Metrol. Meas. Syst., Vol. 30 (2023) No. 2, pp. 305–321
DOI: 10.24425/mms.2023.144873

PAN POLSKA AKADEMIA NAUK

METROLOGY AND MEASUREMENT SYSTEMS
Index 330930, ISSN 0860-8229
www.metrology.wat.edu.pl

M&MS

ACCURACY OF DRYING SELECTED PRODUCTS USING A MOISTURE ANALYZER METHOD BASED ON INFRARED RADIATION

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Abstract

Trueness and precision of a method for determining the water content (%) of food and chemical products based on infrared radiation with a wavelength in the range (2.70÷7.21 μm) was evaluated. The most accurate measurements for food products were obtained when the heat source was a radiant heater with a radiation wavelength of 7.21 μm, a trueness deviation of 0.01%. When heated with radiation with wavelengths (from 3.32 μm to 7.21 μm), the trueness of the measurement ranged (0.03%÷0.13%) for chemical products. The shortest analysis time for food products was found when the analysis was carried out using an IR source with a wavelength of 7.21 μm, while for chemical products, a heat source with a wavelength of 2.70÷7.21 μm was optimal. According to the results of the analysis, the use of IR radiation with a wavelength range of 3.32÷7.21 μm is an alternative for accurate measurements.

Keywords: moisture analyser, infrared heating, food quality, mass measurements.

1. Introduction

The quality of many products is closely dependent on the amount of water content found in their structure. Excess water is usually unfavourable because it leads to hydrolytic and oxidative changes [1], which result in a reduction in the quality of the product, especially food, and a reduction in its shelf life. A partial solution to this problem is dehydration, which reduces the water activity and slows down the changes taking place in the product. However, the quality of the product obtained in this manner is depends heavily the technology used during the process. The best results are obtained in the processes of freeze-drying [2,3] and microwave drying [4–6], but also using hybrid drying methods [7–9]. Undoubtedly, the cheapest method of dehydration is the convection drying process [10], but according to the authors [11, 12] it leads to a significant loss of quality. On the other hand, the expected quality of the products is achieved by mixing many ingredients, including water. These activities require the addition of specific amounts of water in order to achieve the desired quality characteristics of the product or to allow for further

Article history: received December 9, 2022; revised January 24, 2023; accepted February 9, 2023; available online June 18, 2023.

processing. According to [13], such a procedure is used in the production of processed cheese. Water in this process acts as a plasticiser.

Similar quality relationships can be found not only in natural products, but also in processes involved in the manufacture of industrial products. One example is biodiesel, which as an alternative fuel has excellent biodegradability with a low carbon footprint [14]. The amount of water used in its production, which determines the superiority of the transesterification process over the hydrolysis reaction, should be kept to a minimum [15–18]. Strict control of the amount of water is also required in the moulding of plastic granule components. According to the authors [19], an excess of it can cause visible quality changes in the product, such as material discontinuities or discolouration. For this reason, despite the so-called pre-drying of the granules, an additional quick check of the water content of the granules just before they are fed into the injection moulding machine is often of order. Information on the actual water content of a product is also important for hygroscopic products [20], especially in the context of their storage, shipping, or determining commercial grade.

Furthermore, information about the content of water is critical in other areas of the food industry, particularly in the storage and processing of cereal grains and cereal preparations, as well as in the pharmaceutical industry, where microbiological stability and purity are important [21] as well as in the chemical industry, where water is a destructive agent for many chemical reactions [22]. It must be stated that regardless of the technological processes used or the areas of operation, there is a need to accurately and quickly test the water content. To make such measurements, the electrical method [23] using the relationship between the electrical conductivity and dielectric constant value of the product and the water content of the test product, the Karl-Fischer method [24–26], or the convective drying method [12] can be used.

Any of these methods can be used but their practical limitations stem from insufficient measurement accuracy, the method's complexity [7], its high cost, and time-consuming analysis. A universal method that does not have such disadvantages is the thermo-gravimetric method [27, 28] in which the mass of the product before and after drying (LOD) is recorded. An ergonomic development of this method is the continuous-controlled product mass loss method, additionally using infrared radiation [29, 30]. The test product is measured until it reaches a stable mass, equivalent to the complete removal of water from its structure. It should be noted, however, that the accuracy of this method depends on the phenomena that occur in the product's structure when heated. The increase in product temperature in this method is due to the convective movement of hot air and the effect of infrared radiation [31–34]. Too high a temperature during testing usually results in the product's surface burning, while too low a temperature prevents water from being removed from the deeper layers [35]. Therefore, for many product groups, the validation process of the infrared method should be carried out as a first step [36–39]. The testing time for the water content in this method is between a few and several minutes, which allows it to be used not only for laboratory work but also for quick inter-operational checks and during water content control in B2B (Business-to-Business) relations [39]. Undoubtedly, evaluating a method using infrared radiation beyond the realm of temperature-product interactions should also include an analysis concerning heat migration from the drying chamber into the measuring system. Prolonged drying can be a significant factor affecting the accuracy of the test being conducted. A commonly used piece of infrared drying equipment is the moisture analyser. It consists of a weighing module, which performs the mass measurement, and a drying chamber, which ensures a stable analysis temperature. Information on the difference in mass between the wet and dry product is sufficient to determine the water content. This work aimed to evaluate the accuracy of drying food and chemical products using a moisture analyser method based on infrared radiation of varying infrared radiation wavelengths.

