

## Performance Evaluation of the Karl Fischer Method for Determining Water Content in Food Products

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### ABSTRACT

Water content is a crucial parameter in determining the quality of food products, as it influences physicochemical properties, microbial growth, and shelf life. Therefore, reliable methods for water content determination are essential. This study evaluated the performance of the Karl Fischer method through several parameters: linearity, limit of detection (LOD), limit of quantitation (LOQ), precision, accuracy, and measurement uncertainty. The method showed strong linearity with the regression equation  $y = 0.9719x + 0.5062$  and a correlation coefficient ( $r$ ) of 0.9998 across 1.0–10.0% water concentration. The LOD and LOQ were found to be 0.10% and 0.13%, respectively. Precision (%RSD) at 5, 60, and 100% water concentrations yielded values of 0.88, 0.26, and 0.09%. Accuracy (% trueness) ranged from 92% to 105%, while the mean relative uncertainty was 6%. These results demonstrate that the Karl Fischer method offers acceptable performance and is suitable for accurate water content determination in food products.

**Keywords:** Karl Fischer titration; water content; method validation; food analysis; measurement uncertainty.

### ABSTRAK

Kadar air merupakan parameter penting dalam menentukan mutu produk pangan, karena memengaruhi sifat fisikokimia, pertumbuhan mikroba, dan umur simpan. Oleh karena itu, metode yang andal untuk penentuan kadar air sangat diperlukan. Penelitian ini mengevaluasi kinerja metode Karl Fischer melalui beberapa parameter, yaitu linearitas, limit deteksi (LOD), limit kuantitasi (LOQ), presisi, akurasi, dan ketidakpastian pengukuran. Metode ini menunjukkan linearitas yang sangat baik dengan persamaan regresi  $y = 0.9719x + 0.5062$  dan koefisien korelasi ( $r$ ) sebesar 0.9998 pada rentang konsentrasi air 1.0–10.0%. Nilai LOD dan LOQ masing-masing diperoleh sebesar 0.10% dan 0.13%. Presisi (%RSD) pada konsentrasi air 5, 60, dan 100% menghasilkan nilai 0.88, 0.26, dan 0.09%. Akurasi (% ketepatan) berada pada rentang 92% hingga 105%, sedangkan rata-rata ketidakpastian relatif sebesar 6%. Hasil ini menunjukkan bahwa metode Karl Fischer memiliki kinerja yang dapat diterima dan sesuai digunakan untuk penentuan kadar air secara akurat pada produk pangan.

**Kata Kunci:** titrasi Karl Fischer; kadar air; validasi metode; analisis pangan; ketidakpastian pengukuran.

## INTRODUCTION

Water is a non-nutritive component that can indicate the quality of a food product (Fuquay, McSweeney, & Fox, 2011; Le Maguer, 2017). Water content in food products has an important role in determining their physicochemical properties, including viscosity, emulsion structure, and thermal properties, and contributes relatively to the processing and manufacturing requirements of food products (McSweeney, O'Mahony, & Kelly, 2022). In addition, water has a key role in controlling microbial growth, product's shelf life, palatability, and the overall quality of a food product (Barbosa-C, Fontana Jr, Schmidt, & Labuza, 2020; Nollet, 2004; Tapia, Alzamora, & Chirife, 2020). Therefore, it is very important to determine the water content using a valid method.

Determination of water content in a sample is usually conducted by drying using an oven. However, this method has a major drawback, namely it cannot distinguish the water content from other relative components (Bozkir, Tekgül, & Erten, 2021; Quirino, Lima, Palma, Franco, & Detmann, 2023). In the drying method, water content is calculated as the lost weight after drying. In food products with high lactose content, the drying method becomes unsuitable for use, because there is a clear difference between the lost weight and the water content in the product (Melnikova, Bogdanova, Paveleva, Saranov, & Pandiselvam, 2023; Quirino et al., 2023; Shivanna & Rao, 2025). One method of directly determining water content is the Karl Fischer (KF) instrument. This instrument is an alternative method used to determine the water content in liquid, solid, and gas samples (Chohan & Sykes, 2024; Shen et al., 2020; Surangsrirat et al., 2022). The KF instrument is widely used to determine the water content in food samples, such as bread (Popescu et al., 2020), honey (Khan et al., 2024), plant-derived (Vilbaste, Pawade, & Leito, 2025), and biomass and their derivatives (Jędrychowska & Kowalczyk, 2022).

Determination of water content using the KF method is based on the principle that iodine is reduced by sulfur dioxide in the presence of water. The end point of the titration is indicated by a brown color, a marker of an excess amount of iodine. The titrant used is KF reagent, which is a mixture of iodine, sulfur dioxide, and pyridine in methanol solution (Chohan & Sykes, 2024; Rivera-Quintero et al., 2024). Thus, the KF method is a more selective method for determining water content in food samples.

