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# DETERMINATION OF THE MOISTURE CONTENT IN INFRARED DRIED APPLES

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The objective of this work was to find parameters for determining a moisture content of dried apples by means of infrared analyzer (MA 35 from Sartorius, Germany), such ones that the results correspond to those determined by classical vacuum oven method. Apples were available in seven batches with moisture contents from 2.5 to 28%. The drying parameters were established using the automatic mode. Temperature was varied in a range from 80 to 112˚C. Two various temperatures 82 and 98˚C were defined as the most suitable to apples with moisture content less and more than 7.5 % respectively. The statistical evaluation of the accuracy of the results of measurements was derived.

*Keywords*: infrared analyzer, rapid method, drying temperature, statistical estimation

## 1. Introduction

The moisture content of dried fruit is a very important indicator of their quality, which influences their sensory, textural and mechanical properties, microbiological stability and stability to undesirable biochemical changes. Rapid methods for determining the moisture are needed for a proper management of the technological process and to obtain the dried product of a standard quality.
Various methods for determining moisture content in foods, based on different measurement principles are used in the world. The thermogravimetric method is a very common analysis of dried fruit. The thermogravimetric method determines the moisture content as a loss of product weight during drying, under certain conditions. The advantages of this method are the relative simplicity and no chemicals.

The problem with all drying techniques is that they do not measure water specifically. All the compounds volatile under the analytical conditions contribute to the mass loss, even compounds that are not originally contained in the sample but are formed by chemical reactions during the analysis, particularly by decomposition reactions at higher temperatures. But, on the other hand, strongly bound water may escape detection (Isengard, 2008).

Standard Layout (2008) has established two methods for determining the moisture content in dried fruits: method 1 - laboratory reference method; method 2 - rapid method. This rapid method is based on the method prescribed by AOAC (AOAC Official Method 972.20) and serves to determine the moisture for dried fruits, especially such as prunes and raisins. The measurement principle is the determination of the conductance and temperature of a test portion by the moisture meter. The moisture meter has to be calibrated according to the laboratory method for each kind of dried fruit, taking into account the variety or commercial type and the type of presentation and, when necessary, the crop year and/or the origin. Conventionally moisture content is the correlation between moisture content and meter's readings specified in AOAC Official Method 972.20. According to the Standard Layout, it is also possible to employ other rapid methods based on the principle of loss of mass by heating with apparatus including a

halogen or infrared lamp and a built-in analytical balance, always with the condition that the method and the apparatus have to be calibrated according to the laboratory method.

Thermogravimetric infrared analyzers with integrated scales are widely used to analyze the moisture content of chemical products, pharmaceutical, paper, cosmetic, textile, food and animal feed industry. Sartorius AG application database for moisture determination includes about

100 items of food products; however, very limited number of them belongs to the dried fruit. Dried fruits are characterized by a significant amount of volatile substances, sugars, polyphenols, which have been modified in the process of drying and tend to decompose under the employed drying conditions. Consequently the measured mass loss is not very likely to match the water content of the sample. A calibration of the device has therefore to be carried out.

The objective of this work was to find parameters for the infrared drying of dried apples, such ones that the results correspond to those determined by classical oven method.

## 2. Materials and methods

### *2.1. Samples preparation*

Two varieties of fresh apples, Golden Delicious and Prima, were purchased from the local market.

Each of the experiment series was carried out, using the same lot of fresh apples. Apples were washed, cored, sliced, placed onto perforated stainless steel trays and dried in a pilot-plant tunnel air drier at the temperature of 62 ± 2ºC. In the course of drying, certain quantities of the product (7 portions) with different moisture content in a range from 4.2 to 28 % were selected. The range of moisture taken covers the dried apples commercial quality which is regulated by UNECE STANDARD DF-16.

Before application each portion of dried apples was ground in an electric coffee mill of 160 Wt power to obtain uniform bits with the size of 2-4 mm. Samples with low moisture were ground with care, avoiding large amounts of fine powder to be formed. Ground samples were packed and tightly closed.

### *2.2. Reference method for determining moisture in dried apples*

The test portions (2.5000±0.1000 g for brittle product and from 3.5000 to 4.0000 g for product with elastic texture) were placed in non-corrosive metal containers, provided with well-fitting lid, about 45 mm of diameter and dried in an electric vacuum oven at 70 ± 1˚C under pressure below 100 mm Hg (13.3 kPa). The drying was proceeded until, practically, constant mass was reached (if the reduction in weight over 2 hours of drying is less than 0.0010 g).