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2. Materials and Methods

2.1. Material

Testing the water content was conducted for food products (wheat flour type 450; granulated garlic; Mexican sauce; curry seasoning; horseradish mustard sauce; grains: peas, buckwheat, barley, oats, wheat). Cosmetic and household chemical products, *i.e.*, hair gel, cleansing milk with microcrystals, liquid soap, liquid floor polish, and washing gel, were also tested for water content (Table 1). Prior to testing, the products were stored in sealed containers under laboratory conditions (temperature 23°C, relative humidity 40%). Granular products were mechanically crushed to be tested in this form, while products with a semi-liquid structure were mixed to achieve homogeneity.

Table 1. List of products – reference method. Characteristics of the products and parameters to which the product samples were submitted when determining the water content with the reference method.

Product name	**Product structure, density**	**Drying temperature**	**Sample mass**	**Drying time**
	g/cm^3	(°C)	(g)	(hour)
Food products				
Wheat flour, type 450	*p*	105 ± 0.5	6.03 ± 0.12	3
Granulated garlic	*p*	105 ± 0.5	5.12 ± 0.14	3
Mexican sauce	*p*	105 ± 0.5	7.55 ± 0.46	3
Curry seasoning	*p*	105 ± 0.5	2.44 ± 0.15	3
Horseradish mustard sauce	*s*/*l*	105 ± 0.5	5.81 ± 0.34	3
Agricultural products				
Peas	g	135 ± 0.5	5.16 ± 0.14	2
Buckwheat	g	135 ± 0.5	5.11 ± 0.14	2
Barley	g	140 ± 0.5	5.23 ± 0.06	2
Oats	g	140 ± 0.5	5.07 ± 0.03	2
Wheat	g	130 ± 0.5	5.39 ± 0.19	2
Cosmetic and household chemical products				
Hair gel	*s*/*l*, 0.95	105 ± 0.5	4.58 ± 0.17	3
Cleansing milk with microcrystals	*s*/*l*, 1.03	105 ± 0.5	4.54 ± 0.12	3
Liquid soap	*s*/*l*, 0.91	105 ± 0.5	2.38 ± 0.08	3
Floor polish	*s*/*l*, 0.85	105 ± 0.5	3.19 ± 0.43	3
Washing gel	*s*/*l*, 0.94	105 ± 0.5	2.96 ± 0.47	3

where *p* is a powder sample, *s*/*l* is a semi-liquid sample, *g* is a grain sample.

2.2. Methods

Testing the Reference Water Content of Food, Cosmetic, and Household Chemical Products by Convection Drying

The water content was determined for each product using the convection drying method. The sample was placed in a glass vessel and dried in a temperature-controlled oven. After drying, the samples were placed in a silica gel desiccator to bring the sample temperature to ambient temperature. Based on the mass of the wet product (before drying) and the dry product (after

drying), the water content was calculated according to Relation (1).

$$mc = \frac{m_w - m_d}{m_w} \cdot 100\%, \tag{1}$$

where: mc is the product water content, m_w is the wet product mass, and m_d is the dry product mass.

The mass of each product before and after drying was determined using an AS 220.X2 balance, designed by Radwag Wagi Elektroniczne, Poland. The parameters that were determined during the reference test for the water content of the products selected in the study are summarised in Table 1.

Testing the Water Content of Food, Cosmetic, and Household Chemical Products by Infrared Radiation as a Heating Source

The equipment used to test the water content were three MA 50.X2 moisture analysers from Radwag Wagi Elektroniczne, Poland, equipped with heat sources respectively:

- IRL, infrared heater, wavelength 7.21 µm,
- IRM, infrared heater, wavelength 3.32 µm,
- IRS, infrared heater, wavelength 2.70 µm.

The principle for using the moisture analysers was to continuously record changes in product mass during heating [30]. The water content was calculated via a moisture analyser algorithm according to relation (1). The use of infrared radiators with different wavelengths did not necessitate any changes in the moisture analyser's design. Stable temperatures inside the drying chamber were achieved by convection and radiation phenomena. Convection is the transfer of heat between media due to the movement of warm air inside the drying chamber [12]. During this process, the heat was initially absorbed by the upper layer of the sample and then transferred to its deeper layers. In the case of infrared radiation, the heat was transferred to the sample structure in the form of infrared radiation. The amount of heat delivered depended on the wavelength of the radiation (IRL, IRM, IRS). Consequently, the percentage of convection and radiation in the drying process varied depending on the type of infrared heater used. The final value of the water content of the test product depended not only on the temperature of the test, but also on the criterion describing the constancy of the dry product mass.

The parameters in Table 2 were used when testing the products regardless of the type of heat source (IRL, IRM, IRS). The trueness of the method using a moisture analyser was determined

Table 2. Adopted drying parameters for the method using an IR moisture analyser.

