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Water determination in products with high sugar content by infrared drying

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Abstract

Instead of measuring the real water content, very often drying mass loss is determined. Not only water contributes to this mass loss, but all volatile materials under the applied drying conditions. On the other hand, very tightly bound water may elude detection. The mass loss by drying does therefore not necessarily correspond to the water content of the sample. To accelerate analyses, in comparison with the classical oven method, more efficient heating sources have been introduced. Infrared dryers belong to this category. This more vigorous heating tool involves a higher risk of decomposition of the sample with production of further volatile compounds. The danger of obtaining erroneous results is particularly high for heat-sensitive products, such as sugars. The objective of this work was to find parameters for the infrared drying of sugars and products with a high sugar content such that the results correspond to those determined by the Karl Fischer titration which selectively measures the water content.

Keywords: Water determination; Products with high sugar content; Infrared drying; Karl Fischer titration

1. Introduction

Infrared drying is a rapid method aimed at the thermogravimetrical determination of water content. It has been shown that the true water content of dairy products can be obtained by infrared drying (Brack & Isengard, 1995). The principle of infrared heating is the transformation of radiation energy into movements of the molecules in the absorbing matter. In contrast to heat transfer by convection in a drying oven, thermal radiation supplies an efficient heating of the sample with the consequence that water is liberated faster. However, the measured mass loss includes, not only water, but all substances that are or become volatile under the drying conditions. Particularly sugars and sugar-containing foodstuffs 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 following aspects have to be taken into account for this calibration:

- A reliable method of water determination is used as a reference (here: Karl Fischer titration).
- The sample preparation and application is standardised.
- The visible sample change, due to decomposition reactions, should be as small as possible.

2. Materials and methods

The reference water content was determined by Karl Fischer titration (KF Titrino 701 from Metrohm, Herisau, Switzerland) with a one-component system using Hydranal-Composite 5 (Riedel-de Haën, Seelze, Germany) as titrating solution.

Necessary modifications of the standard Karl Fischer procedure are

- working at elevated temperature (50 °C) (Isengard & Schmitt, 1995),
- addition of formamide,
- internal homogenisation using the Ultra-Turrax (IKA, Staufen im Breisgau, Germany) (Isengard & Nowotny, 1991).

The samples were then analysed for mass loss by infrared drying (MA 40 from Sartorius, Göttingen,

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Nomenclature

A	Automatic mode
IRD	Infrared drying
KFT	Karl Fischer titration
ME	Working medium methanol
ME/FA	Working medium methanol/formamide (2:1 by volume)
n	Number of replicates
s	Standard deviation
UT	Internal homogenisation using the Ultra-Turrax
(+)	Sample with the addition of water
I	Batch I
II	Batch II

Germany). Mixing and grinding the samples thoroughly was sufficient for sample preparation. Candies were ground in a mill, with a cooling device, before application (IKA Laboratory mill A 10 from Janke & Kunkel, Staufen im Breisgau, Germany). Honey and fruit jellies were applied using glass-fibre pads. The drying parameters were established using the automatic mode. In this mode the analysis is terminated when, at a set temperature, the mass loss within the last time interval of measurements is below the minimum. Temperature and sample size were varied. Before starting an analysis, the dryer was brought to the temperature set as parameter, because it has been shown that the state of the device at the beginning of a measurement has a great impact on the results (Isengard & Färber, 1999).

To ensure that the established drying conditions are suitable, samples differing only in their water content were analysed. Candies were available in two batches, with different water contents. Cookies were moistened by keeping the sample in a humid atmosphere. To honey and fruit jellies, a small amount of water was added.

The results were evaluated in comparison to the reference method focusing on:

- correspondence with reference method,
- variation of the results,
- duration of the analysis.

