Practical aspects of moisture analysis

Determination of moisture content in a substance seems to be quite a simple measurement, at least at the first sight. As the experience and practice show, the above assumption may be false and result in incorrect measurement results.

The problem with drying relates to the drying device, which is burdened with an error, as there are no ideal instruments. Next error may occur as result of analyzed sample, specifically by methods of its collection and analysis parameters.

Janas Sławomir

Testing Laboratory Manager

e-mail: janas@radwag.pl

RADWAG WAGI ELEKTRONICZNE – TESTING LABORATORY

/ TECHNICAL SUPPORT /

26-600 RADOM, 28 Bracka Street

tel.+48 (0-48) 38 48 800 ext. 536, tel./fax. +48 (0-48) 385 00 10

http://www.radwag.pl

1. Sample preparation for drying

Sample preparation covers two activities. The first one is sample collection, and the other is its storage. The sample storage location is very important here, as it influences the reliability and traceability of measuring results. A sample is a reflection of the analyzed substance as a whole. Both stages (collection and storage) should be performed with identification of sample characteristics.

1.1. Sample characteristics

Sample mass - (depends on substance) should be prepared in such a way, that weighing pan of a moisture analyzer is completely covered with a thin layer. In case of substances that do not release water easy, sample mass has very big influence on weighing result. As humidity is an indirect measurement (calculated from mass reading), it is not recommended to use too small samples, i.e. below 1 g.

Colour – if a substance is dark than its colour may absorb heat, and sample may get overheated. In such case it is recommended to use infrared emitters as source of heat.

Granularity – it is recommended to dry samples in grinded form. Drying results from grinded samples have much lower dispersion and lower drying time.

Substance consistence – it is a characteristic feature of a substance. It should be remembered, that substances with dense consistence (clay, moulding mass) do not release water easily.

1.2. Collection and preparation of various kinds of samples

At this stage, it should be considered how to collect an uniform sample, which will be a reflection of substance as a whole. The substance can be prepared by grinding or mixing before collection of the sample. Common practice gives hint to collect several samples from various areas of a substance or from its defined sections, and calculation of an average value from all samples. Another means of making sure that a sample is homogenous in its from is to mix several samples collected from various sections of a substance.

In case of loose substances like grains, pastas, it is recommended to mix its large portion. Such sample should be crumbled manually (mortar) or mechanically (mill). Due to such sample preparation, measuring results from series of drying processes are repeatable. During milling or mortaring activities heat may be created, and it may influence the sample causing

possible evapouration of moisture. As practice shows, in case of substances with moisture content of dozens of percents, this problem does not influence the sample in a way which may change drying result. At each stage of sample preparation, it should be protected from contact with ambient environment. It can be obtained by storing a sample in hermetic containers. This form of large portion sample should be used for collection of actual sample for drying process. When performing each stage of sample preparation, the operator should be aware of the fact that a sample may absorb humidity from surrounding air or emit humidity to air.

Semi-fluid substances like yogurts, homogenizing cottage cheese, should be mixed, as in most cases they are formed from a few ingredients with various density. More dense ingredients fall down to the bottom of cottage cheese container, and if sample is collected from the cheese top layer, dry mass content will significantly vary from the one collected from the cheese bottom layer.

Liquid substances should be also mixed, especially of their density is different, like in case of oils, concrete additions. Actually, it is assumed, that medium density is homogenous in its all volume, but only mixing provides a kind of warranty of its uniform density.

1.3. Sample storage

Sample preparation process is very important, as the operator has to make sure that the sample does not loose its humidity in contact with ambient air. Sample storage requires usage of hermetic containers. However, the personnel has to check whether ambient area conditions do not affect the sample. In case of hygroscopic substances, humidity must not condensate on container walls. Before analyzing such sample, humidity that has condensed in the container has to be mixed with the sample. Container dimensions have to be adequate to sample size.

2. Moisture analyzer preparation

Generally, it is preparation of a workstation for the scope of performed activities. A moisture analyzer, as any other electronic precision balance, should be positioned is a location that is free from vibrations, are protected from breeze of air. Temperature changes in weighing room in which the moisture analyzer is positioned should be absent or very small. The moisture analyzer should be plugged to mains at least 30 minutes before measurement start. This conditions comes from general rules on operation of electronic balances.

