

ESTIMATIVA DE INCERTEZA PARA A MEDIÇÃO DA CONDUTIVIDADE ELETROLÍTICA POR MÉTODO SECUNDÁRIO USANDO CÉLULAS TIPO D

UNCERTAINTY ESTIMATION FOR THE MEASUREMENT OF ELECTROLYTIC CONDUCTIVITY BY SECONDARY METHOD USING CELL TYPE D

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RESUMO

A medição da condutividade eletrolítica é um dos parâmetros importantes para avaliar a concentração de substâncias dissolvidas em uma solução e tem sido amplamente utilizada em vários campos da vida, ciência e tecnologia. Estava sendo usado para controle de processo e garantia de qualidade. A rastreabilidade dos resultados da medição é um requisito crucial para garantir a confiabilidade. O laboratório de eletroquímica - metrologia química da Indonésia começou a desenvolver um método secundário para medição de condutividade eletrolítica. O objetivo deste trabalho é fornecer uma cadeia de rastreabilidade para medição de condutividade eletrolítica na Indonésia por meio de material de referência secundário de cloreto de potássio (KCl) 1M. O material de referência secundário de KCl 1M pode ser usado como padrão para calibrar o medidor de condutividade, especialmente na análise da água do mar. O material de referência secundário que é desenvolvido tem um valor de condutividade eletrolítica de cerca de 111 mS/cm e foi medido usando o tipo de célula D de ZMK, Alemanha. Esta célula é feita de vidro com dois eletrodos de platinização no interior. A distância dos eletrodos é de 60 mm e o diâmetro do eletrodo é de 20 mm. A medição da incerteza foi estimada de acordo com a recomendação ISO GUM, identificando todas as fontes possíveis de incerteza no processo de medição da condutividade eletrolítica. O resultado mostrou que a incerteza expandida da medição de condutividade eletrolítica para KCl 1M por um método secundário usando o tipo de célula D foi de 0,33% ($k = 2$) para um nível de confiança de 95% com uma constante de célula e um desvio de temperatura das medições como as maiores fontes de incerteza, que contribuíram em 80% e 17%, respectivamente.

Palavras-chave: rastreabilidade, constante celular, diagrama de causa e efeito

ABSTRACT

Electrolytic conductivity measurement is one of the important parameters to evaluate the concentration of dissolved substances in a solution and has been widely used in various fields of life, science, and technology. It was being used for process control and quality assurance. The traceability of the measuring results is a crucial requirement to guarantee reliability. The electrochemistry laboratory - chemical metrology Indonesia has started to develop a secondary method for electrolytic conductivity measurement. The objective of this work is to provide a traceability chain for electrolytic conductivity measurement in Indonesia through secondary reference material of Potassium Chloride (KCl) 1 M. The secondary reference material of KCl 1 M can be used as a standard for calibrating the conductivity meter, especially in seawater analysis. The secondary reference material that is developed has an electrolytic conductivity value of about 111 mS/cm and was measured using cell type D from ZMK, Germany. This cell is made from glass with two platinization electrodes inside. The distance of the electrodes is 60 mm, and the diameter of the electrode is 20 mm. The uncertainty measurement was estimated according to the ISO GUM recommendation by identifying all possible uncertainty sources in the electrolytic conductivity measurement process. The result showed that the expanded uncertainty of electrolytic conductivity measurement for KCl 1 M by a secondary method using cell type D was 0.33% at $k=2$ for 95% confidence level with a repeatability of the measurements and cell constant as the biggest sources of uncertainty which contributed as 80% and 17%, respectively.

Keywords: traceability, cell constant, cause and effect diagram

1. INTRODUCTION

Electrolytic Conductivity (EC) is a measurand of the ionized substance in the solution (Isabel C. S. Fraga et al., 2008). EC is the most frequently measured in analytical measurement using conductivity meter, because of its low cost and easy to perform. The EC is indicated in several standards and test instruction as a control parameter for monitoring water purity and water quality in many fields such as pharmaceutical, semiconductor, powerplant, food industries, health care, environmental monitoring, and biotechnology (Orrù, 2014; S. Seitz et al., 2010). The result of EC measurement will affect decisions in the related field because it can directly related to product integrity, food safety, human health or environmental protection. In order to guarantee reliable and precise measurement results, the conductivity meter must be calibrated with traceable reference material (Freek Brinkmann et al., 2003).