The mass loss determined by weighing the test portion and the container before and after the drying process (after cooling in desicators) is defined as moisture content. Five parallel determinations on one analysis were carried out on the same test sample. Their average arithmetic value was accepted as a final result.

### *2.3. Rapid method for determining moisture in dried apples*

An analyzer MA 35 from Sartorius AG, Germany, was used for infrared drying. The test portions were placed in one layer in aluminium dishes that were set on the pan of the built-in balance (resolution 1 mg). The apparatus offers two different drying modes. In both modes a drying temperature is chosen. In the automatic mode the analysis is stopped when no change in mass is registered during a certain short time interval. In the time mode the drying time is chosen, after which the determination is ended. The mass or value of moisture content is continuously monitored.

In the case of using the automatic mode, the temperature must be chosen so that the results correspond to the moisture content determined by a reference method. When decomposition reactions with formation on further volatile material occur, resulting in values too high for the mass loss, a lower temperature has to be applied.

In the case of using the time mode, the duration of the analysis has to be fixed in such a way that in the end the mass loss just corresponds to the moisture content. Of course, this procedure is only possible if the drying curves are well reproducible so that the same mass loss is reached after the same determination time.

### *2.4. The statistical analysis of measurement results*

The statistical evaluations of accuracy of the measurement results were established according to the standard ISO 5725 (1994).

## 3. Results and discussions

The automatic mode of the rapid method was used because in the preliminary experiments it had been shown that the determination of moisture in the dried apples, using the time mode led to negative results.

Each first result of measurement was rejected as very different from the other replicates, obtained in the conditions of repeatability. The two-minute intervals between measurements in the conditions of repeatability were maintained. The first result was excluded each time as a change of drying parameters or interruptions in the operation occurred. The fact that the status of the device at the beginning of the measurement has a great impact on the measurement results was indicated in the literature (Isengard & Präger, 2003). It has also been reported in the literature that the duration of intervals between measurements is important and when the time intervals are longer than 10 min., a too high rate of moisture content is resulting (Isengard et al., 2001).

The relationship between temperatures of measurement and moisture content obtained by infra-red analyzer for seven portions of dried apples with various moisture content (first series) are presented in Figure 1. The means of moisture content measured according to the reference method are shown on the schedule in the form of horizontal dashed lines. The lines cross corresponding curves in the certain points, at which both measured values of moisture coincide. Each point of intersection marked as a, b, c, d, e, f and g, corresponds to certain most suitable temperatures (vertical dashed lines).

It can be observed (Figure 1.) that the rising of the drying temperature, set as a parameter, leads to increasing the value of moisture obtained as a result of infrared drying; the most suitable temperature tends to grow with increasing the moisture of the tested sample. Thus to obtain results with a high accuracy, the measurement temperature should be established in dependence of the test sample moisture. As the moisture of product is not known previously, then the establishing of drying temperature in dependence of sample moisture is little suitable in practice. At the same time, it was noticed that slices of dried apples with moisture below 7-7.5% were brittle, and above this value had an elastic texture. The found textural difference in dried apples, allowed us to divide the studied range of moisture on the two corresponding zones. For each of the selected zone, their most suitable temperature as 82 º C and 98 º C, respectively, was established.

Further the next problem was put forward, namely the testing of dried apples at the selected temperatures and determining of statistical characteristics of the measurement results. Other seven portions of dried apples with varying moisture in a range from 2.4 to 25.9 % were prepared (second series). Two brittle samples were analyzed at a temperature of 82ºC. Five samples with an elastic texture were analyzed at a

temperature of 98ºC. A statistical estimation of measurement results (first and second series) are presented in Tables 1 - 2. For symbols and abbreviations see the Nomenclature.