Good practice gives hints on checking the moisture analyzer for its proper operation. Usually it is performed by placing a load with specific mass (e.g. 20 g mass standard) on

analyzer's weighing pan. It is highly recommended if analyzed sample mass is corresponding to mass standard. Another aspect refers to workstation ergonomics. If a moisture analyzer will dry several kinds of samples, it is recommended to set drying parameters for each sample. This option is accessible in moisture analyzers series MAC and MAX offered by RADWAG.

3. Selection of drying parameters

Drying parameters is a list of moisture analyzer settings which includes drying temperature, sample size, drying profile and means of drying process finish. The documents including list of drying parameter settings for various kinds of samples are usually prepared by manufacturer laboratories which made proper tests on each sample drying method. It is quite obvious, as standard regulations and norms refer to traditional drying methods, and not to moisture analyzers. Thus, samples have to be tested by comparison method. Such tests are subject to **qualification process** of drying procedure with use of moisture analyzer.

3.1. Drying profiles

Drying profile determines the way in which drying temperature should be reached. As standard, there are 4 drying profiles, which are described as standard, fast, mild and step. They way each profile is defined is presented on below figure no.1. It should be additionally mentioned, that standard drying profile is set as default one, as it is utilized in most cases.

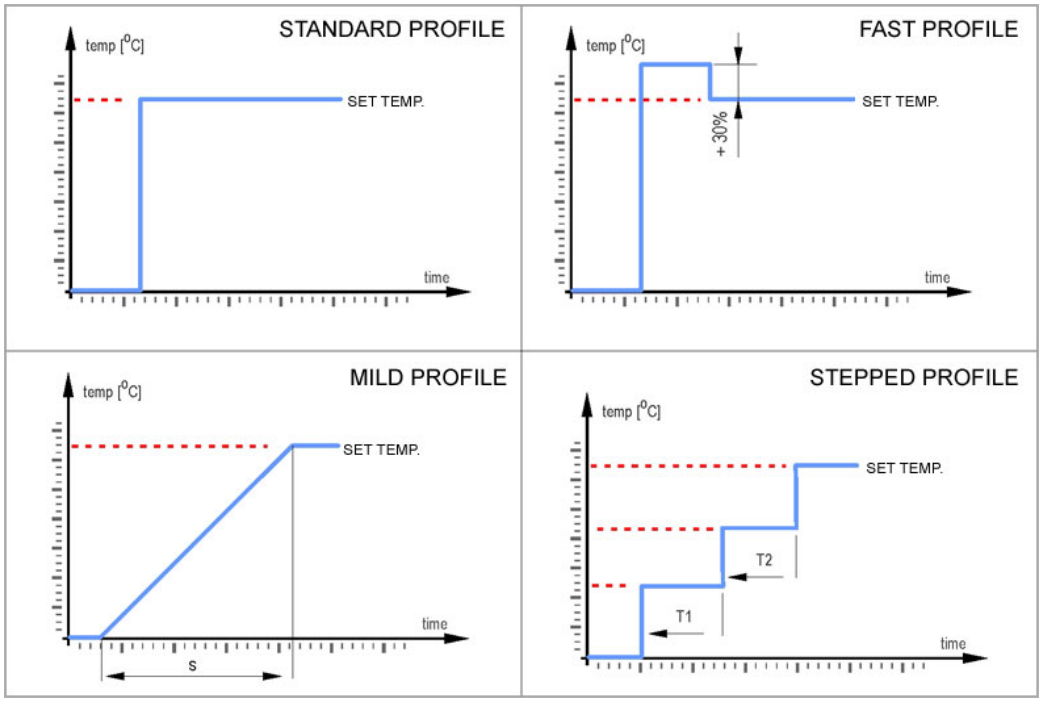

Fig. 1. Drying profiles

3.2. Selection of drying method – drying till obtaining dry mass

Drying procedure with dry mass finish mode is completed if mass of a sample does not change in selected range. In such case sample mass is constant – all humidity has been removed from the sample. This is the most often applied drying procedure in a moisture analyzer. In this drying mode it does not matter if the operator wants to determine dry mass content of humid content. The difference between the two is other means of calculation of obtained drying result. Below please find formula for calculation of drying procedure result:

– Humidity content is the mass that evapourated divided by initial sample mass

$$W[\%] = \frac{(M_P - M_K)}{M_P} \cdot 100\% \qquad [1]$$

where: *W- humidity*
M_P – initial sample mass
M_K – final sample mass

– Dry mass content is current sample mass divided by initial sample mass

$$Ms[\%] = \frac{M_K}{M_P} \cdot 100\% \qquad [2]$$

where: *Ms- dry mass*
M_K – final sample mass
M_P – initial sample mass

Graphic interpretation of drying process with determination of dry mass is presented on figure no.2 below.

