytical solutions in the laboratory



9 News

Packaging Coatings
Dry Matter Matters

Packaging coatings provide color and protection. Whether solvent or water based, an important criteria for constant product quality is the dry matter content. The coating experts from Schekolin in Lichtenstein have used the Halogen Moisture Analyzer from METTLER TOLEDO for many years to achieve the fast and efficient determination of dry matter in lacquers and paints.

Fast and efficient

Schekolin provides coating solutions for cans, tubes and lids produced from metal and plastic. Dependant on function and application technique, the formulation of coatings varies widely and is reflected in the solids content from approximately 30% to up to 80%. According to DIN, the norm solid content of paint is determined by oven drying. At Schekolin, the same results are obtained within a fraction of the time with Halogen Moisture Analyzers from METTLER TOLEDO. The HR83's graphic user guidance makes it easy for Shekolin's lab technician to select one of the pre-installed drying methods. If none of the currently accessible methods prove appropriate for a new coating, the built in test function assists in finding the optimal analysing parameters.

Typical drying temperatures for solvent based coatings are between 180-200 °C with drying times varying between only 3-6 minutes. For the moisture analysis, a sample of the pasty or viscous paint is distributed on an absorbent glass fiber filter in order to prevent any film building and to ensure homogenous sample distribution. The fast halogen heating technology, with its highly precise temperature control, ensures that the target temperature is quickly reached and that the sample is evenly heated ensuring accurate and repeatable dry matter data.

Thanks to the HR83 Moisture Analyzer, Schekolin can now develop and test new coatings far more efficiently in comparison to using a traditional drying oven.

▸ **www.mt.com/moisture**

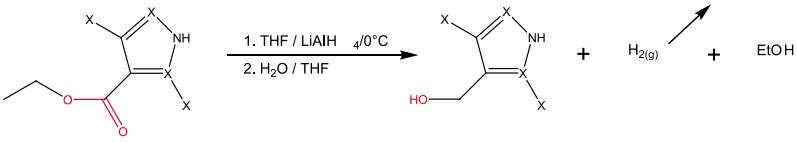
METTLER TOLEDO

Safe Process Design
Using Different Techniques

At Schering-Plough in the Netherlands, the Department of Process Chemistry studied a reduction of an ester into an alcohol with lithium aluminum hydride to determine the design of a safe process. Because the original reaction was not safe for large scale production, small process changes were introduced. Different techniques were used in order to evaluate safety: RC1e® with gas flow measurement, DSC and adiabatic calorimetry. Furthermore, the simulation programs of Dynochem® and AKTS were used.

Reaction

In a 1L AP01 reactor, the ester was dissolved in THF and cooled to 0 °C. Lithium aluminum hydride ($LiAlH_4$, 2.4M in THF) was then added with a dosing funnel - this addition was exothermic (ΔT_{ad} = 102 °C). Heat and gas flow were simultaneously monitored (Figure 2) which was a remarkable phenomenon, since gas release was expected only during the work up procedure. After the addition was completed, a significant accumulation of 65% was observed. Following 20 minutes of stirring at 0 °C the reaction temperature was increased to 20 °C (Figure 3).



For the safety study, the reaction was performed in the RC1e® (Figure 1) based on laboratory experiments.

The accumulated heat, which can cause an adiabatic temperature rise of 66 °C, is released during the temperature increase to 20 °C. The TMRad24 for the reaction mixture was determined as 49 °C by a PhiTec adiabatic calorimeter.