The performance test of the Karl Fischer method for determining the water content in a food product is very important to ensure the quality of the test results. The KF instrument is expected to provide precise and accurate measurement results in accordance with the instrument's specifications. In this study, the performance test of the KF method was carried out by testing several parameters: linearity, precision, accuracy, limit of instrument detection (LOD), limit of quantitation (LOQ), and estimation of measurement uncertainty. This water content determination method refers to ISO 4317 and the acceptance requirements of the KF method refer to Association of Official Analytical Chemists (AOAC) 2013.

## METHODOLOGY

### Materials

The materials used in this study were main material and chemicals. The main material used was aquabidest. The chemicals used were propylene glycol, anhydrous methanol, and Karl Fischer (KF) titrant (a mixture of iodine, sulfur dioxide, and pyridine in methanol solution).

## **Research Method**

The main tool used was Karl Fischer Metrohm 703 Ti-Stand instrument. This study was conducted by testing the performance parameters of the Karl Fischer method, namely linearity, precision, accuracy, limit of instrument detection (LOD), limit of quantitation (LOQ), and estimation of measurement uncertainty. Determination of water content using the Karl Fischer method refers to AOAC 2013. There were three stages of research, namely preparation, testing, and data processing. The preparation stage comprised the preparation of standard materials being tested. The testing stage comprised the testing of all performance parameters. The data processing stage was conducted by processing the data obtained from the test results relatively. The data obtained were then compared with the acceptance requirements referring to AOAC 2013.

### ***Preparation Phase***

Before measurement, aquabidest was used to determine the Karl Fischer factor. Aquabidest was weighed  $\pm 0.005$ – $0.07$  gr using an analytical balance and put into the Karl Fischer container containing methanol, then the "start" button was pressed. Determination of the Karl Fischer factor was conducted by seven replications. If it meets the precision criterion, namely the percentage of relative standard deviation (% RSD) of  $\leq 2\%$ , the mean results then were input into the Karl Fischer Metrohm 703 Ti-Stand instrument.

### ***Preparation of Aquabidest Standard Series Solution***

Aquabidest was weighed as much as 0.1, 0.2, 0.4, 0.6, 0.8, and 1 gr, then propylene glycol was added until the total volume reached 10 gr, then the mixture was homogenized. The concentrations of aquabidest made were 1, 2, 4, 6, 8, and 10% (w/b). After that, the water content of each standard series was measured using the Karl Fischer Metrohm 703 Ti-Stand instrument and transformed into a relationship curve of theoretical water content vs measured water content.

### ***Preparation of Precision and Accuracy Testing Solutions***

Precision testing solutions were made by making aquabidest with concentrations of 5, 60, and 100% in propylene glycol. Aquabidest was weighed as much as 0.5, 6.0, and 10.0 gr, then propylene glycol was added until the total weight reached 10 gr, then the mixture was homogenized.

### ***Preparation of LOD and LOQ Testing Solutions***

LOD and LOQ values were determined by measuring a blank solution (propylene glycol) using the Karl Fischer Metrohm 703 Ti-Stand instrument. The blank solutions were made through seven replications, then the mean and standard deviation were calculated. The measurement results were statistically processed to determine the theoretical LOD and LOQ values.

### ***Preparation of LOD and LOQ Confirmation Testing Solutions***

Confirmation of LOD and LOQ values was conducted by making a standard solution using the concentration of the theoretical LOD and LOQ values, then the water content was measured using the Karl Fischer Metrohm 703 Ti-Stand instrument. This test was conducted on seven blank solutions, then the data obtained were processed relatively.

### Precision

Next, the mean value, standard deviation (SD), and relative standard deviation (RSD) of the measurement results for three replications of each aquabidest concentration (5, 60, and 100%) were calculated using the following formula:

$$\text{Mean } (\bar{x}) = \frac{\sum_{i=1}^n x_i}{n} \quad (1)$$

$$\text{SD} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad (2)$$

$$\% \text{ RSD} = \frac{\text{SD}}{\bar{x}} \times 100\% \quad (3)$$

Description:

$x^i$  =  $i$ -th theoretical content (%)

$\bar{x}$  = mean of theoretical content (%)

SD = standard deviation

RSD = relative standard deviation (%)

$n$  = amount of replication

$i$  = 1, 2, 3, ...,  $n$

### Accuracy

The comparison value of the measured content and the theoretical content was obtained by measuring the concentrations of aquabidest (5, 60, and 100%), each with three replications. The theoretical water content was calculated using the following formula:

### Limit of Instrument Detection (LOD) and Limit of Quantitation (LOQ)

The LOD and LOQ data were repeated through seven replications, then the mean and standard deviation were calculated using the following formula:

$$\text{Theoretical water content } (\% \text{ (w/w)}) = \frac{(C_{\text{H}_2\text{O}}(\%) \times M_{\text{H}_2\text{O}}(\text{g})) + (C_{\text{propylene glycol}} \times M_{\text{propylene glycol}}(\text{g}))}{(M_{\text{H}_2\text{O}}(\text{g}) + M_{\text{propylene glycol}}(\text{g}))} \quad (4)$$

$$\% \text{ Recovery} = \frac{\text{Measured water content}}{\text{Theoretical water content}} \times 100\% \quad (5)$$