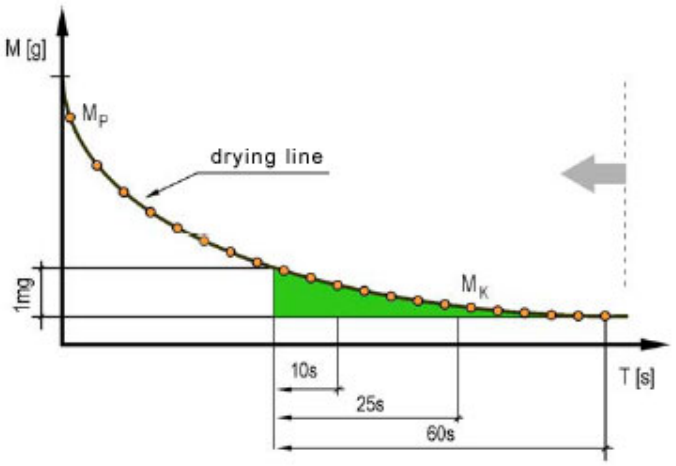

Fig. 2. Drying process with determination of dry mass

3.3. Selection of drying method – drying process in determined time

Some analysis requires control of mass change in a specific time interval and in specific temperature. The result of such analysis may be humidity content, dry mass content or ratio between dry and humid mass. The time interval is set by the operator, who also selects drying profile (means of temperature accruing: fast, standard, etc.).

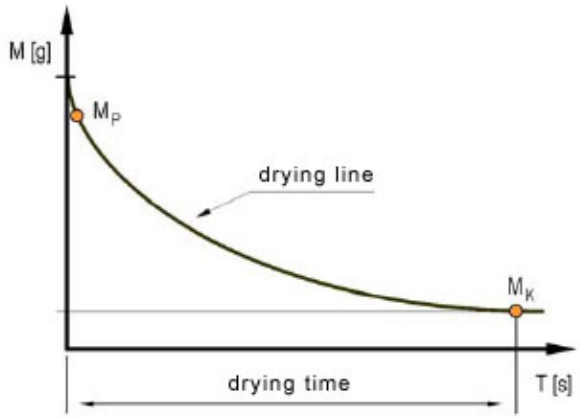

Fig. 3. Drying process in set time interval

3.4. Drying temperature

Temperature is one of the factors which influence final result of drying process. Generally, it is assumed, that drying temperature is the temperature at which a sample is analyzed. Actually, this statement is only partially true. It is a result of moisture analyzer design, which have the control thermometer installed in the upper part of drying chamber. Controlling temperature is performed in a form of feedback:

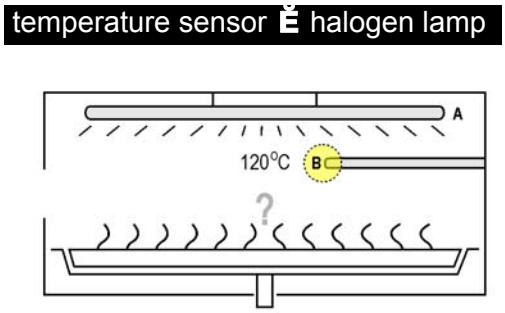

Fig. 4. Distribution of temperature in drying chamber (A - halogen lamp; B – temperature sensor)

Then, what the temperature is during drying process of a sample. At the first stage of drying process, it is definitely lower than set temperature, but within certain time interval is adjusts to set temperature. This delay is the effect of heating process of metal and plastic elements of drying chamber (heat capacity of components).

Nevertheless, sample temperature may differ from drying temperature, and it is an effect of:

- its location (it is lower than the temperature sensor that controls halogen lamps operation)
- its colour and structure (heat absorption)

Thus, when preparing a moisture analysis, the moisture analyzer operators should base on laboratory tests and publications offered by device manufacturer. Manufacturers perform multiple comparable tests and adjust drying parameters, such as drying temperature, to specific sample and type of moisture analyzer. Such approach does not exclude operator's own tests, if there is access to adequate equipment and knowledge.

Independently on type of an analysis and moisture analyzer model, below statements are correct:

- Too low drying temperature causes only partial evapouration of water from a sample (sample is not dry).
- Too high drying temperature causes burning of the sample (sample overheating) and its possible chemical reaction.

