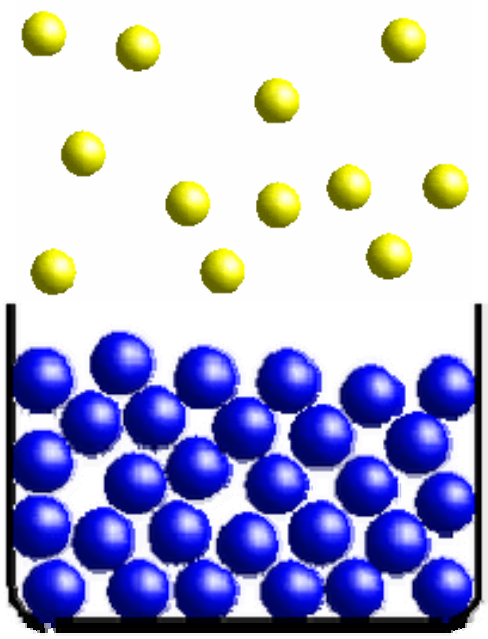
sartorius
mechatronics

Infrared drying in practical application

Moisture determination in material – the basics



- Definition of terms
- Physical principles
- Functional principles

Why is it important to measure the moisture content?

Moisture is a component of nearly all raw materials and end products. It has an important impact on the quality of products, for example it can affect the.:

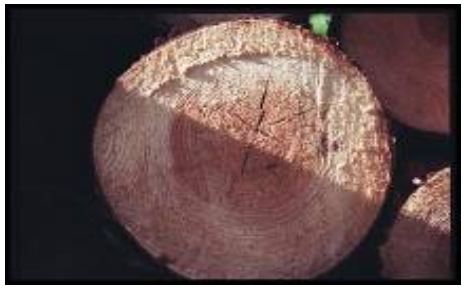
- Perishability (microbiological stability)
- Shelf-life
- Physical and chemical properties
  (flow properties, viscosity)
- Commercial grade
- Price

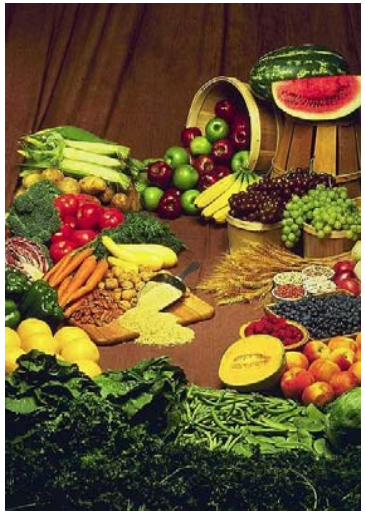
Areas of application for moisture analyzers

Used in the monitoring of time-critical processes:

- As an easy-to-use method for analyzing solid or pasty materials and solutions with moisture contents between 0.1 – 100 %.
  - Incoming inspections
  - Production process
  - Quality control
  - Trade & commerce

Areas of application for moisture analyzers





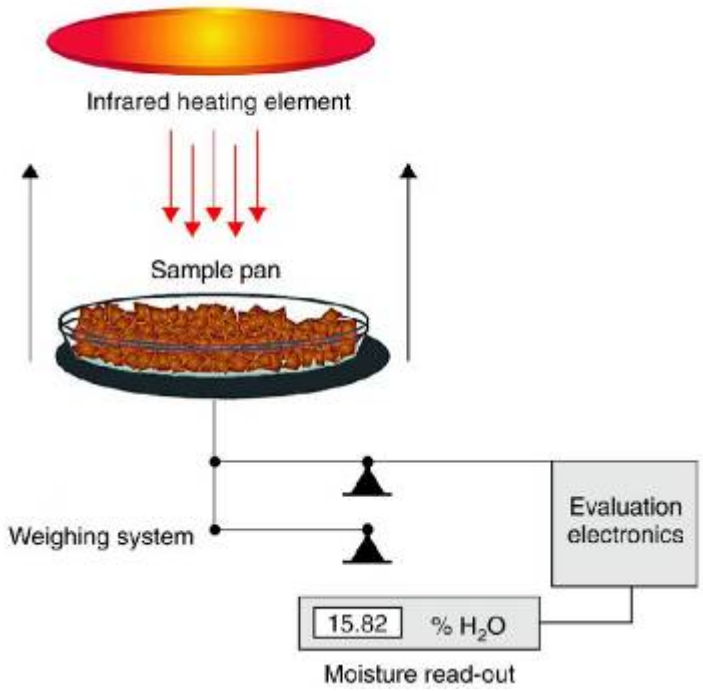
- No industry-specific limitations
- Universal in use:
  - Trade & commerce
  - Chemicals & pharmaceuticals
  - Environmental protection
  - Building materials
  - Food & beverage industry

