

Moisture and
Water Content
Determination in
Polymers and
Plastics

Moisture Guide for Plastics

An Overview of Methods

METTLER TOLEDO

Editorial

Both manufacturers of plastic parts and their clients focus on quality and rapid production processes. Ensuring the right moisture or water content of materials ensures trouble-free compounding and injection molding, as well as smooth surfaces and ideal mechanical properties of the final injection-molded part or product.

This guide provides an overview of the most common methods for moisture and water determination in plastics and polymers, and a review of sample applications. It also contains tips for achieving good results with typical moisture or water determination methods.

A comprehensive array of METTLER TOLEDO enabling products and service solutions can also assist you with the determination of moisture and water in polymers, pastics and related compounds. A description of these products and their benefits follows as well.

References, resources, and links to further education conclude the guide. We hope you find that this overview offers you useful information to improve your water/moisture determination processes.

Mettler-Toledo

Disclaimer

We carefully developed the described methods with METTLER TOLEDO HR83 Halogen Moisture Analyzers and volumetric and coulometric Karl Fischer titrators. However, you should still test the information provided for its suitability to your intended purpose. As the use and transfer of an application is beyond our control, we cannot accept responsibility for results. The general safety rules and precautions of the manufacturer (e.g. for chemicals or solvents) must be observed.

The maximum permissible moisture content of pellets for processing must always be based on manufacturer instructions. Values noted herein are only typical values and may not be suitable for your specific material or process.

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1. Why Determine Moisture in Plastics?

The moisture content of many resins, such as polyamides or polycarbonates, is important for product processing, product appearance and product properties. Injection molding plastic pellets that are too moist results in processing problems and loss of quality in the final product. Typical symptoms of this kind of trouble are well known in the plastics manufacturing arena: splaying, flash formation, and impaired mechanical properties such as reduced impact and tensile strength. Add to this the fact that many plastics absorb moisture during transportation and storage, and it is easy to understand how critical moisture content control is to the manufacturing of high quality plastic products.

Moisture is defined as the relative mass of liquids in the total mass of the sample. Usually, moisture is related to the amount of water and water vapor contained in a sample. For most samples, moisture content can vary with weather (i.e. humidity) and temperature. Since moisture is often determined based on the loss of weight during a heating procedure, other volatiles are also counted for moisture (1, 2, 3). Such volatiles can be organic solvents, esters, acids and other low boiling compounds. Typical compounds are ethanol, isopropanol, ethyl acetate, acetic acid, and flavors or fragrances, to name just a few. Water content, however, is determined with methods that specifically analyze only water content (H₂O).

Knowing the amount of moisture in a sample can help determine if that sample is suitable for a specific use. This applies to a wide variety of samples such as soil, ceramics, fruit, wood, vegetables, fibers, oils, polymers and plastics. Moisture content can range from practically zero (i.e. completely dry) to high values depending on the sample's saturation (4). Quite often, moisture content is a pass/fail criterion for processability, storage suitability, taste and/or feel. For plastic injection molding, rather low moisture content limits apply. Methods used for moisture determination in plastics, therefore, must be highly accurate to ensure safe, effective molding and final product quality.



Fig. 1: Injection molding process. Moisture is an important pass/fail criterion.
(Photograph courtesy of Wild & Küpfer AG Switzerland)

- (1) Dictionary of weighing terms, Springer-Verlag Berlin Heidelberg, 2009
- (2) Weighing the right way, METTLER TOLEDO Guide Book, 720906, 2012
- (3) WiseGeek.com
- (4) Wikipedia.org

2. Methods and Principles for Determining Moisture and Water Content

Several principles and methods of moisture and water determination are in common practice today. Depending on presumed moisture content, sample type, application needs, regulatory requirements and further criteria specific to product or manufacturing process the most suitable method is selected.

Ideally, the selection also considers whether moisture determination is sufficient or if water content must be measured specifically.

Two principles widely applied in laboratories and on production floors are most familiar to persons concerned about moisture: Loss of weight and Karl Fischer's chemical reaction.

The most commonly used methods for testing plastics and polymers using either a Moisture Analyzer or Karl Fischer titration, respectively. Each method offers an easy way to execute the tests and delivers results quickly. Chapters 3 and 4 focus on these methods.

Tab. 1: Overview of some moisture determination methods

Method	Principle	Determination	Key points	TTR (1)
Moisture Analyzer	Loss of weight	Moisture	Quick method Simple procedure Suitable for production floor	10–20 min
Karl Fischer titration	Chemical reaction	Water	Selective determination of water content Down to 1 ppm Needs specific reagents	5–30 min
Drying Oven	Loss of weight	Moisture	Big sample size possible Several samples processed simultaneously	>2 h
Thermo-gravimetry	Loss of weight	Moisture, other volatiles	Applies to small samples only Also determines filler contents Delicate sample preparation	30 min
Spectroscopy	Physical	Water	Needs spectrometer (IR, H-NMR) For liquid samples Solid samples with ATR for IR Delicate sample preparation ATR: Attenuated total reflectance	few minutes
Chromato-graphy	Physical	Water	Needs gaschromatograph with suitable detector For liquid samples only	15–30 min

(1) TTR: typical time to result

Chapter 5 provides some hints that help to obtain reliable results with the drying-oven method, which is also a well accepted method in many industries for many samples types. Other methods mentioned in Tab.1 will not be covered because of their low relevance for plastic and polymer moisture content determination.

3. Moisture Determination Using a Moisture Analyzer

The measurement principle of moisture analyzers is loss of weight. The heat sources for heating samples are halogen or infrared lamps. Compared to the heating-by-oven technique, a moisture analyzer produces results much quicker and easier. Results can be obtained in as little as 10–20 minutes.