Drying temperature in case of traditional method, with usage of an "oven" is specified by corresponding norm. It is too low for drying a sample in a moisture analyzer, and this temperature should be adjusted by experimental tests.

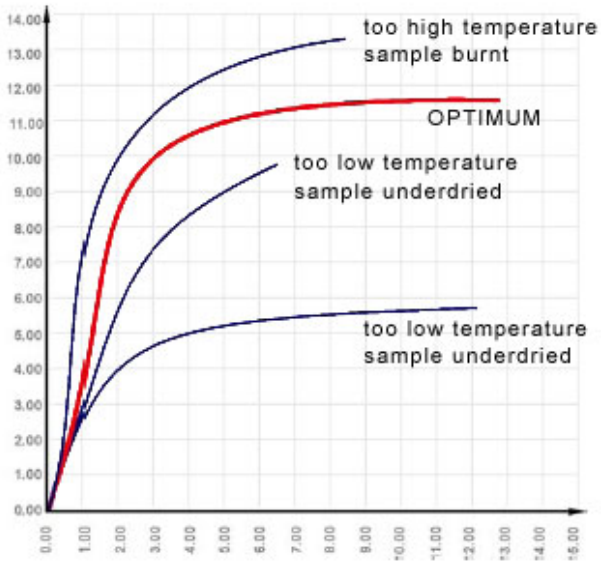

Fig. 5. Temperature influence on result of humidity measurement

3.5. Influence of automatic finish of drying process on result moisture analysis

Drying procedure, as a process, may be conducted semi-automatically (operator initiates the process manually, and the process is finished automatically, by set time interval of mass change), or manually (the operator initiates the process and finishes it by pressing a button).

In manual mode each stage of drying process is clear: start time → stop time → result. In semi-automatic mode, the finish mode should for drying process be determined. It is a relatively important parameter of drying process. Its importance is presented on figure 6. below. By

selection of semi-automatic mode, the operator should choose criteria which will determine the finish of drying process.

Depending on selected criterion, the operator can obtain lower or higher result of moisture content in a sample. The other aspect refers to operator own acceptance criteria for tested sample, e.g. a sample is considered as correct, if moisture content rates between 5% ± 0,3%. If the test refers to a laboratory sample, than result of prying process can be only incorrect. However, in case production procedure, incorrect drying result may lead to sequence of incorrect decisions, and unpredictable economical consequences.

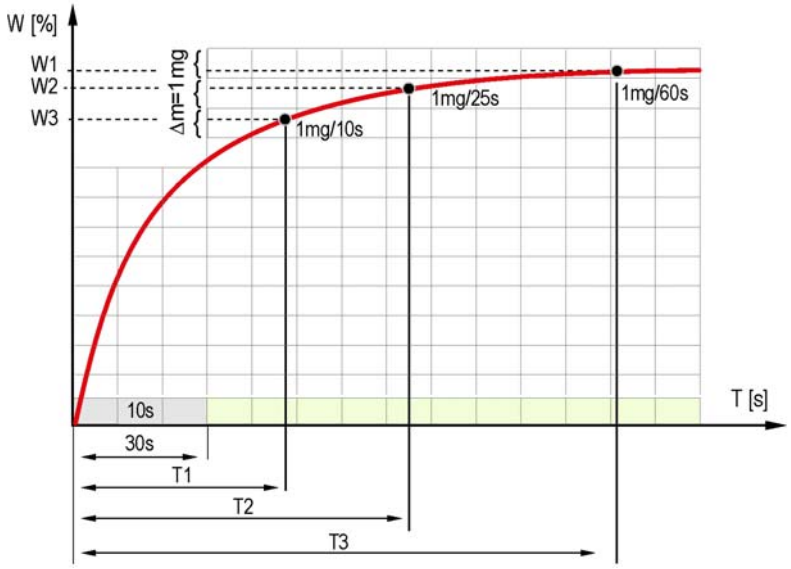

Fig 6. Influence of Auto Finish mode on drying process result

The first stage of drying process includes a 30 seconds interval, which is known as start time, and this time interval is not included in evaluation of auto-switch off mode. Any other measurements are taken into consideration, according to below specification.

- Auto 1 (change of 1mg / 10s)
- Auto 2 (change of 1mg / 25s)
- Auto 3 (change of 1mg / 60s)
- Auto 4 (change of 1mg / 90s)
- Auto 5 (change of 1mg / 120s)

The software controls the changes and check the switch off criterion, for instance if mass change does not exceed set threshold of 1 mg in selected time interval. If this criteria is met, than drying process is stopped. In most cases, auto switch off modes 2 and 3 are applicable.

