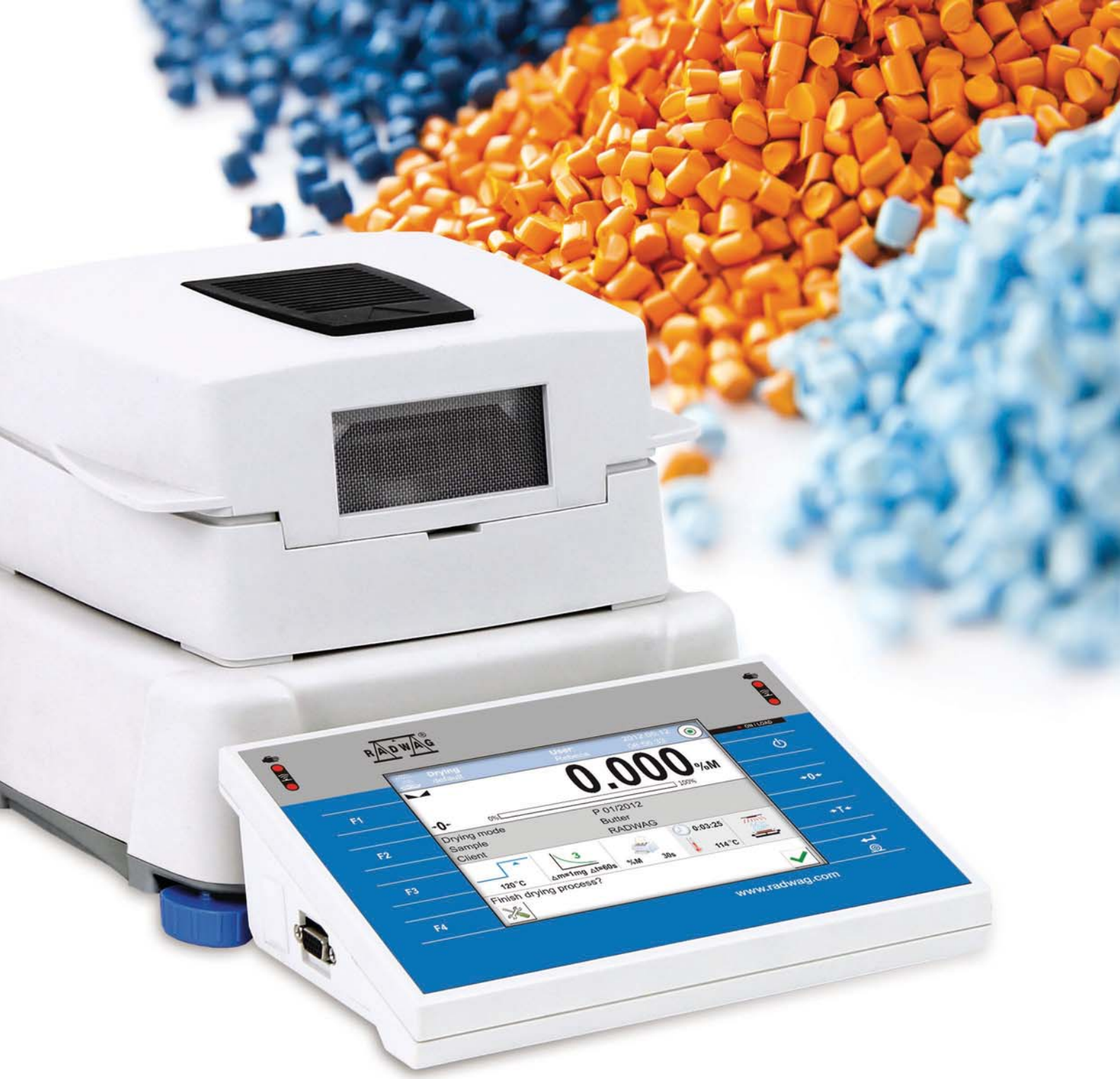

RADWAG moisture analyzers
and moisture content measurement

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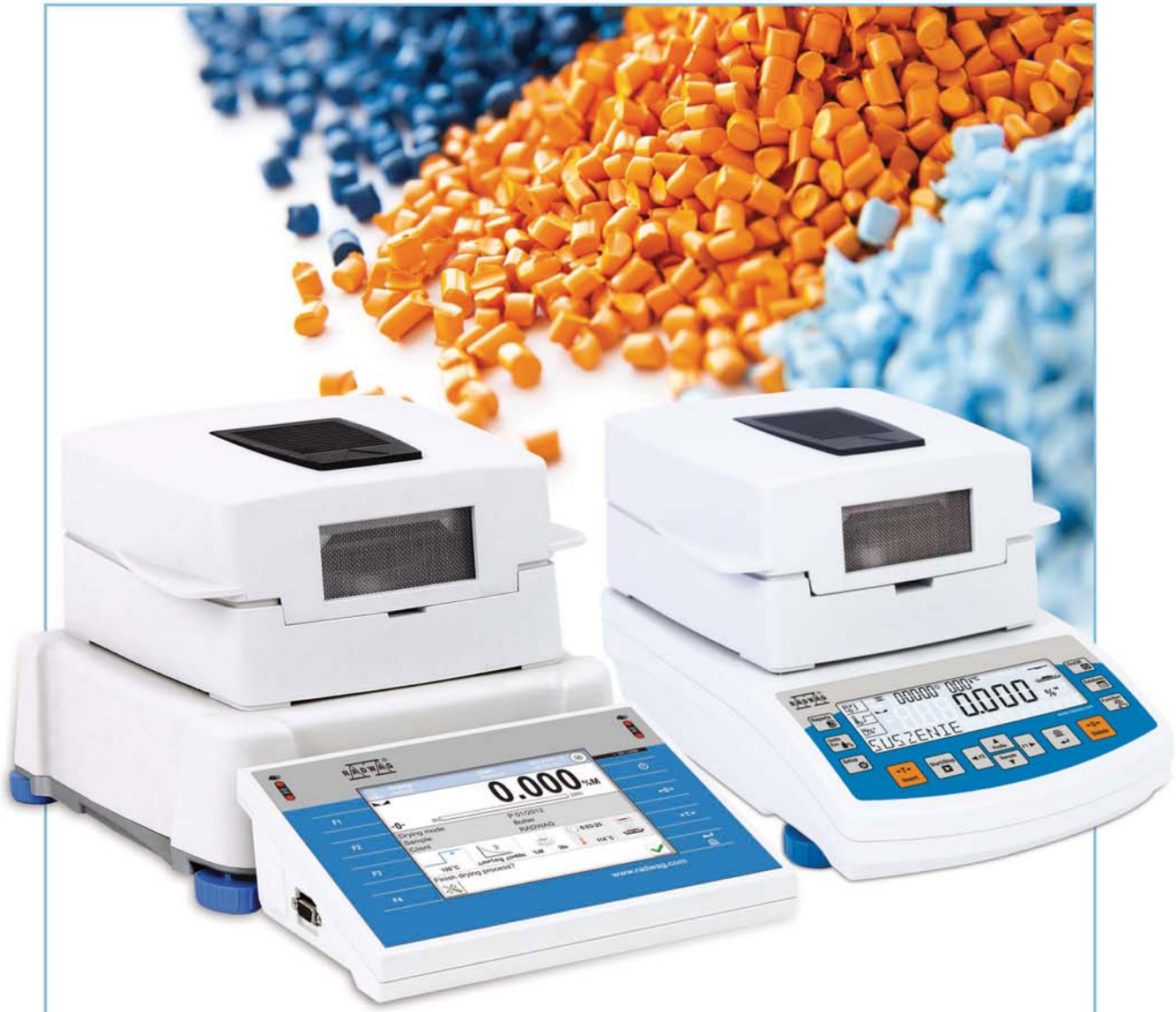


RADWAG moisture analyzers of MA.R, MA.X2.A, MA.3Y series

and moisture content measurement

THE COMPENDIUM OF KNOWLEDGE ON MOISTURE ANALYZERS

addressed to engineers and employees of laboratories, universities of technology and humanities, manufacturing departments and research and development institutions

Radom 2017

Edition I

RADWAG Wagi Elektroniczne
26-600 Radom, Bracka 28
Phone: +48 48 3848800, fax: +48 48 3850010
E-mail: radom@radwag.com
http://www.radwag.com
TIN: 796-000-03-27

AUTHOR'S NOTE

This very publication aims to summarize gained experience and knowledge on moisture content measurements carried out using gravimetric method. Most of the users operate moisture analyzers intuitively, which is quite natural these days when working with devices of this type. The moisture analyzer is an instrument, basic characteristic of which should be the precise measurement. The demanded accuracy is guaranteed when appropriate methods are used. A great variety of tested samples makes it impossible to follow one method only. The measurement method has to be designed with reference to a particular sample type or a product group. This is quite a challenge especially when taking into account the fact that the results have to be referred to tests carried out using reference methods (considered as representative: standard specified, branch enforced). I hope that this publication will provide the readers with detailed information on drying processes, not only those who start to learn how to operate moisture analyzer but also the experienced users.

Sławomir Janas
Testing Laboratory Manager

SŁAWOMIR JANAS - Testing Laboratory Manager who has been working in RADWAG Wagi Elektroniczne for over 20 years. He is an expert on methodology concerning both the mass measurement carried out with adherence to OIML, USP, GMP regulations and the measurements performed by Radwag. Janas organizes and supervises non-automatic scales certification (EN 45501). When it comes to moisture content analysis he is responsible for validation of testing procedures. He is the author of numerous publications on optimisation of the weighing processes, ambient conditions for high resolution scales and moisture content measurement methodology.

CONTENTS

1. Introduction

These days it is recommended to be kind of reserved when trying to select a weighing instrument. Though the particular model may amaze with an interesting design or variety of offered functions (often useless), it is the measurement speed and accuracy that really matter. In case of some weighing devices the most crucial issue when it comes to the said accuracy is methodology, often being developed for years. When the users purchase hi-tech weighing solution, they are also supplied with the right methodology, provided either in a form of a document or highly effective face to face training allowing mutual exchange of information and experience. The trainings help to win customers trust and result with strong bond between the purchaser and the manufacturer. Apparently, distributor is obliged to have thorough knowledge of all technical aspects.

Moisture analyzer is an example of a complex device requiring use of the right measurement methods. Combining weighing and drying processes seems to be an uncomplicated operation however lack of knowledge on the methodology may bring about extremely surprising results. Needless to say, such results make the operator draw erroneous conclusions concerning the used instrument. This calls for the need to explain how such an advanced device as a moisture analyzer operates. It is necessary to focus not only on usage but also on metrological and legal aspects.

2. Significance of the Moisture Content in the Industry

Water is a substantial chemical compound of many raw materials and food products. Water content determines quality, nutritive value and shelf-life period of a particular product. This, along with the fact that food flavour and consistency also depend on accurate water content, is the reason why there are respective standards enforcing the right amount of water and in consequence also food monitoring procedures. Various products hold water taking two basic forms:

Unbound water, i.e. water that is not bound with the product subsurface, it appears mainly on the very surface. Unbound water influences rate of chemical reactions occurring within the particular product simultaneously being the environment for the said reactions.

Bound water, i.e. water that is quite permanently bound with the product. In order to make it evaporate, higher drying temperatures are required. Its characteristics differ from the characteristics of unbound water. In order to test bound water content it is possible to use plaster stone.

Information on moisture content of a particular product is barely an issue of interest itself, nevertheless analysis of it may lead to interesting conclusions concerning the following features:

- clump and cake properties (powders),
- microbiologic stability,
- flow properties, viscosity,
- dry mass content,
- product concentration and purity,
- class (conformity with standards),

- possibility to use the material in a manufacturing process ,
- product's nutritive value,
- conformity with technical, standard and legal regulations.

In industry various methods are used for determination of particular sample's moisture content. Regardless of the method, speed and efficiency are both required, this enforces specific technical solutions. When it comes to food products it is possible to employ:

a. thermal drying methods,
b. azeotropic distillation method,
c. densitometric methods,
d. refractometric methods,
e. chemical methods,
f. electrical methods,
g. nuclear magnetic resonance method (NMR).

Diversity of methods is a result of technological development and innovations. Results obtained using two different procedures should be comparable to the real value no matter how much the mechanical designs of the used devices differ. In order to test and prove that a particular procedure is correct, the obtained results must be compared with results provided using reference methods (standard-specified). Such approach is a part of the validation process. The tests are carried out for one and the same sample, prepared and stored as specified by respective regulations. Among standard-required methods used for moisture content determination there are:

- moisture analyzer method (convection heating of the sample),
- Karl Fisher titration method

Moisture analyzer method is a process consisting in weighing, drying and reweighing of the analysed sample. This requires both weighing device and oven with adjustable temperature. The method is characterized with its own set of rules regarding samples preparation. In order to determine quantity of water molecules using chemical reaction, coulometric or volumetric titration is applied here.

Titration – chemical type of quantitative analysis consisting in controlled adding of a titrant (solution of known concentration) to the tested solution containing analyte. The titrant may be added using burette for example. Observation of changes occurring in the course of the process enables to determine concentration of the particular substance in the analyte.

Apart from the above described methods there are many solutions using microwave. Drying process carried out by means of this method is based on rotation of sample's dipolar molecules (water mainly). The molecules align parallel to the electric field forces. Enforced rotation of the sample or electric field alternation results with dipolar molecules relocation, which in turn leads to molecular friction. As a consequence thermal energy is emitted within a sample and moisture content gets reduced.

Regardless of used moisture content analysis method, all the obtained results shall be comparable and likewise. This leads to a conclusion that method-related technique may be a crucial issue. And indeed it is so especially when it comes to moisture analyzers, which as portable universal devices

are operated in the laboratory, production hall and numerous institutes, organisations and places.

3. The Notion of Moisture Content

Depending on the used method there may be plenty of various approaches to the notion of moisture content. Methods based on thermogravimetric analysis are characteristic for the process of volumetric heating of the sample. Due to the said heating, components such as water, fat, aromatics, organic dis-solvents, chemical additives and components being a result of thermal decomposition are released. Practically speaking there is no possibility to set apart loss of unbound water from loss of other substances. Therefore it may be concluded that for this method the notion of moisture is defined as group of all sample components which evaporate in the course of the heating process. Realising the above enables to objectively assess usefulness of the moisture analyzer operated as a device intended for moisture content analysis.

When determining moisture content it is actually necessary to determine dry mass content. Dry mass is a substance obtained upon evaporation of water and other components from the sample and it informs on the real state of the sample upon performed drying process.

4. Moisture Analyzer as a Versatile Device

Thermogravimetric analysis used for moisture content determination requires mass measurement to be carried out at least twice. In case of some of the standard-specified methods, the balance and the oven are approached as separate devices. Such balance-oven set is rather a stationary than portable instrument, which might be a great disadvantage for some users. Moisture analyzer being a device comprised of two integral modules, i.e. a weighing module and a heating module, may be prised as a dual-function solution. By means of it, the operator can precisely determine mass of any sample, the one that is to be dried and the one that is to be weighed without being subjected to the drying process. However, the drying chamber influencing the moisture analyzer design, makes the device drastically different form typical weighing instruments offered on the market, and it imposes some limitations. With the heating module turned on, it is possible to determine moisture content automatically. The moisture analyzer enables simultaneous performance of two interrelated operations - mass measurement and temperature measurement. Such functionality is desired by numerous specialists operating in many areas of economy: chemical industry, agriculture, etc.

Photo 1 Moisture analyzers of MA.R, MA.3Y series
(in the background: IR – emitter, HAL – halogen lamp)

4.1. Mass Measurement

In the course of mass measurement carried out using the moisture analyzer, or any other weighing device comprising an electromagnetic converter, it is necessary to calculate gravity force:

$$F = F_G = m \cdot g$$

m – sample's mass
g – gravitational acceleration

Usually upon shipping, the moisture analyzer requires readjustment. This is conditioned by a huge difference between values of gravitational acceleration for place of production and place of use.

$$F_P = mg_P \neq F_U = mg_U$$

g_P – gravitational acceleration for the place of production
g_U – gravitational acceleration for the place of use

In most cases the adjustment is carried out by means of an external mass standard of appropriate accuracy class. Successfully completed readjustment is a warranty of precise mass measurement.

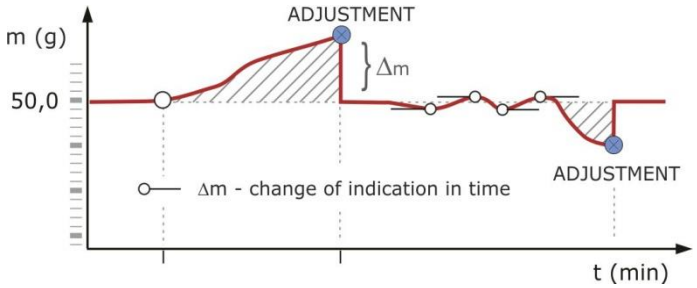


Figure 1. Moisture analyzers of MA.R, MA X2.A, MA.3Y series

NOTE

Each weighing instrument must be subjected to an adjustment process in a cyclic manner. Otherwise certain indication deviation may occur (Δm). Regularly carried out adjustment helps to completely eliminate potential deviation occurrence, often caused by ambient conditions variation. Due to this the balance can display precise indication all the time. Most balances of high resolution (more than 2 000 000 divisions) feature fully automatic adjustment mechanism. This solution ensures accuracy regardless of the adjustment frequency. Because of differential nature of the measurement (M2 – M1) there is no need to provide such high accuracy in moisture analyzers.

If the moisture analyzer is to be used for precise measurement of sample's mass then it is necessary to subject it to the adjustment procedure repeatedly within specified time intervals. One last issue concerning moisture analyzer adjustment are mass standards. While selecting the mass standard (adjustment weight), the operator must make sure that he/she pays attention to the reading unit [d], also referred to as a scale interval or readability. Mass standard - scale interval relation is presented in table 1.