Description:

$C$  = concentration (% (w/w))

$M$  = mass (g)

$x^i$  =  $i$ -th content

$\bar{x}$  = mean of content (%)

SD = standard deviation

RSD = relative standard deviation (%)

$n$  = amount of replication

$i$  = 1, 2, 3, ...,  $n$

Theoretical LOD and LOQ values were calculated using the following formula:

$$\text{Theoretical LOD value } (\%) = \bar{x} + 3 \text{ SD} \quad (6)$$

$$\text{Theoretical LOQ value } (\%) = \bar{x} + 10 \text{ SD} \quad (7)$$

Description:

$\bar{x}$  = blank mean (%)

SD = standard deviation

## RESULTS AND DISCUSSION

The results of the Karl Fischer method performance test on the parameters of linearity, precision, accuracy, LOD, LOQ, and estimation of measurement uncertainty are discussed and compared with the acceptance requirements referring to AOAC 2013. Linearity test was performed by measuring aquabidest standard solutions at the concentrations of 1, 2, 4, 6, 8, and 10% in propylene glycol using the Karl Fischer Metrohm 703 Ti-Stand instrument. The linearity test results and the regression equation obtained are presented in Fig. 1.

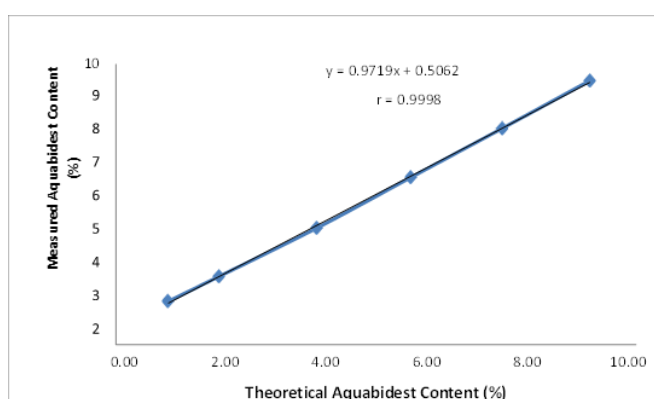


Figure 1. Relationship between the theoretical and the measured aquabidest content.

The regression equation above has a correlation coefficient ( $r$ ) of 0.9998. This correlation coefficient value indicates that the concentration of analyte in the range of 1.0–10% is able to give proportional results to the Karl Fischer method. The analyte level and instrument response has formed a straight line proportionally. This means that the correlation coefficient value generated from this study has met the requirements set by the provision of  $r > 0.99$  based on the referred acceptance requirements. The proportionality of the curve was determined by calculating the angle value formed on the curve using the formula of  $\tan \alpha = \text{slope: } (y/x)$ . In this study, the slope value obtained is 0.9719, and the  $\alpha$  value obtained is  $44.18^\circ$ . This  $\alpha$  value is close to the ideal value, namely  $45^\circ$ . Thus, it can be concluded that the slope value in the water content determination curve using the Karl Fischer method is acceptable. The slope value of the curve shows the sensitivity value of the measurement. Thus, it can be concluded that the measurement of water content using the Karl Fischer method demonstrates a good sensitivity.

LOD is a parameter that indicates the lowest concentration of analyte that still can be detected using the test method performed, but the detection result does not need to be quantitated (Akay Sazaklioglu et al., 2022; Kim et al., 2024; Snow, 2021). In contrast, LOQ is a parameter that indicates the lowest concentration of analyte in a sample that can be determined using an acceptable level of precision and accuracy in a defined experimental method (Jia, Sun, & Zhang, 2021; Kaviani, 2025; Lambarki et al., 2025). LOD and LOQ measurements were conducted by measuring propylene glycol solvent through seven replications using the Karl Fischer Metrohm 703 Ti-Stand instrument. The results of the LOD and LOQ tests are presented in Table 2.

Table 2 Test results of theoretical LOD and theoretical LOQ

Replication	Water Content (%)
1	0.08
2	0.09
3	0.08
4	0.08
5	0.08
6	0.08
7	0.09
<b>Mean</b>	0.08
<b>Standard Deviation</b>	0.0049
<b>Theoretical LOD</b>	0.10
<b>Theoretical LOQ</b>	0.13

Based on the results in Table 2, the LOD and LOQ values obtained are 0.10% and 0.13%, respectively. These are theoretical values. The theoretical LOD value was determined from the blank measurement, which is the mean concentration of the blank solution of seven replications plus the standard deviation of three replications. The theoretical LOQ value was determined from the mean concentration of the blank solution plus the standard deviation of ten replications. The theoretical LOD value of 0.10% means that the lowest concentration of analyte that still can be detected by the instrument is 0.10%. The theoretical LOQ value of 0.13% means that this is the lowest concentration of analyte that can be determined quantitatively using an acceptable level of precision and accuracy. Sample with concentrations below 0.13% will reveal a very high lack of confidence. For this reason, it is recommended that the sample concentration being measured is above 0.13%. In addition, a confirmation is required to ensure the correctness of the theoretical LOD and LOQ values obtained from the calculation. This confirmation was conducted by making aquabidest standard solution with concentrations that match the theoretical LOD and LOQ values, namely 0.10% and 0.13%, each with seven replications. The confirmation test results of the theoretical LOD values are presented in Table 3.