3.1 Halogen technology

Halogen technology provides an efficient heat source and is key to moisture analyzers. Thanks to halogen lamps samples are heated very quickly and evenly. Thus, halogen technology delivers fast results and high reproducibility. The HX204, HS153, and HB43-S moisture analyzers from METTLER TOLEDO are fitted with this innovative halogen heating technology.

3.2 Use of moisture analyzers

The handling and operation of moisture analyzers are principally very easy in principle. Both lab staff and production operators typically appreciate the simple operation steps of this method. The user is guided through the determination step by step. Instructional messages on a display avoid handling errors and promote safe, reliable results.

METTLER TOLEDO moisture analyzers apply the One Click™ user interface. The determination is started with just one easy click on the touchscreen—easy to do and easy to remember. Most modern moisture analyzers also allow the setting of individual user rights to ensure that the right methods are always applied.

Moisture analyzers can accommodate large samples of up to 30–50 g which are an important factor with low moisture samples such as plastics and resins, and inhomogeneous samples. METTLER TOLEDO's HX204 which provides readability of 0.001% is best suited to test samples of low moisture content. Low moisture—on the other hand—is prerequisite for high quality finished plastic goods.

Results are evaluated and calculated automatically. Results and corresponding data are shown on the color display and can also be printed on a local or network printer, sent to file servers via Ethernet, or transferred via USB and RS232 interfaces.

They can also be automatically compared to a target value for a pass/fail evaluation, ensuring unbiased results. The pass/fail status is shown on the display of the moisture analyzer.

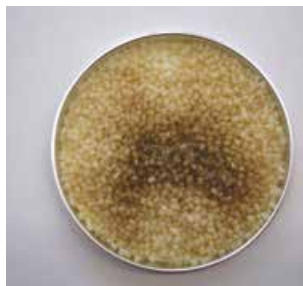
Material specific drying temperature (T_1)

For accurate measurements, it is best to select a temperature such that moisture evaporates and results match the reference value. Melted material or color changes are indications of temperatures that are too high. However, sometimes sample browning cannot be avoided as in the case of polyamides.

Typically a drying temperature in the range of 100–160°C is applied.



Left photograph shows a well chosen drying temperature. Material is neither melted nor changed in color.



The photograph on the right clearly shows signs of burning and melting. The sample is processed at temperatures that are too high.

Fig. 2: Pictures of samples after moisture determination

Measurement parameters (instrument setting)

Results from moisture analyzers are frequently compared to reference values. Typically, Karl Fischer is used as the reference method. Thus, measurement parameters of the moisture analyzer, such as temperature, heating time and switch-off criterion, are set accordingly to meet the referenced content. These parameters are then entered into the moisture analyzer and stored as a distinct user method. Such user methods can be recalled anytime for application.

Use for process control

METTLER TOLEDO instruments are easy to clean (see Fig. 3) Simple operation reduces the risk of handling errors. Thanks to their automatic calculation and documentation of results, these moisture analyzers are ideal for both in-lab and at-line process control.

Generally recommended instrument settings

- Standby temperature 100°C
- Final drying temperature 130°C
- Switch-off criterion 5



Fig. 3: Large sample pans and easy cleaning make the halogen moisture analyzers perfect for quick tests on polymers and plastics

3.3 Sample handling

Analyze samples immediately. If sample transfer and storage are required, samples should be stored in an airtight container to avoid exposure to ambient moisture. The container should be filled completely to reduce the amount of air and minimize moisture uptake during storage.



Fig. 4: Filling of sample containers. Left: Ideal filling. Right: Fill level too low.

3.4 Correlation of moisture determination versus Karl Fischer titration

For many polymers a rather linear correlation between moisture determination values and Karl Fischer titration results can be found.

The example of polyamide 6 is shown in Fig. 5. Samples of Schulamid 6MV (PA6) were analyzed at 3 moisture levels. The analyses have been executed with a halogen moisture analyzer HR83 and a Karl Fischer titrator DL39 with Stromboli oven sample changer.

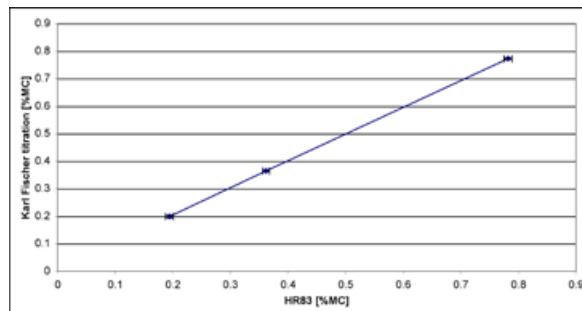


Fig. 5: Correlation between moisture determination and Karl Fischer titration
Horizontal (HR83) and vertical (Karl Fischer titration) bars represent standard deviation

3.5 Results

A selection of polymer examples follows. Samples of 2 or 3 different moisture contents have been measured with a halogen moisture analyzer and compared to Karl Fischer results.

These examples show how quickly the halogen moisture analyzer reaches the reference values. The close the drying curves explain the high reproducibility. Typical moisture limits applied to material for injection molding are indicated.