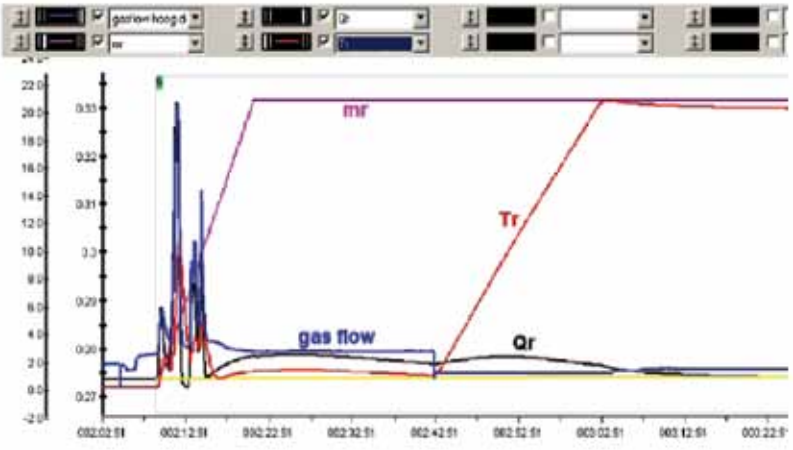


Figure 2. Monitoring gas flow and heat simultaneously.

Publisher
Mettler-Toledo AG
Laboratory Division
Im Langacher
CH-8606 Greifensee, Switzerland

Production
Segment Marketing LAB
Global MarCom Switzerland

Subject to technical changes.

Printed in Switzerland.

Work up procedure:

Lab experiments: The complete reaction mixture added to a small amount of water/THF which is not possible in RC1 due to a minimum starting volume 275 mL. Therefore, the addition was inverted and the water/THF mixture was added to the reaction mass. This resulted in a very thick suspension that could not be stirred, dosing was, therefore, interrupted due to thick immiscible layers and an uncontrolled gas release. Consequence: The process needs to be adapted.

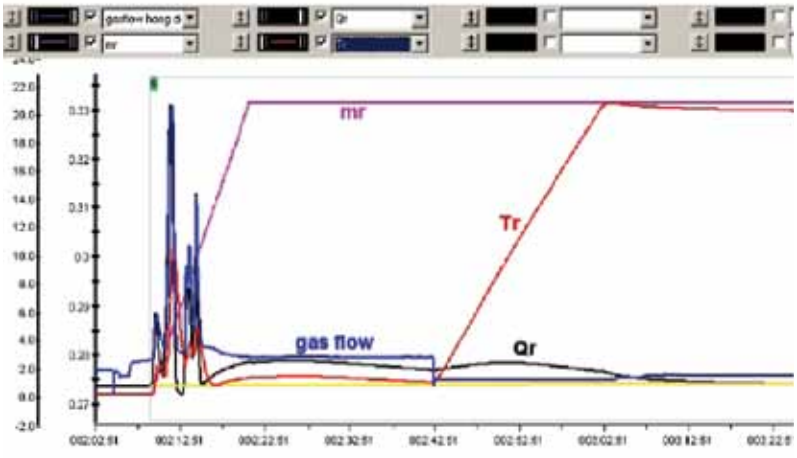
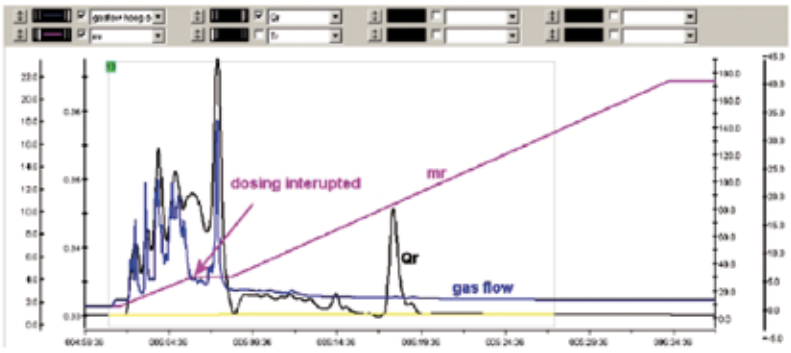


Figure 3. Reaction temperature increased to 20 °C.

