Moisture Determination in Test Laboratories

Today, modern test labs offer a wide range of analyses for numerous industries. Whether it’s in-process inspections, tests concerning product quality or declaration and labeling examinations – in many cases, moisture determination is included as part of the standard inspections. As independent test labs work under intense commercial pressures, quick, precise, and secure measurement methods are especially high in demand. This article highlights two common thermogravimetric methods for determining moisture content: the 'loss-on-drying' method in a drying oven and moisture determination with a moisture analyzer.

The thermogravimetric moisture determination method

The thermogravimetry procedure or method is also designated the 'loss-on-drying' technique. Here the change in mass is interpreted as a releasing of moisture. The drying ends when a state of equilibrium is reached; i.e. when the vapor pressure of the damp substance is equal to ambient vapor pressure. For reproducible thermogravimetric moisture determinations, the drying temperature and the duration of the drying are of great importance. These also influence the measurement result. The influence of atmospheric pressure and ambient humidity is of secondary importance. Loss-on-drying methods are the traditional approaches. They are often stipulated by legislation and as a reference method of the respective national food regulations. The moisture of materials includes all those substances which volatilize when heated and cause the sample to lose weight. The loss of weight is detected with a balance and interpreted as moisture content. In addition to water, therefore, this definition of the term also covers other losses in mass such as evaporating organic solvents, alcohol, oils and aromatic components. Also volatiles from compounds easily decomposed at applied temperatures e.g. carbohydrates, are counted.
Water binds in different ways
One must first understand that water can bind in a great variety of ways. A huge number of substances contain water. In the simplest case, water is adsorbed on the surface of solid substances. However, it is also stored deep in the pore systems of particles or is present as chemically bound water. In addition to well-defined products such as ointments, perfumes or all-purpose sugar, water must also be examined in complex cellular structures such as in dried fruits or meats, in which water is bound both by sorption on the surface and by capillarity between particles. This being the case, the choice of analysis and sample preparation methods must be heavily based on the properties of the products being inspected.

Drying losses with the oven
A wide variety of approaches can be selected for determining a sample’s moisture content. Loss-on-drying or LoD is one of the most important, and also one of the oldest, methods. However, chemical or spectroscopic approaches, gas chromatography, density determination, and refractometry are also suitable for determining moisture. In any event, it is often more than just the water content that is examined in these methods – usually other chemical substances are included. Every one of these methods has its own advantages and disadvantages. Up until now, dry content determination in a drying oven has established itself as the reference method. The moisture contents defined in many of the regulations for the cosmetics, pharmaceutical and food industries are based on this procedure. Values determined by other methods must, therefore, always be referenced against the LoD method in the drying oven.

The measurement principle of LoD
A sample is weighed in and the ‘wet weight’ is thus determined. Then the water is removed through heating. The residual weighing gives the dry weight, and so the difference between the initial weight and the residual weight tells us the mass of the moisture content. Based on the dry weight, this then ultimately allows us to calculate the moisture content. In this process, measuring accuracy and the resolution of the balance are extremely important.

Moisture determination in a drying oven
The two important advantages of drying ovens in moisture analysis are the large sample throughput and the flexibility when it comes to sample volumes/sizes. However, the handling is very laborious and, without technical support, this must be calculated with a range of potential risks of error when performed manually. For example, some of these include:

- A risk of mixing up the samples
- Errors when calculating the moisture content
- Errors with the documenting of the weighing results

Handling Calculations Documentations

Fig. 1: Typical steps during manual processing: mixing up samples, miscalculation, and mistakes when transferring data

The central element in all thermogravimetric methods is the weighing cell (balance). Many of the risks mentioned above can be significantly reduced, or even excluded, using clever technical solutions – such as with the Excellence Balances from METTLER TOLEDO.
Clever solutions reduce the risk of error

A simple version of the differential weighing technique applied for the LoD method is integrated into the XP balance models. After beginning the procedure, the user is partially led through the individual weighing-in and residual weighing steps. This allows computer-free deployment on a production line, for example. The dry content is calculated immediately after weighing, and this can be printed out via external printers and documented. As the balance’s firmware offers multiple languages – such as Russian, Japanese and Chinese, for example – operation is really simple and the print-outs can easily be read by the user. This straightforward yet powerful, integrated application, combined with an external printer, is an excellent solution.

Networking and central control with LabX

Oftentimes customers have greater requirements that can be better solved with the balance’s remote function – the LabX Client. For example, using this function the balance can be connected to and operated on the company network from further distances. The optional, external lab software LabX Balance allows this function. All measurement data is automatically saved and can be accessed for years afterwards. A connected label printer can automatically print out heat-resistant labels with barcodes, with the result that each sample can be clearly identified. After heat treatment in the drying oven – e.g. three hours at 105°C – and then cooling in the desiccator, the sample is again identified via the barcode, the second weighing result is assigned to this code, and all calculations are carried out automatically by the software. The result appears on the balance and this can be printed out through a connected printer with all measured values for complete documentation. These technical solutions provided by the Excellence Balances from METTLER TOLEDO significantly reduce the risk of error, particularly with large sample volumes and high throughputs. In addition to being easy to use, the balances also offer several clever solutions that provide more user comfort and measuring reliability in and around the weighing chamber.

