

## COMPARATIVE ANALYSIS OF OVEN-DRYING AND KARL FISCHER TITRATION WITH DIFFERENT SOLVENTS FOR WATER DETERMINATION IN EVAPORATED MILK

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### Abstract

A crucial aspect of every food product's quality is its water content. The possibility of sample degradation or chemical transformation, as well as the possible loss of volatile chemicals during heating, make it extremely difficult to determine with accuracy. Karl Fischer (KF) titration is one of the most extensively used analytical procedures since it is based on a well-established chemical reaction and has specificity for water. However, in complicated matrices, particularly those that contain both sugars and proteins, even KF titration might produce unreliable findings. Foods often have complex compositions. In many cases they are composed of proteins, sugars, fats, water, and many other constituents. This study evaluates the suitability of two different methods for water content measurement in evaporated milk samples. Two different extraction solutions were used with KF method: solvent - (KF) and boiling methanol -KF (BM). Measuring with solvent as extraction solution was made on two temperatures: room temperature - (KF) and 50 °C - KF (50 °C). Classical heating oven (Oven) was used as reference. Every measurement by KF titration was made in ten replications. Measurements with heating oven were made in five replications. A total of 105 determinations were conducted. For statistical evaluation, descriptive statistics (maximum, minimum, average and standard deviation), correlations and Student's t-test were used. All methods showed high precision. Comparisons were tested and differences were evaluated at a given confidence level ( $p < 0.05$ ). There is statistically significant difference between KF (room temperature) and KF (BM) and KF (50°C) and KF (BM). KF and KF (50 °C) did not show statistically significant difference. All methods are in good agreement with heating oven. Classical KF at room temperature is recommended due to comparable precision and shorter analysis time.

**Keywords:** *Water content; Karl Fischer titration; evaporated milk; extraction solvent; analytical comparison*

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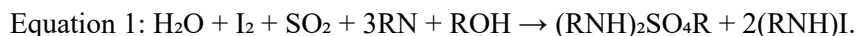
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## INTRODUCTION

Water constitutes anywhere from as little as 1% to as much as 80–90% of the total weight of food materials, highlighting its essential role in food systems (Joardder *et al.*, 2019). The unique physicochemical properties of the water molecule continue to captivate chemists, physicists, and food scientists alike. Accurately quantifying water content in various substances remains a significant analytical challenge. Despite appearing straightforward, this task is often complex and can yield inconsistent or conflicting results. This complexity arises from water's diverse interactions with a wide range of molecular species, leading to variable physicochemical behaviors depending on the specific chemical environment (Isengard, 2006).

The conventional approach to determining water content is based on the weight loss technique, which involves first weighing a sample and then heating it to a specific temperature in a drying oven to promote water evaporation. The sample will have lost water by evaporation after a predetermined amount of time, at which point it is weighed once more. Water loss is the cause of the weight difference if water is the only volatile component in the sample (De Caro *et al.*, 2001). Water and other evaporative fluids are not distinguished by drying techniques such as hoover drying, freeze drying, infrared drying, microwave drying or conventional oven drying. These methods provide the mass loss that the product undergoes under the given circumstances, not the water content (Felgner *et al.*, 2008). One chemical method for determining the water content of different materials is Karl Fischer titration (KFT). Only the water content of a sample may be detected using this technique; other components cannot. The following reaction equation (eq. 1) describes the chemical reaction that produces this selectivity and involves the water in the sample, an alcohol, imidazole as a base, sulfur dioxide, and iodine (Scholz, 1984).



KF is a practical technique for determining the amount of water in a variety of materials, including samples that are insoluble or less soluble. In complex food matrices such dairy and meat products, honey, sweets, chocolate, fruits and vegetables, spices, and cereal items, the water content can be readily and specifically ascertained (Hădărugă *et al.*, 2016; Hădărugă *et al.*, 2012). The extraction of water during KF titration in complex samples could be a limitation of this method. One complex dairy product is evaporated milk. Evaporated milk is produced by evaporating water from milk, without adding sugar or any preservatives. The canned version undergoes heat sterilization at temperatures between 118 and 122 °C for a few minutes (Hess, 2003). Evaporated milk has lower amount of water (around 75 %), in comparison to milk (around 88%) (Hariono *et al.*, 2024; Nouh *et al.*, 2017; Fox *et al.*, 2015).