3. Results and discussion

In Figs. 1–11 the mean values, the standard deviations and the average analysis times are given. The grey rectangles behind these results symbolise the confidence intervals (*p*-level 0.05%) of the respective method, thus showing

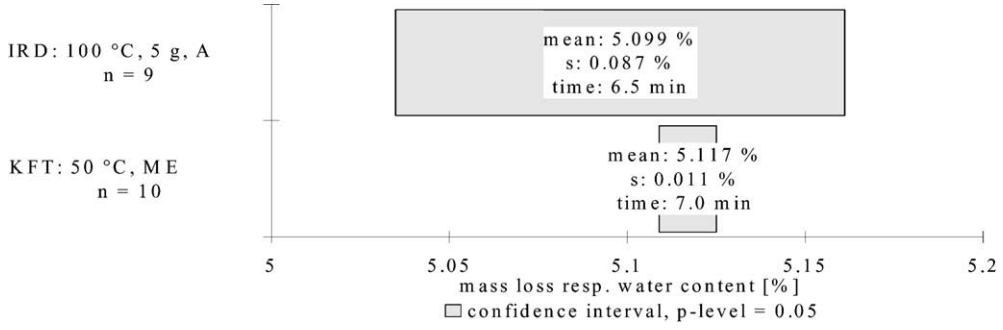

Fig. 1. Water determination in lactose.

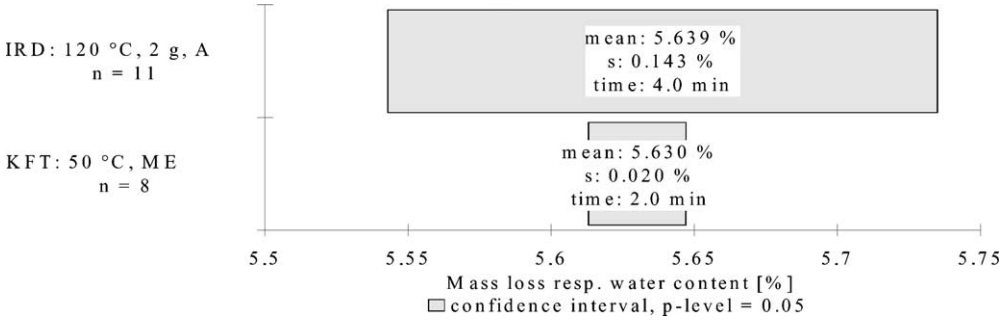

Fig. 2. Water determination in maltose.

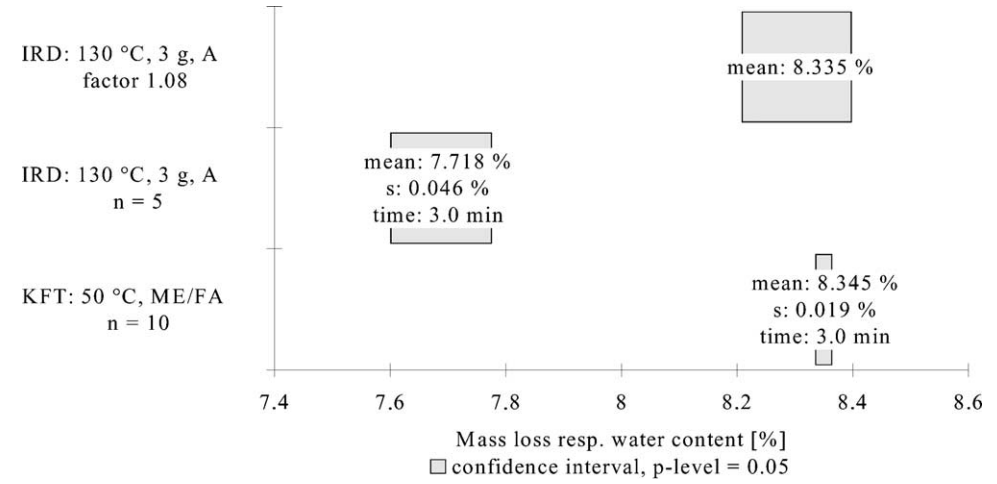

Fig. 3. Water determination in glucose monohydrate.