Table 3 Confirmation test results of theoretical LOD value of 0.10%

Replication	Water Content (%)
1	0.09
2	0.10
3	0.10
4	0.11
5	0.10
6	0.11
7	0.09
<b>Mean</b>	0.10
<b>SD</b>	0.0072
<b>Acceptability Requirements</b>	positive response

Based on the results in Table 3, the theoretical LOD value after confirmation shows a positive response, which means that the analyte can still be detected at a concentration of 0.10%. Therefore, it can be stated that in the use of the Karl Fischer Metrohm 703 Ti-Stand instrument on water samples, if the water content value obtained is smaller than the LOD value, then this

cannot be trusted as a response from the analyte, as the value obtained is only considered a noise. Next, the confirmation test results of the theoretical LOQ value are presented in Table 4.

Table 4 Confirmation test results of theoretical LOQ value of 0.13%

Replication	Water Content (%)
1	0.13
2	0.13
3	0.14
4	0.13
5	0.13
6	0.14
7	0.13
<b>Mean</b>	0.13
<b>SD</b>	0.0061
<b>% RSD <math>\leq</math> 10%</b>	4.56
<b>% Recovery</b>	100.00–106.94%
<b>Acceptability Condition</b>	90–108%
<b>%Recovery</b>	

The confirmation test results of theoretical LOQ value in Table 4 show that the precision test resulted in a % RSD of 4.56% and the accuracy test resulted in a % recovery range of 100.00–106.94%. Thus, it can be concluded that the data obtained meets the referred acceptance requirements. The smaller the LOD and LOQ values of an analyte indicate the more sensitive the capability of the instrument used to determine the concentration of the analyte.

Precision is defined as how close the measurement results are among replications of measurement samples taken at about the same time and under relatively the same conditions (Crucho, Margaritis, Tanghe, Vansteenkiste, & Vanelstraete, 2024; Razumić, Runje, Alar, Štrbac, & Trzun, 2025; Takeshita & Suzuki, 2025). Precision shows how exact a measurement in producing the intended result through replications. The precision test in this study was carried out by testing the aquabidest sample at the concentrations of 5, 60, and 100% in propylene glycol. Each concentration was measured three times. The precision test results are presented in Table 5.

Table 5. Precision test results of water content determination using the Karl Fischer method.

Theoretical Water Content (%)	Measured Water Content (%) n = 3	% RSD	Acceptability Requirements (AOAC 2013)
5%	5.10 ± 0.04	0.88	% RSD $\leq$ 2%
60%	59.00 ± 0.15	0.26	% RSD $\leq$ 1.5%
100%	98.32 ± 0.09	0.09	% RSD $\leq$ 1%

The Karl Fischer instrument used in this study can measure water content until a maximum limit of 100%. Precision test was conducted at low, medium, and high concentration levels, namely at the water content below 10%, close to 50%, and the maximum level that can be measured by the instrument, namely 100%. The precision test results show that the % RSD values for the aquabidest standard at the concentrations of 5, 60, and 100% are 0.88, 0.26, and

0.09, respectively. The precision test results for these three concentration levels are declared to meet the acceptance requirements referring to AOAC 2013. Based on these results, it is revealed that the smallest % RSD value was obtained at aquabidest concentration of 100%. The smaller the % RSD value indicates the more precise the data obtained. This is because the aquabidest sample concentration of 100% only consists of pure aquabidest without any other matrix that could interfere with the measurement. Meanwhile, the aquabidest sample concentration of 5% produced the highest % RSD value. This could be due to the fact that the sample contains propylene glycol matrix with higher concentration. In addition, in analytical chemistry, the smaller the concentration of an analyte in the sample, the higher the random error generated.

The accuracy test aims to determine the closeness between the test results and the actual value, in other words, to show the accuracy of a method (Biyao, Lingtian, & Ping, 2024; Wang, Wang, Wei, Yu, & Shen, 2025; Yang et al., 2021). The accuracy test was conducted by measuring the water content at the concentrations of 5, 60, and 100% in 10 gr of propylene glycol. The accuracy test results were evaluated by calculating the trueness value against the theoretical content value. The accuracy test results are presented in Table 6.