- Blue curves** Drying curves measured with the halogen moisture analyzer
- Green lines** Value obtained by Karl Fischer titration
- Red line** Typical limit of maximum moisture content for processing

3.5.1 Polycarbonate (PC)

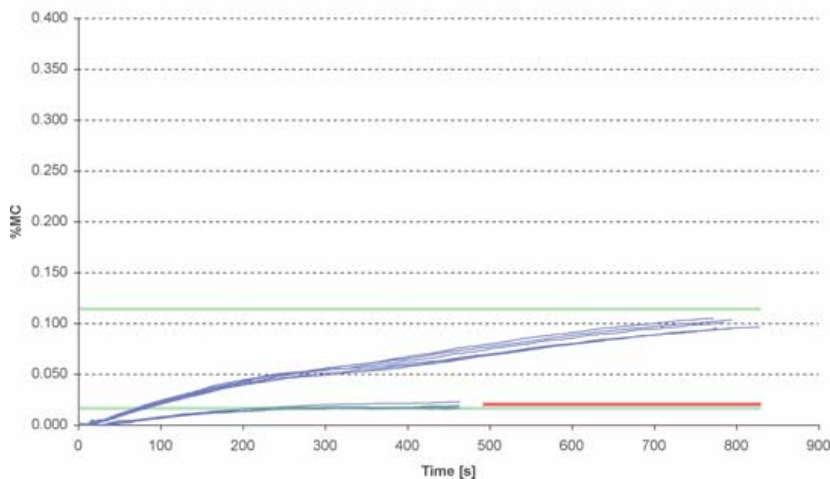


Fig. 6: Drying diagram PC, measurement of samples with 2 different moisture contents

3.5.2 Acrylonitrile butadiene styrene (ABS)

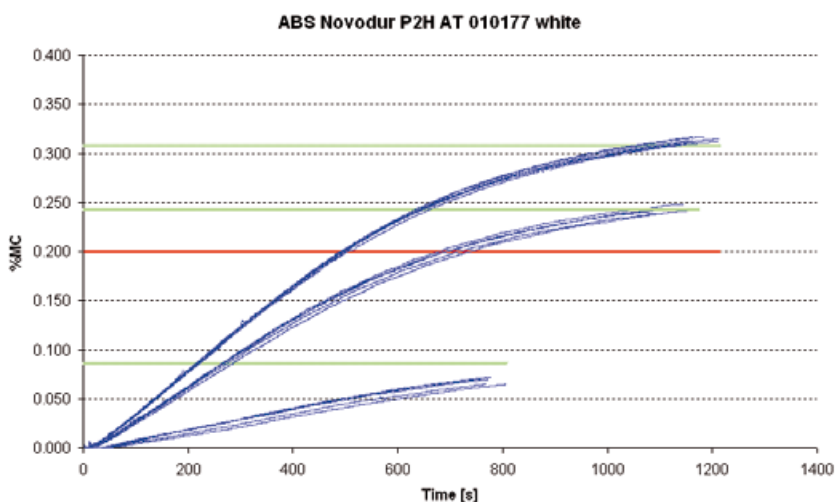


Fig. 7: Drying diagram ABS, measurement of samples with 3 different moisture contents

3.5.3 Polyamide 6 (PA6)

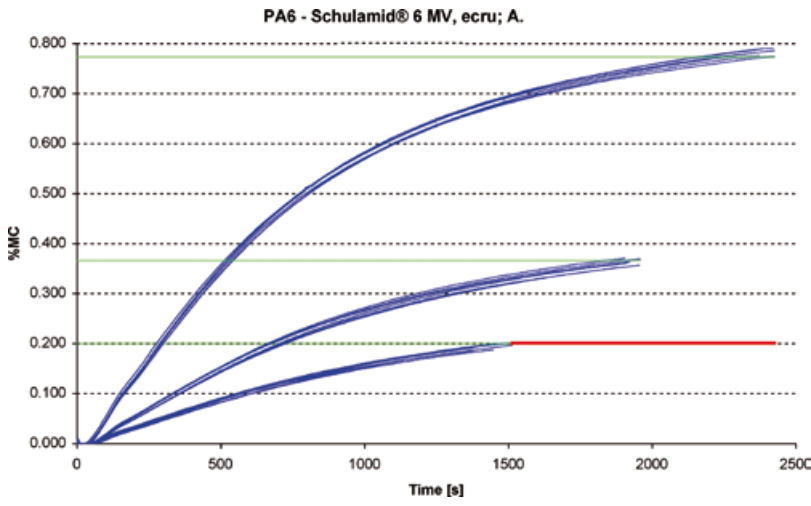


Fig. 8: Drying diagram PA6, measurement of samples with 3 different moisture contents

3.5.4 ABS + PC (Blend)

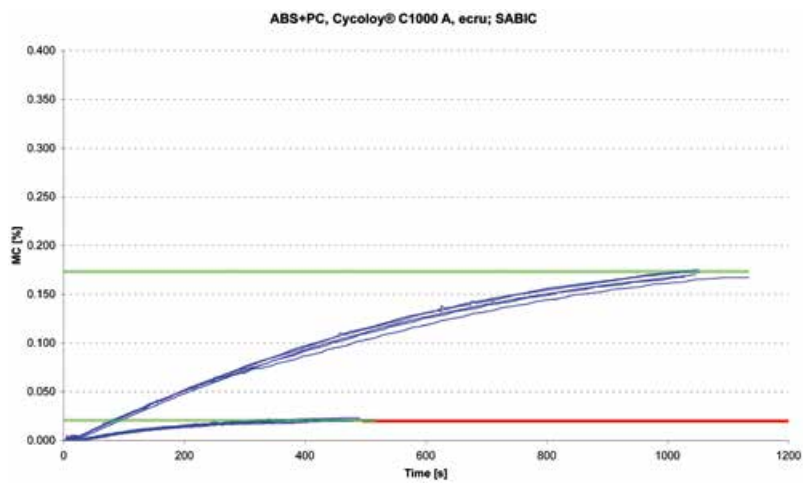


Fig. 9: Drying diagram ABS-PC blend, measurement of samples with 2 different moisture contents

4. Water Determination by Karl Fischer Titration

In Karl Fischer (KF) titration, special chemical reagents containing iodine and other compounds react directly with water. Thus, the water content is determined specifically.