Criticality Classification

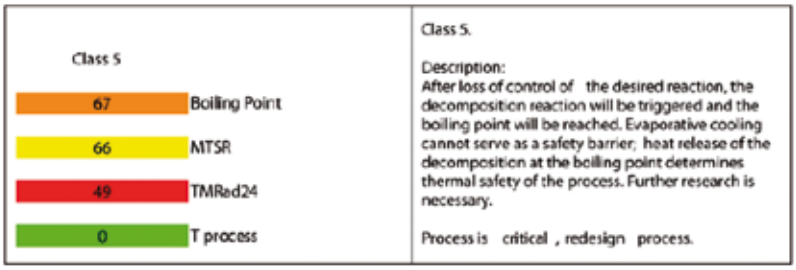
The safety classification of processes according to F. Stoessel is based on 5 classes with class 1 being the least critical and class 5 being the most critical. The classification includes four relevant temperature parameters: Tprocess, Maximum Technical Temperature, MTSR and the TMRad24 (often referred as TD24). The relative position of these four temperatures defines which class the reaction belongs to from a safety point of view. To summarize, class 1 & 2 reactions are typically assumed to be safe, while class 3 and higher reactions need a more thorough investigation. Class 5 reactions are typically unsafe and need to be redesigned.



[1] This is the temperature where it takes 24 hours to a maximum heat rate (runaway)

Figure 4. Reaction work-up procedure.

The original process (class 5) needs to be modified in order to become a class 1 or 2 process. In this case, the MTSR has to be decreased.

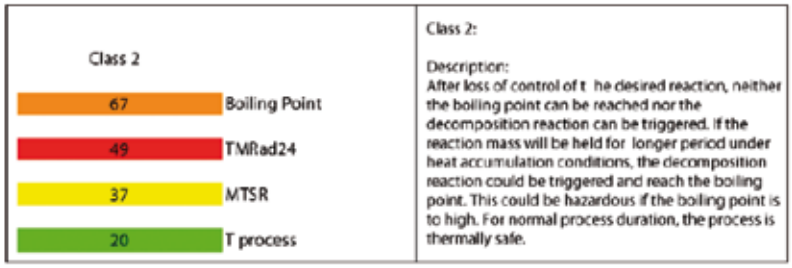


Stoessel Safety Classification of original process.

MTSR = Tprocess + (ΔT_{ad} x heat accumulation)

Different ways to lower the MTSR:

- More solvent (-> lower ΔT_{ad})
- Increase Tprocess (faster kinetics). Only if the purity remains the same
- Increase of the addition time at same Tprocess.



Stoessel Safety Classification of improved process.

Process changes:

1. Reaction: the addition of $LiAlH_4$ was performed at 20 °C instead of 0 °C.

2. Work up procedure: addition of the reaction mixture into a large amount of water/THF.

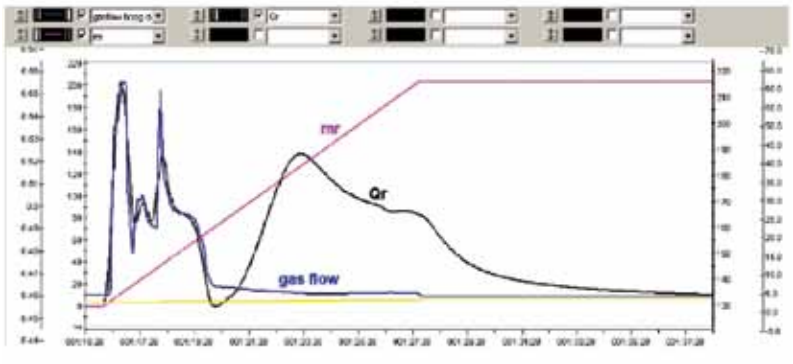


Figure 5. Improved reaction control.

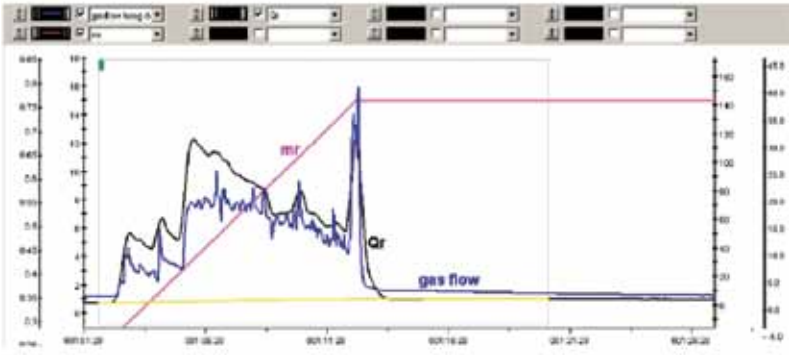


Figure 6. Improved process - no stirring issues and no gas or heat accumulation.