Definition of the term “moisture”

- In thermogravimetry, the term “moisture in materials" refers to all the constituents that evaporate when the sample is heated, thereby leading to weight loss. These constituents include:
  - Water
  - Oil/Fat
  - Alcohol
  - Flavorings
  - Decomposition products
- Errors in handling (use of the wrong operating parameters or improper sample preparation) have an effect on the result. However, these errors are often reproducible so that a “wrong“ value is still be considered valid!

What is thermogravimetry?

- Thermogravimetry is the method for extracting moisture from a sample by heating (thermo) it and determining the resulting loss of weight (gravimetry) using a balance or scale.



Equations

$$\frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \cdot 100 = \%\ \text{Humidity}$$

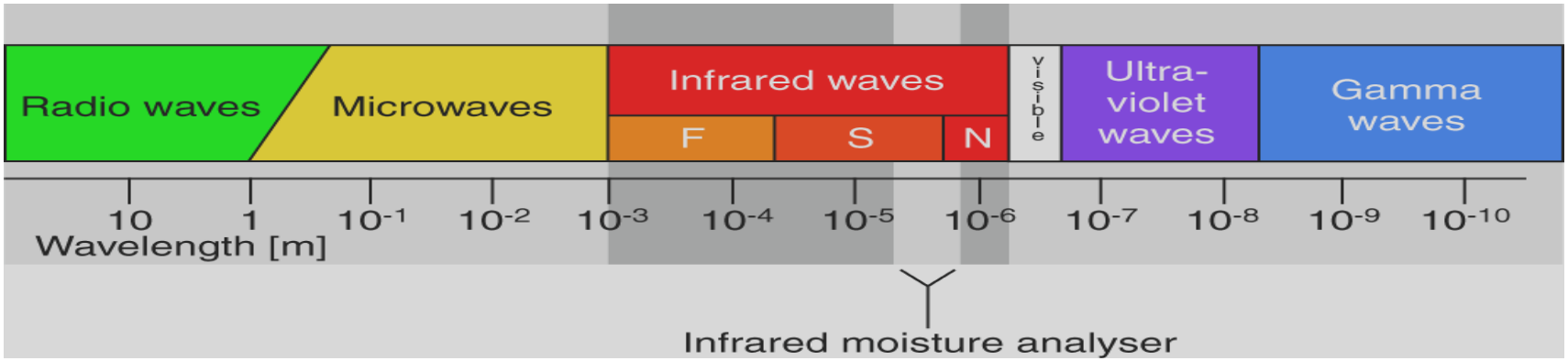
$$\frac{\text{Final weight}}{\text{Initial weight}} \cdot 100 = \%\ \text{Dry Weight}$$

$$\frac{\text{Initial weight} - \text{Final weight}}{\text{Final weight}} \cdot 100 = \%\ \text{RATIO (1)}$$

$$\frac{\text{Initial weight}}{\text{Final weight}} \cdot 100 = \%\ \text{RATIO (2)}$$

What are infrared rays?

- Infrared rays are part of the electromagnetic spectrum.
- This invisible heat radiation generates wavelengths within the long-wavelength limit of visible red light.



- Infrared rays are governed by the laws of optics and can be focused using a concave spherical mirror or a concentrating reflector.

Wavelenght of various IR radiators

Radiator or heat source	Wave length in µm Measured from peak to peak	Surface temperature (°C)	Spectral properties
Metal rod radiator	2.8 – 4.4	400 - 750	Standard range to far infrared radiation
Infrared lamp	> 1.3	< 1950	Near-infrared radiation
Ceramic surface radiator	2.8 – 5.0	310 - 750	Standard range infrared radiation
Halogen lamp	< 1.4	< 2200	Near-infrared radiation
Quartz rod radiator	2.1	1100	Standard range infrared radiation

What is a halogen heating element?