The electrochemistry laboratory - chemical metrology Indonesia has developed an activity to prepare the traceable reference material of potassium chloride (KCl) 1 M. This EC value was determined by the secondary method using cell type D. Therefore, it is called as secondary reference material. According to OIML R56, KCl 1 M is a recommended standard solution for EC measurement with value 111 mS/cm at 25 °C (OIML, 1981). This standard solution can be used for calibrating the conductivity meter in monitoring quality of seawater with EC value about 50 mS/cm. (Breuel *et al.*, 2009). If the conductivity of the seawater is higher than 50 mS/cm, it indicates that more chemicals dissolved, including salts and heavy metals. The high concentrated substances in seawater will affect the fitness and survival of the organism and ecosystem (Miguel Cañedo-Argüelles et al., 2019; Staff, 2010).

The cell type D that used in the electrochemistry laboratory - chemical metrology Indonesia was purchased from ZMK, Germany. It is a glass tube with two platinization electrodes with a diameter of 20 mm and the distance of the two electrodes 60 mm. A cover plate of the cell was made from metal and had grips to set the cell into the thermostatic bath (Ulrich Breuel et al., 2008).

All measurements result, including the EC measurement of KCl 1 M is an estimation of the concentration of ionized substances in a liquid sample (Freek Brinkmann et al., 2003). It can not be known exactly how near the measured value with the true value. Many factors influence the EC

measurement process. Therefore, uncertainty of measurement must be estimated. This paper will present the uncertainty estimation for the measurement of the EC by the secondary method using cell type D.

2. MATERIALS AND METHODS

All chemicals were analytical grade and used as received without any further purification. A certified reference material (CRM) of the primary standard solution with EC value 100 mS/cm at 25 °C (code CRM1714 batch 17101201JA, bottle 18) which purchased from DFM (Danish Metrology Institute-Denmark) was used to calculate the cell constant. A KCl with 99.5% purity was purchased from Sigma-Aldrich. Demineralized water 0.055 µS/cm was produced from Thermo Scientific Barnstead Smart2pure water purification system.

In this experiment, an analytical balance with accuracy 1 mg (PR5003 DualRange, Mettler Toledo Switzerland) was used and also the clean glassware, volumetric flask 2 L, and HDPE (High-Density Polypropylene) bottle 250 mL were used. For determination of the EC value of KCl 1 M, a cell type D was used. In addition to the cell, the measurement system also contains the following equipment (describe in Figure 1), such as precision thermostatic bath (water bath Proline PV36 and Chiller DLK25, Lauda Germany), temperature measuring device (MKT50, Anton Paar Germany) and precision LCR meter (8105G, GW-Instek Taiwan).

2.1. Preparation of secondary reference material solution

Secondary reference material was made from KCl 1 M. A certain amount of KCl (149.1 g) was dissolved with a few of demineralized water in the beaker glass. Then the solution was transferred into a clean volumetric flask (2 L) and the demineralized water was added until the mark etched on the neck of the flask. The procedure was similarly repeated until 7 times. Then the prepared KCl 1 M was transferred into 250 mL of HDPE bottles. This solution is called secondary reference material because the EC value was determined by secondary method.

2.2. The procedure of EC measurement

The EC measurements in these studies were carried out by the secondary method using cell type D. This cell has a conductivity measurement capability ranging from 20 to 100

mS/cm. Before used in the measurement, the cell must be rinsed with a small volume of the sample solution. The amount of 80 mL solution (KCl 1 M) was put into the cell type D. The cell was closed and placed in a thermostatic bath to maintain the temperature at 25 °C. When the temperature was stable, the resistance (R) measurements were measured in the frequencies (f) range from 120 until 480 using LCR meter. To evaluate the results, the data from R vs $\frac{1}{f}$ were interpolated by regression line ($y = mx + c$) where m is the slope and c is the intercept. The intercept is expected as the mean value of R . This value was used in the calculation of conductance using Eq. (3). After the measurements finished, the cell is emptied, and then rinsed with demineralized water for three times.