Reaction:

The addition of $LiAlH_4$ was significantly exothermic (ΔT_{ad} = 92°C) and almost dosing controlled. There was an improved control of the reaction compared to the LiAlH4 addition of the original process (Figure 5). The first heat generation peak, including the observed gas flow, was a result of the reaction of $LiAlH_4$ with the -NH functional group. The second heat flow peak was caused by the reduction reaction of the ester.

Work up procedure:

The addition of the reaction mixture to a large amount of water/THF proved medium exothermic (ΔT_{ad} = 49 °C) and almost dosing controlled. Neither stirring issues nor a significant accumulation of reactants, heat or gas production were observed. (Figure 6)

Criticality Classification of the modified process

Due to the modifications of the two process parameters (Tprocess and adding the reaction mixture to a large amount of water/THF), the process became a class 2 process and therefore, inherently safe.

Conclusions

The investigation of a critical process by the RC1e®, DSC and the adiabatic calorimeter PhiTec, combined with the simulation and evaluation software Dynochem® and AKTS, accounted for these significant improvements and substantially reduced the risk of an exotherm process. Thanks to the process-like operation of the RC1e®, process modifications were carried out quickly and resulted in the successful production of three product batches.

www.mt.com/rc1

Determination of Liquid Crystal Phase Transitions


DSC 1

Liquid crystals play an important role in the advancement of modern technology. They are unique in their properties and are widely used for technical innovations including LCD displays for monitors, portable computers, cell phones, digital clocks and other electrical appliances.

Visual characterization of phase transitions

The liquid crystal state is a distinct phase observed between the crystalline (solid) and isotropic (liquid) states. Several different transition phases occur with liquid crystals which include the Nematic, Smectic and Cholesteric phases.

Differential scanning calorimetry (DSC) is an excellent tool for investigating the phase transitions of liquid crystals. It can quickly and easily measure the temperatures at which melting and phase transitions occur.

Hot-stage polarization microscopy is an advanced technique that is widely used for the visual characterization of phase transitions. This technique allows the user to directly observe morphological changes in a sample as it is heated or cooled. Changes in the shape, structure and color of crystals can be observed along with their size and number. This is very valuable information within research and development and quality control.

Achieving excellent results with the right combination

The following example shows how cholesteryl myristate can be easily investigated and characterized using the METTLER TOLEDO DSC 1 combined with the FP82 hot-stage microscopy system.

The DSC curve in Figure 1 illustrates the phase behavior of cholesteryl myristate. Three liquid-liquid transitions can be observed upon heating. If the sample is directly observed under polarized light then the individual transitions can be identified.

When the FP82 hot-stage microscopy system is used in combination with the DSC 1 it is possible to directly observe and identify the individual phase transitions which correspond to each DSC peak. Figure 2 shows that the sample has changed from the solvent-crystallized form to the smectic phase at 75.5 °C. Figure 3 shows that at 79.1 °C the crystals have changed to the cholesteric phase and present a structureless gray image.

The DSC 1 combined with the FP82 hot-stage microscopy system is a powerful tool for identifying and characterizing liquid crystal phase transitions. The FP82 hot-stage microscopy system yields a wealth of information that is complementary to the data obtained from the DSC 1. Furthermore, polarization microscopy and DSC measurements provide a complete thermal picture of each sample.

