
Determination of food products' water content
performed using **MA.X2** moisture analyzers (IR radiation)
and **PMV** moisture analyzers (microwave radiation)



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Introduction

Massive food production is an effect of consumerism of the contemporary society. Food is often manufactured on either automatic or semi-automatic production lines. As a result, mass-consumption product of particularly specified qualitative features is produced, quality and price of which is accepted by the consumer. An important thing to be noted is that development of technology goes in pair with consumers' awareness (Kicińska 2009). They expect both, a relatively low price and a durable product of good quality and long shelf life. In order to manufacture product of high quality, process-type approach is required (ISO 9001:2015). Following the process-related rules results with an "in-built product quality" (Total Quality Management, TQM). A consequence of TQM approach is a necessity to verify product quality at different stages of production. The monitoring aims to check microbiological safety, stability and content of water in a product.

Water is one of the most crucial components of many food products (Bradley R.L. Jr. 2010). According to authors (Isengard 2001, Pałacha 2011) quality, shelf life and sensory features of the product depend on quantity of water stored in it. Therefore the water content should be precisely determined and controlled during the product manufacturing process. Uncontrolled increase of water content in a food product causes hydrolytic and oxidative changes which in turn lead to modification of physiochemical features of the product. As a result the product quality deteriorates and its shelf life shortens. With regard to this, there is a need for fast and precise methods allowing verification of amount of product water content, i.e. for methods offered by moisture analyzers of **MA** and **PMV** series by **Radwag Wagi Elektroniczne**.

Reference methods for determination of water content (dry mass) are provided by respective standards (EN). Standard-specified test methodology allows to obtain very precise results, however it cannot be applied in food industry during the inter-operational control due to the fact that it takes too long (ca. 2– 4 hours). An effective, fast and modern solution used during the inter-operational control of water content are **MA** and **PMV** moisture analyzers of **Radwag** production.

In order to use these devices efficiently for control of product water content, validation of the test method is required. Validation as a result gives fast, cheap and uncomplicated method for determination of water content which is comparable to reference method when it comes to precision. The validation can be carried out on one's own or in cooperation with Radwag. In the second case test product information is required, it must be delivered to the Testing Laboratory along with the very product. Validation performed by Radwag is documented by product card featuring test results and hints regarding the designed test method.

Product water content analysis may be realised using the infrared radiation – moisture analyzers of MA.R2, MA.X2, MA.3Y series or using the microwave radiation – moisture analyzer of PMV series. Both of the aforementioned methods are radiation water content determination methods, they can be applied in food industry for testing of solid, semi-solid and liquid products (Sakai and Hanzawa 1994; Shrama, Verma and Pathare 2005; Soysal Y. 2004; Togrul 2006). Water content determination test was performed for the following products using both reference method

and moisture analyzer method, the latter of which was carried out via **MA. 50 X2** and **PMV** moisture analyzers:

- natural yoghurt,
- pasteurised milk of 2 % fat content,
- vanilla quark,
- creamy quark with olives,
- margarine.

REFERENCE METHODS

a. Natural yoghurt

Prior sampling, the yoghurt was stirred, this allowed to provide homogeneous structure over the whole sample's volume. Next, about 3 gram of the product was placed in a glass container on a pre-dried silica sand. Temperature during analysis was 105°C, the analysis took 3 hours. Water content was calculated using the following equation:

$$W_w = \frac{m_w - m_s}{m_w} \cdot 100 \%$$

where: m_w – mass of a wet sample,
 m_s – mass of a dry sample.

b. Pasteurised milk

Procedure for determination of water content in milk and procedure for determination of the water content in natural yoghurt are likewise.

c. Vanilla quark

The water content determination was carried out with reference to PN-EN ISO 5534. It was calculated using the following equation:

$$100 - w_t = \frac{(m_2 - m_0) - (m_3 - m_4)}{(m_1 - m_0)} \times 100 \%$$

where: w_t – overall dry mass content of the sample, expressed as mass fraction in percent,
 m_0 – mass of the container, given in gram,
 m_1 – mass of the analysed sample and container prior drying, given in gram,
 m_2 – mass of the analysed sample and container after completed drying, given in gram,
 m_3 – mass of the container used in a blank test, given in gram, determined for the same drying time as m_2 ,
 m_4 – mass of the container prepared to be used in a blank test, given in gram.