Due to the problematic water determination in complex foods (difficult water extraction), the aims of this research arise:

- Comparing two different Karl Fischer solvents and temperatures for water determination.
- Suggestion of the right method for determination of water content in evaporated milk.

## MATERIALS AND METHODS

### Samples

Samples of evaporated milk for analysis were commercially available, from same producer. They were labeled as samples 1, 2 and 3. To test the method's performance for different sample matrices, all samples had different amounts of proteins, fats and sugars (Table 1.)

Table 1. Basic composition (%) evaporated milk samples stated on the product label

| Sample       | 1    | 2    | 3   |
|--------------|------|------|-----|
| Proteins (%) | 2.8  | 5.8  | 5.8 |
| Fats (%)     | 12.0 | 12.0 | 3.0 |
| Sugars (%)   | 4.0  | 7.6  | 8.1 |

### Methods

For determination of water content in samples of evaporated milk the volumetric Karl Fischer titration was used. The measurements were conducted by means of 905 Titrando (Metrohm) instrument. Prior to the measurements the samples were tempered by the room temperature. Samples were taken with syringe with needle. The mass of the syringe with needle and sample was determined before and after the introduction of sample into the titration cell. For extraction medium two different substances were used: commercially available solvent (KF) and methanol KF (BM). Karl Fischer solvent (Sigma Aldrich) is often used for extraction of water from samples; it is two component solvent based on methanol. The extraction procedure with solvent was conducted at two different temperatures: room temperature - (KF) and 50 °C - KF (50 °C). Pure methanol (Sigma Aldrich) is also used for water extraction. In this research the extraction of water was also conducted in boiling methanol. Titrating with Karl Fischer in boiling methanol is a practical method that increases the speed and accuracy of the analysis. This is because heating to the boiling point of methanol (65 °C) enhances the solubility and extraction of water from the sample. The stoichiometry of the Karl Fischer reaction remains unchanged (Isengard & Striffler, 1992). As reference method a classical heating oven (Oven) was used.

A single titration typically takes 1-2 minutes, making the KF titration a quick, accurate, and precise analysis method. Another advantage of this method is its broad measurement range, which essentially covers the entire water content range of 1% to 100%. This allows samples with varying water contents to be titrated volumetrically using a single instrument (De Caro *et al.*, 2001). The difference between coulometric and volumetric method is that in volumetric method, the amount of reagent consumed indicates the amount of water. It is normally used for higher amounts of water ranging from 0.1% to 100%. Prior to sample measurements the instrument was standardized with standard with known water content. All measurements were made in ten parallels. Instrument parameters are presented by Table 2.

Table 2. Instrument parameters

|                 |                                                 |
|-----------------|-------------------------------------------------|
| Stop criteria:  | Absolut drift: 20 $\mu\text{g}/\text{min}$      |
| Titrant         | Titrant 2 (Sigma Aldrich)                       |
| Working medium  | 20 mL solvent + 20 mL formamide (Sigma Aldrich) |
| Temperature:    | 50 $^{\circ}\text{C}$                           |
| Measuring cell: | Volumetric                                      |
| Sample mass:    | 0.1500-0.2500g                                  |

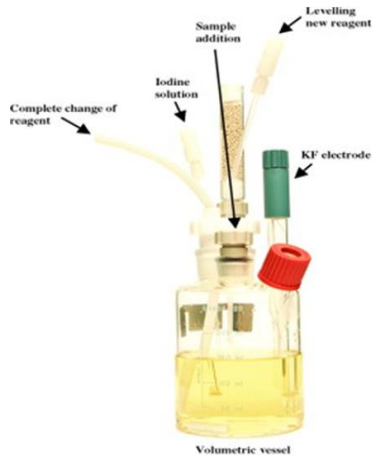


Figure 1. Volumetric reaction chamber setup (Felgner *et al.*, 2008), and Classical Karl Fischer titration device

As reference method the classical oven was used. The heating process was conducted on 105 °C for four hours. Sample mass from 2-4 g was weighted with precision of  $\pm 0.0001$  g, mixed with silicate sand and heated. After the heating the sample was weighed again. The heating and weighting process continued until the constant mass was reached.