Selection of long switch of time intervals (e.g. 120 seconds) in drying process, may result in undesired lengthening of drying procedure. In such case oscillations may appear. The oscillation do not affect the drying process result.

4. Auxiliary methods in drying process

Introduction of auxiliary methods is closely related to sample characteristics, like high surface tension, etc. The surface of such sample may create a form of impermeable shell, which result in drying process error. In result, surface of a sample is dried, and its interior is still humid.

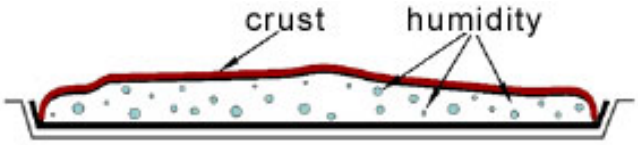

Fig. 7. Sample capping

The only possible solution to avoid such process is to increase evapouration surface. Such effect is obtained by application of high-silica sand or glass fiber. As auxiliary elements also take part in drying process, they have to be initially dried.

5. Drying process

5.1. Solids

Depending on substance structure (compact, loose), the process of humidity determination takes shorter or longer time. Size of solids is the main factor influencing drying speed and measurement reliability. Thus, solid surface should be as big as possible, and for this reason a sample should be evenly distributed on all weighing pan. As solids release humidity through its surface, sample thickness is also very important.

5.2. Liquids and semi-liquids

Semi-liquids are dried in a form they exist. Large amount of grease that occurs in some substances makes drying process difficult. In such cases additional elements should be used for increase of active surface of a sample through which humidity is released.

5.3. Sample location on weighing pan

In order to obtain repeatable drying result, samples should be distributed on all weighing pan in a form of thin layer. A sample should be placed on a weighing pan quickly enough so that there is no humidity loss before drying process. If a sample is distributed randomly, unevenly, than it may result in non-uniform heat conduction to dried substance. In such case a sample **will not dry completely**, and drying time will be undesirably extended.

Fig. 8. Sample distribution
A – uniform (correct); B – non-uniform (incorrect)

6. Sources of errors

Drying process, as any other measuring process is burdened with an error. There are two sources of errors. The first one is the sample, its uniformity, ability to absorb humidity, evapourate during its preparation or capping during an analysis.

The other source of errors is the moisture analyzer, which requires repeatable indications, can be affected by sensitivity and zero drift on temperature change, and requires correct temperature indications from drying chamber. Matching of all above mentioned factors gives an objective assessment of moisture analyzer usability in a specific analysis. Such approach is a must in case of materials having low humidity content, like ABS (Acrylonitrile-Butadiene-Styrene) or PA (Polyamide) and such, which have very low dry mass content (liquids).

7.Moisture analyzer drying method qualification process

It is commonly known, that determination of humidity content with application of moisture analyzer is different process than described in branch norms. This statement gives a clear hint, that it is not possible to use the same settings in two methods specified above. On the other hand, a moisture analyzer is a much faster device, and norms do not specify such case. Thus, which option should be selected?

The solution is **qualification process of drying method**, that is comprehended as activities which aim at confirmation in a documented and compatible with assumptions way, that procedures, processes, devices, substances, and activities do lead to planned results. In practice, the operator should:

- Perform drying procedure with standardized procedure (according to EN norm or any other valid norm) with consideration of sample preparation method, sample size, etc.
- Perform a series of tests of the same substance with application of moisture analyzer, so that obtained drying result is as close as possible to result obtained by standardized method. During tests, the operator should control and adjust:
    - Drying temperature
    - Sample size
    - Means of drying process finish (usually Auto 1- Auto 5)

Sample preparation method is practically the same in case of standardized and moisture analyzer method. Differences may occur in case of loose or compact substances. Generally, it is assumed, that standardized method is more precise than moisture analyzer method. It is a result of static measurements process of dried sample, which is thermally stabilized before weighing. In case of moisture analyzer, weighing takes place in dynamic sample status (it is hot). Additionally, there are air drifts in drying chamber (due to moisture analyzer design).

Bearing in mind above statements, it is not recommended to stick to temperatures specified in standards and norms for moisture analyzer drying procedure. The operator should use publications which have been created during qualification process of drying method with application of moisture analyzer.