▸ www.mt.com/dsc

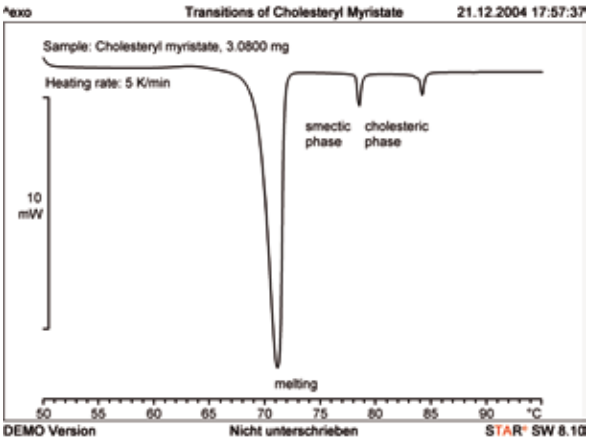


Fig 1. DSC cholesteryl myristate curve

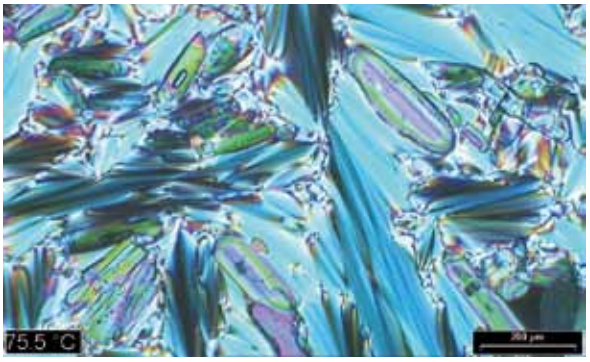


Fig. 2. Smectic phase of cholesteryl myristate at 75.5 °C

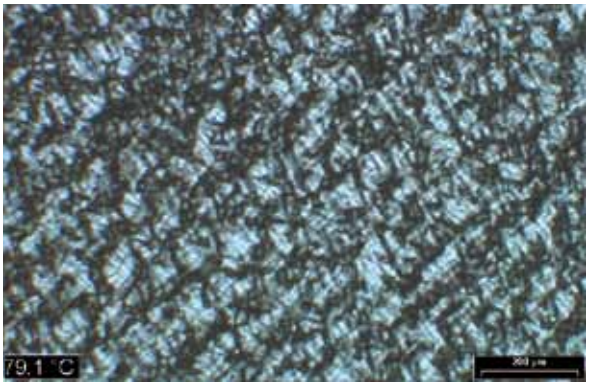


Fig. 3. Cholesteric phase of cholesteryl myristate at 79.1 °C. (Source: The FP-Microscopy System)

New Loss on Drying
Easily Meets Customers' Regulations

A leading international manufacturer of bulk chemicals recently explained that their final products must meet the regulations which apply to their customers' processes e.g. USP, ISO. The declaration of dry content requires using the standard reference method with a drying oven. The new One Click™ Loss on Drying application offers a much easier process and ensures compliance with the strictest regulations.

The traditional reference method

USP 731 precisely describes the method for analysis of volatile content. Our bulk chemical customer explained how this is put into practice: For each test a "Working List" is generated by their internal Enterprise Resource Platform (ERP). This defines the batch and the number of samples for each test. All results are handwritten onto this list. Much care must be taken to ensure container IDs, sample IDs, tare weights and sample weights are logged correctly. Samples are dried in the oven according to the monograph of the particular substance e.g. Saccharin sodium, typically used as a sweetener in pharmaceutical formulations, must be dried at 120 °C for 4 hours. After cooling, the samples are re-weighed. The dry content is calculated by manually entering the weights into a spreadsheet. The overall results are recorded on the Working List and manually entered back into the company ERP. The method is very time-consuming and there is a high possibility of recording the wrong result and obtaining an incorrect dry content. Research indicates typical transcription errors may be between 4% and 10%.

One Click™ optimizes three key points

METTLER TOLEDO's new One Click™ Weighing Solutions are complete product bundles for dedicated applications consisting of LabX software and an appropriate balance equipped with the corresponding accessories. One Click™ Loss on Drying is a full solution for an-

One Click™ Start


Samples with barcodes drying in the oven


Customized reports

One Click™ Loss on Drying

alyzing moisture / dry content and addresses three critical control points:

Transcription error – eliminated

No manual transcription is required. The lab technician enters the number of samples and barcode labels are automatically printed for the tare containers. After preconditioning in the drying oven, a barcode reader uniquely identifies each container and all weight results are automatically recorded.