2.3. Procedure for determination the cell constant and conductivity value of KCl 1 M

EC (κ) is defined as the inversion of the resistance (R) multiplied by the cell constant (K_{cell}). The equation is described in Eq. (1) (Isabel Cristina Serta Fraga, 2013).

$$\kappa = K_{cell} \times \frac{1}{R} \quad (\text{Eq. 1})$$

The cell in which the conductivity is measured has its cell constant. The cell constant is a function of the electrode areas, the distance between the electrodes and the electrical field pattern between the electrodes (Slovacek, 1998). It is influenced by geometrical factors, such as electrode distance and the active electrode surface. (Ulrich Breuel et al., 2009). The cell constant is defined in Eq. (2) (R. H Jameel et al., 2000).

$$K_{cell} = \frac{l}{A} \quad (\text{Eq. 2})$$

where l is the effective length between the electrodes and A is the effective cross-sectional area. In the primary method, the cell constant was obtained through the physical dimensions of the cell geometry and the observed value of R is directly traceable to International System of Units (SI) (Freek Brinkmann et al., 2003). Any changes in the cell constants can be measured precisely by dimensional measurements. The measurement of conductivity using primary method has been widely developed during the last decade. But this system is not easy to handle. Therefore the secondary method can be used as an alternative measurement of conductivity (Ulrich Breuel et al.,

2007).

In the secondary method, cell constant was determined using the primary standard solution that traceable to SI (Ulrich Breuel et al., 2007). In this case, CRM1714 from DFM was used as the standard reference solution with conductivity value 100 mS/cm. Consequently, the EC measurement of secondary method is traceable to DFM through CRM1714.

The resistance of primary standard solution (CRM1714) then converted into conductance (G). Conductance in Siemens (S) is reciprocal of the resistance (R) in ohm (Ω). The equation is described in Eq. (3) (Ulrich Breuel et al., 2007):

$$G = \frac{1}{R} \quad (\text{Eq. 3})$$

Combination of the Eq. (1) and Eq. (2) produces an equation of the cell constant (K_{cell}), written as Eq. (4):

$$K_{cell} = \frac{\kappa}{G} \quad (\text{Eq. 4})$$

where κ is the conductivity value of CRM1714 from the certificate. After the cell constant is known, this value can be used for calculation of the secondary reference material solution (KCl 1 M) by using Eq. (5):

$$\kappa = K_{cell} \times G \quad (\text{Eq. 5})$$

2.4. Procedure for uncertainty estimation in EC measurement

A result measurement will be meaningless without the statement of measurement uncertainty because it represents the quality of a result measurement (Günther Meinrath and Spitzer, 2000; Vicki Barwick and Pricard, 2011). Similar to other experiments, EC measurement is affected by the measurement process.

The estimation process of measurement uncertainty is started by the specification of the measurand. Measurand is a particular quantity subject to be measured (JCGM, 2008). In this case, measurand is EC. Then all sources of uncertainty are identified and quantified. The final step is the calculation of the combined uncertainty (S. L. R Ellison and Williams, 2012).

All sources of uncertainty can be listed using a cause and effect diagram. It shows their relationship and indicates their influence on the uncertainty of the result (S. L. R Ellison and Williams, 2012). Figure 2 illustrates the cause and effect diagram for EC measurement using the secondary method. The procedure for uncertainty

estimation in EC measurement by the secondary method using cell type D is similar to the procedure for uncertainty estimation for measurement by a secondary method using cell type C (Nuryatini Hamim et al., 2019).

One of the uncertainty sources that contributes to the uncertainty is cell constant because it is an important factor of EC measurement. (SAS, 2004). Other factors that contribute to and influenced the EC measurement are LCR meter, temperature, carbon dioxide (CO₂) effect, and temperature coefficient (Tangpaisarnkul, 2017; Ulrich Breuel et al., 2009). The CO₂ from the air may form hydrogen carbonate ion (HCO₃⁻) in the water and it will change the conductivity of the solution (SAS, 2004). Then, the temperature coefficient can influence the result of EC measurement because there is some correction of EC measurement at various temperatures for conductivity at a standard temperature (25 °C). It reflects the rate of conductance changes per degree of temperature (Smith, 1962).

3. RESULTS AND DISCUSSION

Every measurement cell of the EC system has its cell constant because the dimension of the two platinum electrodes is not the same, such as the distance between the electrodes and surface area of the electrodes. Different cell constants will be used in a different area of EC measurement. (Crison, 2004). The cell constant of the secondary method was determined using known standard solutions with traceable conductivity standards (Ulrich Breuel et al., 2009). In this study, CRM1714 from DFM was used to calculate the cell constant of the cell type D. Measurement results were tabulated in Table 1.

From the calculation, the cell constant of the cell type D is 1.5167 cm⁻¹. This value is then used to calculate the electrolytic conductivity value (κ) of secondary reference material solution (KCl 1 M). The measurement results for calculation K of KCl 1 M are tabulated in Table 2.