Sample name	**Analysis temperature (°C)**	**Auto switch-off criterion**
Food products	105	1mg / 60 sec
Cosmetic and household chemical products		
Hair gel, cleansing milk with microcrystals, liquid soap	105	1mg / 60 sec
Floor polish (Sidolux)	120	1 mg / 10 sec
Washing gel (Persil)	120	1 mg / 25 sec
Agricultural products – grains		
Peas, Buckwheat	135	1 mg / 60 sec
Barley, Oats	140	1 mg / 60 sec
Wheat	130	1 mg / 60 sec

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from Relation (2):

$$A = \overline{wc}_{\mathrm{REF}} - \overline{wc}_{\mathrm{TEST}}\,, \tag{2}$$

where A is the trueness of product drying using the moisture analyser method, $\overline{wc}_{\mathrm{REF}}$ is the average water content determined by the convection method, and $\overline{wc}_{\mathrm{TEST}}$ is the average water content determined by the moisture analyser method.

3. Statistical Analysis

The Shapiro–Wilk test was used to check whether the water content results obtained using the infrared drying method meet the assumptions for a normal distribution. Hypotheses were therefore formulated for this purpose:

- Hypothesis H0: the measurement data has the characteristics of a normal distribution,
- Hypothesis H1: the measurement data does not have the characteristics of a normal distribution.

The value of the statistic was calculated from Relation (3)

$$W = \frac{\left[\sum_{i=1}^{n/2} a_i(n) \cdot \left(x_{(n-1+1)} - x_{(i)}\right)\right]^2}{\sum_{i=1}^{n} (x_i - \overline{x})^2}\,, \tag{3}$$

where $a_i(n)$ is constant, n is sample size, and $\overline{x}$ is the mean value.

The water content results have the characteristics of a normal distribution if the calculated value of the Shapiro–Wilk statistic is greater than the critical value, which for sample size $n = 6$ and probability $\alpha = 0.05$ was 0.788. The reciprocal correlations between the variables mass/trueness of the analysis were then determined by assuming the value of the r-Pearson correlation coefficient:

- weak correlation, coefficient value in the range 0.0÷0.3,
- moderate correlation, coefficient value in the range 0.3÷0.5,
- strong correlation, coefficient value in the range 0.5÷0.7,
- very strong correlation, coefficient value in the range 0.7÷1.0.

The significance of the correlation coefficients was assessed by checking whether the value of the test statistic calculated according to Equation (4) for probability $\alpha = 0.05$ and sample size $n = 6$ is within the critical area $(-\infty; -2.776 < U < 2.776; +\infty)$, which was determined for quantile $1 - \alpha/2$ and $n - 2$ degrees.

$$t_{\mathrm{emp}} = \frac{r \cdot \sqrt{n-2}}{\sqrt{1-r^2}}\,, \tag{4}$$

where r is the value of correlation coefficients calculated from the sample and n is the sample size.

4. Results and Discussion

The water content results for food, agricultural, cosmetic, and household chemical products that were obtained by convection drying are shown in Table 3. The water content values obtained

with this method were the reference points for determining the trueness of the measurements that were made using the moisture analysers equipped with heating media of different IR wavelengths, 2.70 µm (IRS), 3.32 µm (IRM) and 7.2 µm (IRL), respectively.

Table 3. Reference water content of products dried using the convection method.

Water content (%) ± precision*				
Food products				
Wheat flour	Granulated garlic	Mexican sauce	Curry seasoning	Horseradish mustard sauce
14.35 ± 0.04	7.86 ± 0.04	9.10 ± 0.04	5.92 ± 0.10	56.98 ± 0.08
Agriculture products				
Peas	Buckwheat	Barley	Oats	Wheat
14.11 ± 0.02	16.93 ± 0.01	13.05 ± 0.03	14.86 ± 0.04	14.60 ± 0.09
Cosmetic and household chemistry products				
Hair gel	Cleansing milk with microcrystals	Liquid soap	Floor polish	Washing gel
96.82 ± 0.06	71.42 ± 0.02	89.75 ± 0.16	96.72 ± 0.06	71.46 ± 0.30

* Precision was expressed as the standard deviation of 6 measurements.

The results of testing the water content of food products showed no significant differences between them when dried with infrared IRL, IRS, IRM, especially for those products with homogeneous structure, *i.e.*, wheat flour, granulated garlic. Small deviations in the trueness of the test were obtained for these products (maximum 0.25%, – determination of water content in flour using a moisture analyser with IRS as the radiation source). The precision of the measurements for these products had low values (garlic max. 0.23%, IRL source), indicating the correct choice of test parameters (Table 4). The total drying time for the flour and garlic was between 15 and 20 minutes. The analysis time resulting from the method used was found to be acceptable for acceptance tests performed under laboratory conditions for control samples, but may be too long for analyses performed in the production cycle. Controlling the quality of a mass-produced product requires rapid access to key information related to the technological process, which is the basis of quality management systems using process approaches such as Lean, Sigma 6 [40, 41].