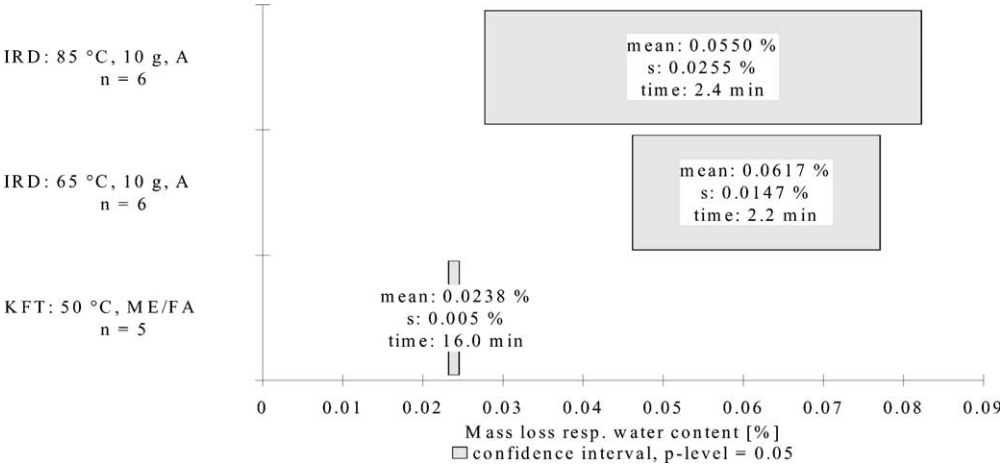

Fig. 4. Water determination in sucrose.

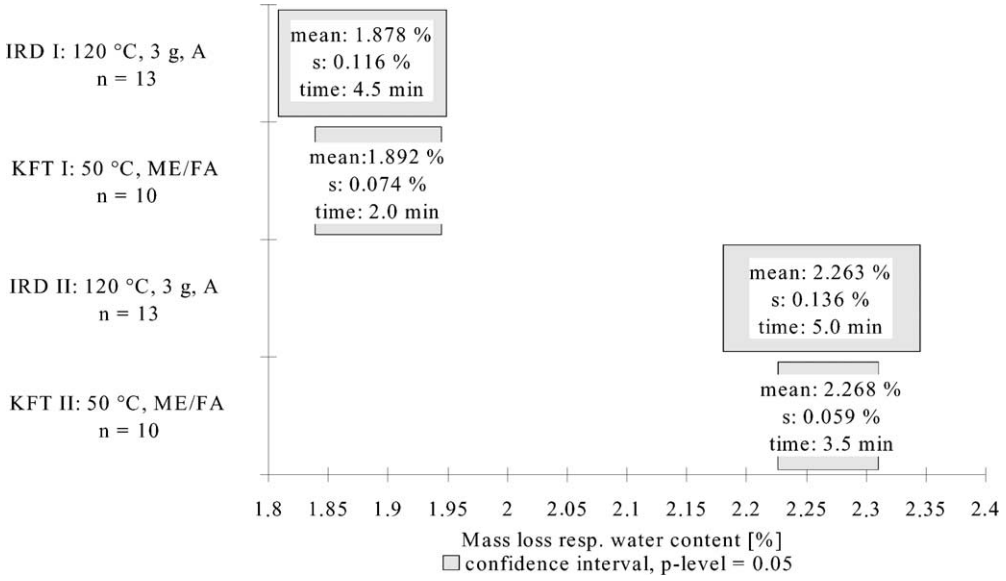

Fig. 5. Water determination in candies on the basis of isomalt.

the precision of the method and indicating significant differences between the methods applied on the same sample. For symbols and abbreviations see the Nomenclature.

Lactose contains 5.0% water of crystallisation and additional water adsorbed at the surface. The Karl Fischer result can be reproduced by infrared drying, even though the standard deviation is eight times higher. For this sugar, infrared drying presents an efficient method for water release (Fig. 1) compared to oven-drying where the correct water content cannot be detected, even after 6 h at 103 °C (1.24±0.09% for four replicates).