4.1 Introduction

The chemical reaction, which this method is based on, was first described by R.W. Bunsen in 1853. Karl Fischer discovered that this reaction could be used for water determinations in a nonaqueous system containing an excess of sulfur dioxide in 1935. Methanol proved to be suitable as a solvent. To achieve completion of the reaction, it is necessary to neutralize the acids that are formed during the process. Karl Fischer used pyridine for this purpose which made his reagents toxic and unpleasant.

Further studies conducted in 1976 by J. C. Verhoef and E. Barenrecht on the subject of the Karl Fischer reaction have revealed that:

- Pyridine is not directly involved in the reaction, i.e., it only acts as a buffering agent and can therefore be replaced by other bases. As a consequence, less toxic and obnoxious organic bases have been introduced.
- The rate of the Karl Fischer reaction depends on the pH value (see graphic below). New generation Karl Fischer reagents react considerably faster which reduces the titration time to a few minutes only. A sharp endpoint is also achieved which contributes to better reproducibility of the results.

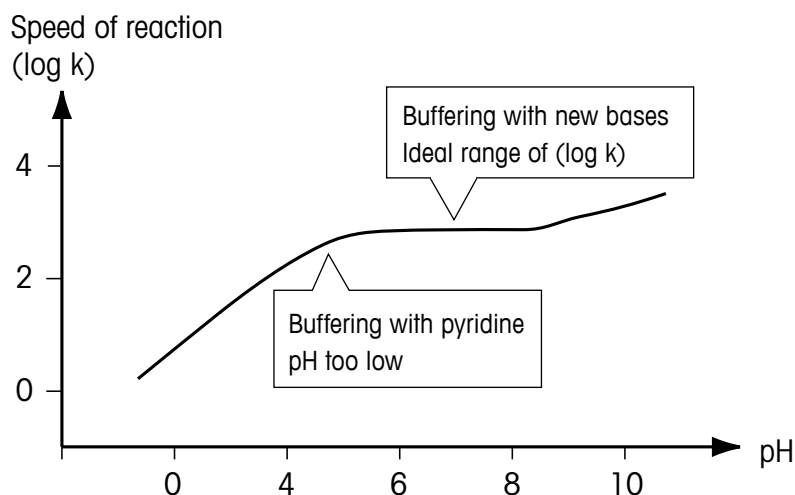


Fig. 10: Rate of the Karl Fischer reaction depends on the pH value

4.2 Modern Karl Fischer reagents

On the basis of this knowledge, E. Scholz developed a pyridine-free KF reagent with imidazole as base in 1980. This reagent not only replaced the toxic, pungent pyridine, but also facilitated faster and more accurate titrations because imidazole buffers in a more favorable pH range than pyridine.

Research experiments in 2000 on the stoichiometry of the reaction showed that methanol can in fact be replaced by other alcohols (e.g. ethanol, 2-propanol, methoxyethanol, diethylene glycol monoethylether). This improves the titer stability and further reduces reagent toxicity.

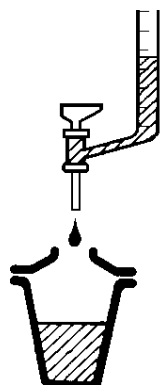
Based on this research new measuring procedures and new KF reagents have been developed. This extended the application of the Karl Fischer titration to such compounds as ketones and aldehydes.

4.3 Karl Fischer techniques

Karl Fischer water content determination is currently performed using two techniques:

- Volumetric Karl Fischer Titration, where a solution containing iodine is added using a motorized piston burette.
- Coulometric Karl Fischer Analysis, where iodine is generated by electrochemical oxidation in the cell.

The selection of the appropriate technique is based on the estimated water content in the sample.

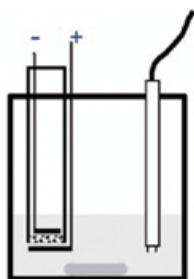


Volumetric Karl Fischer Titration

Iodine is added by a burette during titration

Suitable for samples where water is present as a major component:

100 ppm - 100%



Coulometric Karl Fischer Analysis

Iodine is generated electrochemically during titration

Suitable for samples where water is present in trace amounts:

1 ppm - 5%

However, in spite of the striking advantages of the Karl Fischer method for specific water content determination, low detection limit and fast titration speed, a few inherent difficulties have to be carefully considered when deciding to use this method:

- The influence of atmospheric humidity has to be minimized
- Sample preparation to release the water.

4.4 Drift compensation

Atmospheric humidity represents the most relevant source of error in Karl Fischer titration. Moisture can enter the sample, the titrant and the titration stand. In short, the higher the atmospheric humidity in the laboratory, the greater its influence on the results of Karl Fischer titration.

Drift is the quantity of water that enters the titration stand over a defined period and is presented as μg water/minute. No titration stand is completely water-tight; traces of water will always find a way into the titration cell. This water amount, i.e. the drift, is also titrated during analysis. Therefore, it must be taken into account when calculating the water content of the sample.

Hence, quantification of the drift is required which is achieved by titration of the dry solvent for a defined time (drift determination). Modern Karl Fischer titrators offer several ways to measure drift. It can be taken from the so-called standby titration, or a specific drift determination method can be executed and the result stored internally. Another possibility is to enter a fixed value, found by experience, to the method.

Karl Fischer autotitrators take the drift automatically into account when calculating the results (= automatic drift compensation). The applied formula is:

Sample result = Total amount of water determined – (drift * titration time)

For accurate results, the drift value should be as low and stable as possible before starting the titration.