	Accuracy class		
Mass standard	E_2	F_1	F_2
50 g	± 0.1 mg	± 0.3 mg	± 1.0 mg
100 g	± 0.16 mg	± 0.5 mg	± 1.6 mg
200 g	± 0.3 mg	± 1.0 mg	± 3.0 mg

Table 1 Maximum permissible errors by OIML R 111-1

Model	Scale interval (d)	Mass standard	Option (*)
MA 50/1.R	0.1 mg	E_2 50 g	F_1 50 g
MA 50.R	1 mg	F_1 50 g	F_2 50 g
MA 110.R	1 mg	F_1 100 g	F_2 100 g
MA 210.R	1 mg	F_1 200 g	F_2 200 g
MA 60.3Y	0.1 mg	E_2 50 g	F_1 50 g
MA 200.3Y	1 mg	F_1 200 g	F_2 200 g

Table 2 Mass standards used in the course of adjustment

Table 1 provides maximum permissible errors for weights. Real error values for a particular mass standard are to be found on a calibration certificate, they are much lower than MPE. Table 2 provides two class variants. In case of the first variant, influence of mass standard's error on the adjustment result is eliminated. For the second variant (optional), relations occurring in the course of adjustment are referred to.

Mass value of the adjustment weight in most cases is comparable to max capacity value. Potential error resulting from mass standard’s deviation practically does not influence accuracy when weighing loads within range of up to maximum capacity.

Photo 2 Adjustment of MA 110.R moisture analyzer (100 g mass standard)

When selecting mass standard for the adjustment it is necessary to take into consideration moisture analyzer's weighing range. Weighing tolerance is another crucial information to be considered. Relation between these two has been presented in figure 2.

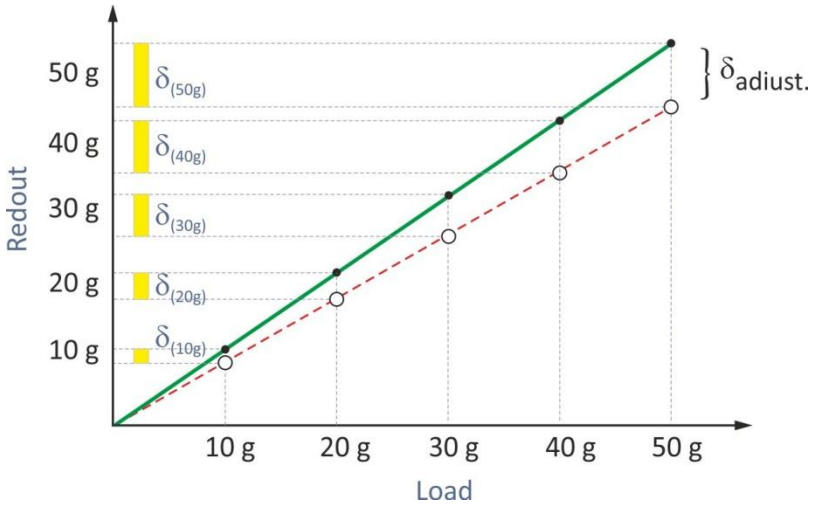


Figure 2. Influence of adjustment deviation on indication accuracy

Figure 2 presents theoretical effect of adjustment inaccuracy being a result of use of mass standard of too great deviation. Yellow rectangles inform on indication error value for various loads. The value decreases linearly. It is greater for heavier loads and smaller for light loads. If the moisture analyzer is to be used for precise weighing of the samples this relation may be used to estimate measurement accuracy. Analysis of the above graph lead to one important conclusion that is true for all electronic devices. Namely, the lighter the sample is the less significant influence of adjustment deviation on measurement accuracy. Therefore in case of light weights the decisive parameter when it comes to measurement accuracy is repeatability not sensitivity.

NOTE

Legal metrology regulations concerning MPE (OIML R 111-1) refer to weights only. They specify characteristics such as shape, dimensions etc. The legal metrology gives no limitations for mass standards. These are defined by stability of mass over time, deviation and uncertainty. When speaking of an external adjustment weight used in the course of adjustment process, nothing else is meant but a weight of particular accuracy class with a calibration certificate. An internal adjustment weight (in-built), contrary to the external one, is a typical mass standard. Its features such as shape and dimensions do not adhere to OIML R-111-1 regulations.

Maximum capacity (Max)	50 g ÷ 200 g
Readability [d]	0.001 g or 0.0001 g
Tare range	0 ÷ Max
Adjustment	external adjustment weight
Repeatability (SD)	0.001 g or 0.00015 g
Linearity	± 0.003 g or 0.0006 g
Sensitivity stability	2 ppm/°C (15 °C ÷ 35 °C)
Stabilization time	ca. (2.5) sec.

Table 3 Mass measurement related parameters

SUMMARY

MASS MEASUREMENT

Measurement of sample's mass carried out by means of a moisture analyzer requires the device to be subjected to periodical adjustments. Time interval between successive adjustments cannot take too long. Since ambient conditions powerfully influence stability of indications, therefore adjustment should be performed once a day. It is not necessary to adjust MA.3Y and MA.R moisture analyzer by means of the same mass standard as the one that was used in the course of a manufacturing process. Moisture analyzer's menu enables the operator to use freely selected adjustment weight, e.g. instead of 50 g he/she can apply 20 g mass standard. When taking advantage of this option, refer to figure 2 remembering that reverse relation is applied. In case of light adjustment weight, even minor adjustment error may cause risk of significant indication errors for loads greater than 50% of maximum capacity. An exception would be situation where sample's mass is stable all the time and its value is comparable with value of the mass standard which had been used for adjustment performance.

4.2. Moisture Content Measurement

Moisture content of a particular sample is calculated on the basis of two mass measurements, initial (M1) and final (M2 – sample's mass obtained upon drying) using the following equations:

$\%\mathrm{M} = \frac{\mathrm{M1} - \mathrm{M2}}{\mathrm{M1}}\ 100\%$	$\%\mathrm{D} = \frac{\mathrm{M2}}{\mathrm{M1}}\ 100\%$	$\%\mathrm{R} = \frac{\mathrm{M1} - \mathrm{M2}}{\mathrm{M2}}\ 100\%$
sample's moisture content (relative humidity referred to wet mass)	dry mass content	moisture / dry mass (absolute humidity referred to dry mass)

$$\boldsymbol{F = (m_1 - m_i) \cdot g}$$

m_1 – start mass value,
m_i – temporary mass value
g – gravitational acceleration

On the basis of the obtained results it is possible to determine fat content for pork and beef. This is calculated with use of a dedicated algorithm.

According to what has already been mentioned, precise weight value is not required when it comes to start mass of a sample. As for the end mass, it is an effect of sample's thermal state (heat level). Therefore it can be concluded that TEMPERATURE is the factor determining how great the moisture content loss is. Since it is the thermal state that decides on how much moisture evaporates form the sample, therefore the above conclusion should not be a surprising fact.

It is not a secret that the temperature value, chosen experimentally, differs depending on the sample type. In case of some sample types an impermeable layer is formed on the surface in the course of the heating process, it is so called crust. The crust prevents moisture content evaporation which calls for application of respective drying methodology. All sample-related problems, if correctly diagnosed, can be solved through right sample selection, preparation and storage, and with use of respectively designed drying process.

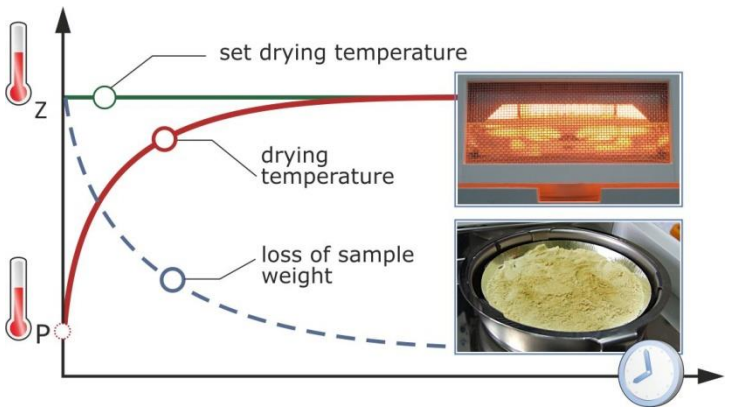


Figure 3. Changes occurring in the drying chamber during the drying process

Graph presented in figure 3 demonstrates typical drying process where sample's mass loss is recorded along with temperature growth. There are two factors deciding on effectiveness of the process: temperature and sample's ability to give up moisture content. Apart from the drying process temperature it is necessary to specify many other parameters: sample size, finish mode, drying profiles, etc. These are commonly known as DRYING PROCESS PARAMETERS and should be optimised either in the course of moisture analyzer validation or during validation of new samples. Optimisation aims to show that results obtained by means of moisture analyzer are comparable to the reference methods results. The way the operator deals with the particular sample is often of great importance. For more detailed information with regard to this read the following subchapters.

Maximum sample weight	Max
Recommended minimum sample weight	ca. 2 g
Moisture content readability	0.0001 % or 0.001 %
Moisture content repeatability	+/- 0.05% (sample ca. 2g), +/- 0.01% (sample ca. 10g)
Disposable pan dimensions	ϕ 90 mm
Drying temperature	40°C ÷ 160°C or 40°C ÷ 250 °C
Drying temperature accuracy	+/- 2°C
Finish mode	automatic, manual, time-defined, user-defined
Heating module	IR emitter (400 W) /option: halogen lamp, metal heater/
Workspace	Maximum sample height - ca. 20 mm
Drying method	drying profiles /standard, fast, mild, step/

Table 4 Moisture content measurement related parameters

When analysing parameters referring to moisture content measurement, it is advisable not to follow some common statements saying that moisture content readability equals scale interval (reading unit [d]). Such a generalization is nothing but a great mistake. There are more than just one factor influencing accuracy of moisture content measurement therefore:

Moisture content readability [%] ≠ moisture content measurement accuracy [%]

NOTE

The above paragraph points to a conclusion that temperature set to heat the sample in the course of the drying process is a crucial parameter. Remaining ones are of minor significance although they can affect the drying result significantly. It is known that mass measurement requires periodical test confirming indication accuracy. Such test consists in comparison of displayed indication with weight value of mass standard put on the weighing pan. As for moisture content measurement, the most critical moisture analyzer's parameter to be subjected to periodical control is of course the drying temperature. It is tested using control thermometer approved with calibration certificate. RADWAG provides customers with both services and control sets comprising control thermometer compatible with RADWAG moisture analyzers. For detailed control procedure refer to section 13 of this publication.

Control thermometer is not one and only option, there are some alternative solutions enabling drying temperature control. One of them is set of samples for with moisture content is potentially known, these are so called zeolites dried at specified temperatures. This method however has drawbacks, namely the result is considered to be correct only if it is comprised within 1% tolerance. Thus, it is rather hard to speak of any control of drying process accuracy but more like of checking whether the devices operates properly or not. From metrological perspective use of zeolites is irrelevant since the zeolite-based test does not allow to clearly determine accuracy of moisture content measurement. Distributors of zeolite samples are bound to oppose the above statement but even brief analysis of documentation (e.g. user manual) leave the reader with no doubts.

Zeolite-based testing of moisture analyzer indication would be possible if certified moisture content standards existed. No such standards are available on the market so far. In countries other than EU Member States the drying accuracy test is carried out using silica sand and distilled water samples, which due to appropriate neutrality level allow the operator to determine the said accuracy. Example:

- silica sand weight = 10 g, distilled water weight = 2 g
- M1 = 10 g + 2 g = 12 g (water-sand mixture)
- M2 = 10 g (water evaporates in the course of the drying process), therefore the moisture content equals:

$$w = \frac{12\ \text{g} - 2\ \text{g}}{12\ \text{g}}\ 100\% = 83{,}3\ \%$$

All that needs to be done is simply to compare the obtained moisture content result with a theoretical value. It is possible to test the whole measuring range, to do it various proportions of the components (water/sand) have to be used. Such test, according to Central Office of Measures methodology, is based on 5 measuring points. In practice there is no need to perform such detailed control.

Photo 3 on the right presents RADWAG-recommended control thermometer set. Such set was introduced in 2012 and it has been applied ever since that time. Older moisture analyzer models offered a bit different solution. The thermometer probe was inserted into the drying chamber through a dedicated opening, which was to be found in a front of the drying chamber.

Photo 3. Control thermometer used with MA.R/MA.3Y moisture analyzers (available from November 2012)

Control process aiming to test drying temperature has individually set tolerances which is nothing unusual. In case of moisture analyzer, the tolerance values are specified with reference to the device's weighing capacity and thermal gradient of a drying chamber. This is demonstrated in figure 4.

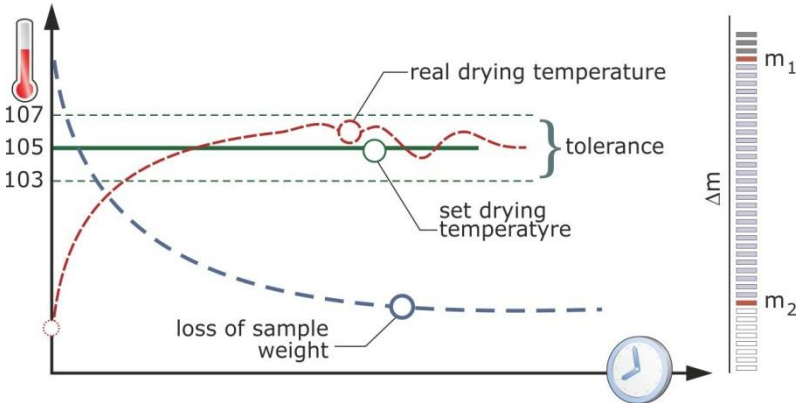


Figure 4. Drying temperature control

SUMMARY
MOISTURE CONTENT MEASUREMENT

If the moisture analyzer is used for moisture content measurement exclusively, then there is no necessity to scale its indications by means of adjustment carried out using mass standard. Error, when it comes to measurement of sample's start mass, even of 0.2 g value, exerts no effect on the final moisture content result. This value is calculated on the basis of difference of mass value indications. Due to the above, majority of moisture analyzers lack so called inbuilt internal adjustment.

Variation of the drying temperature in relation to set temperature, of value no greater than $2^{\circ}C \div 3^{\circ}C$, does not influence significantly the result of sample's moisture content. Because of this fact, also reference methods (EN/PN) provide required drying temperature in the following form 130 $^{\circ}C$ ($\pm 3\ ^{\circ}C$), for example. In order to specify what is the value of drying temperature deviation, which affects moisture content indication, a number of tests needs to be performed. However two facts may be stated, namely:

- too low drying temperature = insufficiently dried sample, moisture content value too low in relation to the true value,
- too high drying temperature = sample burning, moisture content value too high in relation to the true value.

Heat absorption by samples of dark colour is of great significance.

5. Functionality of MA.R / MA.3Y Series Moisture Analyzers

Users of various devices find both accuracy and ergonomics as the most important features when it comes to the operation. Particular application must be accessed quickly and intuitively, the more automated the access the better. Convenient software - hardware combination characterized with all the above mentioned features is a key factor guaranteeing success when speaking of distribution and spread of particular standards regarding functionality.

In case of Radwag-manufactured moisture analyzers we can speak of two different levels of functionality. THE FIRST allows the user to quickly start operation with any type of sample, enabling him or her to select the following:

a. drying profile
b. drying temperature
c. finish mode option
d. units (%M, %D etc.)

Structure of MA.R moisture analyzers menu is of a cascading nature – modification of one of the parameters requires preview of all the others.

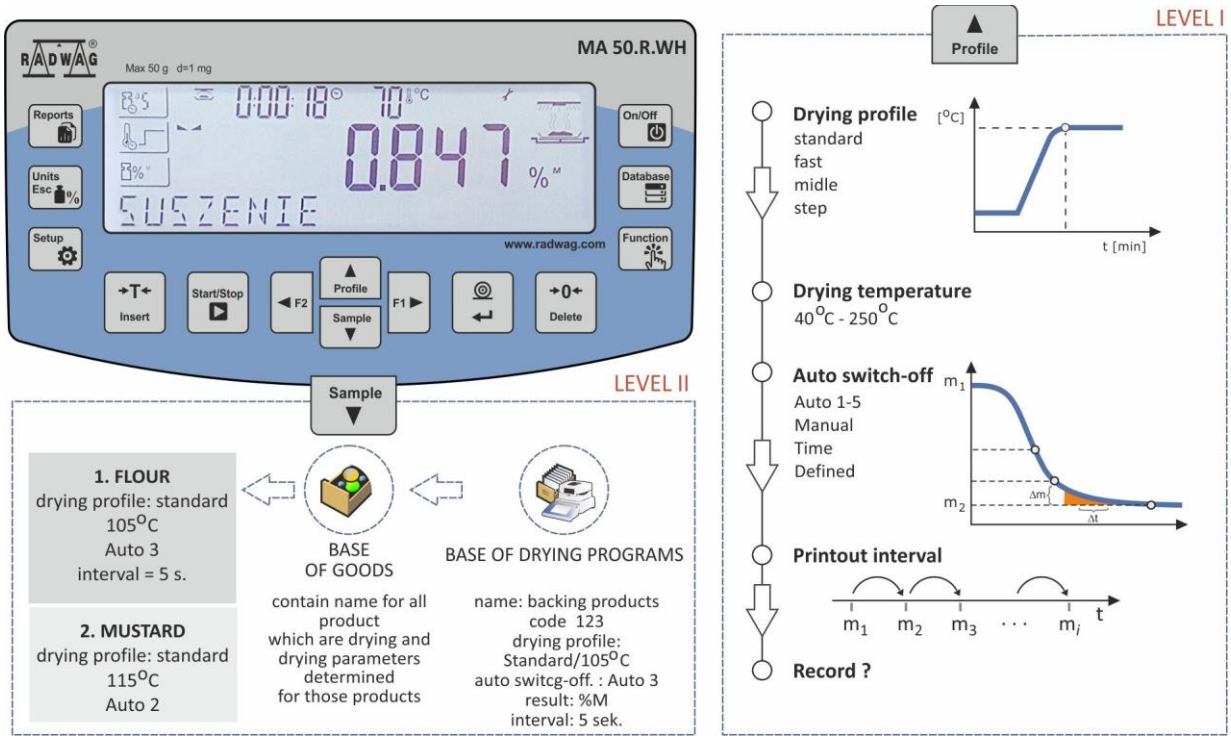


Figure 5. MA.R moisture analyzer – drying operation menu

THE SECOND functionality level is based on the use of moisture analyzer databases such as PRODUCTS and DRYING PARAMETERS. The databases allow the operators to define all products that can be subjected to the drying process, and to set all parameters related to the drying process. Every single data is interrelated one to another which allows automation of vast range of activities. All the operator has to do, is simply to select particular product, the drying parameters are set automatically.

