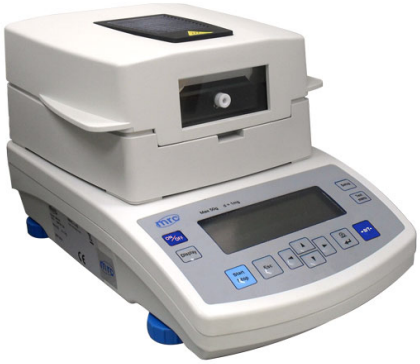
MOISTURE ANALYZERS IN HUMIDITY DETERMNATION PROCESS



It is commonly known, that moisture content may affect various physical features of a substance. Hence, there is a need to match humidity content on selected stages of manufacturing process or control of a product. In most cases the process of humidity or dry mass determination is relatively fast and reliable.

For above reasons on reliability and repeatability, there should be instruments which provide such operation. Such features are offered by moisture analyzers, which are dual function instruments for broad application in laboratories and industry. The aim of this publication is to show directions and hints for optimal application of a moisture analyzer.

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1. Introduction

Substance humidity is one of the criterion which determines substance quality. In most cases, determination of humidity should be performed quickly and reliably. It refers to all laboratories and testing facilities which operate as manufacturing plants.

Traditional drying method which is applicable in such cases concentrates at:

- Weighing,
- Drying for a couple of hours and repeated weighing

Time devoted to determination of humidity with traditional method is too long. An ideal instrument for quick and reliable measurement is a moisture analyzer, which similarly to traditional method utilizes phenomenon of **THERMOGRAVIMETRY**.

Thermogravimetry is a process at which decrement of mass is determined if a substance is heated. In this process, a sample is weighed before and after drying process, and a difference in mass between these two is calculated. The term comes from Latin three sectioned name:

Thermo = heat

Gravi = weight

Metry = method

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2. What humidity of a substance is?

In thermogravimetric methods (as mentioned above also in case of a moisture analyzer) all substance components volatilize if this substance is heated. The components are grease, aromatic substances, organic solvents, chemical additions, water and other component, which may appear as result of thermal decomposition (so called burning products). Thus, humidity of a substance is a composition of all components which appear in a sample, and which evapourate during its heating. Unfortunately, most of the operators do not differentiate humidity and water content in a sample.

Generally it is not possible to distinguish decrement of pure water from other substance components. It should be mentioned here, that the decisive factor here is drying temperature. If it is too high, than sample is burned, and it is not possible to determine decrement of other components. In drying process, there is a commonly used term of “free water”, i.e. water that is removed fro ma sample at temperature of $105^{o}C$.

Bearing in mind the above statements, it is justified to implement a term of dry mass content in a sample.

3. Methods for determination of humidity

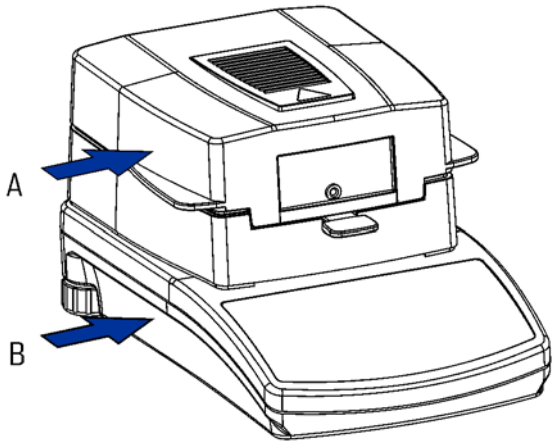
There are several methods for determination of humidity in substances. Usually, all methods are divided into two main categories:

- **Absolute**
  moisture content is matches directly, e.g. as a decrement in mass recorded in standard weighing process.
- **Deductive**
  Moisture content is determined indirectly, physical features that refer to moisture content in a substance are measured (like: absorption of electromagnetic waves or electric conductivity).

4. General data on moisture analyzer design

A moisture analyzer consists of a precision laboratory balance and a drying chamber that is attached to the laboratory balance. The drying chamber provides stable temperature on measuring process. Such design of a moisture analyzer makes the drying process different from traditional method. The functioning of a moisture analyzer is basically:

- Precise determination of mass of a weighed sample before drying process and during this process with no need to take the sample out of a drying chamber
- Automatic finish of a drying process */ drying till dry mass or dryign till elapse of set time interval /*
- Calculation of drying process result by a algorythm for selected drying profile
- Forwarding data from drying process to a pronter or computer if there is a need to prepare drying process documentation.