- Do halogen heating elements really produce no infrared radiation, but give off some other kind of radiation as several suppliers of moisture analyzers like to suggest?

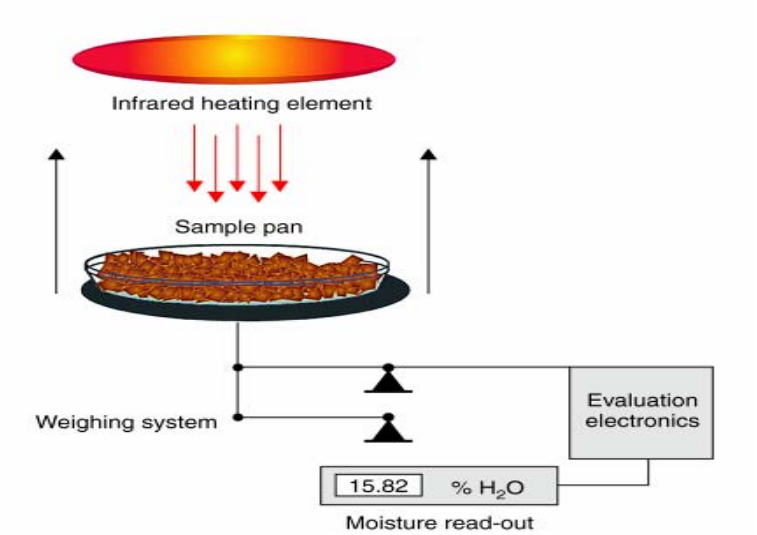
  •No ...

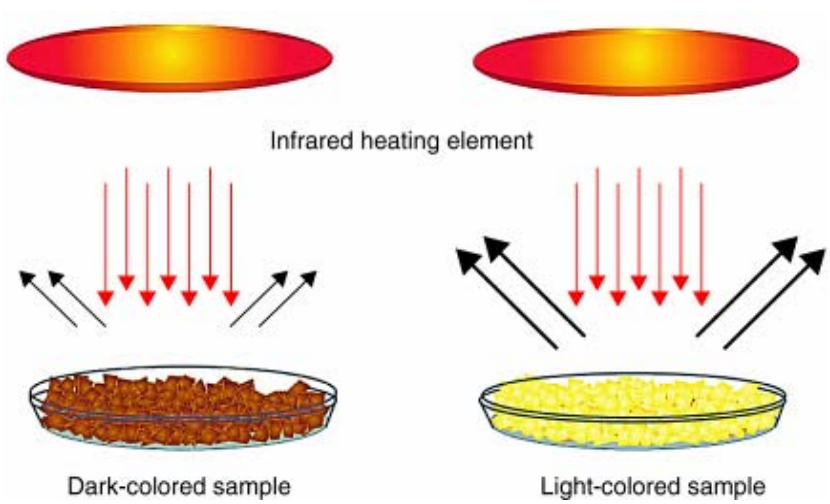
  •Halogen radiation is unknown in the electromagnetic wave spectrum. The use of this term is misleading!

- All warm/hot bodies give off infrared radiation, as does a halogen heating element
- The terms metal rod, ceramic, quartz or halogen heating element only describe the main component that the heating element is made of

Functional setup of a moisture analyzer

- The time it takes to heat the sample depends on:
  - Absorption/reflection/transmission
  - Thermal conductivity
  - Consistency
  - Quantity





Areas of application for a moisture analyzer

- IR moisture analyzers give you more options beyond your routine methods which can sometimes be time-consuming and technically complicated.

Comparison of methods

Drying Oven

- Weigh-in sample pan
- Weigh-in sample
- Dry sample for 1 hour
- Cool sample in a desiccator for 20 min.
- Backweigh sample
- Calculate the results manually
- Post-dry sample for 30 min.
- Cool sample in a desiccator for 20 min.
- Backweigh sample
- Recalculate the results
- Repeat these steps until the sample weight remains constant

IR moisture analyzer

- Tare sample pan
- Place neighed sample on pan
- Lower the hood
- The test shuts off automatically
- The result is calculated automatically

Practical work

Why do different methods result in different moisture content?