For food products whose composition varies (multi-ingredient systems), significant errors were found in the trueness of the result in terms of water content (Mexican sauce deviation −0.88%÷1.49%; curry seasoning −0.63%÷0.98%; horseradish mustard sauce −0.61%÷−1.96%) (Table 4). Using the information provided by Vitali Vişanu [42], it can be assumed that the water content results obtained, which were significantly higher than the reference values (trueness value in Table 4), indicated a probable mass loss of the sample as a result of surface burning of its fine fractions [43], (Fig. 1). Such a phenomenon was observed for Mexican sauce and curry seasoning when these products were subjected to drying with a moisture analyser using IRL and IRM heat sources. In contrast, the use of an IRS heat source resulted in an under-drying effect (Table 3). As reported by Li, Zeng, Zhang [44], such a phenomenon is also detrimental to the quality of the dried product.

The consequence of significant errors in the determination of the water content was a significantly poorer precision of the measurements (Mexican sauce, 0.35%, IRL source). The indicated value clearly showed the need to verify the drying parameters in terms of temperature, which was closely related to the heat source used in the operation (IRL/IRM/IRS). The total testing time for

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Table 4. Water content of food products obtained with the moisture analyser method.

Parameters	**Wheat flour**		
Water content (%) depending on the IR source ± St. dev.	IRL 14.35 ± 0.04	IRM 14.37 ± 0.07	IRS 14.60 ± 0.14
Trueness δ (%)	–0.06	0.02	0.25
Analysis time t (min)	11:46	14:05	15:33
Sample mass (g)	5.01 ± 0.10	5.02 ± 0.09	5.02 ± 0.19
	Granulated garlic		
Water content (%) depending on the IR source ± St. dev.	IRL 7.78 ± 0.23	IRM 7.70 ± 0.07	IRS 7.64 ± 0.06
Trueness δ (%)	–0.08	–0.16	–0.22
Analysis time t (min)	15:39	21:36	15:26
Sample mass (g)	4.99 ± 0.13	4.84 ± 0.28	4.93 ± 0.05
	Mexican sauce		
Water content (%) depending on the IR source ± St. dev.	IRL 9.88 ± 0.35	IRM 10.59 ± 0.13	IRS 8.22 ± 0.31
Trueness δ (%)	0.78	1.49	–0.88
Analysis time t (min)	13:30	20:09	12:07
Sample mass (g)	2.10 ± 0.10	2.21 ± 0.06	2.09 ± 0.07
	Curry seasoning		
Water content (%) depending on the IR source ± St. dev.	IRL 6.31 ± 0.20	IRM 6.90 ± 0.26	IRS 5.29 ± 0.08
Trueness δ (%)	0.39	0.98	–0.63
Analysis time t (min)	04:30	08:28	05:43
Sample mass (g)	1.14 ± 0.12	1.19 ± 0.03	1.13 ± 0.11
	Horseradish mustard sauce		
Water content (%) depending on the IR source ± St. dev.	IRL 55.43 ± 0.22	IRM 56.37 ± 0.17	IRS 55.02 ± 0.16
Trueness δ (%)	–1.55	–0.61	–1.96
Analysis time t (min)	23:10	23:27	27:58
Sample mass (g)	1.79 ± 0.06	1.70 ± 0.15	1.73 ± 0.08

these products varied quite a bit, ranging from 4.5 minutes (curry seasoning, IRL source) to about 28 minutes (horseradish mustard sauce, IRS source). No clear correlations were found between drying time and the infrared radiation source used.

In the context of the cosmetic and household chemical product tests, it should be noted that they all featured liquid consistency. For testing the water content, an approximate mass of product was taken in the form of a thin layer of uniform thickness. The most correct water content results for these products were obtained when the measurement was carried out using a moisture analyser with an IRL or IRM heat source applied (Fig. 2). The difference between the value measured by the above method and the reference value was a maximum of 0.13% for the cleansing milk product, IRL source (Table 5). The use of an IRS heat source in the moisture analyser method resulted in a twofold reduction in correctness of the tests for hair gel, cleansing milk, liquid soap, and floor polish, and approximately fourfold one for washing gel compared to the results obtained when the heat source in the moisture analyser was an IRL (Table 5). From the values obtained,

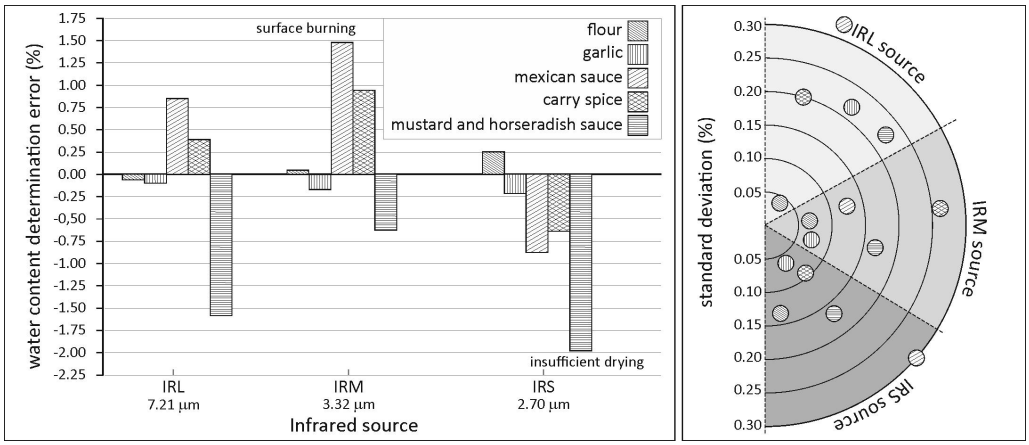

Fig. 1. Trueness and precision of water content measurements for food products.

