# Evaluation of measurement uncertainty for quantitative determination of chlorite and chlorate in fresh-cut vegetables using ion chromatography

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**Abstract**   This study aimed to evaluate the measurement uncertainty for the quantitative determination of chlorite and chlorate in ready-to-eat fresh-cut vegetables using ion chromatography with a hydroxide-selective column. One gram of the homogenized sample in deionized water was sonicated and centrifuged at 8,500 rpm. The supernatant was purified by passing it through a Sep-Pak tC18 cartridge, followed by chromatographic determination using a Dionex IonPac AS27 column. The linearity of the calibration curves, recovery, repeatability, and reproducibility of the method were satisfactory. The method detection limit was estimated to be approximately 0.5 mg/kg. Each uncertainty component was evaluated separately, and the combined and expanded uncertainty values were calculated at the 95% confidence level. The measured concentrations for 3 mg/kg of chlorite and chlorate standard materials were 3.18±0.32 and 3.10±0.42 mg/kg, respectively. These results confirmed the reliability of the developed method for measuring the two chlorine-based oxyanions in fresh-cut vegetables.

**Keywords:** chlorite, chlorate, fresh-cut vegetable, ion chromatography, measurement uncertainty

## Introduction

$ClO_2$, $NaClO$, and $HClO$ are widely used for disinfecting agricultural produce in South Korea (1). When a chlorine-based disinfectant is used as an aqueous solution, it decomposes into chlorite ($ClO_2^-$) and chlorate ($ClO_3^-$), which can decrease thyroid colloid levels and affect the nervous system if consumed in excessive quantities (2-4). Despite the health risks associated with food disinfectants and their decomposition products, systematic methods have not yet been developed to evaluate residual chlorite and chlorate ions in food products, and standards for their residual levels have not been established. Therefore, there is an urgent need for pertinent control measures to be implemented for determining the residual levels of these ions in various foodstuffs.

Analysis of chlorine oxyanions is often been performed for drinking water disinfected with chlorine dioxide or ozone (5-9). However, unlike drinking water, food samples have complex matrices and high organic and inorganic matter content, which necessitates a purification process to remove substances that may interfere with the analysis. Inadequate purification can result in incomplete separation of the analytes and decrease the performance of the analytical column and shorten its service life. Previous studies evaluated disinfectant residues by isolating the relevant analytes using AS9-SC, AS9-HC, or IC-Pak (Dionex, Sunnyale, CA, USA) columns after homogenized seafood samples were shaken with deionized water, extracted, centrifuged, and filtered, without undergoing a separate purification process (10,11). The reliability of the results of these studies cannot be determined because they presented neither raw ion chromatograms nor quality assurance/quality control (QA/QC) data.

A method has been recently established for quantifying chlorine oxyanions in fresh-cut vegetables and dried marine products (12). This quantitative analytical method consists of mixing homogenized food samples with deionized water, extraction by sonication, high-speed centrifugation (8,500 rpm), cartridge purification (Sep-Pak tC18), and chromatographic analysis using an IonPac AS27 (Dionex) column and a solution containing hydroxide ion as the stationary and mobile phases, respectively.

Analytical results are subject to variation due to latent or potential error factors. Therefore, it is critically important to ensure the reliability of the analytical results by evaluating the measurement uncertainties to determine the degree of approximation of the respective true values. Measurement uncertainty is a parameter that characterizes the discrepancy between the actual and measured values (13). The uncertainty is generally estimated in accordance with the guidelines provided by EURACHEM/CITAC and JCGM (14,15). Additionally, given the importance of the measurement uncertainty evaluation process, especially for the components influencing the analytical results, it is essential to provide quantitative indicators of measurement uncertainty of the results.

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This study was conducted to evaluate the measurement uncertainties in the results of the quantitative analysis of chlorite and chlorate (12), which are common residues in food which originate from chlorine-based disinfectants used for ready-to-eat fresh-cut vegetables. The reliability of the analytical method was ensured by determining the measurement uncertainty and considering the errors to which the particular quantitative analysis is susceptible, as well as the QA/QC for the measurements.