A – drying chamber
B – weighing section

Fig. 1. Moisture analyzer design schema

Most of moisture analyzers are equipped with a drying chamber installed above weighing section of the instrument. Such design proviodes easy and reliable analysis of sample mass decrement. Moisture analyzers may differ in shapes and dimensions, but they have one common element, which is separation of drying chamber from weighing section.

4.1. Mass measurement

This process is the same as in case of most electronic balances. It is the process of measuring force with which a sample is drawn by Earth, according to a formula:

$$F = m \cdot g$$

[1]

where: *F- force*
m – sample mass
g – gravitational acceleration force in measuring location

Hence, there is a practical aspect, namely: each moisture analyzer can be adjusted, if it is planed to be used as a precision balance. Adjustment process eliminates errors which occur as result of gravitational acceleration force change. Most of moisture analyzers are instruments that are equipped with adjusting system with use of external mass standard. Adjustment of a balance should only be performed with a mass standard in adequate accuracy class.

Instrument adjustment

It is a collection of activities which ***set a relationship*** *between a value indicated by a balance, and a reference mass (adjusting mass standard), which is a load placed on balance weighing pan, and* ***correction activities*** *of mass indication, if there is such need.*

Systems of automatic internal calibration is practically not installed in moisture analyzers, as the main application of a moisture analyzer is determination of humidity, which is measurement of a difference in masses.

The other aspect relates to continuous operation of a moisture analyzer, during which system of automatic internal calibration could activate, causing incorrect analysis of humidity.

4.2. Determination of moisture content

Determination of moisture content, or determination of dry mass content is a differential measurement, in which the operator measures sample mass before the drying process and after its finish. Thus, there is no need to determine mass extremely precisely. Moisture content is calculated by a formula:

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

[2]

Where: *W- humidity*

M_P *– initial sample mass*

M_K *– final sample mass*

Practically, one of the measured values is known already at the beginning of an analysis. It is the initial mass of a sample. final mass of measuring process depends on a few factors, which are:

- Drying temperature
- Size of analyzed sample
- Auto switch-off criterion, if it is active.

Dependence of final result from above factors requires (mainly from the manufacturers) performance of drying tests. Such tests offer specification with drying parameters of different substances with reference to traditional method and norms. It is a great help for moisture analyzer operators, who use ready schemas for drying substances.

5. Selection of a moisture analyzer

When choosing a moisture analyzer, the operator should focus on applications for which an instrument will be used. If a moisture analyzer is to perform drying procedures only, than purchase of a 50 g capacity instrument is sufficient. Determination of moisture content requires relatively small samples. As for temperature range, the standard offers maximal temperature of 160^{o}C, which

is relevant for 95% of operators. However, in case where samples have to be dried in very high temperatures, the operators should search for instruments equipped with temperature range increased to 250°C.

If a moisture analyzer is to be used also as a precision balance, than an operator should focus on three main factors:

- Maximal capacity
- Necessity have an calibration weight (mass standard) with adequate accuracy class
- Dimensions of drying chamber.

Weighing range in case of most of moisture analyzer is maximum 50 g. however, in case of some substances this may not be enough. Substances with very high thickness may require application of a moisture analyzer with maximal capacity of 100g or 200g.

Calibration weight is a typical weight (mass standard), which is used in case of precision balances. Thus, it is possible to use a single weight for several moisture analyzers or precision balances.

Weighing chamber (drying chamber) in case of moisture analyzers is relatively small, and for this reason its usability is limited, mainly by its height.

5.1. Halogen lamp or infrared emitter?

Halogen lamps and infrared emitters that are applied in moisture analyzers emit waves ranging from 0,76μm to 1000 μm which transfer heat to dried substance. basically, both sources of heat are infrared emitters, but they operate with different wave length. Different names are given to distinguish the two types of heat source.