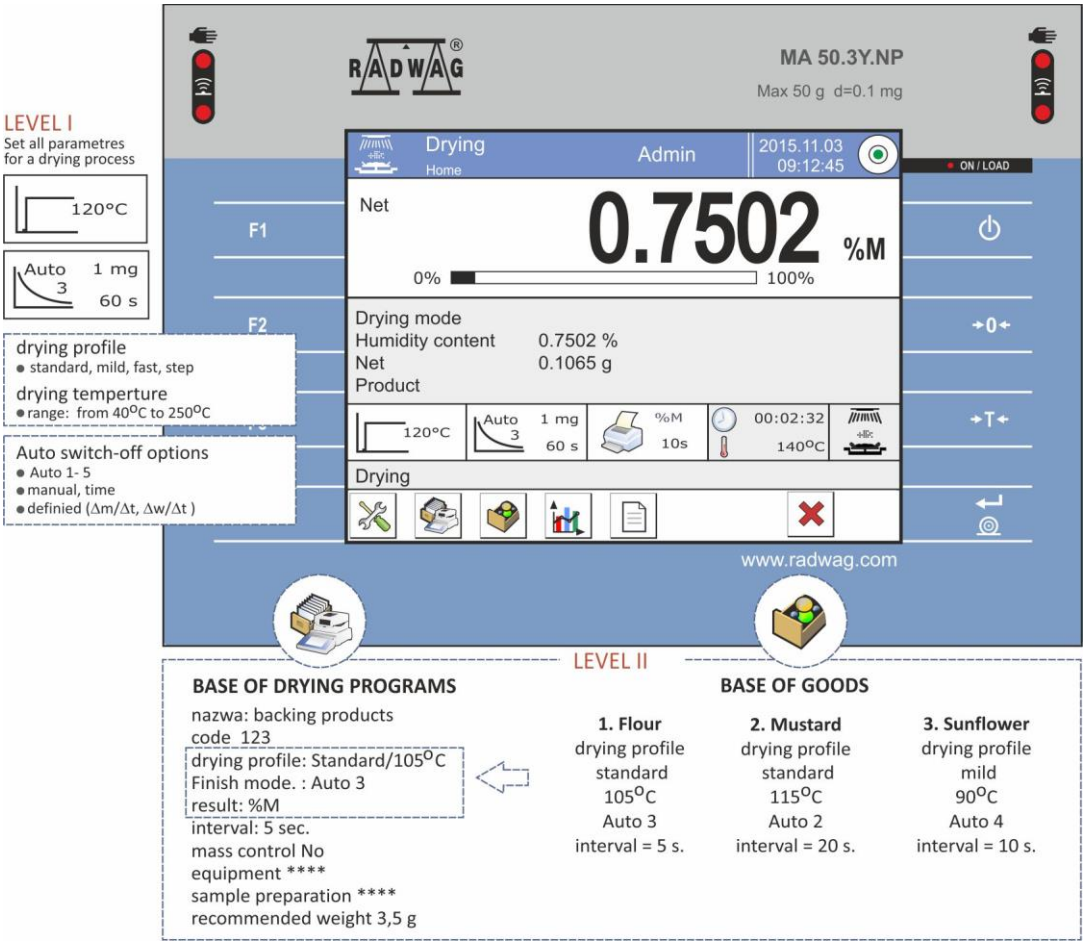


Figure 6. MA.3Y moisture analyzer – drying operation menu

Moisture analyzers of MA 3Y series have been equipped with a graphic interface. It simplifies the selection of drying profile, auto switch-off option etc. Needless to say, it also greatly improves readability, and makes operation of the analyzer intuitive and ergonomic.

As for the MA.3Y, the second level of functionality, just like in case of MA.R series moisture analyzer, also uses databases, drying programs databases among them. Methodology designed for the LEVEL II proceeds as follows:

a. the process of defining a product in the Database by specifying and providing its:
   - name
   - code, EAN code
   - mass
   - description
   - tolerance etc.
   - drying mode name
b. the process of defining drying program in *Drying Programs* database by specifying:
   - drying temperature
   - drying profile
   - finish mode
   - mass control, and by
   - preparing the sample and equipment (description)
c. the process of selecting sample that is to be dried (drying parameters will be set automatically).

The drying process can be supported by software in many different ways. It has already been said that validation of moisture analyzer method, i.e. adjustment of the device parameters aiming to ensure that the obtained results are comparable to results of reference methods, is a good solution. Successfully completed validation as a result gives highly specific drying parameters. The parameters can be permanently saved to moisture analyzer memory, next the operator can assign them to a particular product. Product description provides any information regarding the product mass, weighing tolerance, code, EAN code etc. With this, search for a given sample in the database is easy and takes little time. At the very moment when a given sample is selected, relevant drying parameters are set (drying modes database).

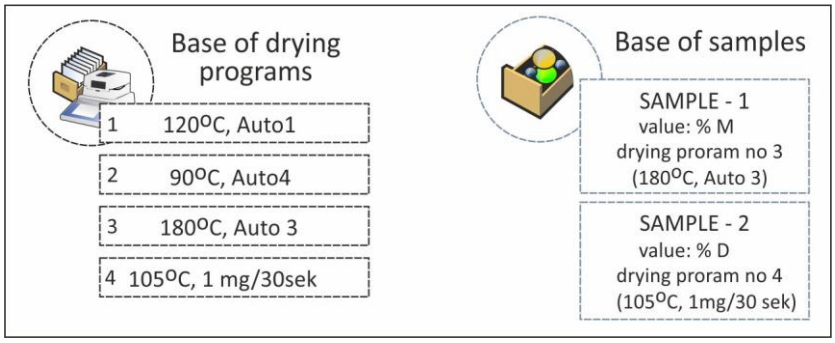


Figure 7. Drying programs and products databases

Drying process results are registered, sent and recorded in the moisture analyzer memory in a real time. On the basis of the registered data, graph and report (MA.3Y) on the drying process are generated. The report is a document which allows to verify the sample and its real moisture content.

----------Drying Process Report----------	
Date	26.10.2015
Time	12:41:03
Operator	Nowak
Product	Sample - 1
Program	Temperature -120
Drying process parameters	
Profile	Standard
	120°C
Finish mode	Auto1
	1mg/10s
Result	%M
Interval	5s
Start mass	2.814 g
0:00:00	0.000 %M
0:00:05	0.000 %M
0:00:10	0.036 %M
. . . . .	
0:08:15	11.052 %M
Status	Completed
End mass	2.503 g
Result	11.052 %M

The report on the drying process is permanently stored in the moisture analyzer memory, therefore it can be printed any time. The operator can preview, print and export any selected report.

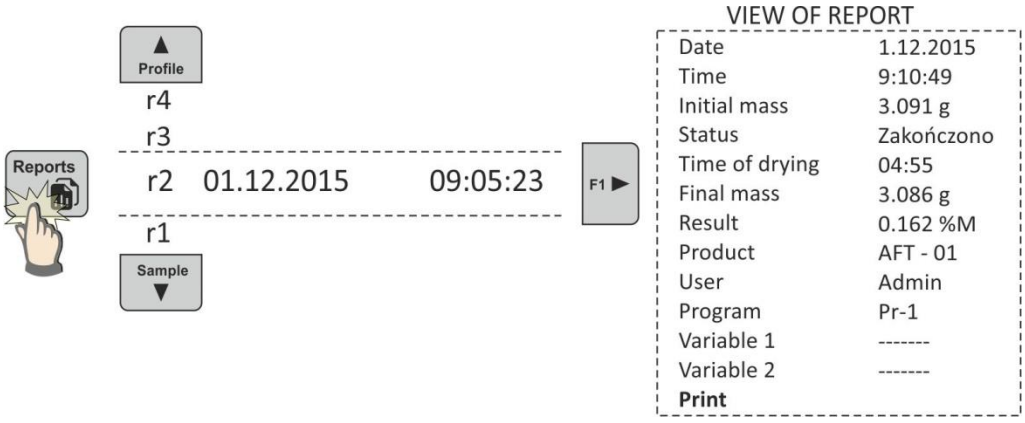


Figure 8. Preview and selection of the drying process reports – MA.R moisture analyzer

The report in preview mode features **Print** option, which when pressed provides the operator with more detailed information.

----------Drying Process Report----------

Date	01.12.2015
Time	9:10:49
Operator	
Product	
Program	
Drying process parameters	
Profile	Standard
	200°C
Finish mode	Auto5
	1mg/120s
Result	%M
Interval	30s
Start mass	3.091 g
0:00:00	0.000 %M
0:00:30	0.065 %M
0:01:00	0.065 %M
0:01:30	0.065 %M
0:02:00	0.097 %M
0:02:30	0.097 %M
0:03:00	0.129 %M
0:03:30	0.129 %M
0:04:00	0.162 %M
0:04:30	0.162 %M
0:04:55	0.162 %M
Status	Completed
End mass	3.086 g
Result	0.162 %M

Example of a report on a completed drying process

If one and the same product is subjected to the drying process numerous number of times then graph of moisture content trend over time is generated automatically (only MA.3Y).

Such graph is one of the components of production cycle monitoring. Automatically generated trend graph is an option for the operator's use only in case he or she selects the product stored in the database.

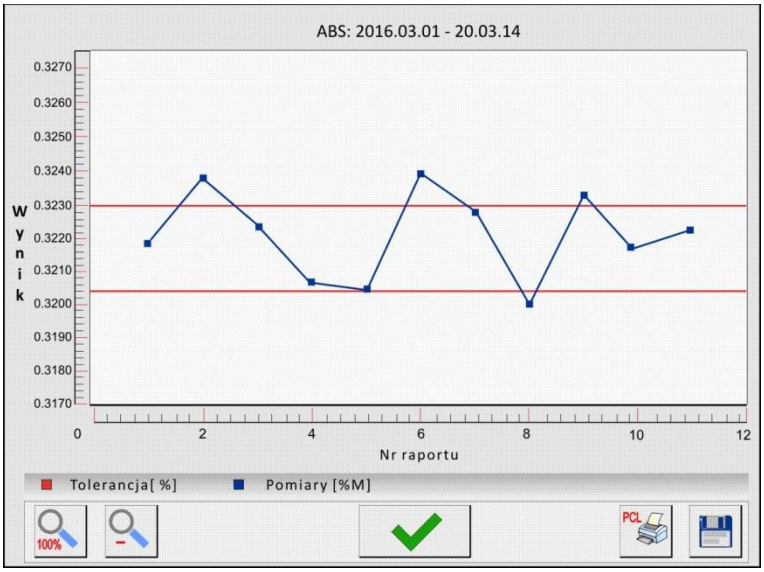


Figure 9. Moisture content trend over time – MA.3Y moisture analyzer

The weighing market provides its users with some more advanced IT solutions that operate moisture analyzers connected to monitoring systems such as ERP. With this it is possible to supervise many operators on-line, visualize all processes and carry out databases update for each device.

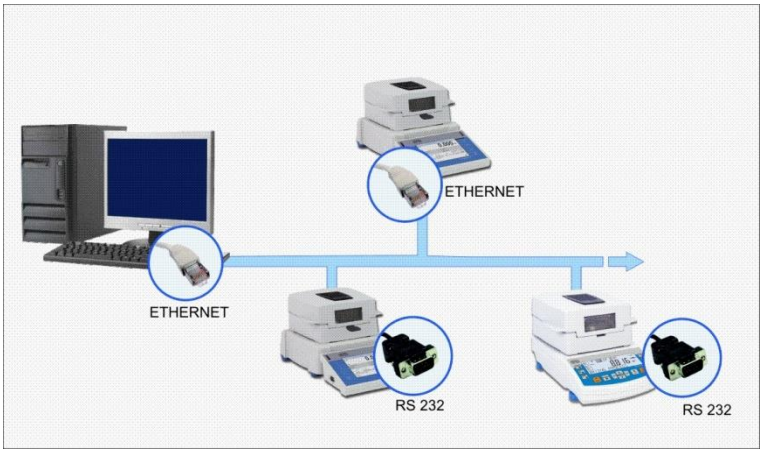


Figure 10. Data transfer

The possibility to copy parameters such as user profiles, and databases of products or drying process parameters for example, makes implementation of a new moisture analyzer an uncomplicated operation. The device to which the above mentioned data has been transferred is compatible with the system and therefore can be operated almost instantly. Settings of both the new moisture analyzer and the one that was used before are the same. Each operator is assigned with a certain permissions level, this allow to prevent occurrence of potential mistakes made by new employees who are still beginners learning how to perform particular tasks.

Additionally the moisture analyzer has been equipped with auto-control mechanisms, which help to monitor correctness of operation when it comes to mass measurement or drying temperature accuracy. This kind of support is especially useful while designing and following both SOP and GMP procedures.

6. The First Test of the Drying Operation

Before performance of the first test of the drying operation, it is necessary to set moisture analyzer parameters. Since the first test aims to develop the right methodology rather than to check the moisture content result, then it is possible to use any freely selected sample. Each MA.R moisture analyzer features START/STOP key, the operator is required to press it and next:

- tare the weighing pan to determine net weight value of the sample unaffected by zero drift of the moisture analyzer (1),
- open the drying chamber and put the sample inside on the weighing pan (2),
- close the drying chamber,
- system of heating components gets activated, the drying process starts (3)

MA.3Y moisture analyzer features touch screen which in the bottom information line displays messages related to the drying process. Sequence of actions for the MA.3Y series is the same as in case of MA.R moisture analyzer.

(1)

(2)

(3)

The drying process is carried out either using default parameters or the most recently set drying temperature values and finish mode options. The parameters must be optimised with regard to a dried sample or a sample type.

NOTE

The first tests aim to make operator familiar with functional possibilities of the moisture analyzer, and to check if the device operation is valid. Carrying out the drying process with default parameters set, may provide a result other than expected. There are users who in accordance with specification provided by reference methods want to dry substances of quite large mass. It is possible to dry large sample of course, however in such case the analysis takes a lot of time. The intention when it comes to use of the moisture analyzer is to provide as accurate result as possible within a relatively short period of time, much shorter than in case of the reference method.

7. Drying Process Accuracy

When speaking of the drying process accuracy it is nothing else but a difference between the obtained result and a true value. In order to be able to assess this parameter, information concerning real moisture content of the sample is needed. The required value may be taken from the tested product certificate (if only such one exists), it may also be obtained by means of tests carried out using the reference method. Practice shows that most of the users lack this kind of knowledge. In such a case it is very hard to speak of drying process accuracy.

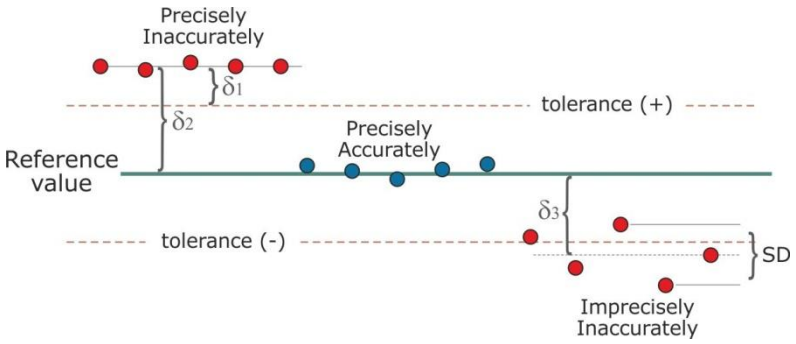


Figure 11. Accuracy and precision in drying processes

NOTE

The ability to obtain the same results means measurement precision, however the notion of precision is not the same what the notion of accuracy is. Errors δ1 and δ2 confirm the above statement. The worst case is the series of measurements characterized with a great dispersion (SD) and a serious error δ3 of average value related to the reference value. Lack of precision is mainly a result of the adapted drying method. Since the cause of the above is wrongly adjusted drying temperature then it is possible to minimize such a defect. Inaccuracy is mainly a result of the adopted temperature value. When the operator knows the reference value the problem can be easily eliminated.

The Best Possible Solution

In many branches of industry, threshold values are specified and referred to when it comes to the moisture content, for example, the maximum permissible value is 7.5 %. Sample for which such a condition is met is regarded as valid. Due to the fact that the said sample is representative for a particular batch, then the whole batch is classified as one that adheres to the requirements. The moisture analyzers are devices used to perform control in the course of the production. In production it is necessary to apply and refer to either threshold (no more than) or tolerance (+/-) values specified with regard to the set quantity. This kind of approach is a reasonable one, providing that correct methodology is used. The right methodology must take into account the following:

- the sampling method ensuring that the selected sample reflects, in the best possible way, a real state of the particular batch; this requires mixing (in case of liquid and semi-liquid substances), or taking up the sampling material from few different points.
- the way of preparing the sample for drying (cutting, milling, crushing, grinding, etc.)