Sample handling – easy

Handling the samples is easy. Due to the unique identification, weighing the samples both before and after drying is greatly simplified. The order in which each sample is weighed is of no importance as One Click™ Loss on Drying ensures all weights are correctly logged against the corresponding container ID.

Calculations – automatic

All calculations are performed automatically. One Click™ Loss on Drying determines the percentage moisture / dry content for each sample and results can be printed out at any time in a customized report to meet documentation requirements. Results are also easily transferred into existing LIMS or other systems.

Meeting customers' requirements

The whole process is easy and error-free and all results are stored securely. Full user guidance is provided on the balance to ensure SOPs are followed exactly, and LabX even ensures balance tests have been performed to provide an even higher level of process security. New methods can be created in LabX to exactly match individual weighing processes and so chemical manufacturers can be sure they meet the precise requirements of their customers.

www.mt.com/1-click-weighing

One Click™ Loss on Drying – See how easy it is

Benefit from this new solution for determining moisture content using the drying oven:

- Full SOP user guidance
- Intelligent sample handling
- Automatic documentation and calculations

Watch the One Click™ Loss on Drying video on our landing page demonstrating how straightforward the whole process is.

www.mt.com/1-click-weighing

Benefit From our Knowledge
for Your Best Practices

Discover 4 selected best practices from METTLER TOLEDO in mt.com. Profit from the useful, valuable yet free know-how from our experts to support your daily laboratory procedures and minimize risk of measurement.

Good Titration Practice™

Discover the 5 major steps to Good Titration Practice™. Comprehensive support and guidelines ensure the financial and time investment are always allocated appropriately whilst minimizing risks.

Risk Checker

Diverse factors can affect titration results. Spend 5 minutes answering 8 questions, determine titration result reliabilty and save money.

Movie

See how One Click Titration™ simplifies daily lab work with its uniform and intuitive user concept.

Brochure

Order a copy of the 'Good Titration Practice™' brochure. The brochure focuses on how to perform optimum titration analysis.

▸ **www.mt.com/gtp**

Good Weighing Practice™

GWP® (Good Weighing Practice™) is a risk-based approach that clearly interprets the regulations of each industry and puts them into straightforward weighing practice. This results in a simple program that assures compliance at any time.

Risk Checker

Find out if your results are continuously accurate. Simply answer 8 questions and the GWP® Risk Check provides expert advice on how to optimize system quality.

Movie

Watch our educational videos and learn more about how GWP® ensures weighing result accuracy.

Webinars

Attend our 'Measurement Uncertainty & Minimum Weight' and 'Routine Testing of Weighing System' interactive GWP webinars to learn more on how to establish and maintain a sustainable quality of weighing processes.

▸ **www.mt.com/gwp**

Good Crystallization Practice

Scale-up of crystallization is notoriously complicated and companies are under pressure to develop scalable crystallization processes faster, at lower costs and with higher quality. Companies must control crystallization conditions in order to improve cycle times and optimize product quality.

Improving Laboratory Crystallization

Focusing on laboratory case studies, key stages in the process development lifecycle where the implementation of FBRM® and PVM® has been successful are demonstrated.

Improving Production Crystallization

Discover five high impact ways to achieve ideal crystallization production conditions. Special attention is paid to the most common 'problem areas' encountered in production crystallizations where the implementation of FBRM® has proven useful.

Crystallization Webinars

A variety of on demand crystallization webinars are available on our internet portal. The webinars support you in development, optimization and scale-up of your crystallization processes.

▸ **www.mt.com/crystallization**

Good Moisture Practice

Moisture determinations need to be carried out reliably and quickly so that any interventions in the production process can be made promptly in order to avoid interruptions. Our website offers a valuable Moisture Guide method database and industry solutions which can help to save precious development time.

Method Database

Explore the database that contains more than 100 methods referenced to an official moisture determination method (e.g. drying oven).

Industry Solutions

Experience complete solutions for moisture analysis in a variety of industries, including sugar, plastics and wastewater.