4.5 The Karl Fischer cell

The titration cell of the KF titrator (see Fig.11) must be sealed as tightly as possible against atmospheric moisture. The following rules should be observed:

- Close all openings in the titration cell. If you need to open it, e.g. for sample addition, close it immediately afterwards.

The coulometric cell should not be opened, however. Sample administration is done through a septum using a syringe.

- Protect the titration cell with a desiccant (3 Å molecular sieve, silica gel). The desiccant absorbs the moisture and protects the titration cell against the ingress of moisture. The drying capacity of the desiccant is limited and can be exhausted after 2–4 weeks depending on the humidity.

When you assemble the titration cell for the first time, there will be moisture on the glass surface of the titration cell and the inserts. The air within the titration vessel also contains moisture. After the analyte (coulometer) or the solvent (volumeter) has been added, it is titrated to dryness during the pretitration until it is absolutely free of water. However, it may take 1–3 hours until the drift is low and stable enough for feasible sample analysis.

4.6 Sample preparation

Because water has to dissolve to undergo the Karl Fischer reaction, dissolution of the sample or extraction of its water content is prerequisite. Thus, insoluble samples such as polymers and plastics are heated in a dedicated oven and the evaporated water is transferred by a dry purge gas to the Karl Fischer cell.

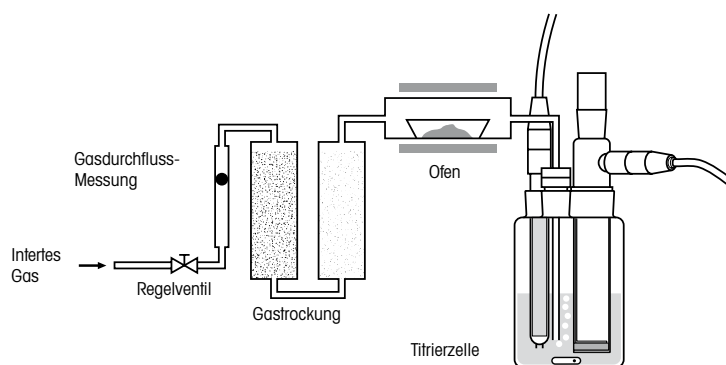


Fig. 11: Water determination in solid samples: System with cell, oven and purge gas drying

The sample is heated in an oven, causing the water in the sample to vaporize. Water is transferred to the titration cell in a current of dry inert gas (purge gas), and the amount of water is determined. The titration itself can be done using both the volumetric or the coulometric techniques.

Nitrogen is frequently used as a purge gas. If air is applied, the oven temperature must not exceed 160°C to avoid oxidation of the sample. In all cases, the purge has to be dried carefully before use.

Several agents are available for drying the purge gas. Depending on the sample and available laboratory equipment, the best, most suitable agents are selected.

Tab. 2: Purge gas drying methods

Method	Residual moisture per L purge gas
Sulfuric acid, 100%	50–80 µg H ₂ O/L
Phosphorous pentoxide, P ₂ O ₅	40–50 µg H ₂ O/L
KF one-component reagent	15–20 µg H ₂ O/L
Silica ge	50–60 µg H ₂ O/L
Molecular sieves 3 A	5–10 µg H ₂ O/L

Due to efficiency and ease, drying the purge gas with a combination of silica gel and molecular sieves is recommended. Start with the silica gel to absorb the bulk of the water, and then use molecular sieves to reduce residual moisture to a minimum.

4.7 Applications

Because the water content of polymers and plastics is usually rather low, the coulometric Karl Fischer method is often preferred. However, since polymer and plastic sample in most cases do not dissolve in KF reagents, an oven is applied to evaporate the water (see chapter 4.4).

The following tables present a short briefing that describes the execution of the Karl Fischer titration.

Tab. 3: Recommended KF methods

Substance	Examples	Method Number
Plastics Polymers	Polyethylene, PVC, Polypropylene, Poly-amides, Polystyrene, Polyurethane, etc.	V30, V31 C31, C32
Dyes (insoluble)	Pigments, dispersion dyes	V2, V8
Adhesives	All-purpose glue, glue stick	V2
Petrochemicals	Waxes, paraffins Lubricating grease, multi-purpose grease	V12 V5, V30
Solvents	Alcohols (propanol, ...) Ethers (dimethylether, ...) Esters (ethyl acetate, ...)	V1, C1
	Ketones (acetone, methylethyl- ketone MEK, ...)	V10, C10

Tab. 4: Volumetric KF procedures

Method Number – Volumetric	Procedure
V1	Direct titration Titrant: two component reagent 5 or 2 mg H ₂ O/mL Solvent: 40 mL KF solvent Stir time: 10 s
V2	Direct titration Titrant: two component reagent 5 or 2 mg H ₂ O/mL Solvent: 40 mL KF solvent Stir time: 60–120 s
V5	Direct titration with added chloroform Titrant: two component reagent 5 or 2 mg H ₂ O/mL Solvent: KF solvent/chloroform 1:1 to 1:2 Stir time: 60–120 s
V7	Direct titration with added toluene Titrant: two component reagent 5 or 2 mg H ₂ O/mL Solvent: 40 mL KF solvent/toluene 1:1 Stir time: 60–120 s
V8	Direct titration with added formamide Titrant: two component reagent 5 mg H ₂ O/mL Solvent: 40 mL KF solvent/formamide 1:1 Stir time: 5–10 minutes
V10	Direct titration methanol free Titrant: two comp. reagent 5 mg H ₂ O/mL for ketone und aldehyde Solvent: 40 mL KF solvent for ketone und aldehyde Stir time: 30–60 s