Table 6. Accuracy test results of water content determination using the Karl Fischer method

<b>Theoretical Water Content (%)</b>	<b>Measured Water Content (%) n = 3</b>	<b>% Trueness</b>	<b>Acceptability Requirements %Trueness (AOAC 2013)</b>
5	5.10 ± 0.04	(99.53 - 101.26) %	(92-105) %
60	59.00 ± 0.15	(98.25 - 98.42) %	(95-102) %
100	98.32 ± 0.09	(98.25 - 98.42) %	(98-101) %

Based on the results in Table 6, the range of % trueness values at the water content of 5% is 99.53–101.26%, at the water content of 60% is 98.25–98.42%, and at the water content of 100% is 98.25–98.42%. This accuracy test results have met the acceptance requirements referring to AOAC 2013. The accuracy test of the Karl Fischer Metrohm 703 Ti-Stand instrument was carried out by measuring the water content of aquabidest at the concentrations of 5, 60, and 100%, which were dissolved in propylene glycol. Propylene glycol functions as a humectant that retains water content (Sharma, Pandey, Tripathi, Upmanyu, & Singh, 2023; Talebi & Armstrong, 2020; Wiegand, 2023). Propylene glycol was used as a solvent in the determination of water content using the Karl Fischer method because this chemical contains water as much as 0.02%. The very small water content in propylene glycol is still acceptable so that it can be used to assess the accuracy of the Karl Fischer Metrohm 703 Ti-Stand instrument.

Determination of the titrant factor is very useful in accuracy test because this test faced difficulties in the practice. The main difficulty is that the actual content of the analyte being tested is unknown. The titrant factor is a correction factor so that from it, the actual analyte content can be known. Accuracy test results are affected by systematic errors, which are analytical results that deviate permanently from the proper value due to systematic errors. Systematic errors occur due to definite causes, such as improperly calibrated instruments and side reactions in titration.

Measurement uncertainty is a parameter that measures the existence of a range of correct values. Measurement uncertainty is determined by combining all possible errors of a complete

method, namely by measuring the error values generated by the method, either systematically or randomly. However, gross error is not included in this uncertainty calculation. In this study, the estimation of measurement uncertainty was carried out using the bottom up method, which calculates all sources of uncertainty that can contribute in causing errors, from preparation to measurement (Beasley-Green & Heckert, 2023; Klu, Puch-Solis, Mudie, Marland, & Nic Daeid, 2025; Ramsey & Rostron, 2024). The estimation of a measurement uncertainty is calculated through several steps as follows: modeling the test system, creating a formula or calculation formula, identifying the sources of uncertainty in the form of a fishbone diagram, grouping these factors in type A and type B uncertainty components, calculating the estimation of each standard uncertainty as a value equivalent to the standard deviation (SD), combining the standard uncertainty components ( $\mu$ ) to produce the uncertainty of the overall test results into a combined uncertainty, calculating the expanded uncertainty (U), and presenting the expanded uncertainty value in the test result report (Gangsarwijaya, Anindhita, & Widaningrum, 2020; Saini, Yadav, Singh, & Ezhilselvi, 2025). In determining the water content using the Karl Fischer method, the sources of measurement uncertainty are presented in the form of a fishbone diagram, as shown in Fig. 2.

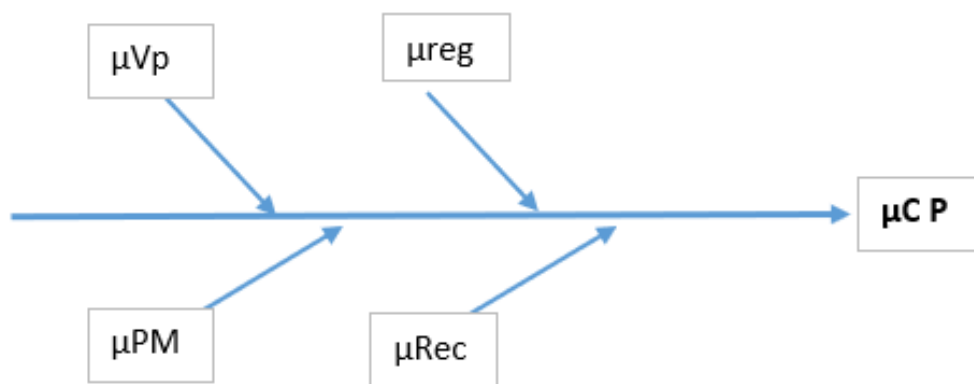


Figure 2. Depiction of the sources of measurement uncertainty regarding water content determination using the Karl Fischer method.

The fishbone diagram above shows that the source of uncertainty in the determination of water content using the Karl Fischer method consists of type A and type B uncertainty. The standard uncertainty sourced from the sample pipetting volume is classified as the component of type B uncertainty. Meanwhile, the standard uncertainty sourced from regression, method precision, and method accuracy is classified as the component of type A uncertainty. Each source of standard uncertainty was calculated, then integrated to form a combined uncertainty. The calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 5.10% are presented in Table 7.