## Materials and Methods

### Samples, reagents, and instruments

Samples of ready-to-eat fresh-cut vegetables (sprouts and salads) were randomly selected, purchased in three supermarkets, and stored at −24°C until analysis. Sodium chlorite (80%) and sodium chlorate (99.0%) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Ethylenediamine (EDA, 99%) and dibromoacetic acid (DBA, 98%) were purchased from Fluka (St. Louis), and a 1 M potassium hydroxide (KOH) solution from Fisher Chemical (Waltham, MA, USA). The instruments used for sample homogenization, extraction, centrifugation, purification, and filtration were a SHG-15A homogenizer (SciLab, Seoul, Korea), Power-Sonic 510 sonicator (Hwashin Tech, Taegu, Korea), 1580 multi-purpose centrifuge (LaboGene, Seoul, Korea), Sep-Pak tC18 cartridge (6 mL, 1 g; Waters, Milford, MS, USA), and 0.2 μm PVDF filters (Whatman, Little Chalfont, Buckinghamshire, UK).

### Stock solution and standard solution preparation

The chlorite and chlorate stock and standard solutions were prepared in accordance with the US EPA method 300.1 (9). A stock solution of 1,000 mg/L was prepared by adding 0.1676 g of sodium chlorite, 0.1275 g of sodium chlorate, and 0.05 mL of a 100 mg/mL EDA solution to a 100 mL volumetric flask and dissolving the mixture in deionized water. The prepared stock solution was diluted with deionized water to prepare the standard solutions at concentrations of 3, 30, 45, 60, and 75 mg/L, which were then used to construct the calibration curves.

Additionally, a 755 mg/L DBA solution was prepared and used as an internal standard (IS).

### Food sample analysis

The ready-to-eat fresh-cut vegetable samples were analyzed based on the method proposed by Kim *et al.* (12). In a glass container, 1 g of the homogenized sample was mixed with the EDA solution and deionized water (15 mL). Ionic components were extracted by sonication and the extract was centrifuged (8,500 rpm). The supernatant was purified through a Set-Pak tC18 cartridge and filtered (0.2 μm). The analytes were determined using an ion chromatograph and a conductivity detector IC-CD. A Dionex IonPac AS27 column and 20 mM KOH aqueous solution were used as the stationary and mobile phases, respectively.

### Quality assurance and quality control (QA/QC)

QA/QC procedures consisted of determining the accuracy, precision, linearity, and method detection limit (12). The accuracy and precision were evaluated by adding 1 g of the chlorite and chlorate standard solution to each of the vegetable samples at two concentrations (3.0 and 6.0 mg/kg) and the recovery rate and relative standard deviation (RSD) were calculated. Each measurement was performed in triplicate. The linearity of the instrumental response was evaluated using dibromoacetate ion as the internal standard and the coefficient of determination ($r^2$) was calculated after constructing calibration curves over the concentration range of 1.7-7.5 mg/kg. All QA/QC procedures including the determination of the method detection limit (MDL) and the limit of quantitation (LOQ) were performed in accordance with the QA/QC Handbook for Environmental Examination and Inspection of the National Institute of Environmental Research (16).