- The oven drying method causes both water and readily volatile components to evaporate from the sample. The slight heating achieved by convection drying does not always dry the sample completely. The result obtained is higher than the water content, but less than the total moisture content.

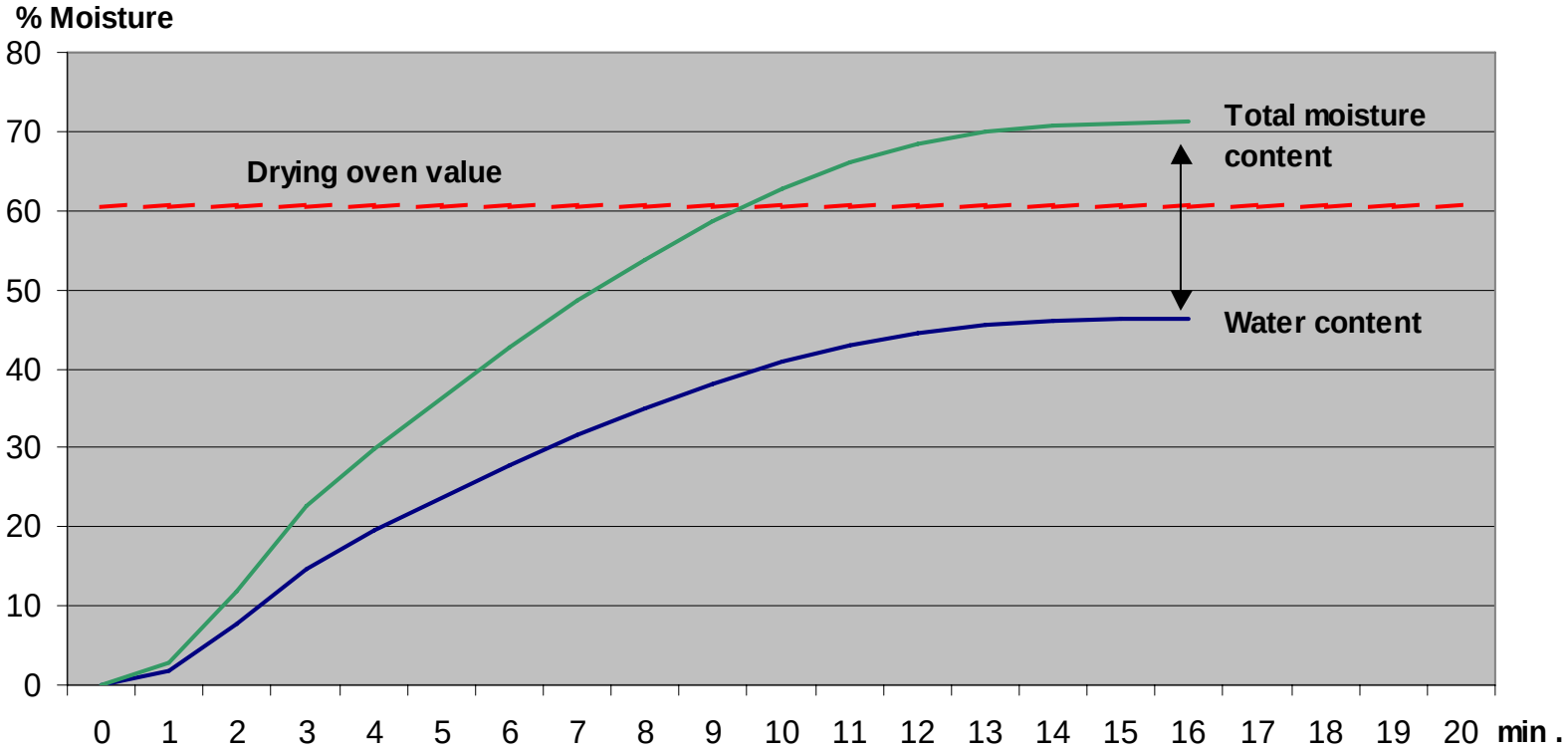
- An infrared dryer causes water, readily volatile and non-volatile components to evaporate. The total moisture content of the sample is analyzed by determining the energy absorption caused by the intense heat. The results achieved are usually greater than with the drying oven reference method.

Why do different methods result in different moisture content?