### Statistical evaluation

Using metrics like the highest and minimum value, mean (average), and standard deviation (STDEV), descriptive statistics provide an overview of a data set's fundamental characteristics and shed light on its central tendency, variability, and distribution (Ibe, 2014). A statistical metric used to define the linear relationship between two variables is correlation. The correlations in this study are used to determine linearity between used methods. The correlation coefficient (r) ranges listed below show how strong the patterns and connections under investigation are: If the association is between 0.0 and 0.19, it is considered extremely weak; if it is between 0.20 and 0.39, it is considered weak; if it is between 0.40 and 0.59, it is considered moderate; if it is between 0.60 and 0.79, it is considered strong; and if it is between 0.80 and 1.0., it is considered very strong (Papageorgiou, 2022). T test: One popular technique for comparing the mean results of two methods is the student's t-test. The means must be near each other in value if the difference is not statistically significant (Rita & Ekholm, 2007). The standard threshold for significance in a t test is set at  $p = 0.05$ , as is the case with other significance tests. A t test result is deemed statistically significant if the p-value is less than 0.05. On the other hand, a result is considered insignificant if the p-value is greater than 0.05. (Krzywinski & Altman, 2013). The basic statistical analysis was performed on ten replications within all used methods. Correlations between methods were performed on all three different samples together.

## RESULTS AND DISCUSSION

### Sample 1

The results of water content measuring of sample 1, by several different techniques are shown within table 3 and figure 2.

Table 3. Results of water measurement of sample 1.

| Sample | Mass   | Water<br>(%) | KF                 |                        | Mass   | KF (BM)      |                        |
|--------|--------|--------------|--------------------|------------------------|--------|--------------|------------------------|
|        |        |              | Water<br>50 °C (%) | Extraction<br>Time (s) |        | Water<br>(%) | Extraction<br>time (s) |
| 1      | 0.0398 | 79.14        | 78.66              | 160                    | 0.0685 | 76.36        | 180                    |
| 2      | 0.0321 | 79.32        | 78.51              | 160                    | 0.0422 | 76.28        | 180                    |
| 3      | 0.0516 | 79.91        | 78.19              | 160                    | 0.07   | 76.19        | 180                    |
| 4      | 0.0564 | 79.91        | 79.25              | 160                    | 0.1011 | 77.41        | 180                    |
| 5      | 0.0348 | 79.86        | 78.10              | 160                    | 0.0601 | 75.36        | 180                    |

|       |        |       |       |     |        |       |     |
|-------|--------|-------|-------|-----|--------|-------|-----|
| 6     | 0.0308 | 78.54 | 78.56 | 160 | 0.0531 | 78.02 | 180 |
| 7     | 0.0322 | 78.64 | 78.81 | 160 | 0.0302 | 77.57 | 180 |
| 8     | 0.0666 | 78.88 | 81.57 | 160 | 0.0311 | 77.72 | 180 |
| 9     | 0.0573 | 81.42 | 78.58 | 160 | 0.1457 | 77.41 | 180 |
| 10    | 0.0348 | 79.92 | 78.16 | 160 | 0.0513 | 78.00 | 180 |
| Max.  | 0.0666 | 81.42 | 81.57 | 160 | 0.1457 | 78.02 | 180 |
| Min.  | 0.0308 | 78.54 | 78.10 | 160 | 0.0302 | 75.36 | 180 |
| Mean  | 0.0436 | 79.55 | 78.84 | 160 | 0.0653 | 77.03 | 180 |
| STDEV | 0.01   | 0.85  | 1.02  | 0   | 0.03   | 0.91  | 0   |