### Measurement uncertainty determination

Measurement uncertainty was determined based on the guidelines found in Quantifying Uncertainty in Analytical Measurement (14) and the Guide to the Expression of Uncertainty in Measurement (GUM) (15). The fishbone diagram in Fig. 1
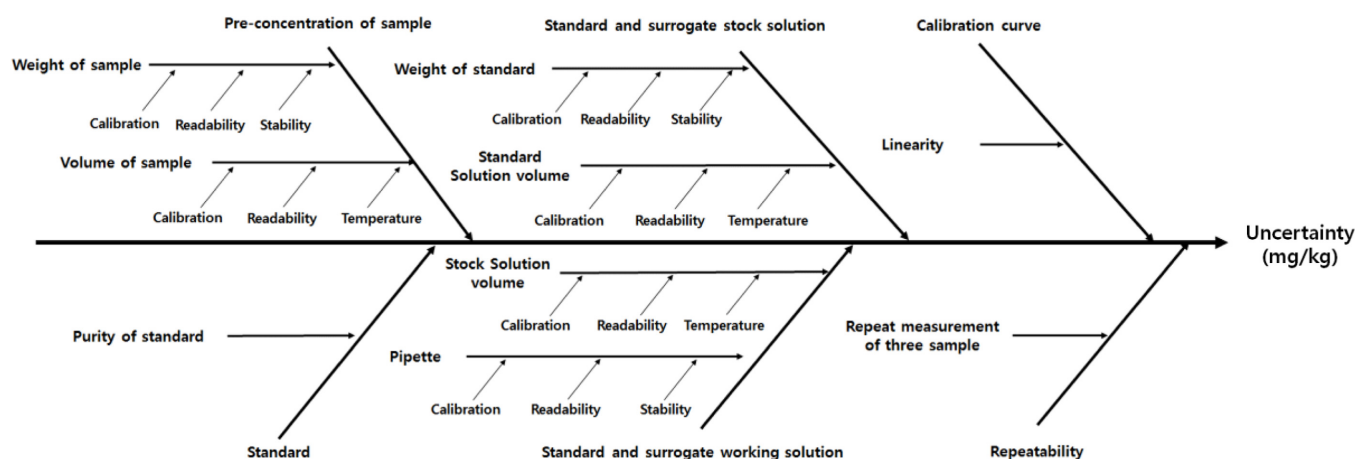


Fig. 1. Fish bone diagram of the sources of uncertainty for the determination of chlorite and chlorate in fresh-cut vegetables.

shows the process for determining the uncertainties related to the purity of the standard, weight of the standard and sample, standard solution preparation, sample pre-concentration, calibration curves, and repeated measurements.

## Standard uncertainty determination

Standard uncertainty was determined using type A and B evaluation methods. The type A method of standard uncertainty determination involves the statistical analysis of observed values based on the frequency distribution of the values obtained by repeated measurements under identical conditions. The type B method of standard uncertainty determination uses theoretically, experimentally, or empirically proven data.

When the mean value of repeated measurements was used as the measured value, the standard uncertainty of the mean value was determined by type A methods using Eq. (1). The component-dependent standard uncertainty related to the test results was also determined by type A methods using Eqs. (2) and (3) according to the pooled standard deviation. The standard uncertainties for the flasks and pipettes were determined based on the data from the calibration certificates. If no repeated measurement data were available, the standard uncertainty was determined by type B methods using Eq. (2). When performing repeated measurements for determining and expressing the measurement uncertainties, $n$-1 was used as the degree of freedom in compliance with the Korea Laboratory Accreditation Scheme (KOLAS) guidelines. When constructing the calibration curves under repeated measurements, $n$-2 was used as the degree of freedom for the calculation of the standard uncertainty of the gradient, as specified in the KOLAS guidelines (17).

$$u(x_i) = \frac{s}{\sqrt{n}} \tag{1}$$

$$u(x_i) = \frac{s_p}{\sqrt{n}} \tag{2}$$

$$s_p = \frac{\sqrt{\sum_{i=1}^{n} v_i s_i^2}}{\sum_{i=1}^{n} v_i} \tag{3}$$

$u(x_i)$: Standard uncertainty
$s$: Standard deviation
$s_p$: Pooled standard deviation
$n$: Number of measurements
$v_i$: Freedom of uncertainty from $y$
$s_i$: Standard deviation of variable $i$
$y$: Measurement results or estimated value