- accessories supporting the drying process, (if required); most often these are filters made of glass fibres, protecting sample's surface against too high temperature rise; the filters may also be used in order to provide increase of the active surface of moisture release (in this case silica sand may also be applied).
- the conditions regarding finish stage of the drying process (the limit value of change of mass over specified time interval, usually $\Delta mass/\Delta t$ or change of moisture content over time $\Delta mstr/\Delta t$).

Figure 12. Processes related with sample preparation

In many situations it is attempted to obtain moisture content of value close to the limit value. This refers especially to those samples that are packed into portion packs. Too low moisture content of a sample calls for more product in order to provide the required net weight value. In case of bulk type of production, this would mean serious loss. With regard to the above it may be concluded that optimisation of the drying process parameters is a must. The optimisation should take into account dispersion of indications, a characteristic feature of each measurement.

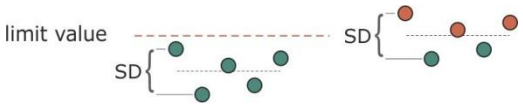
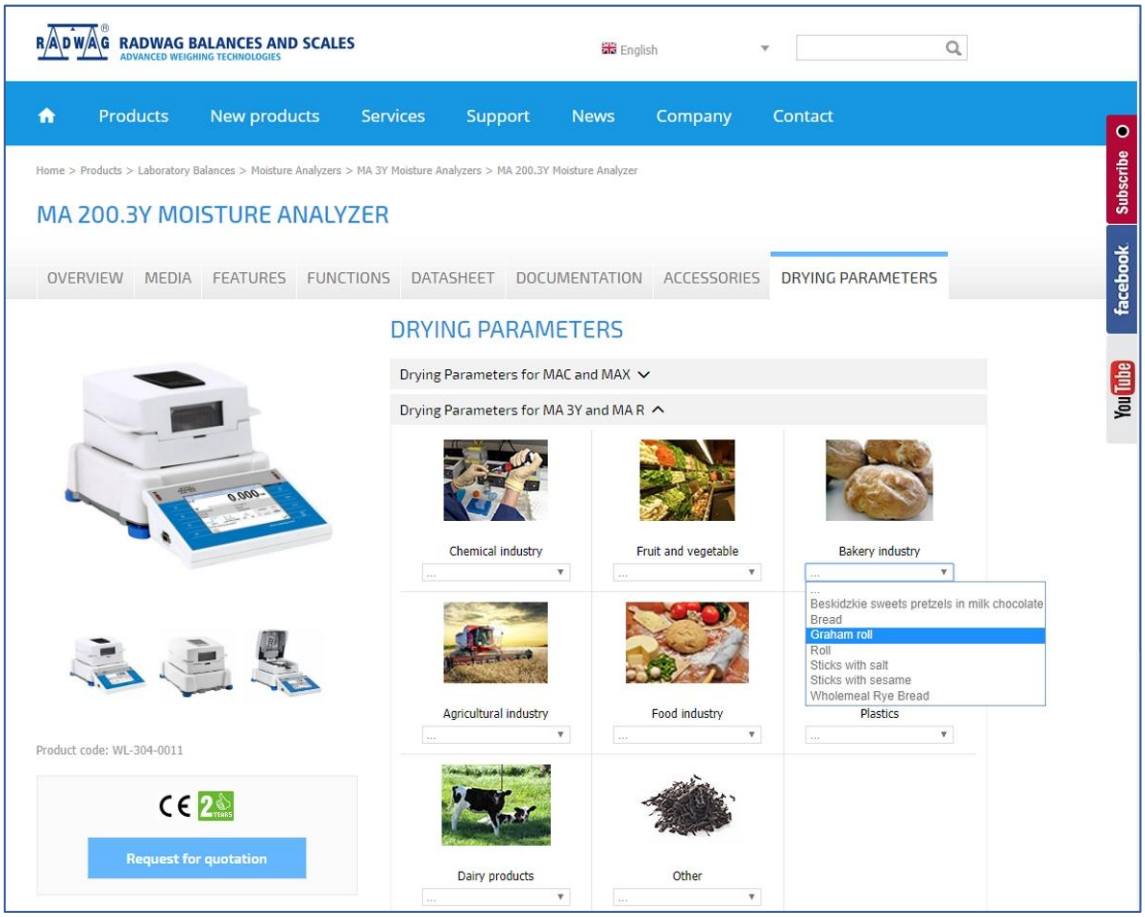


Figure 13. Production limit vs the average value

The manufacturer's experience is of a great value and can be supportive. Radwag has 10-year-long experience in conducting comparison tests for vast range of different samples. As a result of Radwag research activity, drying parameters sheets providing technical information on drying process optimization with regard to particular products, have been developed. They serve as one of many sources of information on moisture analyzers (http://www.radwag.pl).



The best possible accuracy that is to be obtained in the course of the drying process is usually specified in a moisture analyzer's data sheet. The tests are carried out using reference material that is characterized with good properties when it comes to moisture release. In order to determine moisture content readability for a given sample, a series of different tests must be carried out. In the course of the tests both methodology and drying parameters are modified.

The obtained result is often worse than the one that has been provided by the manufacturer. This is due to characteristics of the real sample, which significantly differs from the reference one. In case of the real sample, certain thermal modifications occur, namely: burning, release of highly volatile components, crust formation, potential heterogeneity, absorption of moisture from the environment (powders), inappropriate drying methodology. All the above bring about worsening of the indications repeatability, which in result causes inaccurate indications. Unfortunately, some features of the sample cannot be eliminated anyhow therefore is it advised to be reasonable when it comes to accuracy of the moisture content measurement.

7.1. Sample Size and the Accuracy of the Moisture Content Measurement

The information provided at the very beginning of this chapter concern sample of an optimal mass. Deviation, when speaking of accuracy of moisture content measurement, is an effect of adopted methodology or incorrectly adjusted drying temperature.

Moisture analyzer, like any other weighing instrument, is characterized with certain measurement error that should be referred to in some cases. It is commonly known that in order to calculate moisture content value it is necessary to carry out two measurements of mass (at the beginning and an the end). Even in case of a model measuring cycle there is a high probability that the final result will be encumbered with error being a result of rounding. This is shown in figure 14.

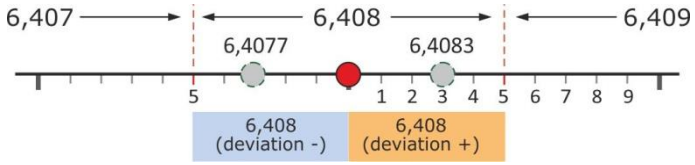
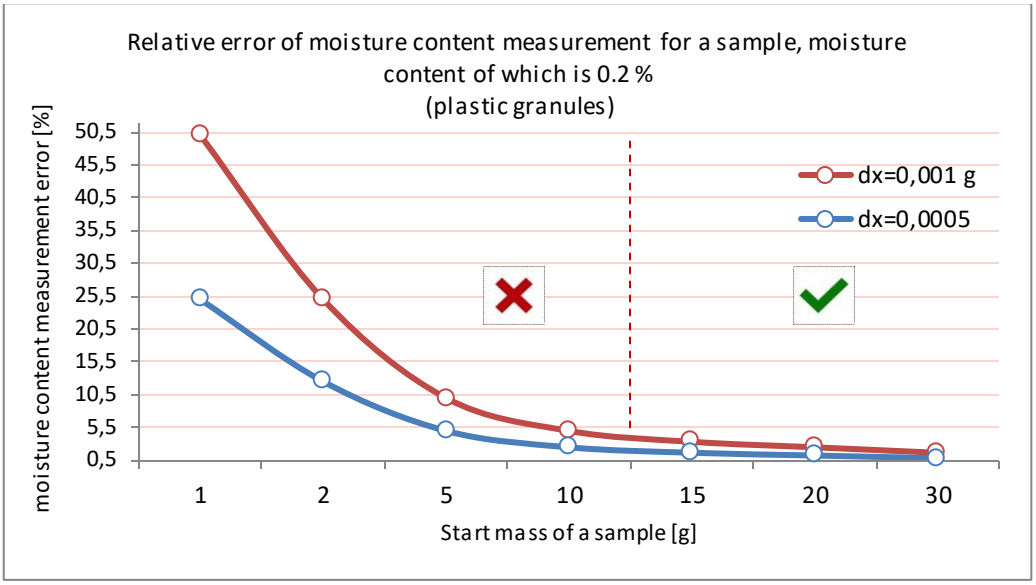


Figure 14. Rounding of the measurement result for digital devices

As it is clearly presented in the figure, the result features certain either positive or negative deviation. The deviation is not comprised within moisture analyzer measuring range that can be observed by the user. Obviously it cannot be definitely stated that the deviation value is not zero, it can be, however such case is rather an incident than a regularity. For a moisture analyzer with a scale interval d=0.001 g, the maximum deviation value being a result of rounding is ~ 0.001 g. It is an absolute error of the device. With this information, it is easy to determine optimal mass for a sample in regard to the required relative error. The above is used wherever sample's moisture content is too low, and wherever too low weight loss is registered upon completed drying. This concerns all different kinds of plastic granules.



Graph 1. Relative error for plastic granules
in relation to mass of the dried sample

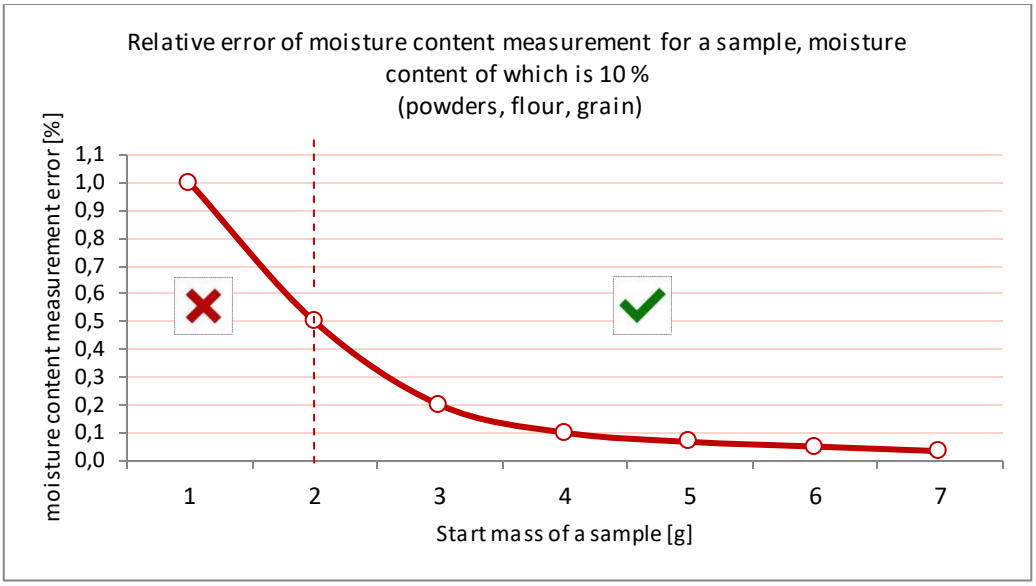
In case of samples of low mass, ranging from 3 to 5 g, high, impossible to be accepted relative error is observed. The greater start mass value, the lower the value of this error. With regard to this, while drying granules of any type, at least 10-gram heavy samples are required. Use of moisture analyzer characterized with a little bit smaller absolute error (dx=0.0005) is not a solution guaranteeing success when it comes to measurement of a sample of small start mass.

In case of most of the moisture analyzers, the drying process is completed using some automatic modes. The automatic mode may be understood as a form of control of stability of dried sample mass in scale of 0.001 g over a certain period of time. This is a cause of a possible error of about 0.001 g being an effect of rounding. Thermal state of the sample is not taken into account here, this refers to the below cases:

- temperature too low where the effect of not enough dried sample occurs,
- temperature too high where the effect of sticking sample particles occurs.

It is possible not to take advantage of these auto finish modes, instead of them definable condition may be applied, e.g. change of mass of the sample by 0.3 mg within 30 sec time interval. This however may result with problems regarding final result oscillation, which drastically lengthens the drying time. Alternative solution is to carry out drying within strictly specified time. Correctly adjusted drying temperature allows to apply constant drying time, within which it is guaranteed that the whole moisture content will be removed. Detailed information regarding the above is provided in later sections of this publication.

The situation is different for samples of average moisture content of about 10%. In such a case, samples of much lower weight value may be used, which is due to the fact that here the influence of relative error on the measurement result is neglected. This has been demonstrated on the below graph.



Graph 2. Relative error size in relation to mass of the dried sample; graph for samples for which the moisture content is 10 %

SUMMARY
MEASUREMENT ACCURACY AND THE SAMPLE SIZE

Drying process accuracy is a complex issue since there are many factors influencing it. Among many, the following are required: relevant drying temperature, sample of optimal weight, adequate methodology. The process of obtaining satisfying results should be based on comparative tests, which leads to a conclusion that it is necessary to validate the used method. Reference methods always provide results showing the real state of the sample. Moisture analyzer is a component completing these methods and requires optimisation of the drying parameters.

7.2. Finish Mode and the Accuracy of the Moisture Content Measurement

The drying process may be finished manually (via the user), it can also be time-defined (the process finishes after elapse of specified amount of time) or automatic. The manual method requires no comment. If too short time interval is set, then too low moisture content value is obtained. Too long drying time is as much objectionable. Drying process duration must be set experimentally, it is strictly related to the weight value of the dried sample, which can be concluded after analysis of the below graph.

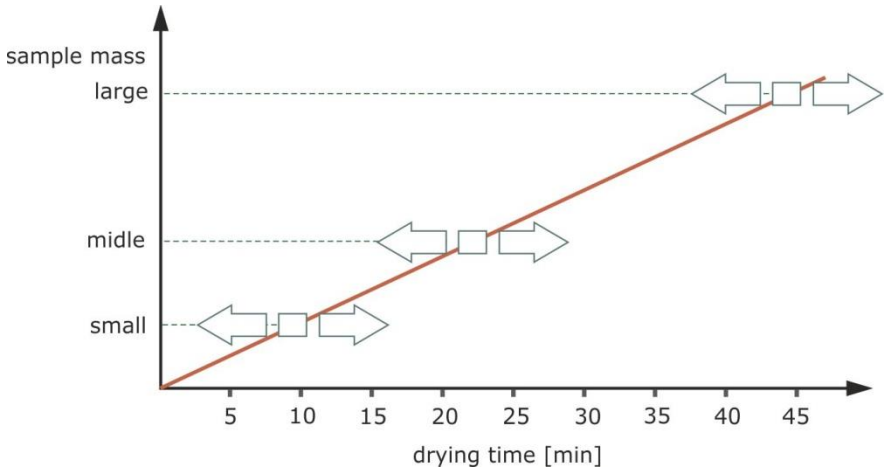


Figure 15. Relation between the constant drying time and the start mass of the sample.

The greater the sample mass the longer it takes to dry it in order to remove the moisture content completely. Sample type and its ability to release moisture is of a great importance here.

When using auto finish mode (Auto 1 – 5) a certain variation when it comes to the final moisture content result may be observed. In the course of the cycle, stability of the dried sample mass over a set time interval is checked. Mass variation is constant by default, and it is 1 mg, as for the time interval, it ranges between 10 - 120 seconds. The longer the drying process end, the more considerable amount of moisture evaporates, which of course influences the final result. Moisture content value obtained using Auto 5 switch-off option is greater than in case of Auto 1 setting.

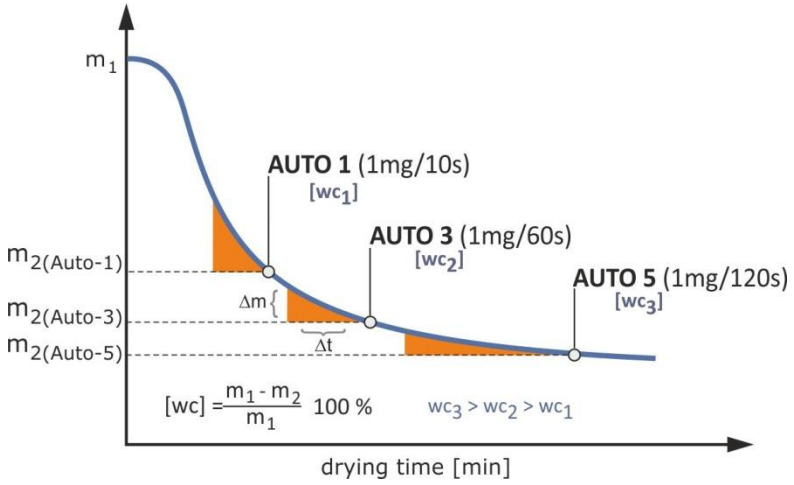


Figure 16. Auto finish mode and the measurement accuracy

Auto finish mode allows the user to define his/her own switch-off criteria. For MA.3Y moisture analyzer the said criterion may take the following form:

- any mass variation within any time interval, $\Delta m/\Delta t$, (MA.3Y, MA.R)
- any moisture content variation within any time interval, $\Delta mc/\Delta t$, (MA.3Y, MA.R)

NOTE

The variation regarding moisture content of a particular sample, which is a result of use of different auto switch-off options, mostly depends on the sample type. However difference of few percent range shall not be expected. Nevertheless should it happen that difference of this size is registered, then it must be stated that the reason for that is either too low drying temperature or formation of impermeable layers over the sample surface.

8. System for Heating Samples in the Drying Chamber

Regular heating process seems to be uncomplicated: the operator just needs to place the sample next to the heat source, as a result the sample's temperature will rise. Simplicity is usually a reliable solution (there is barely anything to be broken) but not always efficient. Presently, the requirements regarding drying processes call for both speed and flexibility. In order to provide that such requirements are met, it is necessary to design device, operation of which is based on the knowledge confirmed by numerous tests. This is how two series of Radwag moisture analyzers have been developed: Moisture analyzers of MA.R, MA.3Y series. Both use infrared radiation for the purpose of sample heating.