Oven mean: 79.60%

Oven STDEV: 0.08

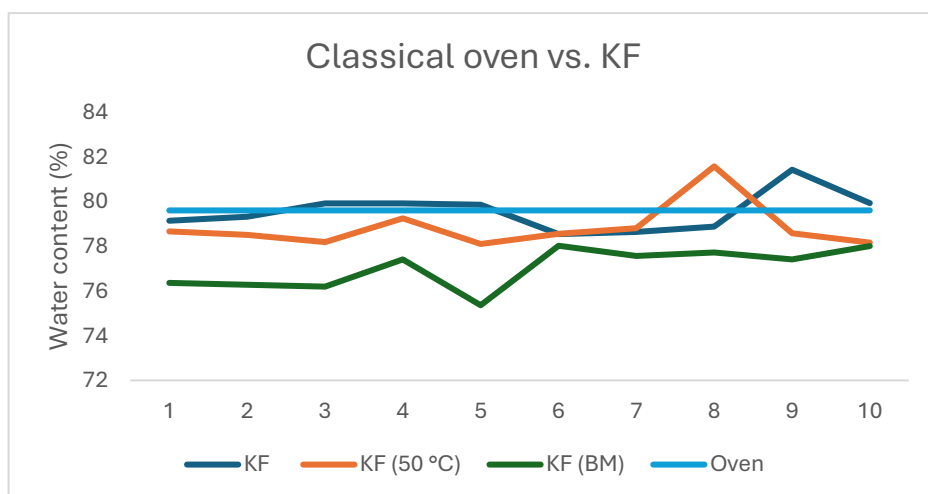


Figure 2. Comparison of reference method with different KF conditions (solvents and temperatures)

The highest mean water content in sample 1 was obtained using the classical Karl Fischer titration performed in solvent at room temperature, yielding a value of 79.55%. This indicates that under these conditions, the Karl Fischer method achieved the most efficient extraction and quantification of water. A comparable average value was obtained using the classical oven-drying method (79.60%), suggesting strong agreement between these two analytical techniques. Similar water content levels have been reported by Lewis (2023), supporting the reliability of these findings. The determination of moisture content in evaporated milk samples by drying techniques is also recommended by *National Condensed Milk Regulation* (2011).

Notably, sample 1 exhibited the lowest concentrations of proteins and sugars among all analyzed samples, which may contribute to its relatively higher apparent water content due to reduced matrix interactions and fewer hygroscopic constituents.

Furthermore, the solvent-based Karl Fischer titration at room temperature yielded the lowest standard deviation across ten replicates, demonstrating excellent precision and reproducibility of this analytical approach.

## Sample 2

Table 4. Results of water measurement of sample 2.

| Sample      | Mass   | Water (%) | KF              |                     | Mass     | KF (BM)   |                     |
|-------------|--------|-----------|-----------------|---------------------|----------|-----------|---------------------|
|             |        |           | Water 50 °C (%) | Extraction Time (s) |          | Water (%) | Extraction time (s) |
| 1           | 0.0359 | 71.68     | 70.68           | 150                 | 0.0469   | 71.87     | 160                 |
| 2           | 0.0509 | 72.38     | 71.18           | 150                 | 0.0369   | 72.12     | 160                 |
| 3           | 0.0354 | 72.01     | 70.97           | 150                 | 0.042    | 71.33     | 160                 |
| 4           | 0.0444 | 73.62     | 70.44           | 150                 | 0.0321   | 70.24     | 160                 |
| 5           | 0.0384 | 73.01     | 70.87           | 150                 | 0.0248   | 70.88     | 160                 |
| 6           | 0.0355 | 72.87     | 72.38           | 150                 | 0.0432   | 71.21     | 160                 |
| 7           | 0.0518 | 72.35     | 71.14           | 150                 | 0.0308   | 71.99     | 160                 |
| 8           | 0.0533 | 72.18     | 71.56           | 150                 | 0.0446   | 70.93     | 160                 |
| 9           | 0.0589 | 73.26     | 71.43           | 150                 | 0.0344   | 72.11     | 160                 |
| 10          | 0.0381 | 72.40     | 71.43           | 150                 | 0.0291   | 72.05     | 160                 |
| Max.        | 0.0589 | 73.62     | 72.38           | 150                 | 0.0469   | 72.12     | 160                 |
| Min.        | 0.0355 | 71.68     | 70.44           | 150                 | 0.0291   | 70.24     | 160                 |
| Mean        | 0.0442 | 72.58     | 71.21           | 150                 | 0.03648  | 71.47     | 160                 |
| STDEV       | 0.01   | 0.59      | 0.54            | 0                   | 0.007431 | 0.65      | 0                   |
| Oven mean:  |        |           |                 | 72.59%              |          |           |                     |
| Oven STDEV: |        |           |                 | 0.08                |          |           |                     |