it was found that the use of different heat sources did not result in significant differences in the precision of the measurements, which ranged from 0.04% (washing gel, IRL source) to 0.30% (liquid soap, IRM source) (Fig. 2).

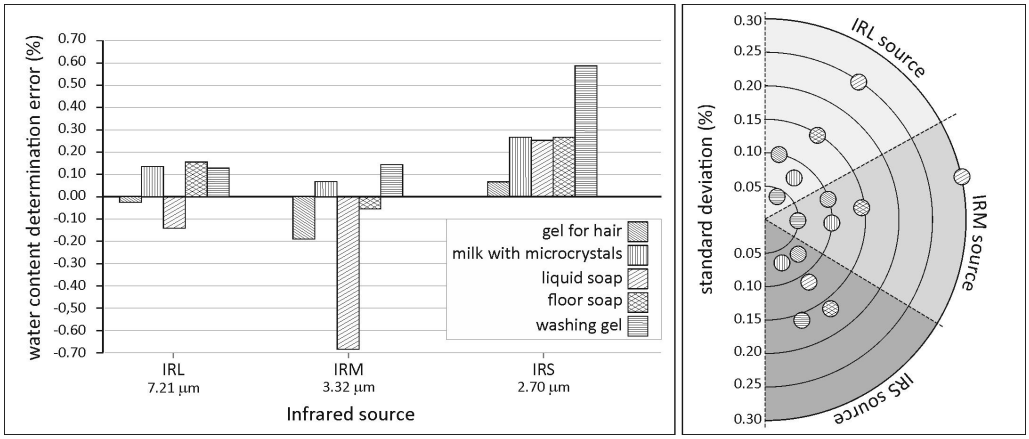

Fig. 2. Trueness and precision of water content measurements for cosmetic and household chemical products.

The longest testing time for water content in cosmetic and household chemical products (approx. 18 minutes) was recorded using IRM and IRS heat sources, while the shortest testing was conducted for floor polish and washing gel when the heat source was an IRL radiant heater (Table 5).

Before testing, the products of the agricultural industry, peas, buckwheat, oats, wheat, and barley, were crushed and their quantities selected so that the product covered the surface of the pan with a thin layer. The study confirmed that, for the samples prepared in this way, the most accurate determination in terms of water content was carried out when the heating medium was an IRL radiator (deviation of 0.01% for peas and oats) (Fig. 3). The largest error in the determination of water content (-0.50%) was registered for wheat when the IRM heat source was used. The use of

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Table 5. Water content of cosmetic and household chemical products.

Parameters	**Hair gel**		
Water content (%) depending on the IR source ± St. dev.	IRL 96.84 ± 0.10	IRM 97.01 ± 0.10	IRS 96.73 ± 0.07
Trueness δ (%)	–0.03	–0.19	0.06
Analysis time t (min)	16:46	15:21	17:29
Sample mass (g)	2.26 ± 0.13	2.25 ± 0.04	2.25 ± 0.04
	Cleansing milk with microcrystals		
Water content (%) depending on the IR source ± St. dev.	IRL 71.42 ± 0.07	IRM 71.36 ± 0.10	IRS 71.18 ± 0.07
Trueness δ (%)	0.13	0.06	0.24
Analysis time t (min)	12:43	12:13	18:11
Sample mass (g)	2.43 ± 0.17	2.42 ± 0.29	2.56 ± 0.20
	Liquid soap		
Water content (%) depending on the IR source ± St. dev.	IRL 89.88 ± 0.25	IRM 90.43 ± 0.30	IRS 89.51 ± 0.11
Trueness δ (%)	–0.13	–0.68	0.23
Analysis time t (min)	13:21	20:25	13:06
Sample mass (g)	2.17 ± 0.09	2.19 ± 0.22	2.19 ± 0.22
	Floor polish		
Water content (%) depending on the IR source ± St. dev.	IRL 96.57 ± 0.15	IRM 96.77 ± 0.13	IRS 96.72 ± 0.17
Trueness δ (%)	0.15	–0.05	0.24
Analysis time t (min)	05:12	05:50	09:25
Sample mass (g)	2.02 ± 0.18	1.95 ± 0.02	1.95 ± 0.06
	Washing gel		
Water content (%) depending on the IR source ± St. dev.	IRL 71.34 ± 0.04	IRM 71.32 ± 0.05	IRS 70.89 ± 0.16
Trueness (%)	0.12	0.14	0.57
Analysis time t (min)	05:12	06:24	06:40
Sample mass (g)	1.07 ± 0.06	1.32 ± 0.19	1.08 ± 0.09

the shortest-wavelength radiant heat source (IRS) for each agricultural product resulted in a water content result slightly higher than its reference value. This result indicated surface superheating of the analysed products, leading to surface combustion. In buckwheat and barley, a slight change in surface colour was observed by Tulej and Głowacki [43], suggesting the possibility of structural damage to the product [33].