As shown in Table 7, at the water content of 5.10%, the expanded combined uncertainty value is 0.31% and the relative uncertainty value is 6.03%. The relative uncertainty value obtained still meets the referred acceptance requirements. Next, the calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 59.00% are displayed in Table 8. As shown in Table 8, that at the water content of 59.00%, the expanded combined uncertainty value is 3.42% and the relative uncertainty value is 5.80%. The relative uncertainty value obtained still meets the referred acceptance requirements. Next,

the calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 98.32% are displayed in Table 8.

Table 7. Calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 5.10%.

Symbol (x)	Value (x)	$\mu(x)$	$\mu(x)/x$	$(\mu(x)/x)^2$
PM (Method Precision)	5.10 %	0.045	0.0088	7.795E-05
Rec (Method Accuracy)	100.41 %	0.86814	0.0086	7.476E-05
Rs (Resolution)	0.01 mL	0.00029	0.0289	0.0008333
Cx (Water Sample Content)	5.10 %			
$\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2$				0.000908
$\mu Cx / Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2}$				0.0301
$\mu Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2} \times Cx$				0.1537
<b>U = 2 x <math>\mu Cx</math></b>				<b>0.31</b>
<b>% Relative Uncertainty</b>				<b>6.03</b>

Table 8 Calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 59.00%.

Symbol (x)	Value (x)	$\mu(x)$	$\mu(x)/x$	$(\mu(x)/x)^2$
PM (Method Precision)	59.00%	0.15172	0.0026	6.614E-06
Rec (Method Accuracy)	98.33%	0.25288	0.0026	6.614E-06
Rs (Resolution)	0.01mL	0.00029	0.0289	0.0008333
Cx (Water Sample)	59.00%			
$\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2$				0.000840
$\mu Cx / Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2}$				0.0290
$\mu Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2} \times Cx$				1.7098
<b>U = 2 x <math>\mu Cx</math></b>				<b>3.42</b>
<b>% Relative Uncertainty</b>				<b>5.80</b>

As shown in Table 9, at the water content of 98.32%, the expanded combined uncertainty value is 5.78% and the relative uncertainty value is 5.80%. The relative uncertainty value obtained still meets the referred acceptance requirements. Based on the results in Tables 7, 8, and 9, the mean value obtained of the relative uncertainty is 6%. Overall, all of these results indicate that the Karl Fisher instrument used in this study has demonstrated a good method performance. The values of random error and systematic error found during the measurement are still within the acceptability limits.

Table 9. Calculation results of the combined uncertainty of water content measurement using the Karl Fischer method at the water content of 98.32%.

Symbol (x)	Value (x)	$\mu(x)$	$\mu(x)/x$	$(\mu(x)/x)^2$
PM (Method Precision)	98.32%	0.08704	0.0009	7.837E-07
Rec (Method Accuracy)	98.32%	0.08888	0.0009	8.172E-07
Rs (Resolution)	0.01mL	0.00029	0.0289	0.0008333
Cx (Water Sample)	98.32%			
$\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2$				0.000834
$\mu Cx / Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2 + (\mu\text{Rs}/\text{Rs})^2}$				
$\mu Cx = \sqrt{\Sigma (\mu\text{PM}/\text{PM})^2 + (\mu\text{Rec}/\text{Rec})^2} \times Cx$				2.8397
<b>U = 2 x <math>\mu Cx</math></b>				<b>5.68</b>
<b>% Relative Uncertainty</b>				<b>5.78</b>

## CONCLUSIONS

The Karl Fischer method performance test for water content determination has revealed that the test results of linearity, precision, accuracy, limit of instrument detection, limit of quantitation, and estimation of measurement uncertainty have met the specified acceptance requirements. The Karl Fisher instrument used in this study has shown a good method performance.

## REFERENCES

- Akay Sazaklioglu, S., Torul, H., Kabadayi, H., Vatansever, H. S., Tamer, U., & Celikkan, H. (2022). Calibration curve approaches for nonlinear data points obtained in Colo 320 exosomes determination. *Analytical and Bioanalytical Electrochemistry*, 14(11), 1027-1043.
- Barbosa-C, G. V., Fontana Jr, A. J., Schmidt, S. J., & Labuza, T. P. (2020). *Water activity in foods: fundamentals and applications*: John Wiley & Sons.
- Beasley-Green, A., & Heckert, N. A. (2023). Estimation of measurement uncertainty for the quantification of protein by ID-LC-MS/MS. *Analytical and Bioanalytical Chemistry*, 415(16), 3265-3274. doi:10.1007/s00216-023-04705-8
- Biyao, S., Lingtian, T., & Ping, H. (2024). Introduction and reflection on the revision of ISO/IEC 17043. *Accreditation and Quality Assurance*, 29(1), 77-83. doi:10.1007/s00769-023-01564-9
- Bozkir, H., Tekgöl, Y., & Erten, E. S. (2021). Effects of tray drying, vacuum infrared drying, and vacuum microwave drying techniques on quality characteristics and aroma profile of orange peels. *Journal of Food Process Engineering*, 44(1). doi:10.1111/jfpe.13611
- Chohan, B. S., & Sykes, D. G. (2024). *Methods and Instruments | Karl-Fischer-Titration*. In *Encyclopedia of Electrochemical Power Sources: Volume 1-7, Second Edition (Vol. 2, pp. V2:119-V112:134)*.
- Crucho, J., Margaritis, A., Tanghe, T., Vansteenkiste, S., & Vanelstraete, A. (2024). Repeatability and Reproducibility of Analysis Methods for Asphalt Mixture Gyratory Compaction. Paper presented at the Lecture Notes in Civil Engineering.