Maltose contains 5.0% of crystallisation water and additionally surface water. Compared to lactose a higher temperature had to be applied. The mass loss corresponds to the Karl Fischer result, but the standard deviation is again much higher (Fig. 2).

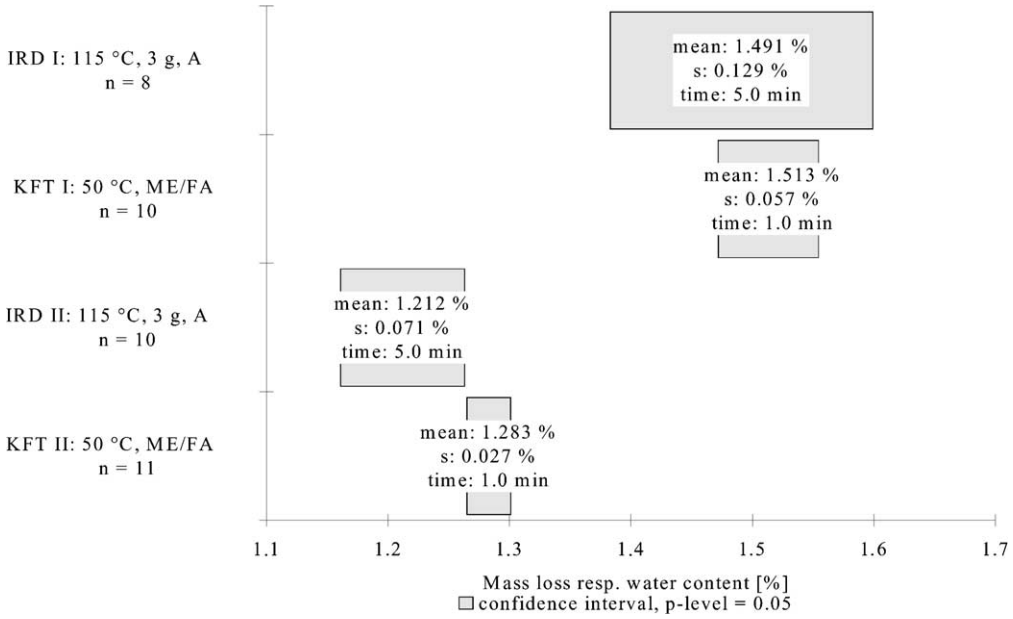

Fig. 6. Water determination in candies on the basis of sorbitol.