Method Number – Volumetric	Procedure
V12	Direct titration with heat (at 50°C) Titrant: two component reagent 5 mg H ₂ O/mL Solvent: 40 mL KF solvent Stir time: 5–10 minutes
V30	With drying oven at 110–150°C Titrant: two component reagent 5 or 2 mg H ₂ O/mL Solvent: 50 mL KF solvent Stir time: 10–20 min
V31	With drying oven at 160–220°C Titrant: two component reagent 5 or 2mg H ₂ O/mL Solvent: 50 mL KF solvent Stir time: 10–20 min

Tab. 5: Coulometric KF procedures

Method Number – Coulometric	Procedure
C1	Direct titration Anolyte: 100 mL anode reagent Catholyte: 5 mL cathode reagent
C10	Direct titration methanol free Anolyte: 100 mL anode reagent for ketones Catholyte: 5 mL cathode reagent for ketones
C31	With drying oven at 140–150°C Anolyte: 100 mL anode reagent Catholyte: 5 mL cathode reagent Titration time: 10–20 minutes
C32	With drying oven at 160–190°C Anolyte: 100 mL anode reagent Catholyte: 5 mL cathode reagent Titration time: 10–20 minutes

4.8 Results

The water content of some selected polymer granule, polymer film and liquid polymeric samples has been determined. Each sample has been analyzed 3–7 times in order to evaluate the standard deviation. The following tables 6 and 7 give an overview of results.

For low water content samples, the coulometric Karl Fischer technique was applied. The volumetric technique with direct sample addition was used for liquid polymeric samples.

Coulometric technique	Volumetric technique
A sample size of 0.1 to 2 g was applied at oven temperatures between 150 and 200°C. The results range from 150–2700 ppm or 0.015–0.27% respectively, which are typical and expected values.	0.05 up to 0.2 g of sample have been directly administered to the titration cell. The water content was <0.3% for the epoxy resin and around 1.3% for the super glue example.

Good values of the standard deviation (s_{rel}) of 1–5% have been achieved. As usual, the lower the water content the higher the s_{rel} value is.

Tab. 6: Results of selected samples with coulometric KF technique

Sample	Sample size g	Number n	Result ppm	S _{rel} %	Coulometric technique Method details
Polyethylene granules	0.8	3	1858	3.1	Automatic oven (1a) 200°C, 80 mL air/min, 1200 s
Polypropylene granules	2.0	6	148.3	5.4	Manual oven (1b) 180°C, 200 mL air/min, 600 s
Polypropylene film	0.5	3	2743	1.2	Automatic oven (1a) 170°C, 80 mL air/min, 600 s
Polystyrene granules	2.0	6	322.2	2.4	Manual oven (1b) 160°C, 200 mL air/min, 900 s
PVC film	0.1	6	638.1	2.2	Manual oven (1b) 150°C, 200 mL air/min, 300 s

Method details:

- (1) Karl Fischer oven
 - a. Automatic oven and sample changer (Stromboli)
 - b. Manually operated Karl Fischer oven (DO307, DO308)
- (2) Purge gas: air, flow of 80–200 mL/min
If air is used, the oven temperature must not exceed 180°C. Polyethylene and other polymers start to oxidize (decompose) at 220°C and water is released.
- (3) Drying time: 300–1200 s respectively 5–20 minutes

Tab. 7: Results of selected samples with volumetric KF technique

Sample	Sample size g	Number n	Result %	S _{rel} %	Volumetric technique Method details
Epoxy resin (liquid)	0.05 g	5	0.258	2.6	Stirring time: 60 s Delay time: 15 s Additional solvent: 20 mL chloroform
Cyanoacryl glue	0.2 g	7	1.28	2.2	Stirring time: 60 s Delay time: 15 s

Method details:

- (1) Sample input: Syringe or tube with needle
- (2) Karl Fischer reagent: Two-component of 2 mg/mL capacity
- (3) Stirring time is applied to dissolve the sample
- (4) Delay time is a switch-off criteria for the titrator

Tab. 8: Sample preparation

Sample type	Sample preparation
Films	A piece of 3 cm ² is cut from the film so that it easily fits in the glass boat of the drying oven. The samples must not touch the side of the oven to prevent sticking.
Granules	When the sample cools, it may stick to the glass boat. Place a piece of aluminium foil in the glass boat when working with the manual oven.
Liquid samples	Fill liquid samples into a tube or syringe for direct input to the titration cell.

5. Moisture Determination with the Drying Oven

5.1 Methods with drying ovens

The two important advantages of drying ovens for moisture analysis are flexibility when it comes to sample volumes/sizes and the ability to determine several samples at once.

On the other hand, the time it takes to obtain a result must be taken into consideration. Extended heating periods and cooling phases must be observed with samples being equilibrated in desiccated environments. A final manual results calculation must also occur.

The loss-of-weight method requires a differential weighing approach, i.e. weighing the same sample before and after the drying. The weight difference is evaluated and the moisture content calculated in percent or g/kg. Such working steps tend to be tedious and error-prone.

5.2 Smart solutions for oven-drying issues

The connection of Excellence XPE balances to LabX software provides many opportunities to support accurate before/after weighing. For example, the entire workflow of the drying oven method can be automated which provides a simplified weighing process and removes several potential sources of mistakes (see Fig. 12). Different users can log in to the balance, each starting his or her own Loss on Drying (LoD) method. The user is then guided through the entire process. Although connected to LabX software, the entire method is smoothly run through the terminal of the balance. All methods can be paused at any time, freeing the balance for other laboratory personnel.