- The microwave method causes water and only minor amounts of readily volatile components to evaporate. Absorption drying which primarily works according to the principle of “dipole rotation“ produces results that very closely approximate the moisture content and are thereby higher than those obtained with the oven drying method.

- The Karl - Fischer titration determines the number of water molecules by means of a chemical reaction. The result gives a very accurate equivalent of the water content.

From water content to total moisture content



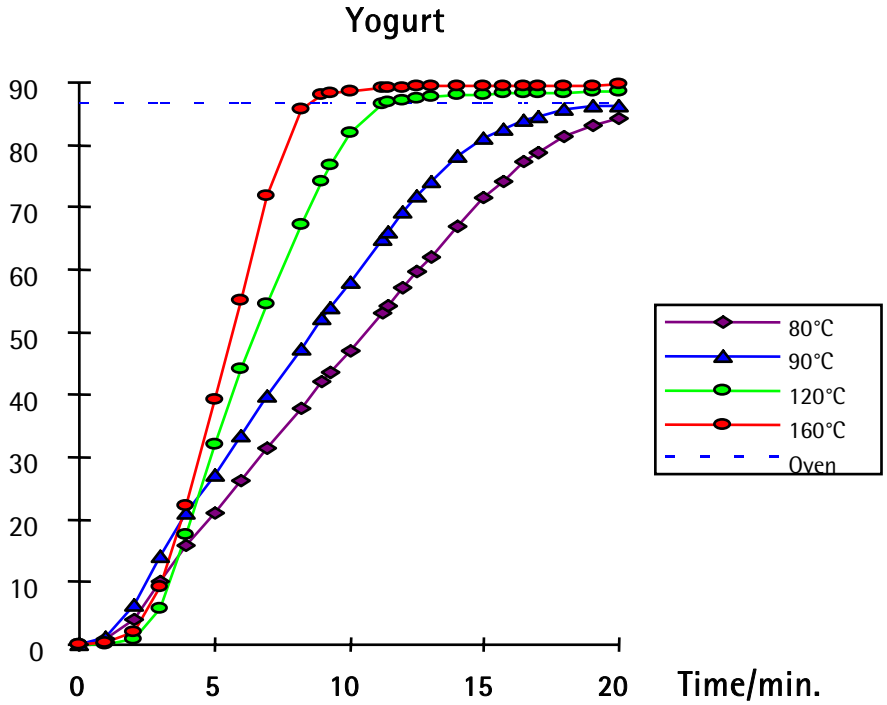
Which test method results in the “right“ moisture content?

- Two reference methods are recognized worldwide:
  - The oven drying method for determining the moisture content of a material.
  - The Karl - Fischer method for determining water content.
  - All other methods must be compared with one of these two methods as appropriate!