- Fuquay, J. W., McSweeney, P. L., & Fox, P. F. (2011). *Encyclopedia of dairy sciences*: Academic Press.
- Gangsarwijaya, N., Anindhita, R., & Widaningrum, D. L. (2020). Decision tree analysis approach to determine factors that affect the quote order lead time fulfillment. Paper presented at the IOP Conference Series: Earth and Environmental Science.
- Jędrychowska, S., & Kowalczyk, A. (2022). Determination of moisture in biomass by coulometric Karl Fischer titration. *Nafta - Gaz*, 2022(5), 393-400. doi:10.18668/NG.2022.05.07
- Jia, Y., Sun, X., & Zhang, F. (2021). Determination of LOD and LOQ with relative standard deviation and relative range or ratio of maximum and minimum. *Yejin Fenxi/Metallurgical Analysis*, 41(1), 1-12. doi:10.13228/j.boyuan.issn1000-7571.011206
- Kaviani, R. (2024). Comparison of Different Approaches for Calculating LOD and LOQ in an HPLC-Based Analysis Method. *Pharmaceutical Sciences*, 31(1), 106-109. doi:10.34172/PS.024.40452
- Kaviani, R. (2025). Comparison of Different Approaches for Calculating LOD and LOQ in an HPLC-Based Analysis Method. *Pharmaceutical Sciences*, 31(1), 106-109. doi:10.34172/PS.2024.40452
- Khan, K., Malik, K., Ahmad, M., Raja, M. N. I., Nazish, M., Kamal, A., . . . Naseem, M. T. (2024). Exploring the nutritional composition and quality parameters of natural honey from diverse melliferous flora. *Scientific Reports*, 14(1). doi:10.1038/s41598-024-79672-w
- Kim, H. R., Kim, S., Jung, J., Lee, H., Ho, K., Kim, B., & Oh, S. (2024). Enhancing LOD determination in gas chromatography: Validating the Hubaux-Vos method for gas concentration measurement. *Journal of Chromatography A*, 1720. doi:10.1016/j.chroma.2024.464764
- Klu, J. K., Puch-Solis, R., Mudie, R., Marland, V., & Nic Daeid, N. (2025). Open-access Measurement Uncertainty Calculator MUCalc and its application to the quantification of Etizolam in tablets using High-Performance Liquid Chromatography (HPLC). *Science and Justice*, 65(1), 70-81. doi:10.1016/j.scijus.2024.11.001
- Lambarki, L. Z., Jhilal, F., Slimani, L., El Hajji, R., Bakkali, F., Iskandar, S., . . . Saffaj, T. (2025). Comparison of approaches for assessing detection and quantitation limits in bioanalytical methods using HPLC for sotalol in plasma. *Scientific Reports*, 15(1). doi:10.1038/s41598-024-83474-5
- Le Maguer, M. (2017). Mechanics and influence of water binding on water activity. In *Water Activity* (pp. 1-25): Routledge.
- McSweeney, P. L., O'Mahony, J. A., & Kelly, A. L. (2022). *Advanced Dairy Chemistry: Volume 3: Lactose, Water, Salts and Minor Constituents*: Springer Nature.
- Melnikova, E., Bogdanova, E., Paveleva, D., Saranov, I., & Pandiselvam, R. (2023). Sucrose, Lactose, Thermogravimetry, and Differential Thermal Analysis: The Estimation of the Moisture Bond Types in Lactose-Containing Ingredients for Confectionery Products with Reduced Glycemic Index. *International Journal of Food Science*, 2023. doi:10.1155/2023/8835418
- Nollet, L. M. (2004). *Handbook of food analysis: methods and instruments in applied food analysis* (Vol. 138): CRC Press.
- Ponomarenko, E. A., Ivanov, Y. D., Valueva, A. A., Pleshakova, T. O., Zgoda, V. G., Vavilov, N. E., . . . Archakov, A. I. (2024). From Proteomics to the Analysis of Single Protein Molecules. *International Journal of Molecular Sciences*, 25(19). doi:10.3390/ijms251910308