d. Creamy quark with olives

For the creamy quark with olives the same test method was applied like in case of the vanilla quark.

e. MR margarine

Water content in margarine was determined in accordance with reference method provided in PN-EN ISO 3727-1 standard „Butter. Determination of moisture, non-fat solids and fat contents. Part 1: Determination of moisture content. Reference Method". Water content w_m was calculated using the following equation:

$$w_m = \frac{(m_1 - m_4) - (m_1 - m_3)}{(m_2 - m_0)} \times 100 \%$$

where: w_m – water content of the sample, expressed as mass fraction in percent,

m_0 – mass of the container, given in gram,

m_1 – mass of the container prepared to be used in a blank test, prior drying, given in gram,

m_2 – mass of the analysed sample and container, prior drying, given in gram,

m_3 – mass of the container used in a blank test, after drying, given in gram,

m_4 – mass of the analysed sample and container, after drying, given in gram.

MA 50.X2 Moisture Analyzer

Water Content Determination Method

Using Infrared Radiation (IR)

Moisture analyzer of MA 50.X2.A series is a modern device intended for determination of water content of various products. It is equipped with a high resolution converter and an intuitive operation panel featuring 5" colour touch screen display. The MA.X2.A series offers an automatic system of opening and closing the drying chamber's lid which enables handy control by means of proximity sensors or an on-screen button. Ergonomics of operation is guaranteed due to a convenient user interface and thanks to quick access keys (hot-Key), info fields and labels. These are programmable elements and as such they allow free configuration of the operation panel. The drying process may be carried out for any temperature value, it is possible to assign the drying process with a particular product using the below listed databases:

- Operators (100 records maximum)
- Products (5000 records maximum)
- Customers (100 records maximum)
- Measurements (50 000 records maximum)
- Packaging (100 records maximum)
- Drying programs (200 records maximum)
- Drying process reports (5000 records maximum)
- Ambient conditions (10000 records maximum)



Figure 1. Functionality of MA 50.X2.A moisture analyzer

Use of USB interface enables fast transfer and copying of results of work (measurements, reports, databases) to a computer or other moisture analyzer. Data management can be carried out online, via remote access to the balance and its databases. Integration with an E2R computer system enables external data management thus maximizing productivity and throughput.

Water content in tested products was determined with use of the differential method, i.e. difference between mass of a wet product and mass of a dry product was referred to. Product mass changes were analysed continuously in a drying chamber. Continuous analysis is a characteristic feature of methods carried out using **Radwag-manufactured** moisture analyzers, and it allows to provide the result within a relatively short time. Figure 2 presents a diagram of MA 50.X2.A moisture analyzer manufactured by Radwag Wagi Elektroniczne, Poland.

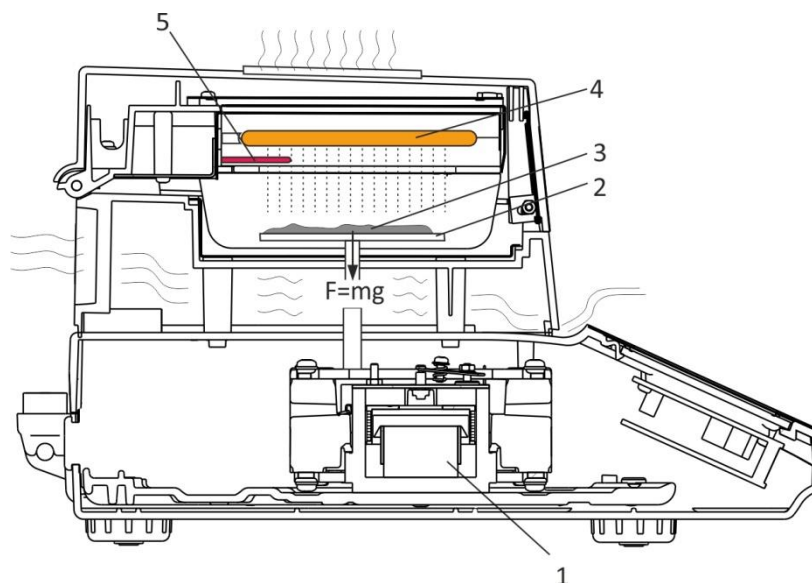


Figure 2. Diagram of MA 50.X2.A moisture analyser; own work

Precise measurement of analysed product mass is guaranteed due to an electromagnetic converter of mass (1). Measurement of the product mass is realised in accordance with the following equation:

$$F = mg$$

m – product mass,

g – on-site gravitational acceleration

Product mass change during heating is analysed by data processing systems operating in accordance with a unique algorithm developed by **Radwag** engineers. Analysis of the measuring signal is carried out fast and precisely, potential signal disturbances that may occur during product mass measurement are eliminated. With this, the measurement of mass is always accurate and ensures precision when it comes to water content analysis.