Heat sources with longer infrared wavelengths (IRL, IRM) caused slight under-drying of barley (water content determination error of –0.09%, –0.36%), oats (error of –0.01%, –0.13%), and wheat (error of –0.26%, –0.50%) (Table 6). The precision of determination of the water content of agricultural products using heat sources in the range of 2.70÷7.21 μm was in the range of 0.02%÷0.27%. For buckwheat, a precision measurement of 0.15% was obtained (IRS source), which was the best precision for this product. By far, the shortest test time was obtained for all grains when the water removal process took place using an IRL heat source. Infrared radiation

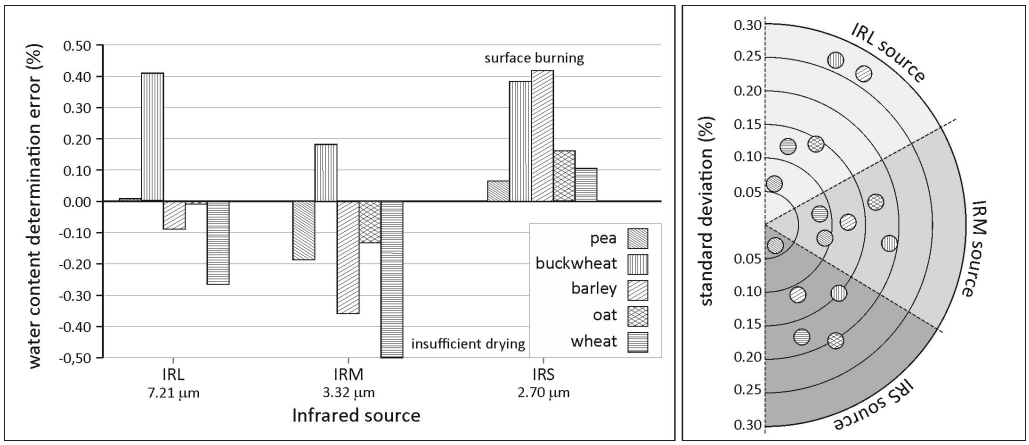

Fig. 3. Trueness and precision of water content measurements for agriculture products.

with a wavelength of 7.21 µm penetrated deep into the sample, through which a volumetric drying effect was achieved [45, 46]. Similar observations were confirmed for flour (Table 4). Through this, the effectiveness of the method using an IRL source to remove water from powder-textured products was confirmed.

The longest testing time for agricultural products was recorded when the IRM source was used. It ranged from about 7 minutes to about 38 minutes. This analysis time is acceptable for laboratory work, but it may be excessive when assessing product quality in a B2B system for multiple contractors at the same time [39].

The statistical analysis carried out to test the normality of the data distribution confirmed that for food, agricultural, cosmetic, and household chemical products, the value of the calculated Shapiro–Wilk test statistic (W) was greater than the critical value (0.788), thus confirming the truth of hypothesis H0 – the water content results obtained for the tested products had the characteristics of a normal distribution. *r*-Pearson correlation coefficients were determined for the relationship between the mass of the sample analysed and the trueness of the analysis, taking into account the heat sources used, IRL, IRM, IRS (Table 7).

Insofar as it is statistically significant in general terms, correlation means that as one characteristic increases, *e.g.*, sample mass, another characteristic either increases or decreases, *e.g.*, trueness of analysis. The assessment of the significance of the correlation consisted of determining the correlation coefficients for each product tested and checking whether the determined value was in the area ($-\infty \div -2.776$ and $2.776 \div +\infty$), which determined the range of data for which there was confidence ($\alpha = 0.05$) that the correlation was statistically significant. Based on the results obtained, it was found that the *r*-Pearson correlation coefficients for the relationship between the mass of the sample analysed and the trueness of the analysis are outside the designated area, so the correlation is not statistically significant. In practical terms, this meant that it was possible to optimise the mass of the sample used during the analysis by increasing or decreasing it, to obtain even better precision measurements and reduce testing time. It should be noted that obtaining a significant correlation between the mass of the sample analysed and the trueness of the analysis would mean, in practice, that the mass of the analytical sample used for the tests would have to be measured very precisely, as its variability would also result in a variability of the water content result. Such a relationship would critically complicate the research method considerably.

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Table 6. Water content of grain agricultural products: peas, buckwheat, barley, oats, wheat.