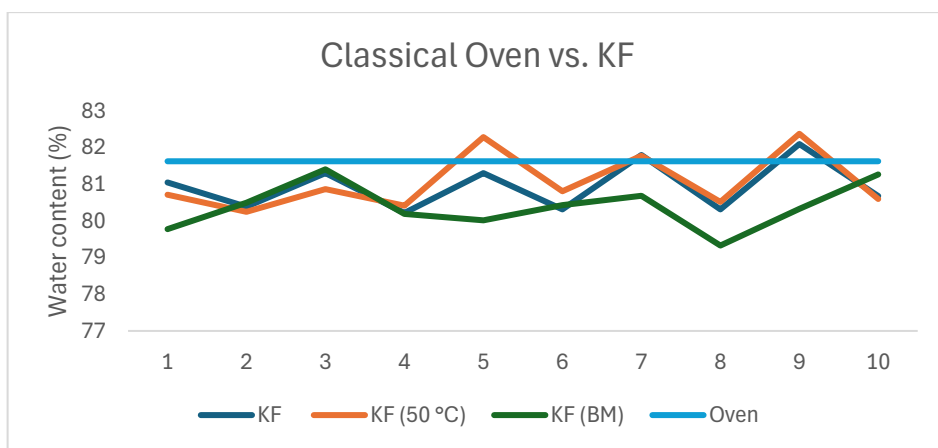


Figure 3. Comparison of reference method with different KF conditions (solvents and temperatures)

When the water content of “Sample 2” was quantified by the classical Karl Fischer titration in solvent at ambient temperature, the highest mean value obtained was 72.58 %. The near-equivalent mean result obtained by the conventional oven-drying method (72.59%) suggests that, under these conditions, both methods extracted essentially the same water fraction.

Importantly, Sample 2 also exhibited higher protein and sugar levels compared to Sample 1, implying that the matrix contained greater amounts of hydrophilic (“water-binding”) constituents. The fact that the KFT solvent-extraction approach yielded the highest mean—and indeed the lowest standard deviation over ten replicates - demonstrates both the efficacy of the extraction protocol in this matrix and the high precision of the method.

This observation is consistent with earlier work indicating that KFT can offer superior repeatability and extraction of “strongly bound” water in complex matrices relative to some gravimetric methods (Thiex & Richardson, 2003).

### Sample 3

Table 5. Results of water measurement of sample 3.