Increase of product temperature aiming to isolate and determine water contained within product structure takes place in the drying chamber. Inside of the chamber there is a weighing pan

(2), permanently fixed to the measuring converter (1). In the course of production the moisture analyzer is calibrated using mass standards, traceability is accounted for. This allows to link the gravitational force with the 1 kilogram mass standard. Due to the above, upon placing the product (3) on the weighing pan (2) it is possible to present the gravitational force ($F = mg$) on a moisture analyzer display as a weighing result expressed in gram.

Increase of product (3) temperature is brought about with use of a precisely controlled heat source, (4), built into the top part of the drying chamber. In case of moisture analyzers of MA.X2.A series, the target drying temperature is obtained within the whole space of the drying chamber. The heat is supplied to the analysed product as a result of convection and IR radiation. According to the authors (Kathiravan, Khurana, Jun, Irudayaraj and Demirci 2008; Nowak 2005) drying of products performed using IR radiation may be realised for quite wide wavelengths range, from 0.78 μm to 1000 μm . It must be however stated that food products absorb IR radiation the most effectively when the wavelength ranges between 3 μm and 6 μm (Figure 3).

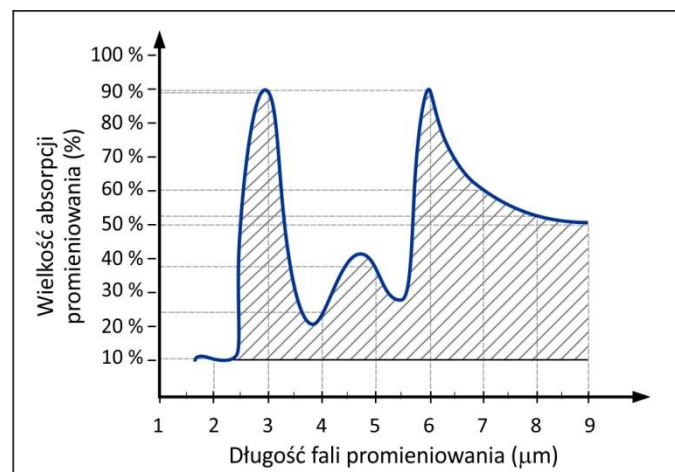


Figure 3. Absorption of radiation by food product

IR radiation wavelength emitted by the infrared radiator is strictly conditioned by the IR radiator temperature (Riadh, Ahmad, Marhaban 2015). With regard to this, a proprietary algorithm for control of the heat source was implemented in **Radwag** moisture analyzers. Such solution provides great stability of temperature in which the product is analysed. In standard models of MA.X2.A moisture analyzers, the heat source emits IR radiation of the wavelength of ca. 3 μm , this makes the product drying fast and effective. Temperature sensor (5) installed in the top part of the drying chamber sends information about the current drying temperature value to the system controlling the heat source. The sensor allows to maintain a thermally stable environment for the product. With time, the temperature of the analysed product increases mainly as a result of absorption of radiation (Togrul 2006; Ertekin and in. 2014). As the authors claim (Ratti, Mujumdar 2006; Adak, Heybeli and Ertekin 2017), speed and effectivity of the drying process depend on the length of emitted infrared radiation wavelength and on the product's ability to absorb the radiation, which must be referred to in the assessment of the process.