## Uncertainties related to the standard reagent preparation process

The uncertainty obtained from the calibration certificate of the balance, the readability of the balance, and the stability of the

repeated measurements were considered in the determination of the uncertainty components related to the measurement of the weight of sodium chlorite, sodium chlorate and the food samples. From the calibration certificate for balance ($U_{calibration}$, type B), the calibration uncertainty ($k=2$) for 1 g of the sample is 0.0003 g at the 95% confidence level. Since this value was obtained using the expanded uncertainty calculation formula, the standard uncertainty was calculated by a type B method using Eq. (2). The uncertainty related to the readability of the balance was calculated by type B using Eq. (2) based on a readability of 0.0001 g taken from the calibration certificate. The stability of the laboratory balance for repeated measurements ($U_{stability}$, type A) was estimated by conducting three measurements every day for four days using a 10 g standard weight, and the standard uncertainty was calculated by type A using Eq. (1).

## Uncertainties related to the sample pre-concentration process

Uncertainties related to the volumetric flask and the analytical balance used for the preparation of the 1,000 mg/L chlorite and chlorate stock solutions were considered in the determination of the uncertainty components related to the sample pre-concentration process. To determine the uncertainty of sample pre-concentration, the uncertainty of the analytical balance from its calibration certificate, stability of the standard weight during repeated measurements, readability of the analytical balance, uncertainty of the volumetric flask from its calibration certificate, repeatability of volumetric measurements, and uncertainty of the volumetric flask due to temperature differences (volumetric stability) were calculated.

The balance-related uncertainties, i.e., uncertainties stated on the calibration certificate, readability, and repeated measurement stability, were calculated using the same methods as those used for the standard reagent preparation process, as described in the previous section.

From the calibration certificate of the volumetric flask ($U_{calibration}$, type B), the calibration uncertainty ($k=2$) of the 25 mL volumetric flask is 0.02 mL at the 95% confidence level. Since the standard uncertainty of the volumetric flask was calculated using the expanded uncertainty calculation formula, Eq. (2) was applied. For the repeatability of volumetric measurements ($U_{repeatability}$, type A), the proficiency-dependent uncertainty was calculated by repeating the measurement ten times for measurements based on the graduation lines of the 25 mL volumetric flask. The uncertainty in the volumetric flask due to temperature differences ($U_{temperature}$, type B) was calculated by multiplying the volume of the volumetric flask by the temperature difference and the water expansion coefficient, and dividing the product by $\sqrt{3}$ under the assumption that the uncertainty of the temperature difference has a triangular distribution, using the type B uncertainty calculation formula. The calibration temperature of the 25 mL volumetric flask used for measurements was 20°C, the water expansion coefficient was 0.00021/°C and volumes were typically measured at 24.8°C.

## Combined standard uncertainty

The combined standard uncertainty is defined as the standard uncertainty of the measurements obtained using various input values. Based on the values of the standard uncertainties calculated using Eqs. (1)-(3), each combined standard uncertainty was calculated using Eq. (4) in accordance with the law of propagation of uncertainty. The combined standard uncertainty was calculated from the measured concentrations of the chlorite and chlorate ions, and the standard and relative standard uncertainties were obtained using all uncertainty components. The sensitivity coefficient (also known as the partial derivative), $\frac{\partial f}{\partial x_i}$, was obtained by differentiating the function, $f$, with respect to $x_i$ (the input amount).

$$u_c(y) = \sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) \tag{4}$$

$u_c(y)$: Combined standard uncertainty

$\frac{\partial f}{\partial x_i}$ : Sensitivity coefficient

$u(x_i)$: Standard uncertainty

## Effective degree of freedom and coverage factor

Calculation of the coverage factor, $k$, should be preceded by the determination of the effective degree of freedom ($v_{eff}$) of the combined standard uncertainty ($u_c(y)$). The effective degree of freedom is commonly understood as the uncertainty measure of the combined standard uncertainty, and expresses the extent to which the combined standard uncertainty estimates the standard deviation of the measured values. Thus, to enable the uncertainty to be calculated, an appropriate value of $k$ was selected from the $t$-distribution table according to the EURACHEM/CITAC (14) and JCGM (15) guidelines. The Welch-Satterthwaite approximation (Eqs. (5) and (6)) was used to determine the effective degree of freedom of the combined standard uncertainty.