Parameters	**Peas**		
Water content (%) depending on the IR source ± St. dev.	IRL 14.10 ± 0.06	IRM 14.30 ± 0.09	IRS 14.04 ± 0.02
Trueness δ (%)	0.01	–0.19	0.07
Analysis time t (min)	10:51	14:25	11:36
Sample mass (g)	5.35 ± 0.22	5.20 ± 0.08	5.19 ± 0.07
	Buckwheat		
Water content (%) depending on the IR source ± St. dev.	IRL 16.52 ± 0.27	IRM 16.75 ± 0.19	IRS 16.54 ± 0.15
Trueness δ (%)	0.41	0.18	0.39
Analysis time t (min)	06:05	07:14	06:50
Sample mass (g)	2.31 ± 0.12	2.33 ± 0.09	2.36 ± 0.20
	Barley		
Water content (%) depending on the IR source ± St. dev.	IRL 13.14 ± 0.27	IRM 13.41 ± 0.12	IRS 12.64 ± 0.12
Trueness δ (%)	–0.09	–0.36	0.42
Analysis time t (min)	12:24	16:53	14:01
Sample mass (g)	2.26 ± 0.23	2.43 ± 0.13	2.19 ± 0.18
	Oats		
Water content (%) depending on the IR source ± St. dev.	IRL 11.55 ± 0.14	IRM 11.67 ± 0.17	IRS 11.37 ± 0.19
Trueness δ (%)	–0.01	–0.13	0.17
Analysis time t (min)	10:58	13:55	11:57
Sample mass (g)	2.28 ± 0.15	2.19 ± 0.11	2.35 ± 0.18
	Wheat		
Water content (%) depending on the IR source ± St. dev.	IRL 11.86 ± 0.13	IRM 12.10 ± 0.08	IRS 11.49 ± 0.17
Trueness δ (%)	–0.26	–0.50	0.11
Analysis time t (min)	22:49	37:15	30:16
Sample mass (g)	5.29 ± 0.12	5.30 ± 0.22	5.33 ± 0.15

The final element of the research conducted in each area should be the analysis and interpretation of the results in the context of their usefulness to science and industry. This approach, on the one hand, allows for the development of research methods demonstrating their weaknesses and strengths and, on the other hand, provides an opportunity to verify the developed research methods through various areas of industry, from quality control departments to technological processes. The obtained results of the water content of food, agricultural and chemical products can be considered in metrological, economic and ergonomic contexts. From a research point of view, the metrology of the process carried out is important, which can be simplified to assess the accuracy of water content determination. The usefulness of the test method for industry can be assessed by the relative error, which allows relating the water content test result not only to technological limits, but also allows estimating gains or losses as a result of too little or too much water in the product. For high-volume production, this is one of the elements of product quality management. The magnitude of the relative error of water content measurements is given in Table 8.

Table 7. Shapiro–Wilk test (W), r-Pearson correlation coefficients (r), correlation significance for the products.

	W / r-Pearson / r-Pearson significance		
Product name / heat source	IRL	IRM	IRS
Flour	0.946 / 0.19 / 0.39	0.956 / –0.10 /-0.19	0.826 / 0.02 / 0.05
Granulated garlic	0.949 / –0.43 / –0.95	0.896 / 0.80 / 2.65	0.918 / 0.24 / 0.49
Mexican sauce	0.975 / 0.33 / 0.71	0.808 / 0.73 / 2.16	0.984 / 0.60 / 1.49
Curry seasoning	0.930 / 0.28 / 0.59	0.980 / –0.20 / –0.41	0.913 / –0.42 / –0.94
Horseradish mustard sauce	0.930 / –0.10 / –0.22	0.885 / 0.65 / 1.71	0.937 / –0.45 / –1.00
Hair gel	0.793 / 0.47 / 1.06	0.948 / 0.44 / 0.98	0.948 / –0.13 / –0.26
Cleansing milk with microcrystals	0.905 / 0.72 / 2.10	0.962 / –0.47 / –1.08	0.831 / –0.42 /-0.93
Liquid soap	0.878 / –0.05 / –0.10	0.811 / 0.64 / 1.66	0.909 / 0.53 / 1.24
Floor polish	0.936 / –0.13 / –0.27	0.846 / 0.80 / 2.69	0.887 / 0.11 / 0.23
Washing gel	0.948 / 0.24 / 0.50	0.906 / –0.71 / –2.03	0.942 / 0.44 / 0.97
Peas	0.967 / 0.36 / 0.78	0.787 / 0.85 / 3.21	0.811 / 0.61 / 1.54
Buckwheat	0.869 / –0.15 / –0.30	0.868 / 0.21 / 0.43	0.910 / 0.58 / 1.41
Barley	0.942 / 0.33 / 0.71	0.827 / 0.47 / 1.06	0.891 / 0.58 / –0.24
Oats	0.980 / –0.22 / –0.46	0.909 / –0.15 / –0.31	0.959 / –0.05 / –0.10
Wheat	0.850 / 0.14 / 0.27	0.883 / 0.86 / 3.33	0.879 / 0.59 / 1.48

Table 8. Relative error of determination of water content depending on the source of IR radiation.