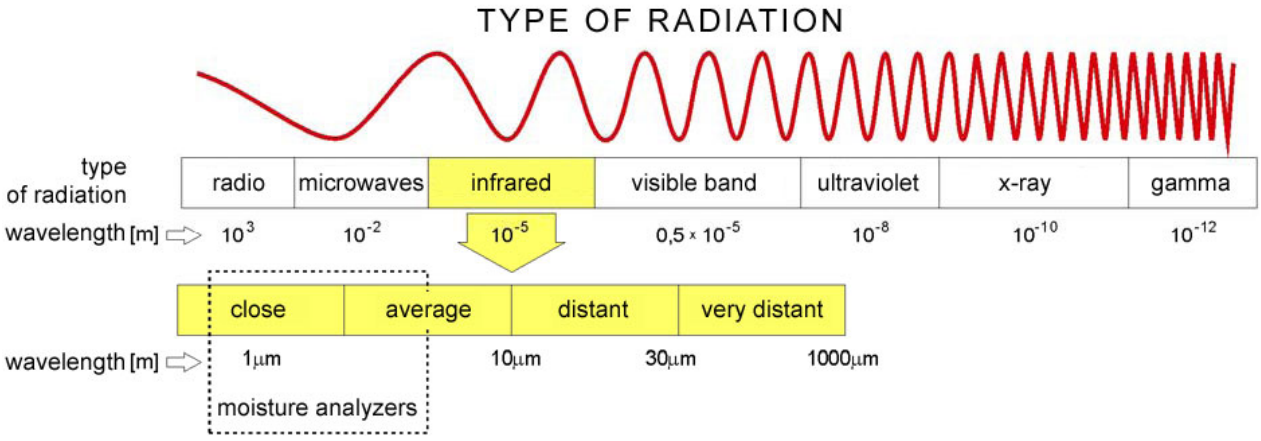


Fig. 2. Division of electromagnetic waves

Halogen lamps – emitters that emit infrared waves, are divided into:

- **IRS** (infrared short), wave length IR 1,2µm.
- **IRM** (infrared medium) - wave length IR 3µm.
- **IRL** (infrared long) wave length IR 5µm.

Above specified division is a conventional one, as wave length overlap each other, and in some application one distinguishes short waves and the same wave length can be considered as wave of medium length. When heating a sample, there are two effects applied:

- Radiation and
- Convection.

Radiation is transferring heat from one substance to another, and amount of transferred heat depends from:

- Difference in temperature between the emitter (sender) and receiver (sample)
- Ambient area
- Length of emitted wave

During radiation process, it is possible that some substances may indirectly influence heat transferring process. In case of a moisture analyzer, the indirect substance is the air.

Convection is transmission of heat with movement of air particles, which are circulating in drying chamber of a moisture analyzer. Heated air, as the lighter one, moves upwards, and cooler air moves downwards. Such circulation allows for transferring energy with use of medium.

Infrared radiation (generated by infrared emitters) is mainly absorbed by water vapour particles. This is due to the fact that the energy of intra-molecular vibration is of the same order as the energy quanta of infrared radiation. The occurrence of this phenomenon is contingent upon the occurrence of radiation of appropriate wave length.

Infrared emitters emit longer waves (wave colour is more red), but less energy. With the increase of wave length, the reflection ratio decreases – more energy is absorbed by a substance (up to 80 %). Such waves are applicable for substances that have high reflection ratio, and substances with dark colour. In such cases, the infrared radiation uniformly penetrates all sample structure.

DEFINITIONS:

REFLECTION — change in the direction of wave propagation on the border of two media that causes its remaining at the medium, where it spreads. Reflection can produce a mirror image, or be blurred, keeping only the wave properties, but not an accurate picture of its source.

REFLECTION DIFFUSION - if the reflecting surface wave is not smooth, there is a scattering reflection. The wave is not reflected in one direction, only scattered in all directions. An example might be the reflection of light from the surface of cards in a book. A lamp illuminates a page, and relflected waves radiate in all directions. This way a reader can see letters. In case of plane photographic paper plain paper, photographs prevents from reading in certain angles of view, because it hampers the light reflected from a mirror smooth surface. Scattering phenomenon is dependent on the ratio of surface roughness to the wave length.

The longer the wave and more smooth surface, the lesser is scattering.

Halogen lamps have bigger energy, but only in case of shorter waves (brighter light). The smaller wave length, the more radiation is reflected. Thus, in case of halogen lamps, reflection constitutes for approximately 50 % of total balance of transferred energy. The ratio between radiation and convection in drying process in expressed in below chart.

	Infrared emitter	Halogen lamp
Radiation	80%	50%
Convection	20%	50%

When discussing this problem, it should be noticed, that halogen lamps heat the drying chamber of a moisture analyzer much quicker (because of higher emission of energy), and thus analysis time is shorter. When deciding on a type of heating source, apart from operation features, one should also consider economical aspect. Infrared emitters are much more expensive than halogen lamps. It can be said, that 99 % of moisture analyzers that are in use, are heated by halogen lamps.