| Sample | Mass    | KF        |                 |                     | KF (BM) |           |                     |
|--------|---------|-----------|-----------------|---------------------|---------|-----------|---------------------|
|        |         | Water (%) | Water 50 °C (%) | Extraction Time (s) | Mass    | Water (%) | Extraction time (s) |
| 1      | 0.0576  | 81.05     | 80.39           | 150                 | 0.0492  | 79.78     | 160                 |
| 2      | 0.0353  | 81.31     | 80.22           | 150                 | 0.092   | 80.50     | 160                 |
| 3      | 0.0315  | 81.31     | 80.32           | 150                 | 0.0266  | 81.41     | 160                 |
| 4      | 0.0297  | 81.80     | 80.32           | 150                 | 0.0495  | 80.19     | 160                 |
| 5      | 0.0364  | 82.10     | 80.68           | 150                 | 0.0386  | 80.02     | 160                 |
| 6      | 0.0313  | 80.72     | 80.25           | 150                 | 0.0598  | 80.43     | 160                 |
| 7      | 0.0363  | 80.87     | 80.42           | 150                 | 0.0308  | 80.69     | 160                 |
| 8      | 0.0353  | 82.29     | 80.81           | 150                 | 0.0787  | 79.33     | 160                 |
| 9      | 0.037   | 81.78     | 80.52           | 150                 | 0.0652  | 80.33     | 160                 |
| 10     | 0.0428  | 82.38     | 80.60           | 150                 | 0.0213  | 81.27     | 160                 |
| Max.   | 0.0576  | 82.38     | 80.81           | 150                 | 0.0920  | 81.41     | 160                 |
| Min.   | 0.0297  | 80.72     | 80.22           | 150                 | 0.0213  | 79.33     | 160                 |
| Mean   | 0.03732 | 81.56     | 80.45           | 150                 | 0.05117 | 80.39     | 160                 |
| STDEV  | 0.01    | 0.59      | 0.19            | 0                   | 0.02    | 0.63      | 0                   |

Oven mean: 81.63%

Oven STDEV: 0.106

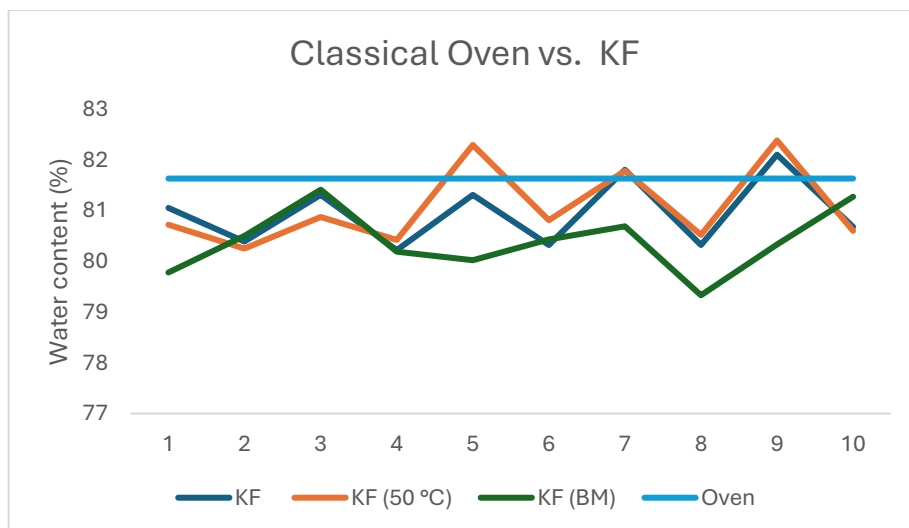


Figure 4. Comparison of reference method with different KF conditions (solvents and temperatures)

The highest mean water content was observed in Sample 3, as determined by the conventional Karl Fischer titration method using a solvent at ambient temperature, with a recorded value of 81.56%. This result indicates that the solvent-based Karl Fischer technique efficiently extracts and quantifies the total water content in evaporated milk. Comparable results were obtained using the conventional oven-drying method, which yielded an average value of 81.63%, confirming the consistency between both analytical approaches. The elevated water content in this sample was anticipated, as Sample 3 exhibited the lowest fat concentration among all analyzed samples. Since fat content inversely influences water retention in milk products, reduced lipid fractions typically correspond to higher relative water contents. Reports concerning water quantification in low-fat evaporated milk remain limited, as most published data pertain to regular evaporated milk formulations (Hariono *et al.*, 2024). Furthermore, the low standard deviation obtained across ten replicates for the solvent-based Karl Fischer method demonstrates its high analytical precision and repeatability for determining water content in dairy matrices.