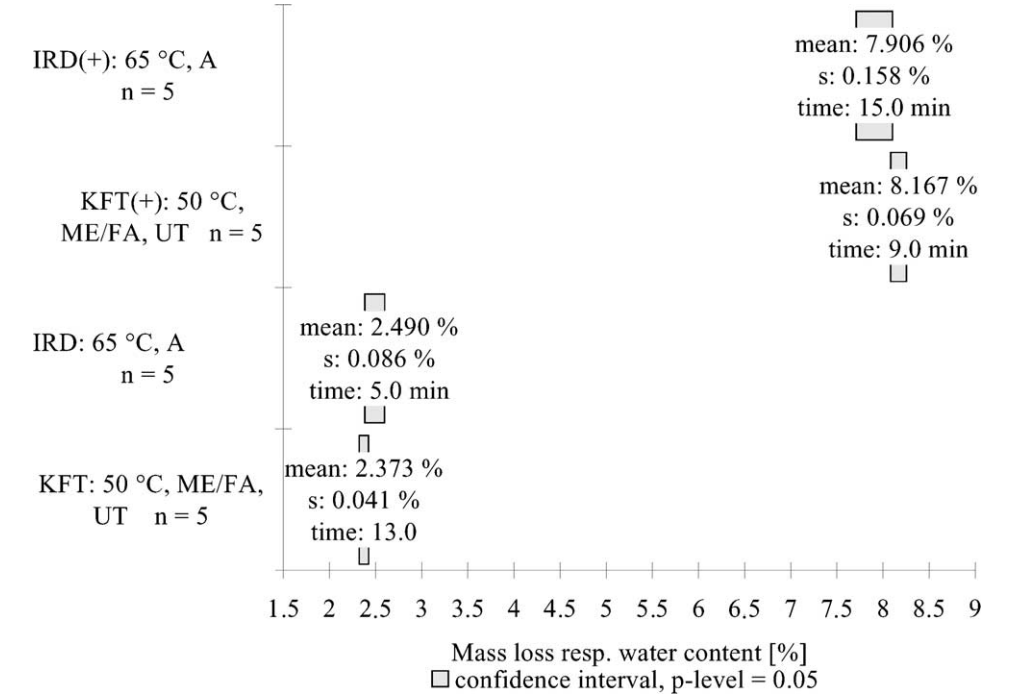

Fig. 7. Water determination in cookies.

Glucose monohydrate contains 9.09% of crystallised water. Usually a lower water content is found because anhydrous glucose is contained in the product (M. Mathlouthi, personal communication). The water of glucose monohydrate cannot completely be released, even at a temperature of 130 °C. Glucose melts during the drying and possibly holds water back. The relatively low standard deviation indicates, however, that the results are well repeatable. In such a case, the infrared result can be multiplied by a correction factor (here 1.08) to obtain the reference value (Fig. 3).

A problematic case is that of sucrose, which has no water of crystallisation. The sensitivity of the device was not sufficient to detect the low water content of 0.02% (Fig. 4). The Karl Fischer method detects the total water content as the sample is completely dissolved in the working medium. The higher values found by infrared drying must therefore be due to a decomposition of the sample. The high standard deviations indicate that this decomposition is not well reproducible.

Other critical product are candies made with sucrose. Decomposition of such products already begins at a very low temperature leading to an unacceptable variation of the results. When drying at low temperature, the energy is not sufficient for total water release. Raising the temperature leads to a continuous weight loss, caused by decomposition reactions, and constancy of weight is not

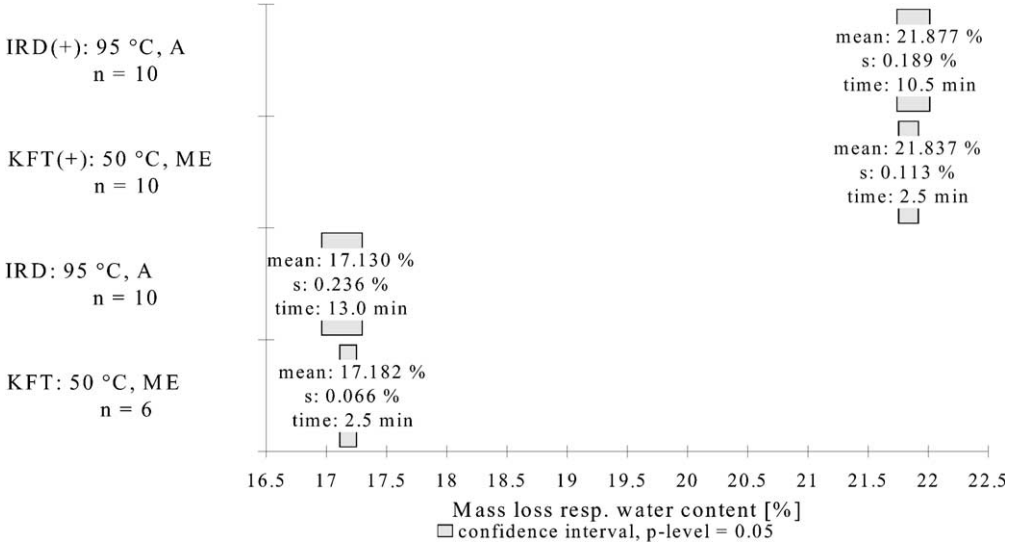

Fig. 8. Water determination in honey.