The value of tested product's water content is presented on the display of MA 50.X2.A dynamically in a form of a result or graph (drying curve). On the basis of wet product mass, and with regard to its mass upon analysis completion, the moisture analyzer's program automatically

calculates water content (dry mass content) in the product, this is done using the following equations:

$$\%M = \frac{m_1 - m_2}{m_1} \cdot 100 \% \quad \text{or} \quad \%D = \frac{m_1 - m_2}{m_2} \cdot 100 \%$$

where: %M – water content (relative humidity)
% D – dry mass content (absolute humidity)
 m_1 – wet product mass, prior analysis start
 m_2 – dry product mass, upon analysis completion

PMV 50 Moisture Analyzer

Water Content Determination Method Using Microwave Radiation

PMV moisture analyzer manufactured by Radwag is the latest solution using the microwave radiation for determination of water content in food and other products which contain 8 % - 100 % water. Product temperature increase is provided as a result of absorption of the microwave radiation by polar compounds of the product (mainly water). In consequence of radiation absorption, reorientation of dipoles of polar compounds occurs which leads to molecular friction (Al-Harahsheh M, Al-Muhtaseb and Magee 2009). This brings about fast increase of temperature within the whole product volume, which is a cause of short analysis duration. Drying process efficiency depends on the power and frequency of the emitted microwaves, and also on both the product structure and its chemical content (Soysal 2004; Kamińska and Ciesielczyk 2011). **PMV** microwave moisture analyzer is a modern and productive weighing equipment, it offers:

- short drying time (2 – 5 minutes maximum),
- products, drying programs, completed drying procedures databases,
- statistical analysis for water content determination of selected product,
- interactive menu with definable buttons, info fields, permission levels, etc.,
- interactive proximity sensors,
- drying process visualisation, presented as a drying curve,
- export / import of products and drying programs databases, remaining data,
- communication via RS 232, USB, Ethernet, Wi-Fi.



Figure 4. PMV 50 microwave moisture analyzer

Diagram of **PMV 50** microwave moisture analyzer, manufactured by **Radwag Wagi Elektroniczne**, Poland, showing the main subassemblies has been presented in figure 5.

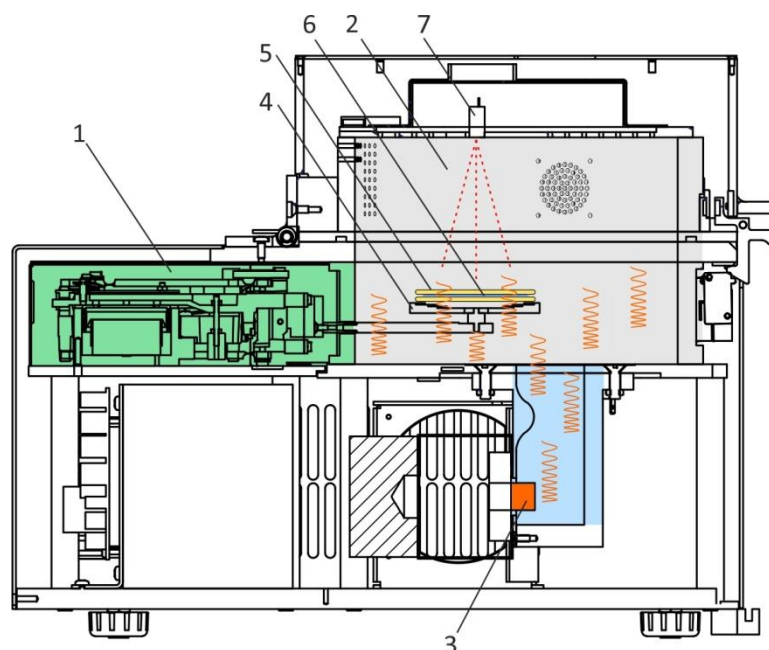


Figure 5. Diagram of PMV 50 microwave moisture analyzer

MEANS OF OPERATION OF THE MICROWAVE MOISTURE ANALYZER

Measurement of the analysed product mass is carried out using electromagnetic system (1), which comprises carefully selected monoblock components. Thanks to such solution the weighing system remains stable even when operating in unstable ambient conditions. Inside of the drying chamber (2) there is a weighing pan (4), it is loaded with a product (6) placed between two glass fibre filters (5). A magnetron (3) emits microwaves that are a cause of dipole effect of water molecules in a product (6). As a result, product temperature increases. The temperature is monitored via an infrared sensor (7) installed in the top part of the drying chamber (2). Maximum product temperature is an individual feature of each product, and it is one of the drying parameters. The temperature sensor (7) registers current product's temperature and the obtained information is used via system controlling power of the magnetron (3). Along with the analysed product temperature drop (desorption of water from the product results with lower molecular friction), the emitted microwaves' power changes, its intensity matches dynamics of the process.