$$v_{eff} = \frac{u_c^4(y)}{\sum_{i=1}^{n} \frac{u_i^4(y)}{v_i}} \tag{5}$$

$$v_{eff} \leq \sum_{i=1}^{n} v_i \tag{6}$$

$v_{eff}$: Effective degree of freedom

$u_c(y)$: Combined standard uncertainty

$u_i(y)$: Relative standard uncertainty of each variable

## Calibration curve uncertainty

For the determination of the uncertainty related to the construction of the calibration curves, standard solutions diluted to the concentrations of 3, 30, 45, 60, and 75 mg/L were first prepared from the 1,000 mg/L chlorite and chlorate stock solutions.

The standard solutions were injected into 1 g homogenized samples to obtain the final concentrations of 0.3, 3.0, 4.5, 6.0, and 7.5 mg/kg, and the analysis was performed based on a predetermined analytical procedure. The uncertainty calculations were performed using Eqs. (7)-(10).

$$A_i = C_i \times B_1 \times B_0 + e_i \tag{7}$$

$$u(C_0) = \frac{S}{B_1}\sqrt{\frac{1}{p} + \frac{1}{n} + \frac{(C_i - \overline{C})^2}{S_{xx}}} \tag{8}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} [A_i - (B_0 + B_1 C_i)]^2}{n-2}} \tag{9}$$

$$S_{xx} = \sum_{i=1}^{n} (C_i - \overline{C})^2 \tag{10}$$

$A_i$: $i^{th}$ measurement of the area of the calibration standard

$C_i$: Concentration of the calibration standard corresponding to the $i^{th}$ area measurement

$B_1$: Slope of the calibration curve

$B_0$: Intercept of the calibration curve

$e_i$: The residual error

$p$: Number of measurements used to determine $C_0$

$n$: Number of measurements used for the calibration

$C_0$: Determined concentration of the analyte in the food sample

$\overline{C}$: Mean value of the calibration standards

$i$: Index for the number of measurements used to obtain the calibration curve

$S$: Residual standard deviation

$S_{xx}$: Residual sum of squares

## Expanded uncertainty

The expanded uncertainty was calculated by multiplying the coverage factor ($k$) and the combined standard uncertainty, as expressed by Eq. (11). The effective degree of freedom, $k=2$, was applied if the number of results obtained using Eqs. (5) and (6) was 11 or higher at the 95% confidence level, and the corresponding value in the Student's $t$-distribution table was used if 10 or fewer results were used.