	Water content (%) reference value	Water content – relative error (%)		
		δ/IRL	δ/IRM	δ/IRS
Wheat flour	14.35	0.42	0.14	1.74
Granulated garlic	7.86	1.02	2.04	2.80
Mexican sauce	9.10	8.57	16.37	9.67
Curry seasoning	5.92	6.59	16.55	10.64
Horseradish mustard sauce	56.98	2.72	1.07	3.44
Hair gel	96.82	0.03	0.20	0.06
Cleansing milk with microcrystals	71.42	0.18	0.08	0.34
Liquid soap	89.75	0.14	0.76	0.26
Floor polish	96.72	0.16	0.05	0.25
Washing gel	71.46	0.17	0.20	0.80
Peas	14.11	0.07	1.35	0.50
Buckwheat	16.93	2.42	1.06	2.30
Barley	13.05	0.69	2.76	3.22
Oats	14.86	0.07	0.87	1.14
Wheat	14.60	1.78	3.42	0.75

The magnitude of the relative error in the determination of water content of less than 1% suggests that for cosmetic and chemical products only, optimization of the test method is not required. However, it is necessary for food and agricultural products especially when the heat source is an IRS heater. The economic aspect of the test method can be considered through

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the amount of energy required to perform the determination and the cost of organizing and maintaining the test stand. The reference method requires several components such as a balance, laboratory dryer, desiccator, and mass standards. Energetically, for one test cycle, the reference method requires power consumption of about 5.10 kWh (dryer power of 1.7 kW × analysis time of 3 hours), while the method based on IR radiation requires only 0.13kWh (IR emitter power of 0.4 kW × analysis time of 20 min). The method using IR weighing machines can therefore be a significant source of savings. Ergonomics and automation of the drying process is also one of the important factors for personnel. Nowadays, simplicity of operation and a certain degree of automation are desirable despite the complexity of the processes that occur during the test. Such features can be sought in a method using IR radiation, however, one should beware of unreflective interpretation of test results. Nowadays, in many cases, the results are obtained automatically, but the evaluation of their accuracy and usefulness requires deeper knowledge, which the authors of this publication have tried to present in a simple form.

5. Summary and Conclusions

There are now numerous moisture analysers that make it possible to determine the water content as a result of the mass loss of the product during its controlled heating. From the viewpoint of the user, such a test method appears simple and intuitive, regardless of the type of heat source that is installed inside the drying chamber. It should be noted, however, that the parameters of the analysis to be carried out, such as the precision of the measurement, the testing time, and the accuracy of the determination of the water content, can be strongly dependent on the characteristics of the heat source that is used to heat the sample. The increase in temperature of the sample is due to the absorption of infrared radiation by all its components. Some, such as amino acids, polypeptides, and proteins, show strong infrared absorption in the 3÷4 μm and 6÷9 μm ranges, carbohydrates in the 3 μm and 7÷10 μm range, and water shows strong absorption of radiation in the 2.7÷3.3 μm and 6 μm range. This clearly indicates that there is no single ideal method for drying products using infrared radiation, but it is advisable to search for a solution that is optimal in terms of the accuracy and precision of the analysis carried out. Such an attempt has been made in this work for food, cosmetic, and household chemical products. Based on the research carried out, it was concluded that the use of a heat source with different infrared radiation wavelengths was a necessity when optimising the test method.

The accuracy of the analysis of the water content of food products was maintained regardless of the infrared radiation source used when the product structure was homogeneous, *e.g.*, flour. In the case of multi-ingredient food products such as Mexican sauce, a lack of accuracy in the analysis was found regardless of the heat source used, with a water content result greater than the reference value when a wavelength of 3.32÷7.21 μm (IRM, IRL) was used, and a lower value when the heat source was an IRS wavelength of 2.70 μm. This indicates the need to verify the amount and method of energy delivery to the test sample. In terms of handling methodology, reducing the drying temperature and placing the dried sample between glass-fibre filters seems to be the right direction, which could be the subject of further research.

Cosmetic and household chemical products can be dried accurately using all IRS, IRM, and IRL heat sources, but the accuracy of the analysis is maintained only if the radiation emission is in the range of 3.32 μm÷7.21 μm (IRM, IRL). The use of a heat source with a shorter wavelength, 2.70 μm, resulted in under-drying of the products while maintaining measurement precision. This deviation can be accepted as a systematic error related to the research method. Agricultural products were best dried using an IRL source (wavelength 7.21 μm), but only slightly worse

results were obtained when the heat source was an IRM (wavelength 3.32 µm). The use of the shortest radiation wavelength, *i.e.*, 2.70 µm (IRS), for agricultural products during drying resulted in surface burning of the sample, which also resulted in erroneous water content values. The time required to analyse the water content of the test products was determined by the mass of the sample being analysed, the heat and mass exchange dynamics of the process, and the radiation absorption capabilities of the ingredients forming the sample. For products with a powder structure, heat and mass exchange took place quickly, but it could be disrupted by surface combustion processes of the sample, which has a significant impact on the total test time. Products with a semi-liquid structure can form an impermeable layer on their surface during drying, limiting the possibility of water desorption from the sample's deeper layers. As in the case of liquid soap, when the heat source was an IRM, the result is significantly lower than the reference value. It should be noted that what appears to be a simple test method using a moisture analyser is actually quite complicated because the sources of potential error are both in the instrument and in the sample being tested. Only a thorough assessment based on prior experience can identify high-risk areas.

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