Moisture Measuring Principles

Learn more about the method of drying (heating the sample using thermal radiation) and the principle of the switch-off criterion.

▸ **www.moisture-guide.com**

Understand and Control
Process Chemistry

Challenged by space constraints and/or complex installation requirements? ReactIR™ 247 is a unique process-hardened FTIR spectrometer designed to fit into nearly any space without sacrificing the proven performance of ReactIR™ technology. The design of the ReactIR™ 247 yields an analyzer with high stability and sensitivity in a product format that delivers extreme space savings with plug-in and measure operation.

Turn-Key
Integrating the Sentinel™ probe technology to a flow cell permits easy integration into existing flow stream and immediate analysis of the reaction as it occurs.

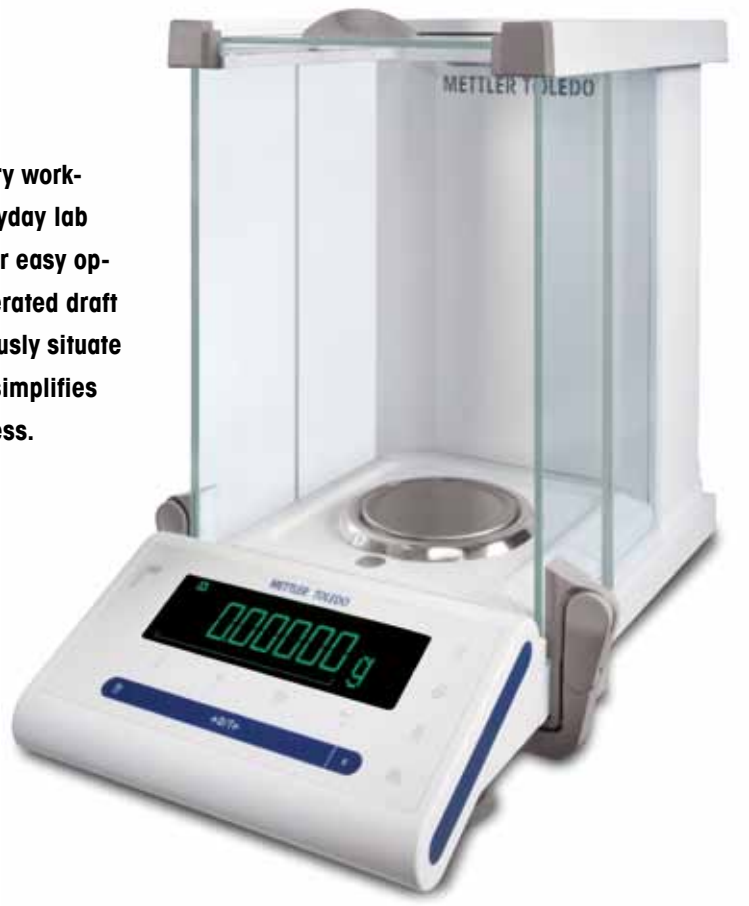
From Lab to Plant
Specifically designed for the production environment, iC Process™ enables the transfer of critical control parameters determined in the lab.

Flexibility
Flexible sampling arrangements allow for the use of longer probes to reach the fill level for small volume starting conditions.

www.mt.com/reactir247

The All-Round
Laboratory Performer

The NewClassic MS105DU Semi-Micro Balance is a laboratory workhorse that delivers quick, precise and reliable results in everyday lab work. It is convenient to operate with tactile keys designed for easy operation while wearing gloves. The ergonomic single-hand operated draft shield doors leave the user's second hand free to simultaneously situate the sample. The NewClassic MS105DU Semi-Micro Balance simplifies routine procedures and accelerates the entire weighing process.



Operable with Gloves
Keys with tactile feedback make the balance easily operable with gloved hands.

Quick and Easy Cleaning
The draft shield can be fully dismantled in a few short steps and all glass panels are dishwasher safe. The drip tray is also easily removable without tilting.

Chemical Resistant
The balance is robust and easy to keep clean thanks to an aluminum housing with IP54-in-use protection resistant to most chemicals, including acetone.

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