EC value for KCl 1 M from the calculation in Table 2 is 111.61 mS/cm. This value is in a good agreement with the conductivity values for KCl 1 M standard solution based on OIML R56 and CRM from ZMK using the same method (Breuel, 2018; OIML, 1981).

After the calculation of EC value for KCl 1 M, the uncertainty measurement can be calculated and quantified in order to involve assigning a statistical confidence level to the measurement result.

In general, the process in EC

measurement is divided into two steps: determination of cell constant and measurement of the sample (John J. Barron and Ashton).

Therefore to estimate the uncertainty of EC measurement, firstly, the sources of the cell constant must be identified. A model equation for the determination of the cell constant follows the Eq. (6) (Tangpaisarnkul, 2017).

$$K_{cell} = \frac{\kappa + \delta_{drift}}{G + \delta_{extrapolation}} \times (1 + TK \times \Delta T) \quad (\text{Eq.6})$$

where κ is EC value of standard reference solution (CRM1714, DFM), TK is temperature coefficient of solution that found in the certificate with value is 1.77 %/°C at 25 °C (Snedden, 2017), ΔT is a temperature deviation of the measurements that consist of bath stability (T stab), bath homogeneity (T hom) and calibration of thermometer (T), and δ_{drift} is a drift of reading CRM1714 from LCR meter.

Drift is a source of uncertainty in measurement that should be included in the uncertainty budget (Hogan, 2019). Then, $\delta_{extrapolation}$ is extrapolated from the graph of R vs $\frac{1}{f}$.

Cause and effect diagram for determination the cell constant is described in Figure 3, and the uncertainty measurement budget for determination the cell constant of cell type D is tabulated in Table 3.

The result showed that κ of CRM1714 is the most significant contribution in the uncertainty budget of the cell constant measurement (93%) because the CRM1714 is used to calculate the cell constant of cell D which is an important factor of EC measurement (SAS, 2004). Besides that, the temperature deviation of the measurements (ΔT) becomes the second-largest contribution in the measurement of cell constant (4%). Since the measurements of EC is temperature dependence (Mäntynen et al., 2011).

From the calculation, the expanded uncertainty (U) of determination the cell constant is 0.0020 at k=2 for 95% confidence level. Then, the uncertainty budget for determination of secondary reference material solution KCl 1 M is calculated using Eq (7). The budget uncertainty for determination of secondary reference material solution KCl 1 M is listed in Table 4. The value of CO₂ equilibrium, CO₂ sensitivity coefficient and CO₂ suppression factor were taken from the literature (Tangpaisarnkul, 2017). The results showed that the biggest contribution was resulted from the repeatability of measurement, which is 80%. It is because there are many factors that affect the measurement process and give the

difference measured value (Kedar A. Upasani and Patkar, 2015). The second-largest contributor is cell constant, which is 17%. Therefore it is recommended to check the cell constant regularly using standard reference solution, especially in platinization electrodes like cell type D, to minimize

4. CONCLUSIONS

The electrochemistry laboratory - chemical metrology Indonesia has developed a secondary reference material for electrolytic conductivity (EC) measurement in the range of 20-100 mS/cm by a secondary method using cell type D. The expanded uncertainty was found to be 0.33% for the EC measurement of KCl 1 M.

These reference materials can be used to guarantee the quality of the EC measurement, establishing the traceability chain, and contributing to reduce the import of these reference materials.

5. ACKNOWLEDGMENTS

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contamination on the electrode (SAS, 2004). From Table 4, expanded uncertainty (U) of the EC measurement for secondary reference material solution of KCl 1 M is 0.36 mS/cm or 0.33% at k=2 for 95% confidence level.

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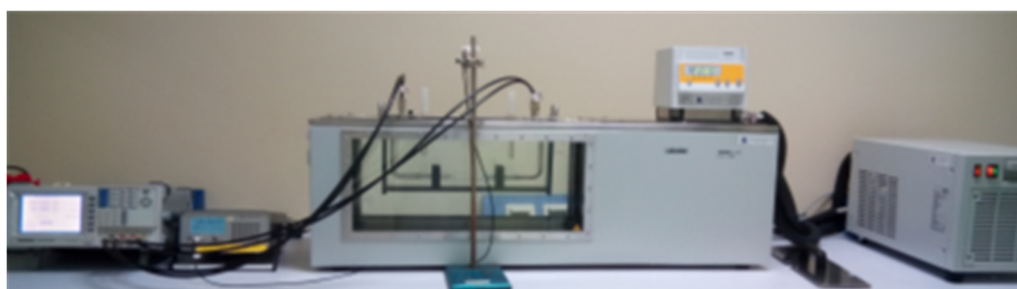