All information, such as sample names, batch or lot numbers, initial and final weights, the calculated values, and a simple PASS/FAIL criteria, is automatically stored and archived in the LabX software database. Results calculation is fully automated, avoiding calculation errors. The final results report can be sent effortlessly to an office printer for authorization or client presentation.

Furthermore, the user can choose to print heat resistant barcode labels for each sample container, avoiding handling mistakes and making sample tracking effortless. This can be particularly helpful when handling large sample volumes at high throughput rates.

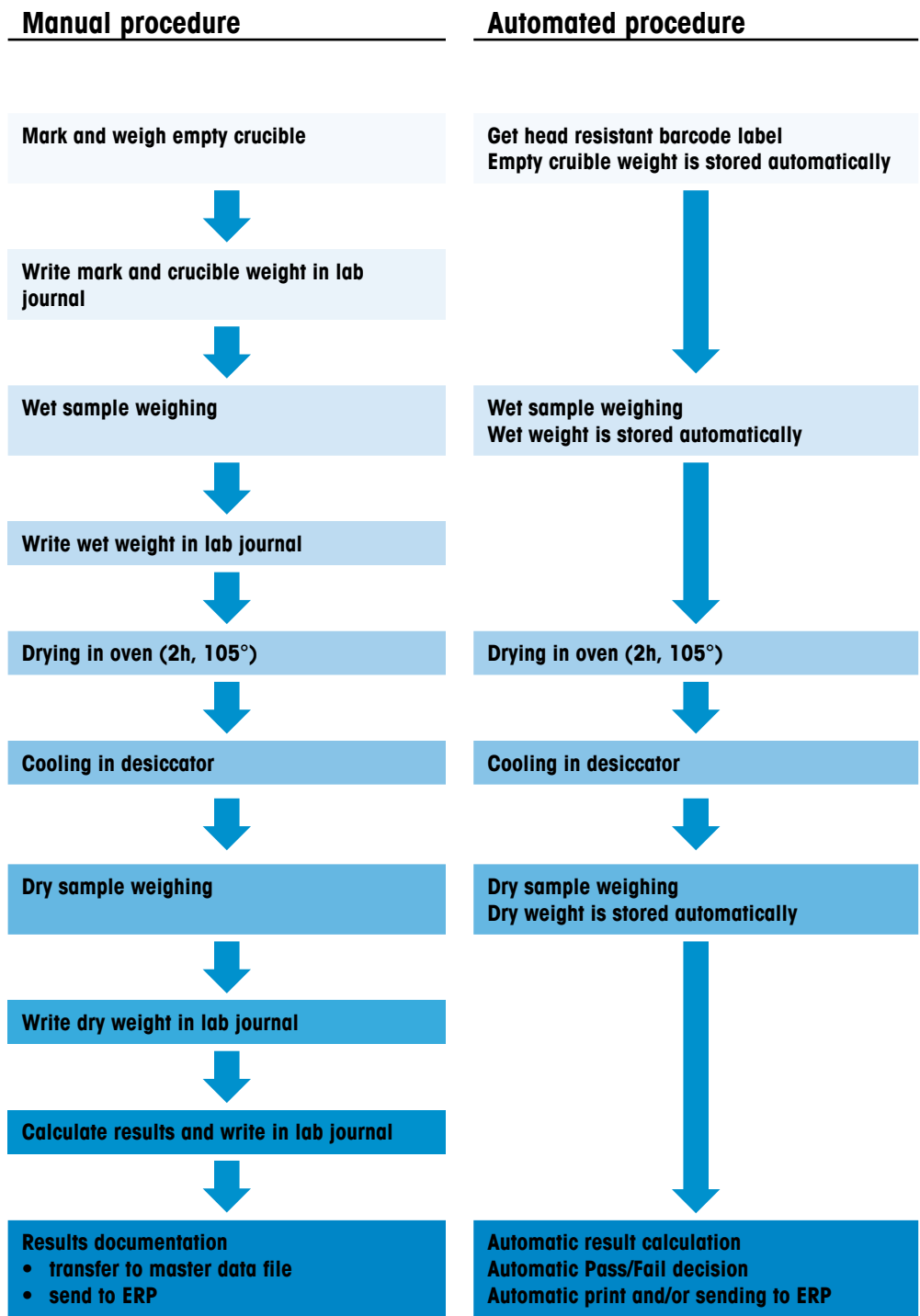


Fig. 12: Comparison of manual with automated procedure. Workflow automation with LabX reduces clearly the number of work steps and eliminates many potential sources of errors.

Other smart solutions can also easily be integrated to provide more user comfort, measuring reliability, and error reduction in and around weighing. The SmartGrid® grid weighing pan is one such solution for Excellence balances. Its design prevents spilled samples falling next to the tare container on the weighing pan and leading to incorrect results.

SmartGrid also ensures that the weighing chamber is extremely easy to clean and that the influence of drafts in the weighing chamber is minimized thanks to its unique open design. Stabilization times, i.e. when measurement data fluctuates before reaching a stable value, are greatly reduced using SmartGrid when compared to conventional balances.

ErgoClips holders nicely amend SmartGrid. ErgoClips are holders that help stabilize sample containers themselves. They in particular are made to fit any tare container imaginable, ensure a secure hold and provide ergonomic, work-friendly container position.



Fig. 13: No measurement errors caused by spills, fast measurement stabilization, and easy to clean: SmartGrid® from METTLER TOLEDO.



Fig. 14: The ErgoClip for crucibles—safe, because crucibles cannot tumble.

6. Summary

6.1 Comparison

The paper provided an overview of the of the most often-used methods for moisture and water determination in plastic and polymer samples. Tab. 9 compares some advantages and disadvantages of the two main methods applied, helping users understand qualification criteria for the method decision and the benefits and potential drawbacks of doing so.