Fig. 3: Starting the method with a click, step-by-step guidance on the display, clear sample identification using barcodes, automatic measured value reporting and calculation, as well as complete documentation, mean the highest level of efficiency, convenience and security.
LabX can do even more. This lab software connects all the balances in a network. Quality supervisors or lab managers can freely define their own methods, user authorizations, read/write rights, and a number of other security parameters and then apply this to specific or all connected balances. The One Click™ ‘loss-on-drying’ method is integrated in LabX as standard and can be started on the balance by One Click™. The user is guided through the process step by step. All entries are shown on the balance display and the measurement results are automatically and digitally calculated. The laborious and error-prone entry of weighing results and the subsequent manual calculation of the moisture content are now a thing of the past. A PC does not have to be installed near the balance, because the process is started by the balance itself. You merely need a connection, either with an RS or network cable (e.g. Ethernet). LabX can be connected with a wide range of lab instruments – for example, titrators, refractometers, density meters, and melting point analysis instruments.

This multifaceted combination of ideas, additional functions, and auxiliary measures significantly reduces the risk of error in the loss-on-drying method. Thanks to the aids described, incorrect weighing results can almost be ruled out. At the same time, you can improve the weighing ergonomics with the help of suitable taring container holders, such as ErgoClips, antistatic accessories, special shield doors, MinWeigh doors, and many others. Without these numerous weighing ergonomics functions, the efficiency of the loss-on-drying method cannot really be improved in the long run. In particular, without these clever solutions, the often large number of samples increases the risk of mixing them up or writing down the wrong measurement results.

**Ideas for improved user convenience**

A large weighing chamber offers lots of space for large weighing containers. A particular advantage in terms of comfort and handling are the hands-free doors on the Excellence Plus XP balances, which open automatically. The control is taken over by a SmartSense infrared sensor which responds as soon as an object approaches the weighing chamber. Another special feature is the SmartGrid grid weighing pan. It holds tare containers securely and simultaneously reduces the influence of air currents in the weighing chamber. The stabilization time, during which the measurement data fluctuates before reaching a stable value, is therefore much faster than with conventional balances. Measurement results can be obtained more quickly as a result. In addition to the SmartGrid® grid weighing pan, METTLER TOLEDO also offers a variety of other holders, called ErgoClips, that are specially tailored for all conceivable tare containers.
Modern functions such as GWP Excellence contribute to even greater weighing reliability. This is an important consideration in strictly regulated fields, in particular. The module not only prompts balance testing and calibration at specified intervals, it simultaneously attends and monitors their correct implementation. Depending on the user-defined setting, use of the balance can automatically be blocked if routine testing is repeatedly skipped or errors occur multiple times.

Moisture determination using the Halogen Moisture Analyzer

Halogen Moisture Analyzers are an advancement in infrared drying. They also work according to the thermogravimetric measurement principle. Thanks to their compact design, the halogen radiators are able to reach the required operating temperature incredibly quickly and can, depending on the manufacturer, be precisely controlled. As a result, measurement duration is usually shorter than with conventional methods and the heat is more evenly distributed in the sample. The combination of consistent heat irradiation of the sample with precise temperature control produces measurement results with extraordinarily good reproducibility. METTLER TOLEDO devices obtain highly precise and reproducible measurement results within an average of between three and ten minutes. Additional advantages are an automatic sequence and the avoidance of manual calculations. Therefore, all moisture analyzers from METTLER TOLEDO are equipped with this innovative heating technology 1). Thanks to the simplest operation and automatic logging inside the instrument, transfer and calculation errors are practically ruled out. Furthermore, the HX204 and H3153 Excellence Moisture Analyzers are fitted with a range of additional functions which further enhance the degree of reliability and operator convenience. Pre-installed methods can be initiated using One Click™. The user is then guided through the process step by step on the display. In the everyday lab environment, where there is the constant change of different measurement jobs and the substances that have to be inspected, this function is a massive help. Similarly to the balances, the moisture analyzers also allow users to set individual user rights to ensure – for example – that quality criteria are met. Results outside the set tolerance limits are clearly marked on the display.

Large library of measurement methods

As the development of a reference-equivalent method for moisture analyzers would require multiple test measurements, METTLER TOLEDO has already built a comprehensive library of measurement methods into its instruments. If a user is examining substances which are not included in this library, comparable components can be selected and then the method merely requires adapting. That saves valuable development time.

1) Exception MJ33
Fig. 6: An innovative solution: the unique suspended weighing pan provides the best results.

**Innovation in detail**

Fig. 7: Small footprint thanks to stand for touchscreen display and printer

The suspended weighing pan is an innovation. It spatially separates the weighing cell from the heat of the sample chamber. Consequently, negative thermal effects on the weighing cell are eliminated and the measurement results are that extra bit more reliable. Drying curves are shown in real time on the color display (see Fig. 8). Users can therefore visually follow and monitor the process. All surfaces underneath the sample pan in the moisture analyzer have been designed to be very smooth and are sealed. Cleaning is therefore child’s play. Even spilled samples cannot damage the weighing cell (see Fig. 9).
All results are documented automatically. Even this reduces the risk of errors. As a result, this method is highly suitable for use in regulated industries. Even for in-process control, which involves rapid results, the moisture analyzers are especially well suited.

Bench space in labs is usually scarce. Many different analytical instruments need to be placed to master the high flow of various samples. To save space METTLER TOLEDO has introduced a special stand with the HX204 and HS153. The stand holds the color touchscreen and a printer and reduces the footprint to a mere minimum (see Fig. 7).

**In summary**
The determination of moisture content is a standard task in a modern test laboratory. Here the instrumental solutions from METTLER TOLEDO ensure greater reliability, flexibility and efficiency. The numerous ideas in these systems help users to protect themselves against errors. Various functions also provide convenience and improve the speed with which measurements can be performed. In the end this means: great reliability and a high degree of efficiency.