Product water content is analysed until the condition of product mass stability over time is met – the same approach concerns MA 50.X2.A moisture analyzer, which in this case means complete desorption of water from the product structure.

Water content (%M) in the analysed products, i.e.: natural yoghurt, milk, vanilla quark, creamy quark with olives, margarine, determined using the PMV 50 moisture analyzers was calculated in accordance with the following equations:

$$\%M = \frac{m_1 - m_2}{m_1} \cdot 100 \% \quad \text{or} \quad \%Dm = \frac{m_1 - m_2}{m_2} \cdot 100 \%$$

where: %M – water content (relative humidity)

% D – dry mass content (absolute humidity)

m_1 – wet product mass, prior analysis start

m_2 – dry product mass, upon analysis completion

THE RESULTS AND A DEBATE

The first stage of the test was determination of water content in products with use of the reference methods. For each method, three samples of the same product were tested. Information about the real water content of the tested products was a starting point for optimization of methods based on infrared radiation (**MA 50.X2.A**) and microwave radiation (**PMV 50**). Results of water content determination obtained using reference methods are to be found in table 1.

Table 1. Water content given in percent - reference method

Primo Yoghurt	Milk 2%	Vanilla quark	Creamy quark	MR margarine
87.09	89.06	72.96	65.06	19.33

Results of water content determination obtained using microwave method and infrared radiation are to be found in table 2 and 3.

Table 2. Water content in products determined using the microwave method (PMV 50 moisture analyzer)

	Water content [%] – the microwave method				
	Primo Yoghurt	Milk 2%	Vanilla quark	Creamy quark	MR margarine
1.	86.66	89.26	72.46	65.79	19.14
2.	86.75	89.21	72.96	65.74	19.00
3.	86.89	89.06	72.98	65.66	19.24
4.	86.53	89.11	73.17	65.86	18.99
5.	86.88	89.07	73.02	65.35	18.93
6.	86.43	88.97	73.60	65.46	18.61
7.	86.12	88.76	72.77	65.56	18.47
8.	86.73	88.84	72.38	65.52	18.87
9.	86.58	89.30	72.15	65.41	18.93
10.	86.91	89.12	72.27	65.47	18.88
\bar{x}	86.65	89.07	72.78	65.58	18.91
SD	0.25	0.17	0.46	0.17	0.23

Table 3. Water content in products determined using the infrared radiation method (MA 50.X2.A moisture analyzer)

Water content [%] – IR radiation					
	Primo Yoghurt	Milk 2%	Vanilla quark	Creamy quark	MR margarine
1.	86.82	89.50	72.84	64.96	19.39
2.	87.01	89.41	72.91	64.92	19.43
3.	87.22	89.42	72.64	65.25	19.35
4.	87.06	89.44	72.79	64.84	19.27
5.	86.91	89.05	72.78	64.76	19.22
6.	86.95	89.39	72.88	65.18	19.34
7.	86.98	89.14	72.89	64.78	19.28
8.	87.22	89.33	72.89	64.95	19.34
9.	87.02	89.40	72.76	65.48	19.26
10.	86.99	89.13	72.87	64.76	19.23
\bar{x}	87.02	89.32	72.82	64.99	19.31
SD	0.13	0.16	0.08	0.24	0.07

One of the most significant parameters was precision of the measurement expressed via standard deviation. The value of standard deviation may be helpful when the obtained water content determination result concerns production limits or when estimating uncertainty of water content determination. The lower the standard deviation value, the greater measurement precision. The lowest precision was observed for vanilla quark (0.46%) and yoghurt (0.25%) tested using the microwave radiation method (PMV moisture analyser) and for creamy quark (0.24%) tested using the infrared radiation method (MA 50.X2.A moisture analyzer). It was however concluded that the difference concerning measurement precision is not crucial in terms of statistics when it comes to assessment of these products quality. In case of the vanilla quark, the difference obtained between the maximum and minimum results for the measurement series was 1.45 %, in case of yoghurt it was 0.79 %.

Accuracy of water content determination performed using microwave and infrared radiation methods was calculated as difference between average values of water content obtained when testing the product via IR radiation and microwaves methods, and the results obtained when testing this product using the reference methods. The results are presented in table 4.