Figure 1. The system of secondary EC measurement

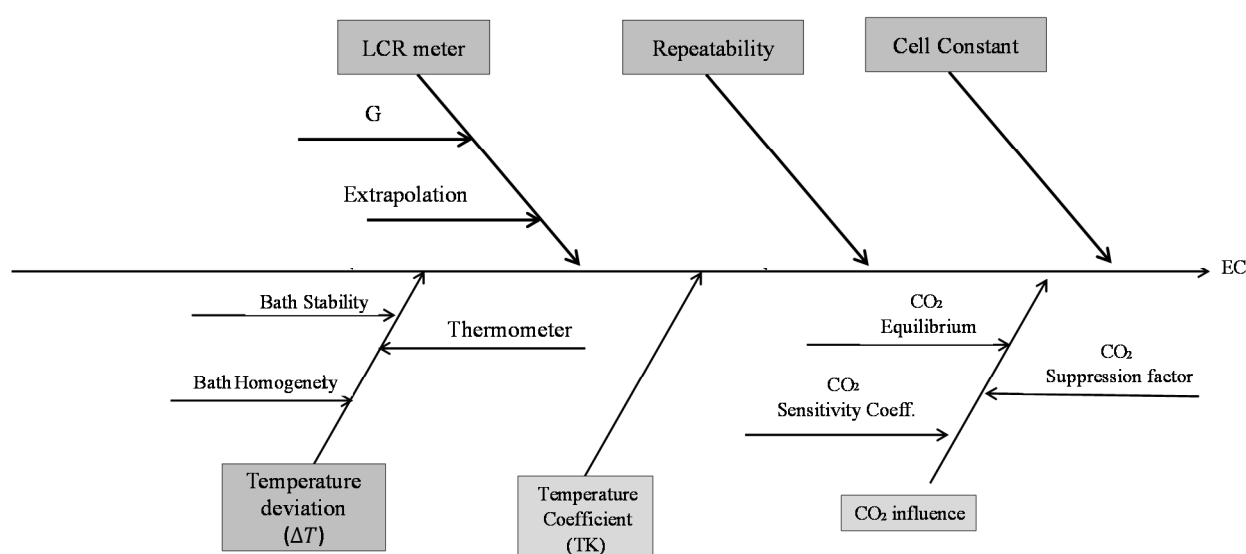


Figure 2. Cause and effect diagram for EC measurement using a secondary method system (Tangpaisarnkul, 2017; Ulrich Breuel et al., 2007)

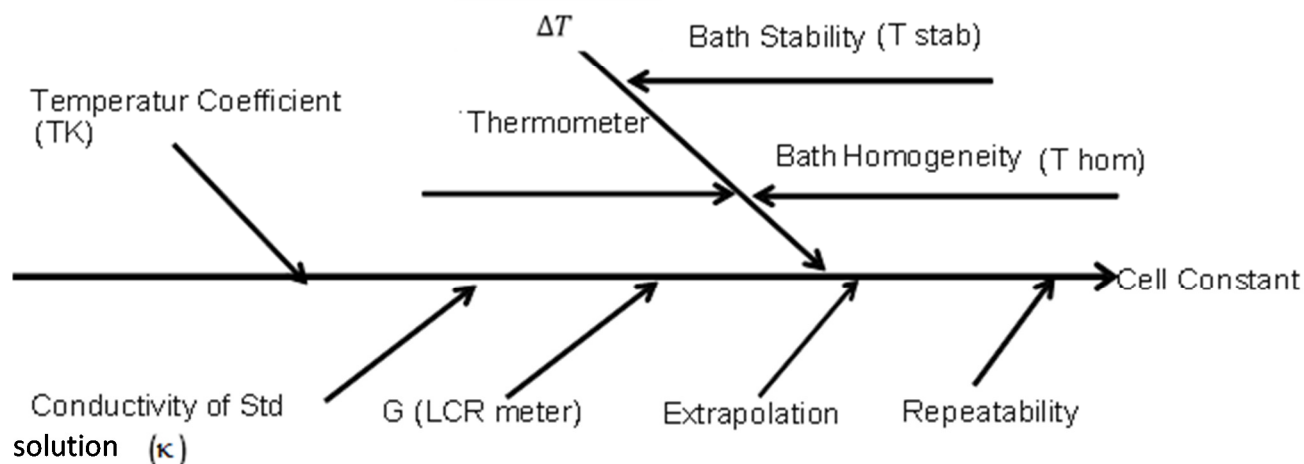


Figure 3. Cause and effect diagram for the estimation uncertainty of determination the cell constant (Tangpaisarnkul, 2017; Ulrich Breuel et al., 2007)