8.1. Infrared Radiation Theory in Brief

One should realise that not all infrared energy emitted onto the sample, or the tested object, is absorbed by it. The IR energy may be partly reflected or transmitted elsewhere, therefore it does not heat the sample directly and may be completely lost in the drying process. The amount of absorbed, reflected or transmitted energy depends on the length of infrared radiator's wave and on type of the surface onto which it is emitted. These, but also some other crucial variables, significantly influence effectiveness of thermal energy.

IR energy gets transferred into heat as a result of getting in touch with the surface, next, due to conductivity, the said heat spreads over the tested object. Materials such as metals are characterized with high thermal conductivity therefore they transmit the heat onto the entire volume quickly and evenly. In case of materials such as plastic, wood, and others, the thermal conductivity is low, these materials' surface may reach high temperature long before the increase of internal temperature gets recorded. This is a reason of formation of crust on some samples' surface.

Most of the materials (except for glass and some types of plastic) are infrared-impermeable, in such case, the energy gets either absorbed or reflected. Glass, see-through plastic films may reflect and transmit major part of radiation. Therefore drying these materials exclusively through radiation seems to be unreasonable.

8.2. Methods for Heating Samples in the Drying Chamber

Standard heat source of MA.3Y and MA.R series is an infrared radiator, operating in a feedback loop with a temperature sensor. This ensures thermal stability in the course of the analysis. Designed by Radwag method of dynamic control of temperature in the drying chamber is one of the elements allowing to obtain short analysis duration and drying series repeatability.

Apart from IR emitter also halogen lamp (HAL) or resistance heating wire can be installed inside of the drying chamber. The substantial difference between these heating modules concerns the method of heat transmission to the sample's surface. In order to transmit heat, IR and HAL lamps emit waves ranging from 0.76μm to 1000 μm. Both the lamps are in fact infrared radiators, however they emit waves of different lengths. They are addressed with different names due to the need to distinguish them one from another.

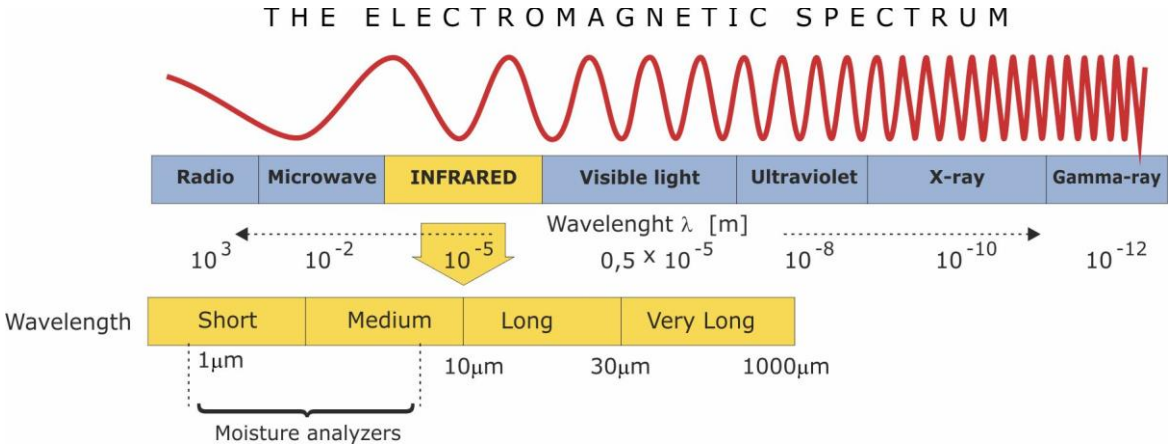


Figure 17. Electromagnetic waves spectrum with range specified for moisture analyzers

General classification of radiators made with reference to wavelengths:

- **IRS** (infrared short) - short-wave radiation, where IR wavelength is 1.2μm,
- **IRM** (infrared medium) - medium-wave radiation, where IR wavelength is 3μm,
- **IRL** (infrared long) - long-wave radiation, where IR wavelength is 5μm.

This kind of classification is quite conventional one since the wavebands cover one another. A certain band, for some of the cases, may be considered to be such one that generates short waves. However in some other circumstances, it can happen that the very same band will be recognized as band generating medium-length waves. When speaking of the drying processes, the crucial information concerning them is the way the heat gets to the sample. We can speak of two phenomena here, the process of radiation and convection.

RADIATION is nothing else but transmission of heat from one body to another, wherein the amount of transmitted heat depends on:

- temperature difference between the emitting element (radiator) and the receiver (sample),
- environment,
- radiated wavelength.

Practically every single body emits radiation, this can be easily noticed using the thermal imaging camera. The above refers also to lamps placed in the drying chamber. The greater the lamp temperature, the more heat is sent to the sample.

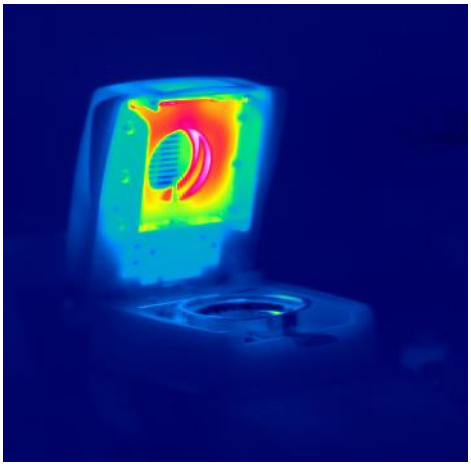


Photo 4.
Infrared lamp radiation

Photo 5.
Radiation spectrum of the hot sample

Each hot body is a radiation emitter, this can be observed in photo number 4. As a result it is not only the radiator that heats the sample but also a major surface of the top panel, due to this the moisture content is released fast.

CONVECTION uses heat transmission, wherein the heat is transmitted as a result of movement of air particles circulating in the drying chamber. The warm air, which is thinner, moves up whereas the cool air goes down. This kind of circulation allows to transfer energy via certain kind of a medium.

It is not just the heater type that decides on the effectiveness of used heating solution. Also sample's absorption properties, i.e. ability to take up radiation, and the level of reflectivity, i.e. ability to reflect radiation, are of a great importance. Therefore the question arises what the optimal choice is.

Infrared radiators (IR) emit wavelengths longer than radiators of HAL type. Light of more intense red colour is visible. The longer the wave, the lower the value of coefficient of reflexion (which depends on the type of sample's surface). This relation means that more energy passes through the material. Thermal energy evenly spreads over the whole samples volume.

Radiators of HAL type emit shorter wavelengths (brighter light) than radiators of IR type. The shorter the wavelength, the more radiation gets reflected. Contribution of radiation when it comes to the whole transferred energy balance may be estimated at 50%. The below table presents comparison of these two radiators.

	Radiator	Halogen
Radiation	70%	50%
Convection	30%	50%

Table 5 Convection and radiation conditioned by the heat source

In case of heat source such as resistance heating wire, the sample is heated via radiation. At the beginning, convection also contributes slightly to the heating process, later its contribution is greater but still almost insignificant. When taking into account physics of the process, this source of heat is the most appropriate when speaking of reference methods (specified by standards) requiring use of the induction heater. Radiator such as the resistance heating wire is not a solution commonly applied in moisture analyzers. However the effectiveness it guarantees in the course of the drying process is satisfactory. Two major disadvantages to be pointed out here are delay concerning the drying temperature increase, and thermal inertia. The above indeed may be a problem wherever flexibility (various drying temperatures) and fast operation (large number of samples) are a necessity. The below pictures present differences between the discussed heat sources.

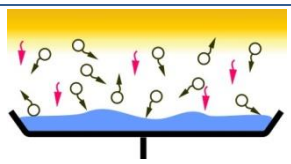
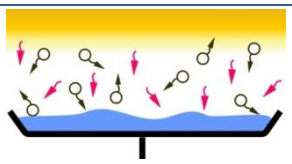
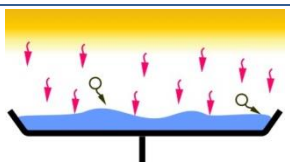
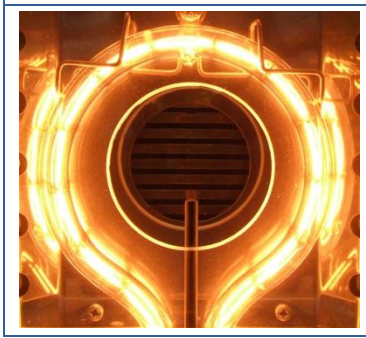
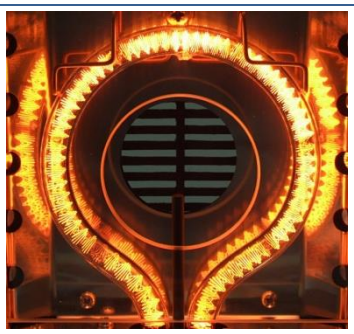
HAL radiator	IR emitter	Resistance heating wire
		
mainly convection surface heating APPLICATION powders, semi-liquid samples, liquid samples	convection and radiation deep layers heating APPLICATION most semi-liquid samples, liquid samples, crushed solids	mainly radiation volumetric heating of the sample APPLICATION thick consistence samples and solids

Table 6 Heat absorption conditioned by the heat source

NOTE

Samples differ in terms of absorption capabilities. Smooth and even surface reflects more radiation, which means that less energy is absorbed and the sample gets heated more slowly. The effect of absorption depends on sample's colour and the sample's reaction to temperature raise (the effect of crust formation). There are no suggestions concerning heat sources that would clearly state which particular solution is preferable.

The photos present infrared radiators that are used in moisture analyzers. Judging by the intensity of light one can easily distinguish IR emitter from the radiator of HAL type.

HAL radiator	IP emitter	Resistance heating wire
		

SUMMARY
DRYING CHAMBER'S HEATING SYSTEM

Before the moisture analyzer start-up, the operator should undergo training regarding safety and functionality issues. Such approach is beneficial for both the distributor and the user. The results of the first drying tests are of no real value when not confirmed by a respective methodology. While attempting to determine the best methods, it is advisable to consult either the manufacturer or the authorised distributor.

It is possible to assess accuracy of drying carried out using the moisture analyzer, however one condition must be fulfilled, namely, the calculated value must be referred to the real (known) moisture content. If such information is unknown, then it must be obtained via test carried out in accordance with recommended procedures. In case of a product for which there are no standards, the test should be carried out with reference to characteristic features of the sample (type, colour, absorption capability, homogeneity etc.). For such objects, initially, drying temperature of 105^{o}C is adapted, however there are cases when the tests must be repeated. The reason for that may be, for example, surface burning of the sample.

It can be easily noted that there are certain differences between infrared radiators, but when it comes to accuracy of the drying process there are two crucial factors: the drying temperature and the methodology (sampling, sample preparing process, etc.). This means that similar moisture content results can be obtained regardless of used heat source type (IR, HAL or resistance heating wire).

9. Samples Preparation

The first factor conditioning 'precise' moisture content result is correct drying temperature. As much significant is every single aspect concerning the sample. Starting from sample selection to the moment of placing the selected sample in the drying chamber. While sampling, it is recommended to take pieces of the material that is to be dried from more than one part of the whole substance or product. Unfortunately, in practice this recommendation is hardly ever followed.

9.1. Sample's Homogeneity

Homogeneity is a parameter characterising semi-liquid and liquid substances. In case of such samples it is highly probable that structure of the surface layers is different than structure of the deep layers (e.g. yoghurt). Prior drying both semi-liquid and liquid samples must be stirred.

For powders and solids such as granules for example, heterogeneity means different levels of moisture content, wherein the difference is an effect of position that a particular granule takes. Stirring may be a solution, however it does not change initial state of a sample, since it still remains heterogeneous. It is advisable to carry out sample's conditioning in constant conditions before the drying process performance. If conditioning is impossible to be carried out, then sample's material must be picked from different shares of the product. This may serve as a constructive advice resulting with use of a different method of samples supplying or storing.

When it comes to solids the fact is that the moisture content of their surface is different than the moisture content of the deep layers. With regard to that, it is hard to speak of homogeneity other than homogeneity of particular layers.

9.2. Samples Storing

The storage method should prevent both evaporation of moisture from the sample and its absorption from the air. Packaging size should be suitable to store given quantity of the sample, i.e. the stored material should fill at least ¾ of the packaging volume.

incorrect correct

Photo 6. Storing technique

The above is possible in case of laboratory applications. When it comes to industry, these requirements are rather impossible to be met. The product that is to be dried is taken directly from the production line, therefore product storing takes no place. Disadvantage standing behind this kind of procedure may be a great dispersion of indications, especially when the manufactured product is characterized with high absorption/desorption capability.

9.3. Optimum Sample's Mass

The main idea about use of moisture analyzer is the possibility to obtain precise results within a relatively short time. This requires weight of a dried sample to be optimal, i.e. heavy/light enough to ensure that for a certain temperature the obtained result is comparable to the reference value. General guidelines regarding sample's mass are presented below.

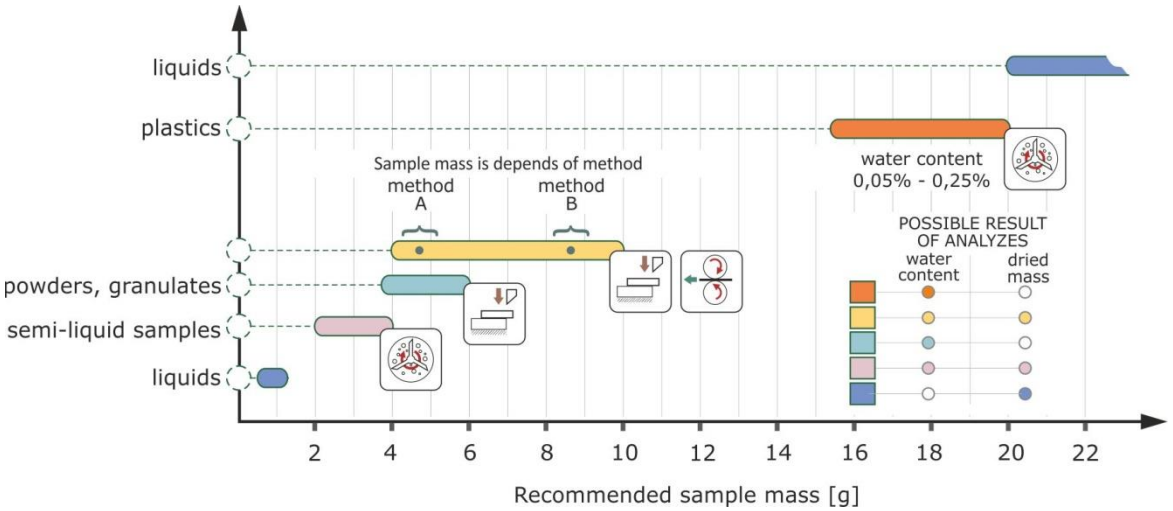


Figure 18. Sample's mass in the drying process

9.3.2. Semi-Liquid Samples

Most semi-liquid samples are characterized with quite significant surface tension. Samples of this kind when applied onto the weighing pan, give accumulation of a concentrated matter. Drying a sample in such a form is ineffective due to limited vaporization surface. It is not advisable to add more product onto the pan and at the same time to increase the sample's mass, however in such a case it would be a wise solution to spread the sample over the weighing pan surface (increase of sample's surface). See the below photos.

Photo 7. Inappropriately prepared sample

Photo 8. Appropriately prepared sample

The thinner the sample layer, the less time the drying process takes. Semi-liquid samples can be dried using an alternative method which requires use of glass fibre filter, for example, or silica sand. In such a case the sample is dried upon being placed on the filter or sand. An advantage of such solution is the ability to dry the sample from the top and the bottom. Due to this active vaporization surface gets increased. It is possible to slightly modify the alternative method. Namely, instead of one, the operator can use two filters between which the dried sample is placed. Such modification is recommended especially when drying samples susceptible to temperature rise in case of which crust formation takes place.

The discussed method is effective for samples containing large amount of sugars, syrup which caramelize as a result of temperature increase. Fondant is a good example of sample of this kind.

Sample placed on a glass fibre filter, before drying

Sample placed on a glass fibre filter, after drying

Sample placed on a silica sand, before drying

Sample placed on a silica sand, after drying

The above photos prove that regardless of the adopted solution, filter or sand, the same result is obtained. Due to economics, most operators will probably go for the silica sand, it is less costly and easily accessible solution. As a professional accessory offering vast range of possibilities (size, type), glass fibre filters are quite expensive.

9.3.3. Powders and Granulates

When drying powders, grain and tiny granules it is recommended to apply samples of about 2 g – 6 g. The operator should make sure that the pan is covered with uniform and regular layer of the sample.

Photo 9. Inappropriately prepared sample

Photo 10. Appropriately prepared sample

In case of large-grain samples at least partial crushing of the grains is necessary. With this, the drying will take less time, additionally dispersion of indications of particular measurements series will be smaller. When crushing (disintegrating) the given material, one must avoid generating too much heat. Examples of samples before and after crushing have been presented below.

Pea - whole-grain sample before crushing

Pea - crushed-grain sample after crushing

Dried raspberry - sample before crushing

Dried raspberry - sample after crushing

In order to crush or grind the sample it is possible to use some electrical or mechanical devices, or any method allowing to provide respectively crushed material. When preparing candied samples (mainly fruits), grinding them with use of devices may turn out to be ineffective. In such a situation prior drying the operator must cut the sample into small pieces.

9.3.4. Solids

If only possible, the solids should be broke into tiny bits of pieces prior the drying process. With this, the chance to obtain a reliable result is much greater. The smaller pieces, the larger the whole vaporization surface. Possible disintegration methods are: cutting, breaking, crushing etc. When it comes to thick solids, it is highly probable that the moisture content of the sample differs depending on the layer. The above refers to products such as boards, wood flooring etc. Here the preparation methodology may be of a great importance.