- Popescu, G., Radulov, I., Iordănescu, O. A., Orboi, M. D., Rădulescu, L., Drugă, M., . . . Riviş, M. (2020). Karl fischer water titration-principal component analysis approach on bread products. *Applied Sciences (Switzerland)*, 10(18). doi:10.3390/APP10186518
- Quirino, D. F., Lima, N. S. A., Palma, M. N. N., Franco, M. O., & Detmann, E. (2023). Evaluation of Heating Times for Loss on Drying at 105°C for Estimation of Laboratory Dry Matter in Animal Feeds. *Journal of AOAC International*, 106(2), 261-266. doi:10.1093/jaoacint/qsad004
- Ramsey, M. H., & Rostron, P. D. (2024). Measurement uncertainty from sampling and its role in validation of measurement procedures. *Accreditation and Quality Assurance*, 29(2), 153-162. doi:10.1007/s00769-024-01575-0
- Razumić, A., Runje, B., Alar, V., Štrbac, B., & Trzun, Z. (2025). A Review of Methods for Assessing the Quality of Measurement Systems and Results. *Applied Sciences (Switzerland)*, 15(17). doi:10.3390/app15179393
- Rivera-Quintero, P., Patience, G. S., Patience, N. A., Boffito, D. C., Banquy, X., & Schieppati, D. (2024). Experimental methods in chemical engineering: Karl Fischer titration. *Canadian Journal of Chemical Engineering*, 102(9), 2980-2997. doi:10.1002/cjce.25295
- Saini, N., Yadav, M., Singh, N., & Ezhilselvi, V. (2025). Estimation of measurement uncertainty in the quantitative analysis of toxic elements in wheat flour using ICP-OES. *Journal of Food Composition and Analysis*, 148. doi:10.1016/j.jfca.2025.108223
- Sharma, T., Pandey, K. K., Tripathi, M., Upmanyu, A., & Singh, R. C. (2023). Ultrasonic attenuation study of liquid mixtures of Propylene Glycol and DD water at various temperatures. Paper presented at the Materials Today: Proceedings.
- Shen, Z. C., Zheng, Y. B., Gao, M. P., Nie, L., Li, G. A., & Du, Z. X. (2020). Determination of moisture content in consumer products by Karl Fischer titration and its interfering factors. *China Surfactant Detergent and Cosmetics*, 50(4), 282-286. doi:10.3969/j.issn.1001-1803.2020.04.012
- Shivanna, S. K., & Rao, P. S. (2025). Challenges and opportunities in processing of lactose hydrolyzed dairy products. In *Lactose Hydrolysis in Dairy Products* (pp. 173-188).
- Snow, N. H. (2021). Going low: Understanding limit of detection in gas chromatography (gc). *LC-GC North America*, 39(5), 232-235. Retrieved from <https://www.scopus.com/inward/record.uri?eid=2-s2.0-85112776007&partnerID=40&md5=c4c9bba992d702cb6ad7572d3cbf56ee>
- Surangsrirat, D., Sridhar, V., Srikun, O., Puanglamjeak, M., Birdi, P., Dumnin, S., . . . Chana, K. S. (2022). Non-destructive measurement technique for water content in organic solvents based on a thermal approach. *RSC Advances*, 12(10), 6181-6185. doi:10.1039/d2ra00352j
- Takeshita, J. I., & Suzuki, T. (2025). A model and method for analyzing the precision of binary measurement methods based on beta-binomial distributions, and related statistical tests. *Quality and Quantity*, 59(2), 1323-1352. doi:10.1007/s11135-024-01998-4
- Talebi, M., & Armstrong, D. W. (2020). Water determination. In *Specification of Drug Substances and Products: Development and Validation of Analytical Methods*, Second Edition (pp. 459-477).
- Tapia, M. S., Alzamora, S. M., & Chirife, J. (2020). Effects of water activity (aw) on microbial stability as a hurdle in food preservation. *Water activity in foods: Fundamentals and applications*, 323-355.
- Vilbaste, M., Pawade, S. S., & Leito, I. (2025). Optimisation of temperature for accurate water content determination in plant-derived materials using vaporisation-coulometric Karl Fischer titration. *Journal of Chemical Metrology*, 19(2), 114-127. doi:10.25135/jcm.126.2511.3730

- Wang, Y., Wang, J., Wei, Q., Yu, L., & Shen, J. (2025). Interpretation of the Disinfection Effects Testing and Evaluation Methods Section in Test Methods for Disinfection Products (WS/T 10009-2023). *Sichuan da xue xue bao. Yi xue ban = Journal of Sichuan University. Medical science edition*, 56(5), 1184-1188. doi:10.12182/20250960102
- Wiegand, T. J. (2023). Propylene glycol. In *Encyclopedia of Toxicology, Fourth Edition: Volume 1-9 (Vol. 7, pp. V7-981)*.
- Yang, B., Mallett, S., Takwoingi, Y., Davenport, C. F., Hyde, C. J., Whiting, P. F., . . . Leeflang, M. M. G. (2021). Quadas-c: A tool for assessing risk of bias in comparative diagnostic accuracy studies. *Annals of Internal Medicine*, 174(11), 1592-1599. doi:10.7326/M21-2234