6. Temperature sensor adjustment

During manufacturing of a moisture analyzer, each model undergoes a process of temperature sensor adjustment. Its correct indication is very important in future drying cycles. Temperature senso scalingis performed in a few points, so that temperature can be correctly indicated during

analysis. It should be mentioned here, that temperature sensors are scaled with no load on the weighing pan of a moisture analyzer. If there is a sample on pan of a moisture analyzer, temperature distribution in drying chamber may be different and changeable. Temperature in drying chamber depends also on sample colour and humidity evapouration process.

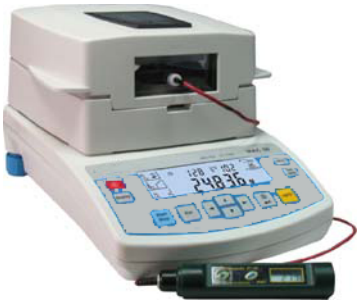
Generally, temperature sensor adjustment is performed as factory setting of a moisture analyzer and it should not be performed by an operator. If own tests give unsatisfactory results on drying temperature values, please contact MRC authorized service or order an expertise.

7. Temperature indication control

It is a procedure which checks whether set temperature is reached and held in drying chamber. Devices applied for such testing procedure may have a form of specific indicators, which are stuck to weighing pan of a moisture analyzer. They are accessible in three temperature ranges, so that they cover all temperature range of a moisture analyzer:

- 88 ÷138 °C
- 143 ÷ 193 °C
- 199 ÷ 260 °C

Control with application of above indicators is an general one, due to indicator design and resolution.



More precise control can be performed with application of electronic thermometers, for instance PT 101 or PT-401 (version equipped with RS 232). It is a very good solution for operators, who apart from sample mass change have to precisely control temperature of drying process (for instance required by ISO quality system certification).

It is important to remember that both thermometers (integral part of moisture analyzer, and test thermometer) should be equipped with probes of the same colour. In case where one probe is light (stainless steel), and the other is dark, there may occur errors souring from:

- Heat absorption by dark coloured probe and
- Reflection of light by light coloured probe

In result difference in temperature indication by both thermometers can reach 20^{o}C.

Control of drying temperature takes place from the moment of process initiation. The first stage of drying process is a dynamic one, in which differences between indicated temperature and actual temperature in drying chamber as shown by control thermometer may occur. It results from heating process of drying chamber, and distribution of temperatures in its interior. In the second stage of drying process the temperatures are leveled. As drying process temperatures interval in moisture analyzer is set for +/- 1°C correct temperature indication is reached if differences between observed temperatures do not exceed +/- 2°C.

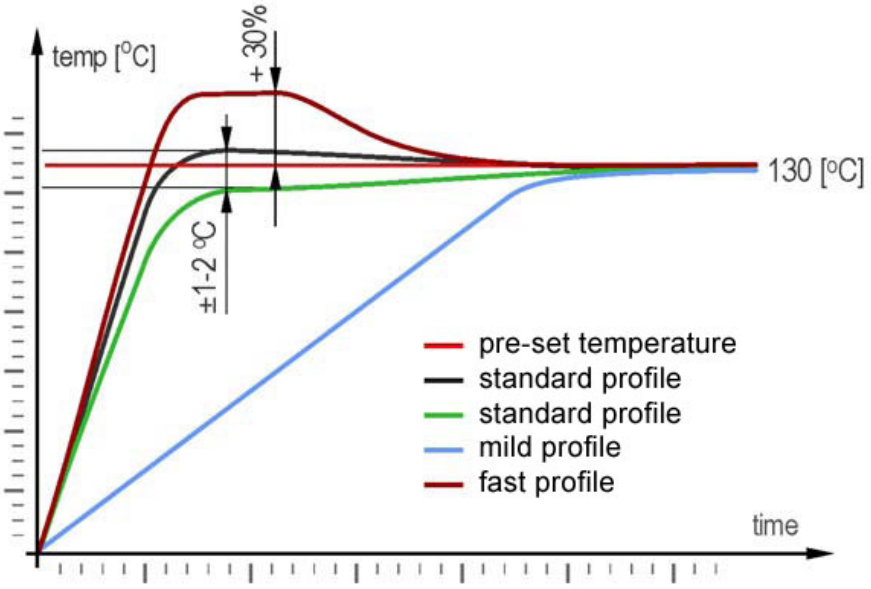


Fig. 3. Drying profiles

When controlling temperature of analysis, the operator should also consider selected drying profile. As presented on picture above, in case of mild profile, such control is possible only after some time from analysis start. It is opposite in case of fast profile, in which the temperature goes beyond set level.

Literature: Internet website: www.mrclab.com