### Correlations

The highest correlation between methods is presented by figure 5.

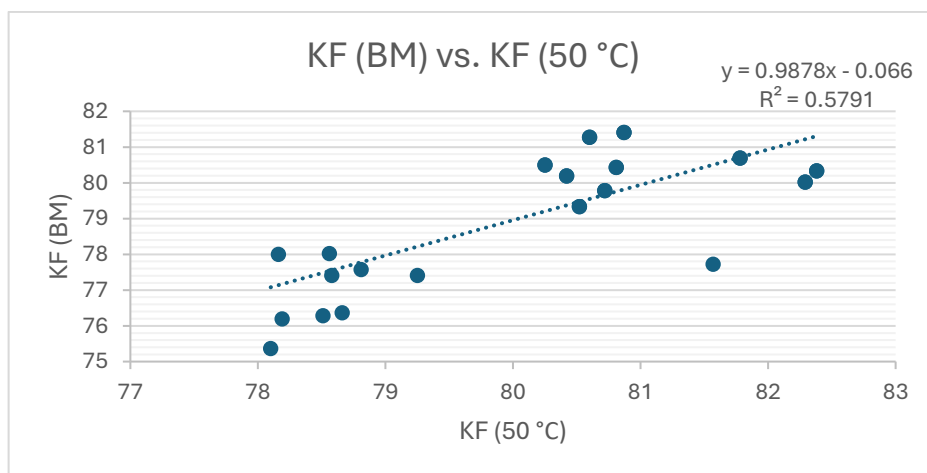


Figure 5. Correlation between results of KF(BM) and KF (50 °C)

All Karl Fischer (KF) titration methods exhibited moderate inter-method correlations, indicating a reasonable degree of agreement among the analytical techniques. The strongest correlation ( $r = 0.760$ ) was observed between the KF method performed at 50 °C and the method employing the BM solvent system. Such a correlation coefficient reflects that these two analytical protocols yield comparable trends in water quantification, suggesting consistency in their relative measurement behavior. Nevertheless, a strong correlation does not inherently confirm the accuracy or trueness of the methods. Instead, it indicates that when one method records a relatively higher or lower water content, the corresponding method tends to exhibit a proportional variation in the same direction, implying systematic alignment in measurement patterns rather than equivalence in absolute values.

### Students t-test analysis

The results of Students t-test analysis are shown by table 7.

Table 7. t test results

| <b>KF vs. KF (50 °)</b>      |            |           |            |                       |
|------------------------------|------------|-----------|------------|-----------------------|
|                              | KF         |           | KF (50 °C) |                       |
| N:                           | 30         | N:        | 30         |                       |
| Mean:                        | 80.485     | Mean:     | 80.322     |                       |
| Variance:                    | 0.95014    | Variance: | 1.8434     |                       |
| Difference between means:    | 0.16233    |           |            |                       |
| Uneq. var. t :               | 0.53197    | p:        | 0.59698    | t value (p=0.05): 2   |
| <b>KF vs. KF (BM)</b>        |            |           |            |                       |
|                              | KF         |           | KF (BM)    |                       |
| N:                           | 30         | N:        | 30         |                       |
| Mean:                        | 80.485     | Mean:     | 79.274     |                       |
| Variance:                    | 0.95014    | Variance: | 3.1059     |                       |
| Difference between means:    | 1.2107     |           |            |                       |
| Uneq. var. t :               | 3.2926     | p:        | 0.0019     | t value (p=0.05): 2   |
| <b>KF (50 °) vs. KF (BM)</b> |            |           |            |                       |
|                              | KF (50 °C) |           | KF (BM)    |                       |
| N:                           | 30         | N:        | 30         |                       |
| Mean:                        | 80.322     | Mean:     | 79.274     |                       |
| Variance:                    | 1.8434     | Variance: | 3.1059     |                       |
| Difference between means:    | 1.0483     |           |            |                       |
| Uneq. var. t:                | 2.581      | p:        | 0.0019     | t value (p=0.05): 2.0 |