Photo 11. Chipboard – integrated sample (inappropriately prepared material)

Photo 12. Chipboard – disintegrated sample (appropriately prepared material)

Some solids which are comprised of various materials are characterized with heterogeneity, for example wood chips. Common feature of all solids that are subjected to drying is a slightly different mechanism concerning release of free water. Separating the free water in case of solids is much more difficult. Usually the drying is carried out for one temperature value set. An exception to this rule are samples for which it is necessary to determine content of bound water, for example minerals that are dried in few different temperatures. Free water content is determined in temperature of lower value, whereas moisture that is bound chemically, in higher temperatures.

9.3.5. Plastics

The nature of plastic samples is quite specific. Generally it is said that plastic materials are moisture-resistant. We may assume that this is true for finished goods, but the test concerns granulates not subjected to plastic working. PS, PVC, PE and PP plastic is not hygroscopic therefore the moisture sets up on its surface exclusively. As for ABS, PA, PC, PET, these may store moisture at the subcapillary or molecular level. Injection moulding (heat forming, blowing) is a process requiring high temperature which is a cause of vaporization of plastic-contained water. Thermal process is very fast, if the plastic contains too much moisture content then it is certain that not all of it will evaporate. Some bubbles are formed both on the surface and inside of the moulded piece. This is a reason for product disqualification because of inappropriate qualities such as look and strength. Applied industrial methods aiming to dry plastics do not provide quantitative value that would indicate quality of the sample prior the shot. This kind of information may be obtained via drying a reference sample, carried out using the moisture analyzer.

In case of majority of plastics, the moisture content is quite low. Depending on the plastic type it ranges between 0.02 % - 0.3 %. Due to this, start mass of the dried sample should be large. In case of small masses, the error when it comes to moisture content measurement is quite serious (chapter 7.1).

Photo 13. Plastic granulate – not enough large sample's mass (inappropriately selected sample)

Photo 14. Plastic granulate – large sample's mass (appropriately selected sample)

9.3.6. Liquids

Liquids drying is a process as a result of which dry substance is obtained. The quantity of the obtained dry mass substance is usually so small that in order to be able to measure it, the start mass must be large. This is typical in case of measurement of dry mass for drinking, surface and waste water, which is drained off to soil and water basins. For the above cases it is advisable to use pans with high brinks allowing to store larger content of the liquid.

Liquids consisting of at least several distinct phases should be thoroughly stirred before drying. Start mass of a sample may be reduced to the amount of few grams, providing that the dry mass obtained in the course of drying is of the order of few milligrams. The drying process duration may be considerably shortened if the active vaporization surface gets enlarged. Ideal solution providing active vaporization surface increase is the silica sand, its weight value should be at least 15 g – 20 g. It can be easily estimated that due to such operation, the sample surface is about 5 times larger.

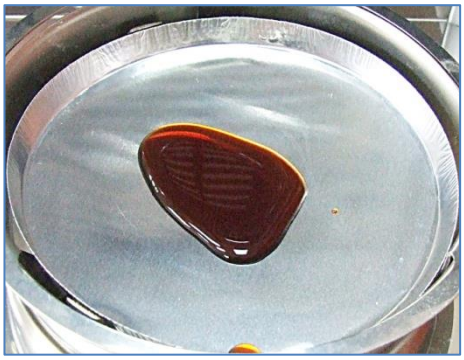


Photo 15. Sebonamine sample applied directly onto the pan

Photo 16. Sebonamine sample dried with use of silica sand
(ca. 2.2 % of dry mass)

10. Thermal Processes Occurring in the Course of Drying

Drying with randomly selected drying parameters may cause unexpected changes of dried sample structure. It is a good idea to inspect the surface state visually after completion of the first tests.

10.1. Crust Formation

Crust formation is a process where an impermeable layer is formed on a sample surface. This makes removal of moisture from the sample impossible. As a result the indication being an outcome of an analysis is lower than the sample's reference value.

Photo 17. Crust formed on the sample's layer

Cases like this require lowering of the drying temperature, which means that the process will take longer. Another good solution is to prevent exposing the sample's surface to the lamp generated radiation. To do that, the sample must be covered with a filter, or placed between two filters prior test start. Sample protected like this will significantly differ from the one presented in the photo.

10.2. Sample Burning

Sample's surface burning is a consequence of too high drying temperature. Usually it results in a change of sample's colour. This can be observed especially in case of samples that are bright. For them the moisture content is greater than the reference value. The drying process can be very long and it may be characterised with a constant tendency of weight change. Moisture analyzers of MA.3Y series offer drying process visualisation, which makes diagnosis of the above incredibly easy.

Photo 18. Surface burning – colour change

Reducing the drying temperature by just a few degrees should help to solve the problem.

10.3. Heat Absorption

Dark samples absorb more heat than the light ones. Due to this a bit lower drying temperatures are applied when drying dark samples. Selection of correct parameters requires tests. Because of quick vaporization, at the first stage of the drying process the sample's temperature may be slightly lower. Quick vaporization is not observed during the second stage of the process. Too high temperature gain may cause surface burning that is not easily noticeable.

When speaking of absorption of heat, one of the problems concerns materials that are not good thermal conductors. Their surface may reach very high temperature, however inside they may stay much cooler.

Photo 19. Samples of dark colour = higher drying temperature

It is necessary to set what is the critical temperature value for which unfavourable changes occur in sample's structure. The applied temperature must be of a slightly lower value than the determined limit temperature. Drying with temperature that is a lot higher than the limit value causes burning of the sample, whereas drying the sample in temperature of much lower value gives incompletely dried material as a result.

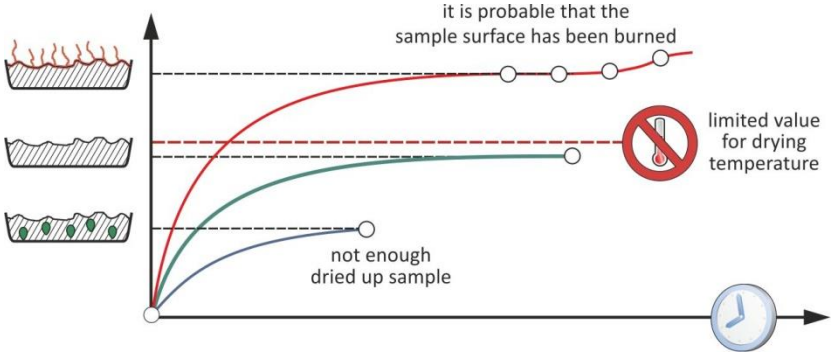


Figure 19. Temperature in the drying processes

NOTE

It is possible to dry the sample in temperature higher than the set limit value, however this requires separation of the sample's surface from the radiation source, for that purpose the sample can be covered with filter for example. If such is the case then the weight value of the filter must be added to the weighing pan's tare value.

11. Test Function, Auto-Selection of Auto Mode

In order to select finish mode of the drying process, at least few tests need to be carried out. The aim of such approach is to compare the obtained results with a reference value. It is the most commonly used method for optimization of this parameter. Moisture analyzers of MA.3Y series offer **Test** function, which simplifies selection of the right auto mode. This function's operation consists in automatically carried out calculation of sample's moisture content in intervals set for finish mode:

- Auto 1, change by 1mg over 10 seconds
- Auto 2, change by 1mg over 25 seconds
- Auto 3, change by 1mg over 60 seconds
- Auto 4, change by 1mg over 90 seconds
- Auto 5, change by 1mg over 120 seconds

The test is performed in a given drying temperature that should be optimal, i.e. not causing sample's burning. Since the cycle is automatic, then it can be carried out for various drying temperatures, which allows to look for the optimum value. This aims to provide the expected result within a possibly short time. Test function is one of the elements of Finish Mode menu.

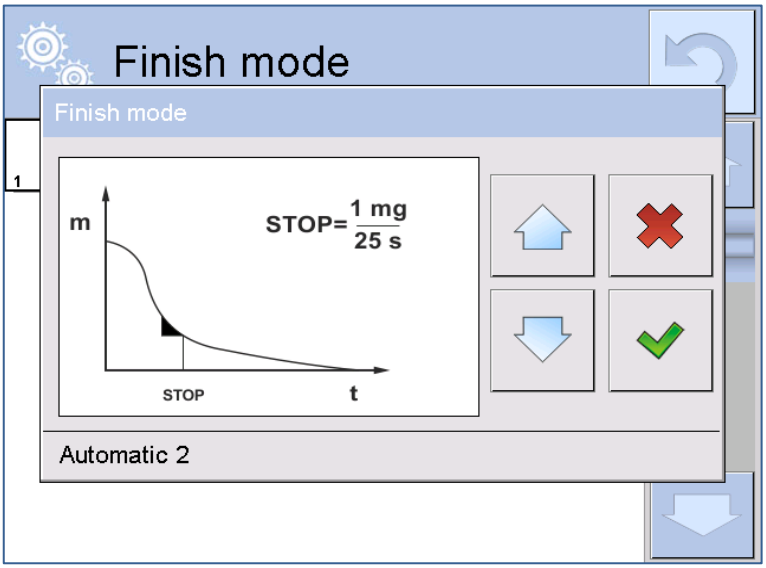


Figure 20. Activation of TEST function in MA.3Y moisture analyzer

Results of Test function operation are printed online via RS 232 interface. In order to analyse the results it is necessary to either print or display them in a special computer application. Exemplary printout, not providing indirect information concerning control of dry mass content (%D), is presented below.

Operator	Admin
Drying program	----------
Drying profile	Standard
Drying profile parameters	120°C
Finish mode	Test
Start mass	4.4699 g

0:00:05	99.9931 %D
---------- Finish Mode ----------	
0:08:29	Automatic 1
Result	89.5246 %D

0:08:30	89.5224 %D
0:08:35	89.5143 %D
---------- Finish Mode ----------	
0:10:04	Automatic 2
Result	89.4027 %D

0:10:05	89.4020 %D
0:10:10	89.3975 %D
---------- Finish Mode ----------	
0:13:09	Automatic 3
Result	89.3047 %D

0:13:10	89.3044 %D
0:13:15	89.3025 %D
---------- Finish Mode ----------	
0:15:04	Automatic 4
Result	89.2753 %D

0:15:05	89.2749 %D
0:15:10	89.2734 %D
---------- Finish Mode ----------	
0:16:58	Automatic 5
Result	89.2539 %D

End mass	3.9896 g

The difference between the results obtained for Auto1 and Auto 5 options is 0.27 %. Of course for samples of different type these proportions will be totally different.

12. Drying Profiles

Drying profile is a set of parameters determining method of control of increase of the drying chamber temperature. There are 4 drying profiles:

- Standard
- Fast
- Step
- Mild

STANDARD profile – applied in 99 % of cases, the temperature is reached fast, it takes about 60 seconds to obtain the set temperature value (for 120°C).

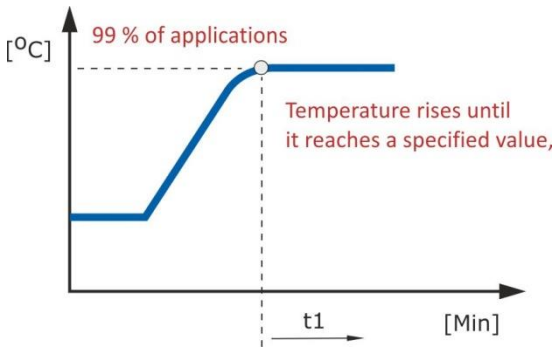


Figure 21. Standard profile

FAST profile – at the first stage the drying temperature reaches about 30% higher value than the set temperature. The higher temperature can be maintained at a constant level up to 59 minutes, the operator has the possibility to adjust the time. This profile is applied when drying samples of high moisture content. Rapid evaporation of large quantity of moisture may result with lowering of the drying chamber temperature, this in turn can prolong time of drying.

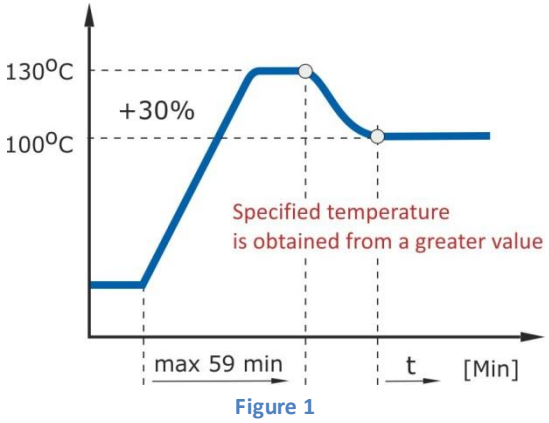


Figure 1

Figure 22. Fast profile

STEP profile – this profile enables drying operation to be carried out in 3 different temperatures. For each of them, time of heating is declared. Such cycle of operation aims to separate sample's free water from the bound water. This profile is applicable when working with minerals for example. It is also useful while testing gypsum purity.

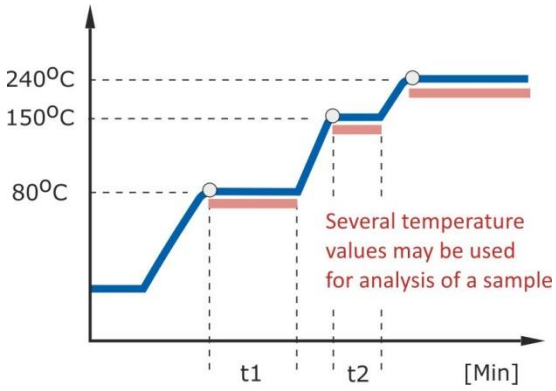
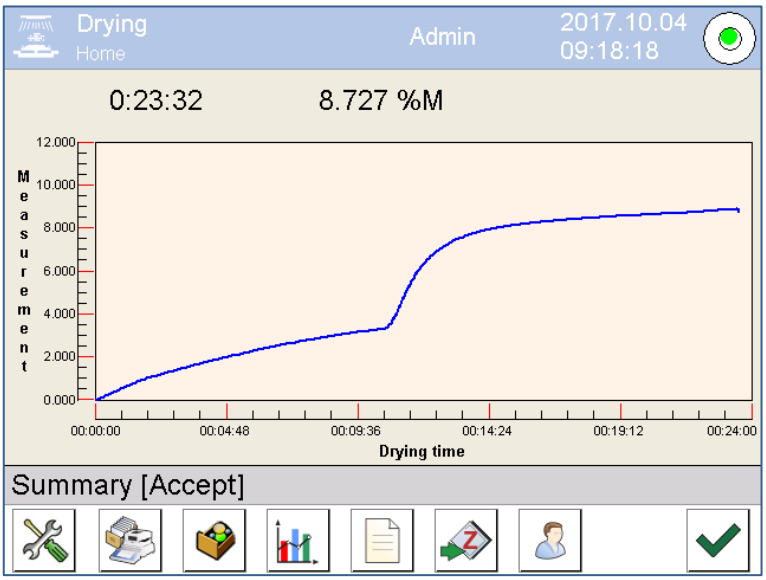


Figure 23. Step profile



MA.3Y moisture analyzer display for two-phased drying process

MILD profile - is used in those cases when too rapid temperature increase causes evaporation of substances other than water. The operator can define time necessary to obtain the set temperature, its maximum value is 20 minutes.

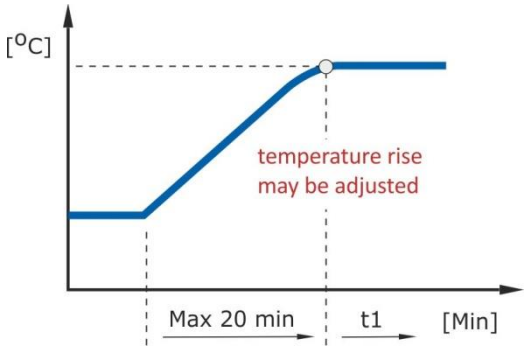


Figure 24. Mild profile

13. Drying Temperature Control

Drying temperature value is an effect of operation of both heating module (IR emitter) and temperature controller. These devices operate in a feedback loop, which positively affects drying temperature stabilisation regardless of analysis duration. Parameters of such system are adjusted in a way providing long-term drying temperature stability, for each profile. Periodically tested drying temperature is not an effect of technological conditions, but rather a requirement specified by respective standards or legal and branch regulations. It is commonly known that each measuring device has to be periodically tested. As for moisture analyzers when testing mass indication, respective mass standards must be used, in case of drying chamber's temperature tests, control thermometer is required. The following assumption is adopted:

If the drying temperature indicated by the moisture analyzer and the value of temperature required to dry the sample are complaint, it means that the whole moisture content will evaporate from the sample. The moisture content result indicated by the moisture analyzer will be precise.

The above assumption is true only if for a given sample, an appropriate drying method is applied. In order to provide precise results it may be not enough just to keep the drying temperature stable. Also drying method and sample preparation are extremely important. With reference to the above a right conclusion can be made that in the course of audits not only results of periodical drying temperature controls should be assessed but also the applied methodology. This may be quite a challenge for auditors who are not specialists within the area of thermo-gravimetric measurements.

13.1. Control Method

The control set comprises a mechanical part and a measuring module. Both components are joined into one unit in the course of the manufacturing process. The thermometer features an additional shield which stabilizes its temperature during the test. Drying temperature test is carried out periodically in an accordance with the adopted control scheme regarding particular drying temperature.