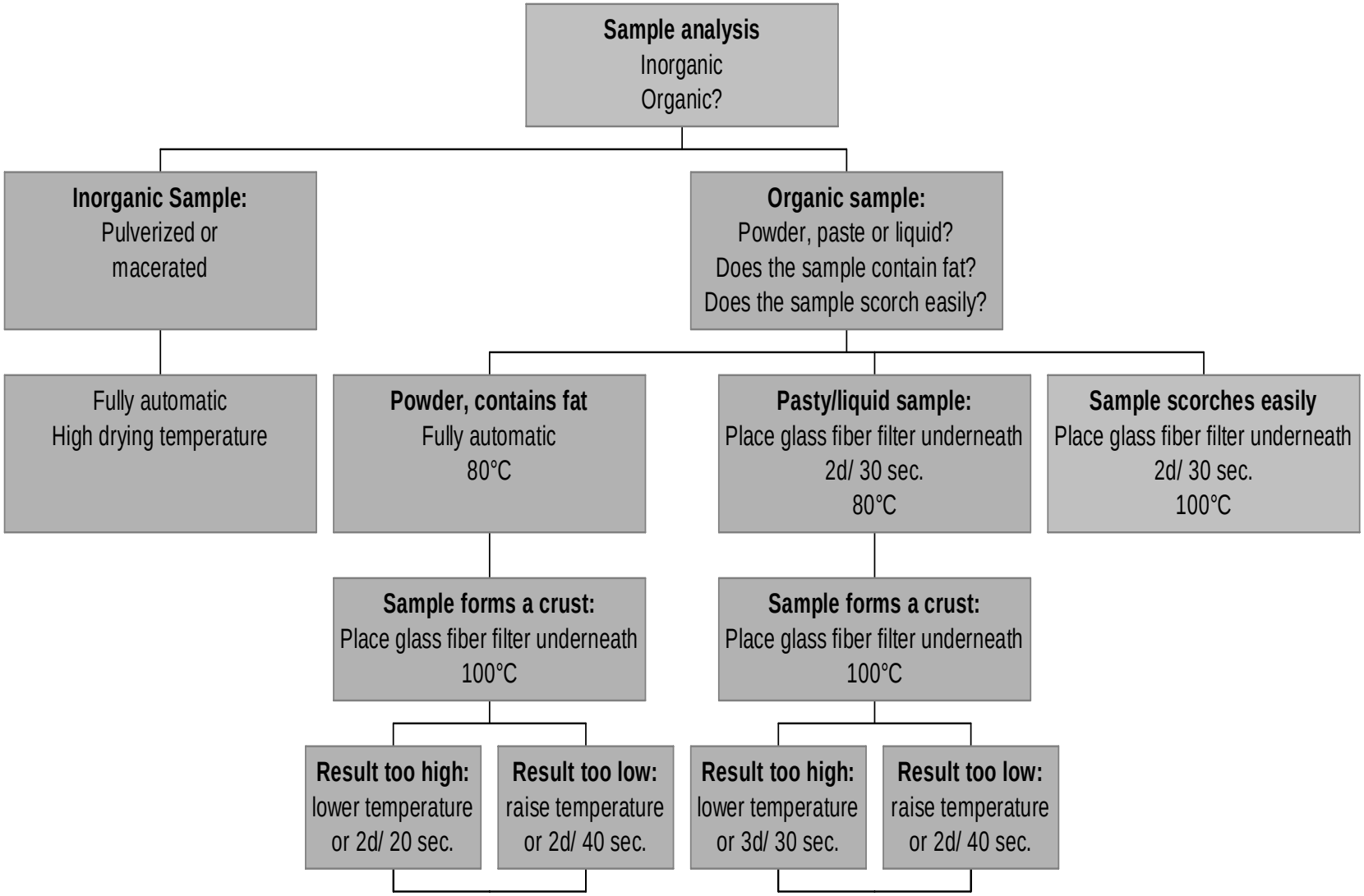
Optimizing parameters based on other reference methods

- Divide the sample in half
- Determine the moisture content using your standard method of analysis
- Use the result of the reference method as the target value or IR drying
- Perform a series of tests with the IR moisture analyzer while constantly optimizing the operational parameters
- Verify the operating parameters by repeating the measurements
- Keep a record of the test setup and results obtained
- Draw up a standard operating procedure (SOP)

Optimizing parameters based on other reference methods



Oven:	5 g, 105°C, 4h	IR:	5 g, 90°C, 20 min.
Result:	86.48 ± 0.01 %	Result:	86.54 ± 0.05 %


Sample analysis
Inorganic
Organic?
Inorganic Sample:
Pulverized or
macerated
Organic sample:
Powder, paste or liquid?
Does the sample contain fat?
Does the sample scorch easily?
Fully automatic
High drying temperature
Powder, contains fat
Fully automatic
80°C
Pasty/liquid sample:
Place glass fiber filter underneath
2d/ 30 sec.
80°C
Sample scorches easily
Place glass fiber filter underneath
2d/ 30 sec.
100°C
Sample forms a crust:
Place glass fiber filter underneath
100°C
Sample forms a crust:
Place glass fiber filter underneath
100°C
Result too high:
lower temperature
or 2d/ 20 sec.
Result too low:
raise temperature
or 2d/ 40 sec.
Result too high:
lower temperature
or 3d/ 30 sec.
Result too low:
raise temperature
or 2d/ 40 sec.

Operational safety

- Due to their design, thermogravimetric IR moisture analyzers are not explosion-proof! These moisture analyzers must not be used
  to analyze products that release flammable or explosive substances!

- On the way from the sample room to outside, the steam comes in contact with the surface of the up to 500°C hot IR radiator

- Evaporation products are emitted directly
  into the surrounding air.

- When analyzing substances that are hazardous to your health, make sure that the room is well ventilated or work is performed under a fume hood!