Table 4. Accuracy of water content determination with regard to the used test method

	Reference method	PMV 50 Microwave radiation		MA 50.X2.A IR radiation	
	Water content	Water content	Readability	Water content	Readability
Primo yoghurt	87.09	86.65	0.44	87.02	0.07
Milk 2 %	89.06	89.07	0.01	89.32	0.26
Vanilla quark	72.96	72.78	0.18	72.82	0.14
Creamy quark	65.06	65.58	0.52	64.99	0.07
MR margarine	19.33	18.91	0.42	19.31	0.02

It was concluded that the best accuracy is obtained using the microwave method for milk (0.01% standard deviation) and vanilla quark (0.18 %). The greatest water content result difference with regard to the result obtained via reference method (0.52 % standard deviation) was observed for the creamy quark with olives. This product, due to the fact that it contains large pieces of olives, was not homogeneous, which influenced the analysis result. When speaking of analysis of water content determination using infrared radiation method (IR) the best measurement accuracy was noted for margarine (0.02% deviation). Much greater deviation of accuracy was obtained for milk (0.26% deviation). A conclusion was made, that from metrological point of view the differences were significant, however they do not matter when the product assessment is carried out in food industry using critical values for the evaluation of product quality.

One of the most significant aspects of the applied methods was time required for completion of water content determination. While analysing time parameter, it was observed that regardless of the tested product type, the determination duration was shorter whenever product temperature increase was a result of microwaves radiation (**PMV 50 moisture analyser**) then in case of product subjected to IR radiation. Results regarding analysis duration are to be found in table 5.

Table 5. Duration of water content determination using microwave and infrared radiation methods

Product	PMV 50 (microwaves)	MA 50.X2.A (IR)
	Analysis duration [min:s.]	
Primo yoghurt	03:47	15:32
Milk 2 %	02:55	12:43
Vanilla quark	04:57	09:35
Creamy quark	03:25	08:35
MR margarine	02:05	10:25

SUMMARY

Use of microwave method for determination of water content allowed to shorten the analysis time when compared to method based on the infrared radiation. The analysis was even 5 times shorter in case of margarine, 4 times for yoghurt and milk, 2.5 times for creamy quark and 2 times in case of vanilla quark.

Analysis of accuracy of water content determination proved that further optimisation of testing methods, when it comes to drying parameters and product preparation procedures, is possible. For a microwave moisture analyser, the modifications should concern shortening of the drying process, wherein acceptable accuracy and precision of carried out measurements must be maintained. In case of MA 50.X2.A moisture analyser it is likely to provide optimisation of drying process with regard to use of different infrared radiation wavelengths. As far as drying is concerned, due to high speed, it is recommended to use PMV 50 moisture analyser. The PMV 50 is a preferable device allowing to determine water content quickly.

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DRYING PROCESS PARAMETERS

Primo yoghurt	MA 50.X2.A	PMV 50
Temperature	drying process: 120°C	product: 100°C
Microwaves power	-	80 %
Drying profile	Standard	Standard
Finish mode	Auto 2	Auto 2
Product mass	ca. 2 g	ca. 1.3 g
Analysis duration (min:s.)	15:32	03:47

Milk 2 %	MA 50.X2.A	PMV 50
Temperature	drying process: 120°C	product: 100°C
Microwaves power	-	100 %
Drying profile	Standard	Standard
Finish mode	Auto 3	Auto 1
Product mass	ca. 3 g	ca. 1 g
Analysis duration (min:s.)	12:43	02:55

Vanilla quark	MA 50.X2.A	PMV 50
Temperature	drying process: 105°C	product: 100°C
Microwaves power	-	100 %
Drying profile	Standard	Standard
Finish mode	Auto 3	Auto 1
Product mass	ca. 2 g	ca. 2 g
Analysis duration (min:s.)	09:35	04:57

Creamy quark	MA 50.X2.A	PMV 50
Temperature	drying process: 105°C	product: 100°C
Microwaves power	-	60 %

Drying profile	Standard	Standard
Finish mode	Auto 3	Auto 1
Product mass	ca. 3 g	ca. 2.5 g
Analysis duration (min:s.)	08:35	03:25

MR margarine	MA 50.X2.A	PMV 50
Temperature	drying process: 105°C	product: 100°C
Microwaves power	-	100 %
Drying profile	Standard	Standard
Finish mode	Auto 2	Auto 1
Product mass	ca. 5 g	ca. 1.8 g
Analysis duration (min:s.)	10:25	02:05

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