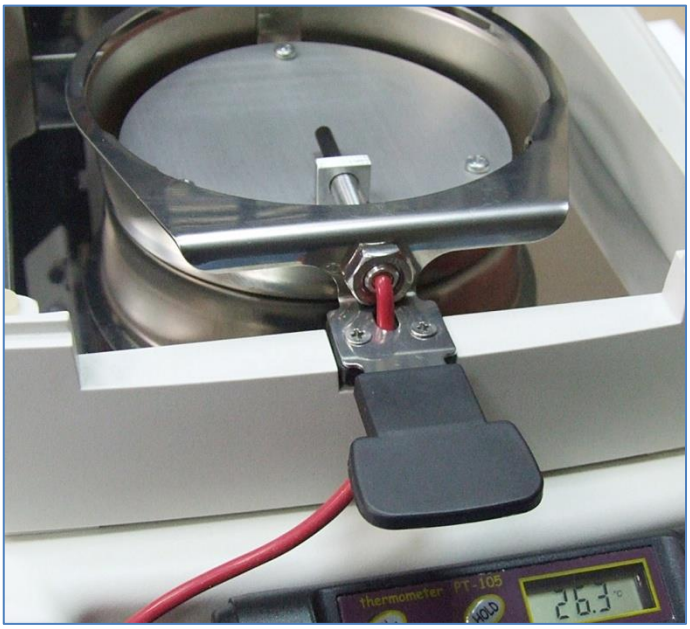
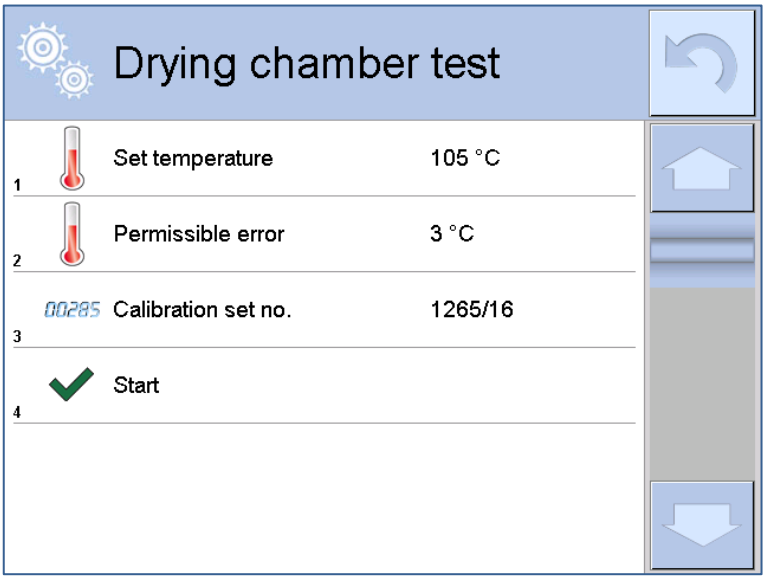


Photo 20. Control thermometer placed in the moisture analyzer's drying chamber

To put the control set into the drying chamber, it is necessary to disassembly particular components that are installed inside of it. The control focuses on the drying temperature exclusively therefore the mass indication displayed in the course of the procedure is of minor importance. Prior to test start, tolerance value, i.e. maximum permissible error concerning temperature indications, must be specified. Additional information is the number of control thermometer set.



MA.3Y moisture analyzer display, drying temperature control screen

Test of drying chamber heating takes exactly 8 minutes, after passage of this amount of time the temperature displayed by the control thermometer must be read and entered into a dialog box displayed on the moisture analyzer screen. The test result is not permanently recorded in the moisture analyzer's memory therefore it must be printed (the printer) or sent to a computer program. Control procedure printout is presented below.

--------- Drying chamber test ---------

Operator	Admin
Start time	2016.03.07 11:15:29
Balance type	MA 3Y.WH
Balance S/N	376820
Control thermometer set no.	87s
Set temperature	105 °C
End temperature	105 °C
Measured temperature	104 °C
Permissible error	+/- 3 °C
Status	OK

.....

(Signature)

NOTE

Possible to be obtained stability of the drying temperature, in reference to the set temperature, is about +/- 2°C. Stability value of this size is efficient enough for typical drying processes. Moisture evaporates from the sample not only when the set temperature is being maintained but also during its rapid increase. Quite a different issue is heating the sample in a constant temperature over a long period of time, however even in such a case the adjustment system ensures thermal stability.

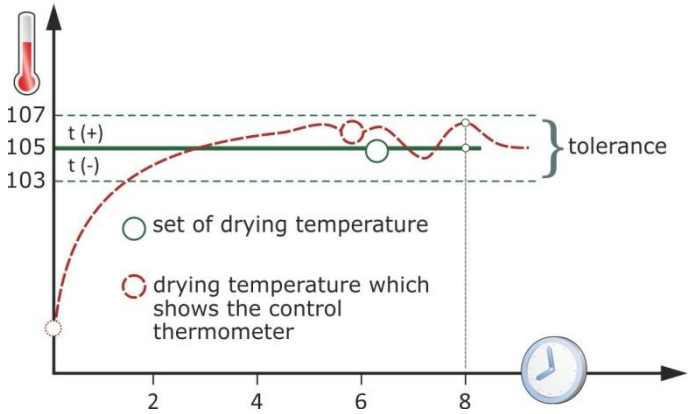


Figure 25. Drying temperature control

13.2. Drying Temperature Adjustment

The control set can be used to adjust the drying temperature. The test is carried out in three different measurement points, the first point is the value of the ambient temperature. Two remaining ones are set by default and can be modified. Heating time aiming to provide stabilisation takes 8 minutes in case of each of the three temperature values. Adjustment consists in entering to the dialog box, the value of the temperature taken form the control thermometer. Control thermometer, GT105K-12/Z, must feature a valid calibration certificate.

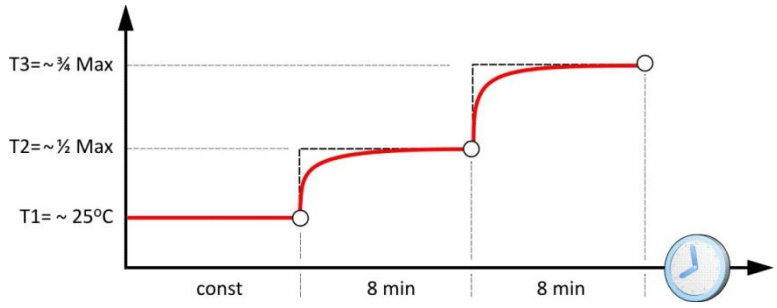


Figure 26. Adjustment of the drying chamber temperature

NOTE

Relation between $\Delta R/\Delta T$ modifications that take place in the thermometer installed inside of the moisture analyzer's chamber is of a linear nature. Periodical calibration of the thermometer is not a necessity. In the course of a long-term usage of the moisture analyzer, change of the thermometer's characteristics is not registered.

14. Methodology-Related Guidelines for Various Areas of Industry

14.1. Dairy Industry

In dairy industry the moisture analyzers are used to access quality of products during a laboratory test. In case of liquid and semi-liquid samples it is necessary to spread a thin layer of the material over a weighing pan surface. Prior to sampling, the material must be stirred. Hard cheese requires grating. Under certain conditions it is possible to use supplementary items such as filters, absorbent paper, silica sand. After the completed drying, the colour of the sample should not change, should it happen otherwise then the change must be minimal. Products of increased level of fat content may be characterized with a greater dispersion of indication within a measurement series than those of low fat content level. A good example of this type of product is powdered semi-skimmed and full-fat milk.

14.2. Food Industry

Vast range of products allows to provide only basic and the most general guidelines concerning the drying methods. Those products that are initially dried in the course of the production cycle must be protected against the impact of humidity, which oscillates within 40 % - 70 % range (EU). Samples of greater size must be either mechanically or manually fragmented. In the course of this mechanical process heat is produced, however its influence on the moisture of a prepared sample is neglected. The smaller the sample, the easier removal of moisture. Mass of the sample must be no greater than few grams (3 g – 6 g), however this is not a strict condition. Exception to the above rule is for example sugar, moisture content of which is low. In case of sugar samples the start mass should be at least 10 g – 15 g. Quite problematic issue here is crystallization of a surface layer of samples characterized with high sugar content. Crust formation is an effect that makes the drying process more complicated. For such an instance it is recommended to protect the surface against overheating, this can be done with use of filters.

Samples characterized with high fat content, for example butter, cannot be dried when placed directly on the drying pan. This kind of sample is placed on filters or absorbent paper, due to which increased active vaporization surface is provided. For liquid samples dry mass content is determined, it is necessary to make sure that they are homogeneous.

14.3. Agroindustry

Moisture analyzers are used in the agroindustry mainly to enable assessment of moisture content of any grains and cereals. If only it is possible the grains should be grinded, crushed, etc. This is done in order to provide sample which is homogeneous as far as moisture content is concerned. To remove moisture from beans may be a complicated issue. Problem seriousness depends on the particular grain or bean structure. Products that have already been processed, such as flour for example, are dried directly on a weighing pan, about 3 g – 6 g sample is applied. Samples of heterogeneous structure such as corn cob mix should be mechanically fragmented into smaller pieces. It is a must since such product is comprised of various materials (corns, leaves, stalk). Large pieces of pre-dried mushroom should be cut into smaller bits. They are dried directly on the weighing pan. Moisture content of mushroom should not be greater than few or a dozen or so percent. Any kind of fodder should be dried as it is, i.e. in its typical form and state.

14.4. Chemical Industry

Chemical Industry means mainly liquid or semi-liquid materials of any type, for which dry mass content is determined after drying. Most users dry the samples directly on the weighing pan. Provided that the sample layer is not too thin, it may be assumed that such drying method is right. However in case of chemical substances it is recommended to increase the active vaporization surface using silica sand or filters. Sample's mass when too great will result with prolonged analysis time. Watery samples may be a bit problematic, their dry mass content is low. After evaporation of the moisture practically nothing is left on the moisture analyzer's sample. For such an instance, the start mass of a sample must be greater. Quite important issue is also the value of scale interval [d].

Example:

In case of a sample of mass 10.000 g, for which the dry mass content left after the drying process is 0.0015 g, use of moisture analyzer with d =0.001 g is insufficient. Potential error of indications due to rounding may constitute even 50 % of the final result, therefore the dry mass indication will be imprecise. For such samples use of moisture analyzers with d=0.1 mg is recommended.

14.5. Bread Industry

In bread industry the moisture analyzers are used to test moisture content of finished goods such as bread, matzo, crackers, buns, gluten-free products etc. The samples must be crushed and cut into small pieces prior drying. It is more than certain that the moisture content in case of a surface layer, subjected to heating, and deeper layers of the sample is different. This needs to be taken into account while preparing the sample, and when analysing the results. Moisture content of bread industry samples amounts to few or over a dozen percent.

14.6. Plastics

Characteristic of plastics requires application of samples of large mass. This is due to low moisture content. The sample can be dried using automatic finish mode, it can also be dried over a particular time interval. The drying time interval should range from 3 to 5 minutes. It is long enough to let the moisture evaporate from the sample completely. While optimising the drying temperature it is necessary to observe sample's surface, too high temperature will result with sticking of the granules one to another, it may also cause change of colour. These days most users apply moisture analyzers with scale interval d of 0.001 g. This is not an optimal solution when it comes to plastic samples. From the perspective of metrology (analysis of errors), use of moisture analyzer with d =0.1 mg sounds more reasonable while drying plastics.

14.7. Fruits and Vegetables

When it comes to fruit or vegetable samples it is checked if certain set threshold value is exceeded or not. This is done mainly for purposes such as potential storage or packing the product into portion packs. Almonds, apricots, pistachios, nuts must be cut or minced into smaller bits. When drying candied fruits, greater dispersion of indication can be expected, this is due to the fact that they contain sugars. Usually the dried samples are not too great, definitely not greater than 10 g. A highly important information is the value of tolerance permissible for the obtained moisture content result.

15. Water Vapour Permeability Test

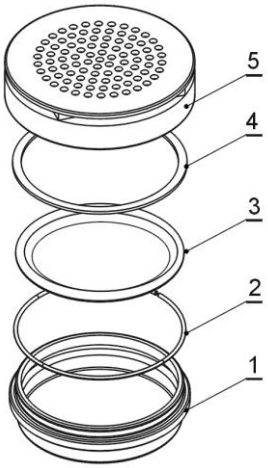
On the basis of mass differences of dried sample, it is possible to determine parameters other than moisture content or dry mass content. A very useful solution is a water vapour permeability determination set. The set is an accessory intended to test permeability to water vapour, which value is used for the purposes of assessment of quality of:

- clothes,
- leather,
- vapour-permeable membranes,
- plastic film,
- laminated materials etc.

The vapour permeability determination set is comprised of two components: a permeability tester and a moisture analyzer. The tester is an accessory dedicated for moisture analyzers and use of it for other testing purposes is impossible. As for the moisture analyser, it is a standard product and can be used either as a precise balance of specified readability, or as an instrument for determination of moisture/dry mass content of various samples.

Photo 21. Tester placed inside the moisture analyzer

The permeability tester is a component of aluminium design, which after assembling forms a hermetic unit from which distilled water is evaporated. Measurement methodology and tester's design are protected by patent right (tester W 116646, measurement method P 381787).



1 body, distilled water is placed inside
2 expanding ring, function of which is to seize sample when it is applied in excess
3 top pan
4 gasket ring, function of which is to ensure that the sample is pressed against tester components
5 lid

15.1. Water Vapour Permeability Testing Method with Use of Moisture Analyzer

Measurement of water vapour permeability consists in precise determination of loss of mass of water which evaporates from the tester through the tested sample. Water vapour permeates as a result of increase of vapour pressure, which is caused by drying chamber temperature raise.

The device measuring permeability is a moisture analyzer, it registers loss of mass of water in the course of the test. The data can be recorded automatically in accordance with a specified time interval. To enable automatic record it is necessary to connect the moisture analyzer to:

- an intended PC software,
- a printer.

It is recommended to use the PC software. On the basis of the recorded data, a graph is generated, which graph visualizes how quickly water vapour permeates through the tested sample, or how quickly the water evaporates for so called blank test.

Measure of the water vapour permeability is the quantity of water vapour permeating through the material, expressed in % in relation to loss of mass of water when on the tester no sample has been placed, so called blank test.

15.2. Test Conditions

When it comes to ambient conditions it is necessary to monitor temperature and the humidity of the room. The temperature should range between 21°C - 26 °C, the humidity must be comprised within 40% - 60% range. Variation of temperature during the cycle cannot exceed ± 2 °C, of humidity ± 5 %. It is assumed that the temperature of the moisture analyzer's drying chamber is stable throughout the test cycle.

15.3. Devices and Materials

Workstation for the water vapour permeability testing must be equipped with the following materials and devices: moisture analyzer with at least 1 mg scale interval [d], permeability tester, thermo-hygrometer, pipette for distilled water, distilled water, computer with respective PC software (optional accessories).

SUMMARY

WATER VAPOUR PERMEABILITY

Methodology regarding water vapour permeability testing, carried out with use of permeability tester, allows to quickly compare materials one to another. Such tests are useful wherever sample structure is modified in order to provide optimal sample composition. Obtained water vapour permeability value allows to assess how the modification-causing component influences the material characteristics. Advantages of such solution are speed, universality and low costs. Since this method is not standardized then it should not be used to assess quality of materials in public trading. Validation and accreditation of testing method for selected materials is open.

16. Moisture Analyzers - Legal Requirements

State authorities supervision over the weighing instrument such as balance is limited to control of indication of mass. Regardless of the displayed indication (% value, pieces quantity, sample's moisture content), in the course of control it is always the result expressed in grams (kilograms) that is being evaluated. Other values are a result of calculations done on the basis of the elementary indication. This kind of an approach is right in case of balances, but not when it comes to moisture analyzers. The moisture content result does not depend on mass indication exclusively but it is affected also by sample size, drying process temperature, etc. Since there are no moisture content standards, then it is not possible to assess accuracy of moisture content measurement in accordance with law regulations. This seems to be a stalemate because due to the universality the moisture analyzers are used also in institutes supervised by the national authorities, e.g. environmental protection. How can the above problem be solved then?

In order to be accordant with the legal system regulations, it would be wise to use moisture analyzers with type approval certificate. However, as it is known, the assessment in the course of certification, next during verification, concerns measurement in grams. The obtained result will provide no information regarding accuracy of moisture content determination. This kind of approach seems to be senseless. Solution to the above may be validation of drying procedures. Validation is a set of operations aiming to prove that the used device meets all the imposed requirements.

The requirements should concern mass measurement, drying temperature stability, accuracy of moisture content determination etc. This is an example showing that technology is one step ahead of the law. Accreditation, which becomes more and more significant, allows to look at the problem optimistically. Maybe the best solution would be calibration of mass indication and the drying chamber temperature. Law regarding weighing equipment subjected to legal metrological control do not concern devices intended to measure moisture content, i.e. moisture analyzer.
(Regulation of the Minister for the Economy (OJ 2008 No 3, item 13, and OJ 2010 No 110, item 727).

17. Drying Procedures Validation

Wherever in the course of the moisture content tests, methods specified by standards are used, there are no greater problems. This is mainly due to the fact that the whole procedure is described in details step-by-step, the description includes equations and relevant notes and guidelines. Similar documents regarding moisture analyzers are also available and can be referred to, however, since they are prepared by manufacturers themselves, not by an international organization, their rank it is not as high as in case of standards. Regardless of this fact, these documents provide valuable information, helpful to many users.

As practice shows, seemingly less complicated solutions such as moisture analyzer are often applied even though the standard-specified methods are provided. As a consequence of the above, the drying procedure should be validated. Validation generally consists in respective selection of drying parameters, such ones which would guarantee that the result obtained using moisture analyzer is comparable to the one provided using standard-specified method. Some of the users delegate performance of standard tests to the accredited laboratories, others relay on

their own experience, internally carried out tests or results of some tests performed externally. These are ways of obtaining information about the real moisture content of the sample.

The first thing required for the purposes of validation is information regarding sample's moisture content. Lack of this information makes it impossible to carry out validation. It often happens that standard-specified tests are performed by the manufacturer's laboratory – like it is in case of Radwag. Such test is followed by a second stage, i.e. optimization of the drying process parameters and designing of methodology that would take into account particular sample's characteristics. General diagram of validation is presented below.