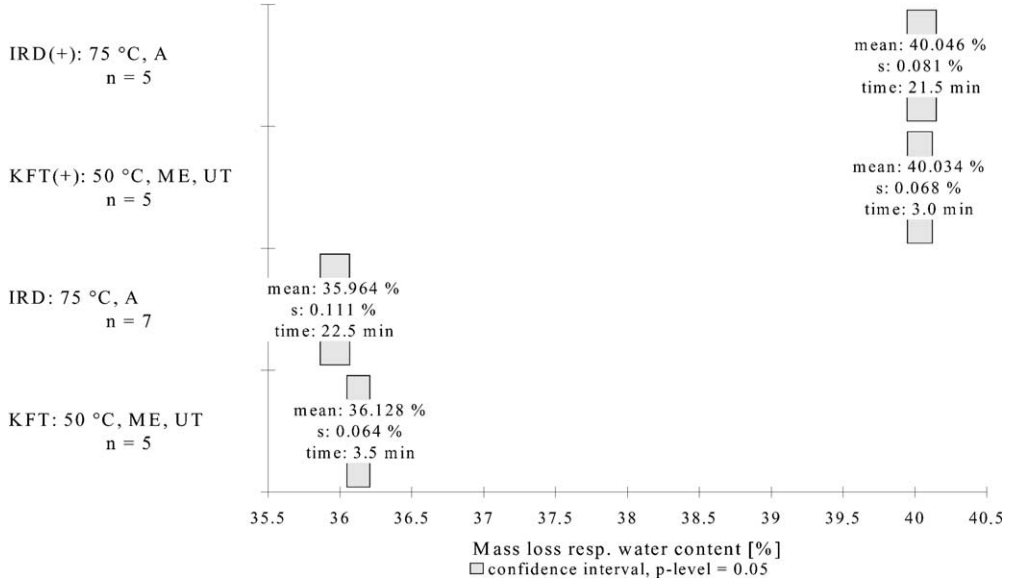

Fig. 9. Water determination in blackcurrant jelly.

achieved. In such cases and under the condition that the sample decomposes reproducibly it may be promising not to use the automatic mode but to fix a certain time and a certain temperature. From the drying curve (mass loss versus time) at the temperature applied, it can be seen after which time the mass loss corresponds to the water content, determined by Karl Fischer titration. This time, and the corresponding temperature can be set as parameters. Such experiments were carried out at various temperatures but were not successful. The standard deviations were unacceptably high, indicating that the decomposition reactions are not well reproducible.

Candies containing the more heat-resistant sugar alcohols, isomalt and sorbitol, show different behaviour. Drying conditions which allow correct determinations of different water contents in each sample were found (Figs. 5 and 6).

In cookies and honey, different water contents could be determined correctly with the same parameter set for the respective product (Figs. 7 and 8).

The infrared method for fruit jellies was worked out for blackcurrant (Fig. 9) and then also applied to quince. The correlation of infrared results with Karl Fischer values was not quite as good (Fig. 10). If the