Tab. 9: Advantages and disadvantages of the two main methods

	Moisture Analyzers	Karl Fischer
Advantages	<ul style="list-style-type: none"> • easy handling • easy cleaning, disposable sample pans • large sample volumes possible • one instrument only (less investment) 	<ul style="list-style-type: none"> • specific for water • very accurate • suitable for trace analysis (coulometric) • applies to all plastics and polymers • option for automatic sample heating and changing
Disadvantages	<ul style="list-style-type: none"> • only one measurement/sample can be performed at a time • no automation possible • not suitable for all types of polymers 	<ul style="list-style-type: none"> • use and storage of chemical reagents • need of analytical balance in addition to titrator

In addition to the two primary methods mentioned in Tab. 9, use of drying ovens was also explored.

6.2 Conclusions

Moisture or water content is a universal factor in plastic and polymer product quality. It can be used to indicate quality and processability of raw materials, intermediates, and end products. For example, substances that are too dry may cause problems in production or affect the structure of the end product. If substances are too moist, they may agglomerate or get stuck inside pipes. Thus, samples need to be checked for moisture in a reliable and efficient way.

Moisture determination for plastics and polymers, which typically have a very low moisture content, must be performed carefully. Karl Fischer titration and Moisture Analyzers are common methods for making these determinations.






Each of the presented methods has its own advantages and drawbacks. Karl Fischer titration offers selectivity for water from low to high water content levels, and makes automation possible. Moisture analyzers are unbeaten when it comes to measurement duration, providing sample analysis in significantly less time and with less manual intervention than other methods. The drying-oven method offers advantages for large sample sizes, however.

Smart solutions from METTLER TOLEDO support customers in the polymers and plastics production and processing industry ensuring high efficiency, user comfort, and measurement reliability. All users can find the right solution to meet their individual requirements in terms of accuracy, resolution, time to result, ease of use, reliability and data handling.

7. Instrument Solutions

METTLER TOLEDO's instruments support customers in the polymers and plastics processing industry, providing the right solution to meet high efficiency, user comfort, measurement reliability and other requirements.

METTLER TOLEDO offers an entire portfolio of solutions for widely-accepted moisture content determination methods. Enhanced with smart accessories, these instruments can meet a broad variety of customer requests from easy operation to fully automated analysis.

Method	Solution	
Moisture Analyzer	Halogen Moisture Analyzers HX 204	
	<p style="text-align: right;">HX204</p> Readability (%) 0.01 – 0.001 Capacity (g) 200 Method memory 300 Lid opening motorized More models available	
Karl Fischer titrators	Volumetric titrators V20 and V30	
	Measuring range 100 ppm and up	
	More models available	
	Coulometric titrators C20 and C30	
	Measuring range: down to 1 ppm	
	Oven options DO308 manual, Stromboli automatic oven sample changer	
Drying Oven	An extended offering of various analytical balances cover any application request or budget needs Excellence line: XPE or XSE	
	Any XPE or XSE analytical balance in conjunction with LabX 2014 provides the complete solution	
	New Classic line: MS Any MS analytical balance covers the basic weighing needs	
Thermo gravimetry	The TGA/DSC 1 and TGA 1 models of the Thermal Analysis instruments offer optimal solutions	
	Temperature range: Room temperature up to 1100°C or 1600°C Sample size: up to 200 mg Readability: 0.1 µg or 1 µg	

8. More Information

Karl Fischer application brochures

Application Brochures describe the use of the Karl Fischer titration with all kinds of samples. Recommended sample preparations, detailed method procedures, many result examples and hints about reagents are presented and explained.

- Application Brochure Nr. 38, KF Compactline, METTLER TOLEDO (2012), 51725075B
- Application Brochure, Good Titration Practice™ in Karl Fischer Titration, METTLER TOLEDO (2009), 51725145
- Karl Fischer Guide 1, Introduction, METTLER TOLEDO (2012)
- Karl Fischer Guide 2, Sample preparation, METTLER TOLEDO (2012)

Applications

We offer comprehensive application support for our solutions. If you have a specific application question, you may find the answer in the respective application database.

- ▶ www.mt.com/titration_applications
- ▶ www.mt.com/moisture

Webinars

We offer web-based seminars (webinars) on different topics. You can participate in on-demand webinars at any convenient time and place.

Live webinars offer the added benefit of allowing you to ask questions and discuss points of interest with METTLER TOLEDO specialists or other participants.

- ▶ www.mt.com/webinars

Good Measuring Practices

Five Steps to Improved Measuring Results

The five steps of all Good Measuring Practices guidelines start with an evaluation of the measuring needs of your processes and their associated risks. With this information, Good Measuring Practices provide straight forward recommendations for selecting, installing, calibrating and operating laboratory equipment and devices.

- Guaranteed quality
- Compliance with regulations, secure audits
- Increased productivity, reduced costs
- Professional qualification and training



Good Weighing Practice™

Guaranteed Better Weighing Results—At Your Fingertips with GWP®. GWP®, the global weighing guideline, provides you with sustainable quality and security in your weighing processes.

Good Titration Practice™

Dependable Titration in Practice—Reliable Results with GTP®. A requirements-based selection of the titration system, as well as professional installation and training form the basis for dependable and risk-free titration.

Good Thermal Analysis Practice™

Fast and secure Thermal Analysis Results—With the help of GTAP™ Good Thermal Analysis Practice™ is an approach to improve your lab workflow and preserve accuracy and precision of results.

Other Good Measuring Practices are available for pipetting, pH measurement as well as density and refractive index determination.

Learn more about Good Measuring Practices program

► www.mt.com/gp

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For more information

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