The results of the independent *t*-tests comparing the different Karl Fischer (KF) titration methods revealed statistically significant differences between the standard KF method and KF (BM), as well as between KF (50 °C) and KF (BM) ( $p < 0.05$ ). Conversely, no statistically significant difference ( $p > 0.05$ ) was detected between the standard KF method and KF (50 °C), indicating that these two conditions produce comparable mean water content values. This outcome suggests that temperature elevation to 50 °C does not substantially influence the quantification efficiency or analytical response of the KF method under the tested conditions, whereas the use of the BM solvent system introduces a systematic deviation relative to the standard protocol.

## CONCLUSIONS

For the determination of water content in evaporated milk, the classical Karl Fischer (KF) titration employing a standard solvent system at ambient temperature is recommended, as it offers an optimal balance between analytical accuracy and operational efficiency. The KF titration conducted at 50 °C produced comparable results, with the *t*-test revealing no statistically significant difference between the two methods ( $p = 0.597$ ). All KF approaches exhibited moderate inter-method correlations, indicating a consistent linear relationship among the measurement trends across the techniques. Although the classical oven-drying method demonstrated the lowest standard deviation, suggesting superior measurement precision, it required substantially longer analytical durations (several hours) compared to the KF titration methods, which typically achieve complete analysis within a few minutes. Consequently, while the oven-drying method remains valuable for reference validation, the classical KF titration represents a more practical and time-efficient approach for routine determination of water content in evaporated milk.

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## USPOREDBA PEĆNICE I KLASIČNE KARL FISCHER TITRACIJE S RAZLIČITIM OTAPALIMA ZA ODREĐIVANJE VODE U UZORCIMA ISPARENOG MLJEKA

### Abstract

Ključni aspekt kvalitete svakog prehrambenog proizvoda je njegov sadržaj vode. Mogućnost degradacije uzorka ili kemijske transformacije, kao i mogući gubitak hlapljivih kemikalija tijekom zagrijavanja, izuzetno otežavaju točno određivanje. Karl Fischer (KF) titracija jedan je od najčešće korištenih analitičkih postupaka jer se temelji na dobro utvrđenoj kemijskoj reakciji i ima specifičnost za vodu. Međutim, u složenim matricama, posebno onima koje sadrže i šećere i proteine, čak i KF titracija može dati nepouzdanu nalaze. Hrana često ima složen sastav. U mnogim slučajevima sastoji se od proteina, šećera, masti, vode i mnogih drugih sastojaka. Ovo istraživanje bavi se mjerenjem sadržaja vode u složenoj hrani koja sadrži masti, proteine i šećere te mnoge druge spojeve. Za ovo istraživanje korištena su tri različita uzorka kondenziranog mlijeka. KF metodom korištene su dvije različite otopine za ekstrakciju: otapalo i kipući metanol (BM). Mjerenje s otapalom kao otopinom za ekstrakciju provedeno je na dvije temperature (sobna temperatura i 50 °C). Klasična peć za zagrijavanje korištena je kao referenca. Svako mjerenje KF titracijom provedeno je u deset ponavljanja. Mjerenja s pećnicom za zagrijavanje provedena su u pet ponavljanja. Na kraju je izmjereno 105 uzoraka. Za statističku evaluaciju korištena je deskriptivna statistika (maksimum, minimum, prosjek i standardna devijacija), korelacije i Studentov t-test. Sve metode pokazale su visoku preciznost. Postoji statistički značajna razlika između KF i KF (BM) te KF (50 °C) i KF (BM). KF i KF (50 °C) nisu pokazali statistički značajnu razliku. Sve metode se dobro slažu s pećnicom za zagrijavanje. Za mjerenje sadržaja vode u uzorcima kondenziranog mlijeka predlaže se klasična KF na sobnoj temperaturi.

**Ključne riječi:** *sadržaj vode, Karl Fischer, rastvarač, ključali metanol, temperatura*