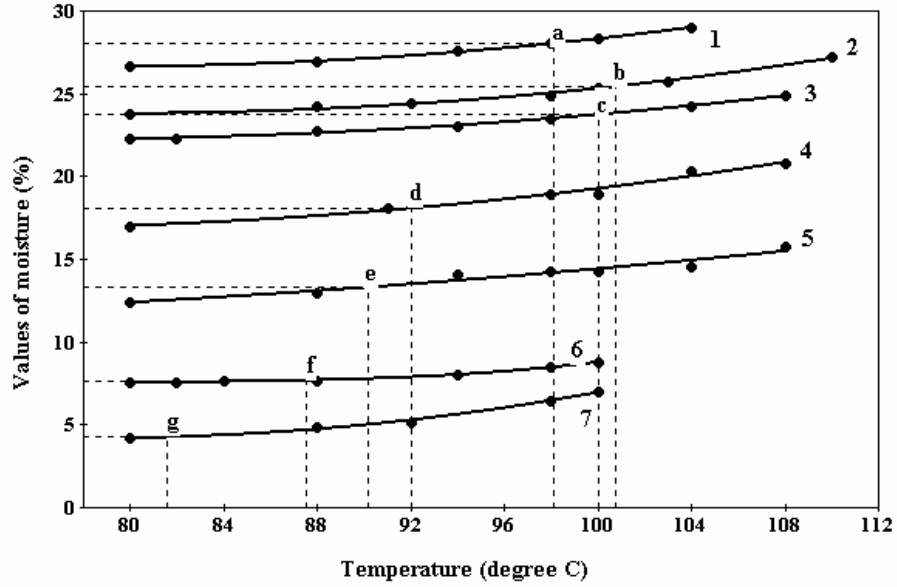


**Figure 1.** Measured values of moisture ( $\bar{y}_j$ ) vs temperature of measuring by infrared drying for apples with different moisture content ( %): 1– 4.24; 2 – 7.67; 3 – 13.29; 4 – 18.06; 5 – 23.73; 6 – 25.42; 7 – 28.02

**Table 1.** Moisture determination by infrared analyser at temperature 82 °C for brittle samples of dried apples. Statistical estimation of experimental values

| Level ($q$) | Statistical characteristics | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $n$ | $s_j$ | $\bar{y}_j$ | $\mu$ | $\hat{\Delta} = \bar{y}_j - \mu$ | $\hat{\Delta} - As_j$ | $\hat{\Delta} + As_j$ | significance of $\Delta$ |
| 1 | 3 | 0.0611 | 4.20 | 4.24 | -0.04 | -0.1091 | 0.02914 | insign |
| 2 | 3 | 0.1305 | 7.57 | 7.67 | -0.01 | -0.2478 | 0.0478 | insign |
| 3 | 5 | 0.1297 | 7.56 | 7.41 | 0.15 | -0.4184 | 0.7184 | insign |
| 4 | 5 | 0.2969 | 3.35 | 2.54 | 0.81 | -0.4912 | 2.1112 | insign |

**Table 2.** Moisture determination by infrared analyser at temperature 98 °C for samples of dried apples with elastic texture. Statistical estimation of experimental values

| Level ($q$) | Statistical characteristics | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | $n$ | $s_j$ | $\bar{y}_j$ | $\mu$ | $\hat{\Delta} = \bar{y}_j - \mu$ | $\hat{\Delta} - As_j$ | $\hat{\Delta} + As_j$ | significance of $\Delta$ |
| 1 | 3 | 0.31953 | 8.46 | 7.67 | 0.79 | 0.43 | 1.15 | sign. |
| 2 | 3 | 0.26514 | 14.17 | 13.29 | 0.88 | 0.58 | 1.18 | sign. |
| 3 | 3 | 0.40004 | 18.92 | 18.06 | 0.86 | 0.41 | 1.31 | sign. |
| 4 | 3 | 0.22605 | 23.49 | 23.73 | -0.24 | -0.5 | 0.016 | insign. |
| 5 | 3 | 0.03214 | 25.03 | 25.47 | -0.44 | -0.48 | -0.40 | sign. |
| 6 | 3 | 0.6354 | 28.01 | 28.02 | -0.01 | -0.73 | 0.709 | insign. |
| 7 | 8 | 0.1487 | 9.30 | 8.70 | 0.60 | 0.50 | 0.70 | sign. |
| 8 | 8 | 0.43398 | 15.60 | 14.75 | 0.85 | 0.55 | 1.15 | sign. |
| 9 | 7 | 0.41457 | 19.26 | 18.49 | 0.77 | 0.46 | 1.08 | sign. |
| 10 | 7 | 0.20254 | 23.82 | 23.70 | 0.12 | -0.03 | 0.25 | insign. |
| 11 | 6 | 0.14774 | 25.46 | 25.90 | -0.44 | -0.56 | -0.32 | sign. |

The bias (Δ) of measurements performed at 82 °C for all four levels, as well as the differences between standard deviations were considered the insignificant values (Table 1), that allowed to unite in one batch all results of measurements and to calculate a value of average standard deviation $S_{n,q}$=0.2099 ($N_{n,q}$=16; $f_{n,q}$=12) and a repeatability limit $r$=0.58 ($p$-level 0.05%).