$$\kappa = (K_{cell} + \delta_{repeatability})(G + \delta_{extrapolation}) (1 + TK \times \Delta T) + \frac{\delta_{CO_2 equilibrium} \times \delta_{CO_2 sensitivity coefficient}}{\delta_{CO_2 suppression factor}} \quad (\text{Eq. 7})$$

Table 1. Measurement result for the calculation of the cell constant

f (Hz)	1/f (Hz ⁻¹)	Resistance (Ω)
120	0.0083	15.1788
130	0.0077	15.1779
140	0.0071	15.1771
150	0.0067	15.1764
160	0.0063	15.1759
170	0.0059	15.1754
200	0.0050	15.1741
210	0.0048	15.1737
330	0.0030	15.1715
360	0.0028	15.1707
480	0.0021	15.1696
Intercept value	(Ω)	15.1668
Conductance (G)	(S)	0.0659
using Eq. (3)	(mS)	65.9335
EC of CRM from certificate (κ)	(mS/cm)	100
Cell Constant (K _{cell}) using Eq. (4)	(cm ⁻¹)	1.5167

Table 2. Measurement results for the calculation κ of KCl 1 M

f (Hz)	1/f (Hz ⁻¹)	Resistance (Ω)
120	0.0083	13.6012
130	0.0077	13.6002
140	0.0071	13.5995
150	0.0067	13.5988
160	0.0063	13.5982
170	0.0059	13.5976
200	0.0050	13.5963
210	0.0048	13.5959
330	0.0030	13.5938
360	0.0028	13.5930
480	0.0021	13.5922
Intercept value	(Ω)	13.5890
Conductance (G) using Eq. (3)	(S) (mS)	0.0736 73.5876
K_{cell} from Table 1	(cm ⁻¹)	1.5167
κ of KCl 1 M using Eq. (5)	(mS/cm)	111.61

Table 3. Uncertainty measurement budget for determination the cell constant of cell type D

Sources (unit)	Value	Divisor	uncertainty	Sensitivity Coefficient (ci)	standard uncertainty (ui)	(ci x ui) ²
$\kappa_{CRM1714}$ (mS/cm)	100	2	0.13	0.0152	0.0650	9.72E-07
G (mS)	65.933	2	0.0158	0.0230	0.0079	3.31E-08
Extrapolation (mS)	1	1	0.0002	0.0230	0.0002	1.28E-11
δ_{drift} (1/cm)	1	2.24	0.0006	0.0152	0.0003	1.69E-11
ΔT (K)	0.0037	2	0.0153	0.0268	0.0076	4.20E-08
TK (1/K)	0.0177	1.73	0.0009	0.0056	0.0005	8.06E-12
					sum of square	1.05E-06
					Combine Uncertainty (root of sum square)	0.0010
					Expanded uncertainty (U, k=2)	0.0020

Table 4. Budget uncertainty for determination of secondary reference material solution KCl 1 M

Sources (unit)	Value	Divisor	uncertainty	Sensitivity Coefficient (ci)	standard uncertainty (ui)	(ci x ui) ²
K_{cell} (1/cm)	1.5167	2	0.0020	73.5927	0.0010	5.67E-03
G (mS)	73.588	2	0.0177	1.5168	0.0088	1.79E-04
Extrapolation (mS)	1	1	0.0001	1.5168	0.0001	3.32E-08
ΔT (K)	0.0023	2	0.0153	3.3483	0.0076	6.54E-04
TK (1/K)	0.030	1.73	0.0015	0.2585	0.0009	5.01E-08
Repeatability (mS/cm)	1	4.47	0.0099	73.5927	0.0022	2.67E-02
CO ₂ equilibrium (ppm)	0	1.73	50	0.00002	28.8675	4.03E-07
CO ₂ sens. Coeff. (mS/cm/ppm-vol)	0.0011	1.73	0.0005	0	0.0003	0
CO ₂ suppression	50	1.73	10	0	5.7735	0
					sum of square	0.0032
					Combine Uncertainty (root of sum square)	0.1822
					Expanded uncertainty (U, k=2)	0.36
					%U	0.33