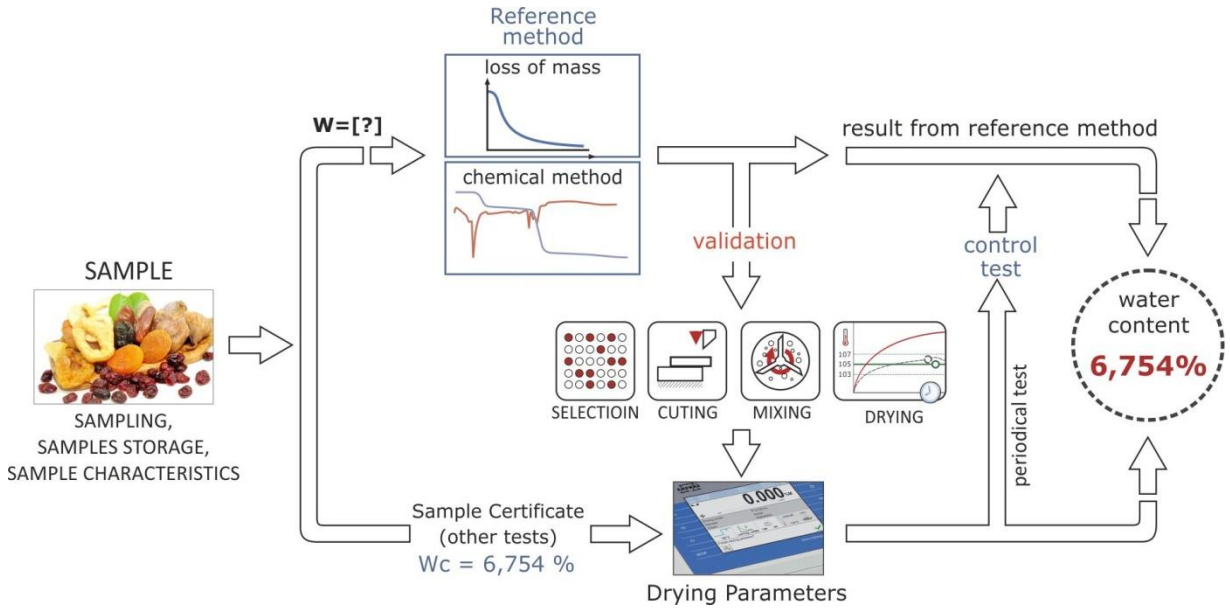


Figure 27. Drying procedures validation

18. Uncertainty of Moisture Content Measurement

Each measuring process is characterised by so called error of indication on the basis of which accuracy of measurement of a particular quantity is accessed. Accuracy is nothing else but a difference between the obtained result and a true value. In case of moisture content measurement the true value is the one obtained using standard-specified method. The obtained result versus the true value is quite obvious metrological relation, characteristic for many supervision systems of the given state over the measuring equipment. It is also crucial in cases where the state supervision is not required or even impossible. One of such examples is a moisture analyzer for which there are no moisture content standards.

Each measurement result, regardless of the measuring device type or the measurand is characterised with uncertainty. Uncertainty specifies range within which, usually with 95% probability, the measured value is comprised. When it comes to error of indications, we speak of comparison of two values, in case of the uncertainty, many factors influencing the measurement have to be referred to. The uncertainty value, although seemingly similar to the error of indication, cannot be used in order to assess the measurement accuracy.

Uncertainty of determination of given sample's moisture content should take into account the fact that the measurement is a differential one. In order to obtain the final result it is necessary to measure sample's mass prior the drying process and after its completion. Additionally the

uncertainty is influenced by the process of sample preparation, drying method and deviation of the drying temperature. The basic budget of uncertainty may include the following: indications repeatability, weighing instrument's resolution, error of indication, and the uncertainty of determination of the indication error.

When all the above are referred to in calculations, then the expanded uncertainty for the measurement of sample's start mass is:

$$m_1 \pm 0.004 \text{ g}$$

However one important thing should be remembered. It can be assumed that the start mass is measured with an uncertainty of such value, but not necessarily the final mass. It has been already said that in the course of drying various unfavourable processes may occur, for example crust may form on the sample's surface, part of the sample may be a subject to thermal decomposition, bottom layer may still contain moisture, etc. With all that in mind it is not hard to conclude that uncertainty value in case of determination of sample's final mass m_2 may be influenced by totally different significant factors. It can be even said that their impact is dominant. Among these factors there are:

- the drying temperature, it oscillates within +/- 2^{o}C in relation to the set temperature. This range of variation does not affect the sample's moisture content result. However incorrect drying temperature, e.g. too high, will be a reason of significant dispersion of indication within one measurement series, caused by surface burning of the sample (solids).

- methodology of sampling, sample preparation and drying. Some of the materials require crushing or grinding, or being dried with other components, etc. Inappropriately prepared sample as a result will give low repeatability of the measurement series. This may be one of the most crucial issues in the course of optimisation of the drying method parameters.

It is practically impossible to estimate how great the influence of these factors is, it will vary depending on the sample type. What the operator can do is to minimize influence of respective factor by means of relevant methodology. In order to design relevant methods it is necessary to carry out many tests, which tests aim to provide better results regarding final mass of the sample, m_2.
Previous chapters provide a lot of details concerning the methodology. It can be concluded that determination of combined uncertainty is a complex issue and may not end up with a success.

Another approach, much less complicated is defining uncertainty of moisture content measurement as UNCERTAINTY of type A - moisture content measurement is a direct measurement. In this case it is necessary to assume that the selected drying method is not affected by any negative factors, in other words that:

- drying temperature is optimal, which means that it is of value allowing to remove moisture from the sample simultaneously not causing burning of the sample,
- sample's mass of respective value is selected, i.e. the drying temperature heats the sample causing evaporation of the whole moisture
- relevant finish mode is set, i.e. process parameters ensure that the drying time is long enough to remove moisture from the sample completely
- additional methods and some supplementary accessories are used, if necessary, to improve moisture release.

On the basis of series of measurements an arithmetic mean is calculated. This value is an estimator for a real sample's moisture content.

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

Dispersion of moisture content indications for a particular sample is characterised with an experimental standard deviation. It can be calculated using the following equation.

$$s(x_i) = \sqrt{\frac{1}{(n-1)}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

Standard uncertainty of the mean value $\bar{x}$ of series of measurements is an experimental standard deviation of an arithmetic mean. It is calculated using the following equation.

$$u(x) = \sqrt{\frac{1}{n(n-1)}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

Although the equation refers to a mean value, $u(x)$ symbol is used instead of $u(\bar{x})$.

19. MA 3Y Moisture Analyzers

The moisture analyzer offers certain measuring functions (metrology) and plenty of functional facilities. Sometimes they are as much important as technologically crucial features, which is due to the fact that they allow smooth, ergonomic operation. These days the users do not read the technical documentation thoroughly from cover to cover, they expect intuitive solutions like the ones offered by RADWAG-manufactured moisture analyzers.

Figure 28. MA.3Y moisture analyzer – functional features

The main functional features of moisture analyzers of MA.3Y series:

1. programmable infrared sensors – moisture analyzer control,
2. communication interfaces: 2 x USB 2.0; 4I / 4O; RS 232; Ethernet 10/100Mbit,
3. wireless communication, terminal – weighing module,
4. customization - day-to-day operation can be customized, each operator can make an individual user profile,
5. auto control of the level, patented LevelSENSING system.

Drying process is documented by means of reports which are comprised of the following sections: header, line and footer. The users can design their own advanced non-standard printouts presenting various information, displayed by means of universal variables (e.g. date, time, sample's mass, etc.) and alphanumeric data. With this, it is possible to provide required printout format.

Technical specifications of MA.3Y moisture analyzers

	MA 60.3Y	**MA 200.3Y**
Maximum capacity	60 g	200 g
Readability	0.1 mg	1 mg
Tare range	-60 g	-200 g
Weighing pan dimensions	ø 90, h= 8 mm	
Operating temperature	+10 - +40 °C	
Power supply	230V 50Hz AC	
Display	5.7'' (touch screen)	
Communication Interfaces	2 x USB 2.0; 4I / 4O; RS 232; Ethernet 10/100Mbit, Wireless Communication	
Packaging dimensions	595 × 395 × 420 mm	
Net weight	6 kg	
Gross weight	10 kg	
Maximum sample weight	60 g	200 g
Moisture content readability	0.0001 %	0.001 %
Moisture content repeatability	+/-0.05% (sample ~ 2g), +/-0.01% (sample ~ 10g)	
Drying temperature range	Max 160 °C (option: 250 °C)	
Maximum sample height	Max 20 mm	
Heating module	IR emitter (option: halogen lamp)	
Heating module power	400 W	
Drying method	4 drying profiles (standard, fast, step, mild)	
Finish mode	4 options (time-defined, user-defined, automatic, manual)	
Additional functions	sample traceability, drying process graph	

20. MA.R Moisture Analyzer and MA X2.A

MA.R and MA X2 series offers similar functionality like the MA.3Y. However due to used display type, access to some of the functions requires greater knowledge on the menu structure of the MA.R moisture analyzer, and a little bit of experience. This concerns especially those users who want to use databases and the drying programs.

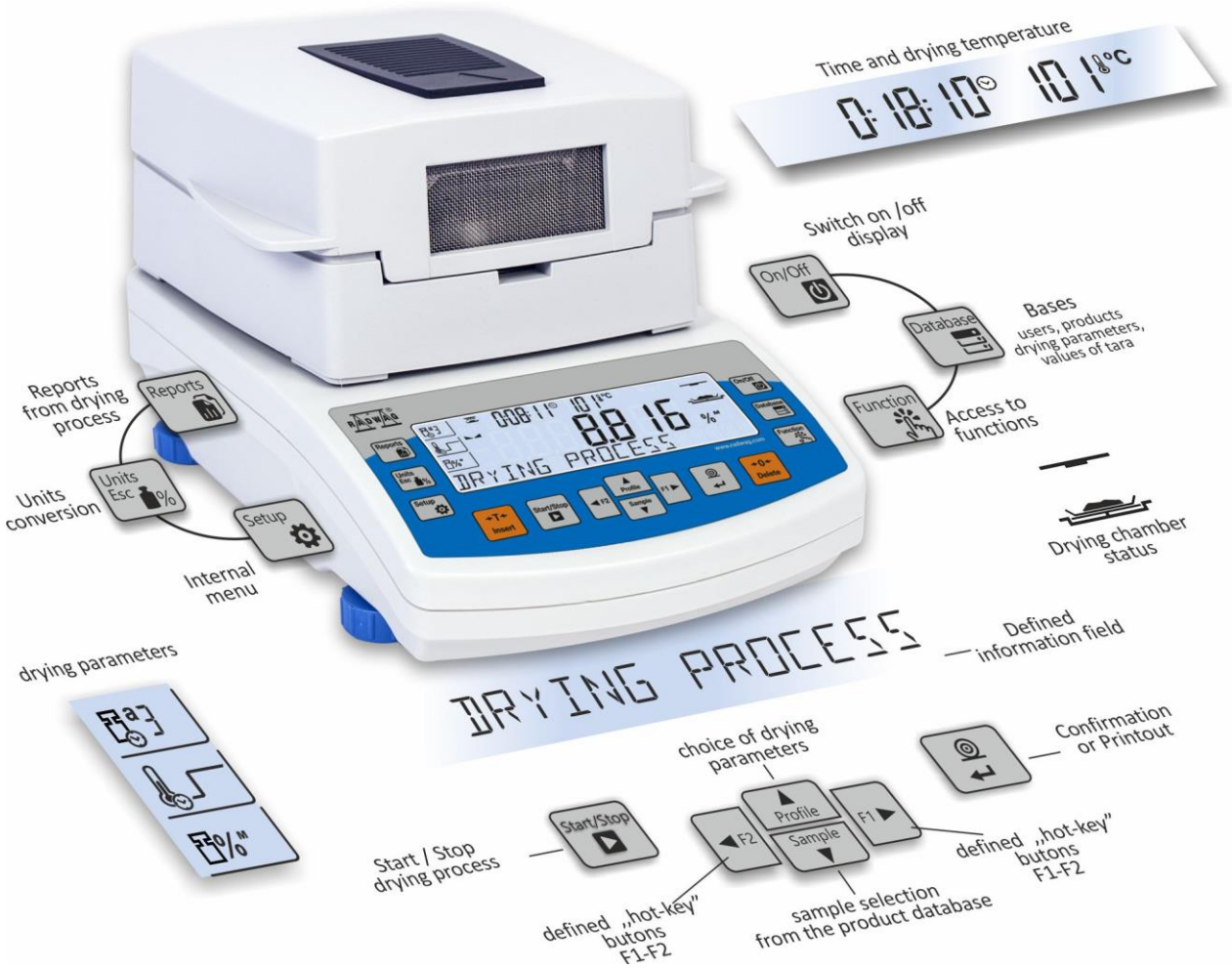


Figure 29. MA.R moisture analyzer – functional features

The information system of MA.R moisture analyzers is based on 6 databases:

- operators (100 records maximum)
- products (up to 1000 records)
- weighings (up to 1000 records)
- tares (up to 100 records)
- drying programs (up to 100 records)
- drying process reports (up to 1000 records)

The information system offers two-directional exchange of data via USB interface. MA.R moisture analyzers allow databases import and export by means of memory sticks.

Technical specifications of MA.R moisture analyzers

	MA 50/1.R	MA 50.R	MA 110.R	MA 210.R
Maximum capacity	50 g	50 g	110 g	210 g
Readability	0.1 mg	1 mg		
Tare range	-50 g	-50 g	-110 g	-210 g
Weighing pan dimensions	ø 90, h = 8 mm			
Power supply	230V 50Hz AC			
Display	LCD with backlight			
Communication Interfaces	1×RS 232, USB-A, USB-B, Wireless Communication (option)			
Packaging dimensions	470 × 380 × 336 mm			
Net weight	4.9 kg			
Gross weight	6.4 kg			
Sample's mass	max 50 g	max 50 g	max 110 g	max 210 g
Moisture content readability	0.0001 %	0.001 %		
Moisture content repeatability	+/-0.05% (sample ~ 2g), +/-0.01% (sample ~ 10g)			
Drying temperature range	Max 160 °C (option: 250 °C)			
Maximum sample height	Max 20 mm			
Heating module	IR emitter (option: halogen lamp)			
Drying method	4 drying profiles (standard, fast, step, mild)			
Finish mode	4 options (time-defined, user-defined, automatic, manual)			

MA.X2.A moisture analyzers are hi-tech measuring instruments intended for quick determination of relative moisture content, dry mass content and other parameters in samples of different substances. MA.X2.A series is equipped with innovative system: the drying chamber can be opened and closed automatically using button or proximity sensors. Highlights of MA X2.A series moisture analyzer:

- Automatically opened drying chamber
- Automated drying process
- DRYING FORECAST – Prognosis of the Drying Process

MA X2.A series moisture analyzers are now equipped with DRYING FORECAST function that enables to shorten the drying process. If you do not care about the highest accuracy and increased measurement error (ranging from 5% to 20% of the end value) you can shorten the drying process up to 6 times. It is an option dedicated for users who require fast and multiple estimation of materials moisture content. DRYING FORECAST method is a prognosis of the end result, carried out before the drying process is completed. Based on characteristics of current drying curve, created online, moisture analyzer estimates the end result of the drying process.

Figure 30. MA.X2.A moisture analyzer – functional features

The information system of MA.R moisture analyzers is based on 6 databases:

- operators (100 records maximum)
- products (up to 5000 records)
- weighings (up to 1000 records)
- tares (up to 100 records)
- drying programs (up to 100 records)
- drying process reports (up to 1000 records)

Technical specifications of MA.X2.A moisture analyzers

	MA 50/1.X2.A	MA 50.X2.A	MA 110.X2.A	MA 210.X2.A
Maximum capacity	50 g	50 g	110 g	210 g
Readability	0.1 mg	1 mg		
Tare range	-50 g	-50 g	-110 g	-210 g
Weighing pan dimensions	ø 90, h = 8 mm			
Power supply	230V 50Hz AC			
Display	LCD 5” capacitive touchscreen			
Communication Interfaces	1×RS 232, 1×USB-A, 1×USB-B, 1×Wireless Connection			
Packaging dimensions	470 × 380 × 336 mm			
Net weight	5.2 kg			
Gross weight	6.7 kg			
Sample's mass	max 50 g	max 50 g	max 110 g	max 210 g
Moisture content readability	0.0001 %	0.001 %		
Moisture content repeatability	+/-0.05% (sample ~ 2g), +/-0.01% (sample ~ 10g)			
Drying temperature range	Max 160 °C (option: 250 °C)			
Maximum sample height	Max 20 mm			
Heating module	IR emitter (option: halogen lamp)			
Drying method	4 drying profiles (standard, fast, step, mild)			
Finish mode	4 options (time-defined, user-defined, automatic, manual)			

21. Summary

Moisture analyzers are universal measuring instruments, due to this they can be used both in spotlessly clean laboratories or workrooms of pharmaceutical companies, and in industry where they facilitate control of an ongoing production. The accuracy of the moisture analyzer results depends on the adopted methodology, which methodology should be designed (verified) in the course of validation. Only this kind of approach guarantees success and correct assessment of the real state of the sample. Consultations with our customers who operate moisture analyzers on a regular basis let us realize that users look for ready drying procedures. In order to meet this expectation, RADWAG has been undertaking research activity for years. We carry out various tests regarding moisture content issues and provide the customers with technical, practical and scientific knowledge. This very publication is one of many examples of our struggle to educate those who deal with weighing. I hope that problems discussed above will be helpful, especially for those whose role is to implement moisture analyzers and take advantage of their expanded functionality on a greater scale. Maybe they will select products manufactured by RADWAG, such products that have been tested, and therefore are recommended, by our Laboratory for years.

Sławomir Janas

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