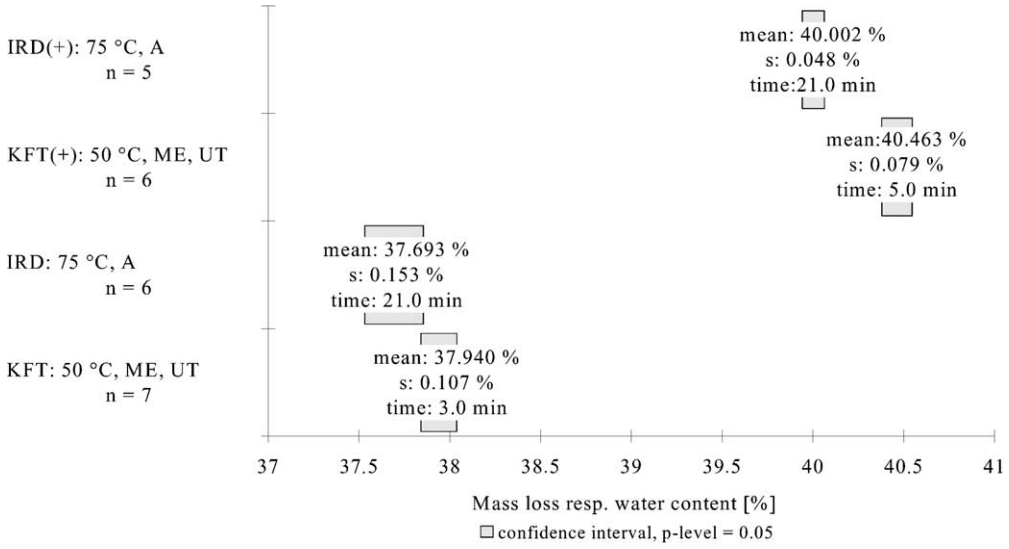

Fig. 10. Water determination in quince jelly, using the method developed for blackcurrant jelly.

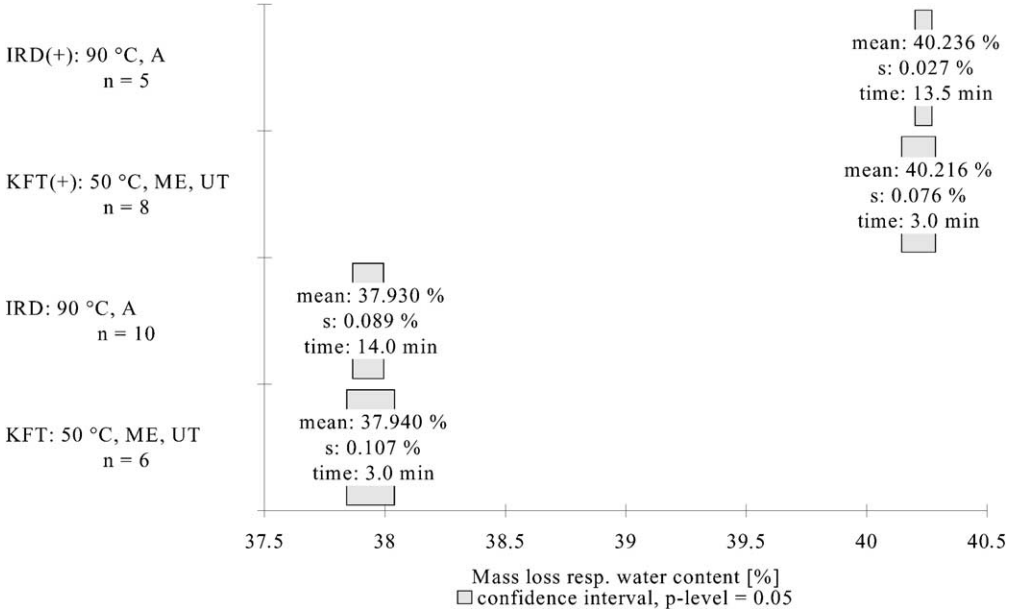

Fig. 11. Water determination in quince jelly with adapted drying conditions.

method is, however, adapted to a specific fruit jelly, the results become better (Fig. 11).

4. Conclusions

The quality of the Karl Fischer titration cannot be matched by infrared drying in terms of precision, reproducibility and duration of the analysis. To obtain exact results for products such as those investigated here, a certain practical experience with the Karl Fischer method and its variations is required. It is, however, necessary to calibrate the infrared dryer against such a reference method. Once the infrared dryer is calibrated, it is well suited for application in the food manufacturing process. Problems arose for sucrose and candies made with sucrose. For these products, no appropriate parameter sets for the infrared dryer could be found to match the mass loss with the water content analysed with Karl Fischer titration.

Advantages of the infrared drying technique are an easy handling, a reliable technology, no chemicals, short time of the analysis and the possibility of analysis of different samples without any change of the device.

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