The biases of measurements performed at 98 °C were considered the significant values for the most of the indicated levels (Table 2). However, since the differences among the standard deviations obtained at each level, proved to be insignificant, $S_{n,q}$ = 0.3212 ($N_{n,q}$=54 ; $f_{n,q}$=43) and $r$=0.89 ($p$-level 0.05%) were calculated.

It is clear from Figure 2, that the values of the moisture content obtained by rapid method ($\overline{y}_i$) and reference method ($\mu$) are closely correlated. The correlation can be expressed by the equation of a linear type: $\overline{y}_j = 0.9379801 * \mu + 1.5114988$; $R^2$=0.9976; $s$=0.3274.

This equation applies to a range of moisture content in apples from 7.5 to 28%.

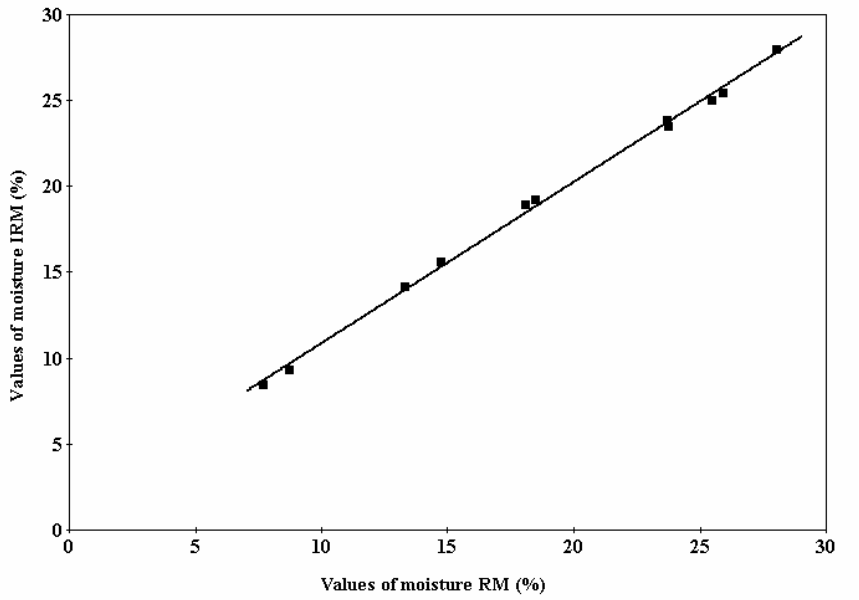


**Figure 2.** Values of moisture measured by IRM ($\overline{y}_j$) vs values of moisture measured by RM ($\mu$) for the range of the moisture content of dried apples from 7.5 to 28%

## 4. Conclusions

Comparison of the determination of moisture content obtained by the rapid method of infrared drying and classical vacuum method are presented in this paper in relation to dried apples with moisture from 2.5 to 28%.

Application conditions of the thermogravimetrical infrared analyzer (MA 35 from Sartorius, Germany) have been established: the automatic mode, the temperature 82˚C for the brittle dried apples, the temperature 98˚C for apples with an elastic texture.

The relationship between moisture content obtained by IRM and by RM is expressed in the form of a linear equation for the moisture range from 7.5 to 28 %. The accuracy of the rapid method has been estimated.

**Nomenclature**

| | |
|---|---|
| $\bar{y}_j$ - arithmetical average value of test results | $\Delta$ - bias of the measurement method |
| $s$ - standard deviation | $\hat{\Delta}$ - estimation of bias |
| $\mu$ - accepted reference value | $A$- index used for calculating the uncertainty in evaluation. A= $1.96/\sqrt{n}$ |
| $q$ - number of levels of the characteristic measured | $r$ – repeatability limit (p-level 0.05%) |
| $j$ - level identifier | $R^2$- coefficient of determination |
| $n$ - number of parallel measurements at each level | IRM – infrared drying method |
| $N$- number of measurements | RM – reference method |
| $S_{n,q}$ - average standard deviation | sign. – significant |
| | insign. - insignificant |

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