$$U(y) = ku_c(y) \tag{11}$$

$U(y)$: Expanded uncertainty

$u_c(y)$: Combined standard uncertainty

$k$: Coverage factor

## Results and Discussion

### Determination of the measurement uncertainties related to chromatographic analysis results

Unlike the Dionex IonPac AS9-HC column, which was not suitable for separating the interfering substances in the food sample
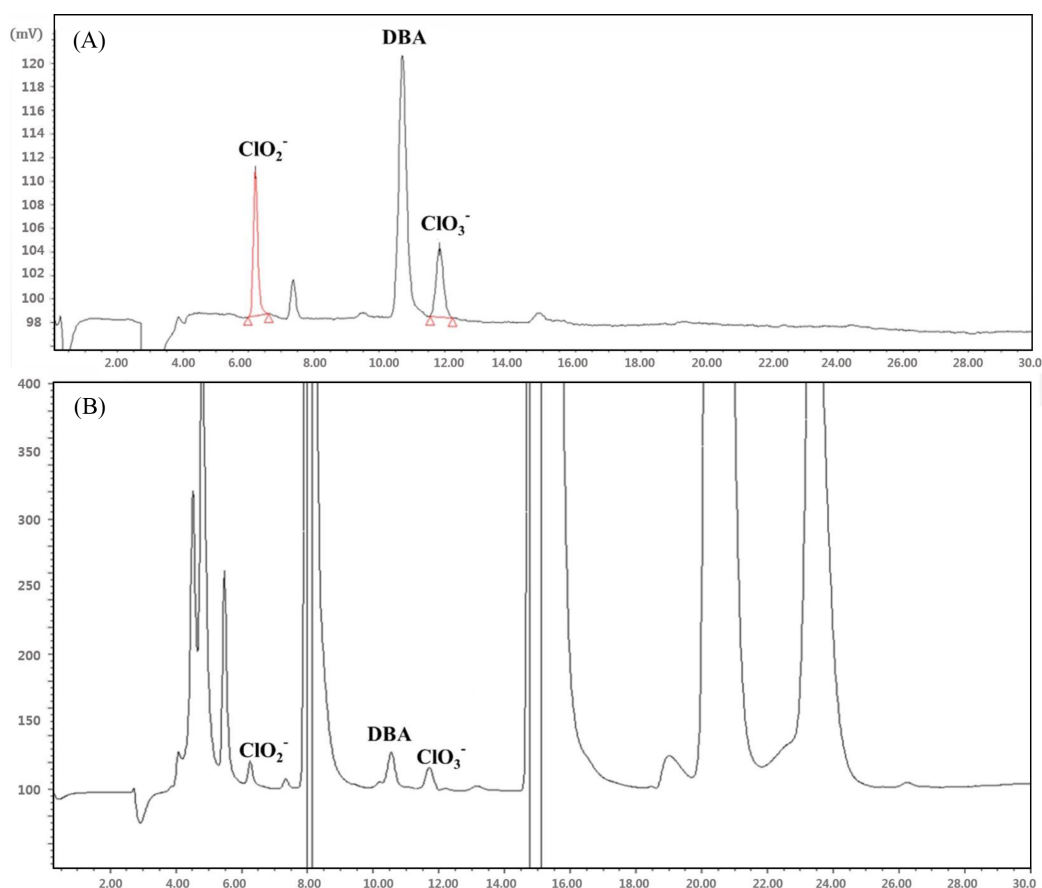
**Fig. 2. Chromatograms for the determination of chlorite and chlorate using an ion chromatograph-conductivity detector: (A) 1 mg/kg of chlorite and chlorate in a standard solution; (B) 1 mg/kg in chlorite and chlorate spiked into a homogenized fresh-cut vegetable sample.**

from chlorite, peaks could be clearly separated on a Dionex IonPac AG27 column, as shown in Fig. 2 (12). In a previous report, chlorite and chlorate residues in vegetables were determined using an ion chromatograph equipped with an integrated conductivity detector (IC-CD) with the instrumental detection limit (IDL) set at 0.01 mg/L, without considering the method detection limit of the two ionic compounds (11). In addition, the analyses of potato peel and some of the seafood samples were not accompanied by proper QA/QC data and thus failed to prove the reliability of the analytical results (10,11,18,19). In this study, two chlorine oxyanions, which are common residues on ready-to-eat fresh-cut vegetables treated with chlorine-based disinfectants, were chromatographically determined. The reliability of the analytical results was ensured by conducting relevant QA/QC procedures and by evaluating all related uncertainties. Uncertainty calculations were performed for each of the components considered to influence the analytical measurements. The overall uncertainty was calculated by applying the coverage factor ($k$) and the combined standard uncertainty at the 95% confidence level.

### Accuracy and precision

Recovery rates were estimated by performing triplicate analyses of the samples prepared by mixing 1 g of homogenized ready-to-eat fresh-cut vegetables with a standard solution and subsequently processed to obtain theoretical chlorite and chlorate concentrations of 3.0 and 6.0 mg/kg. High recovery rates were obtained: 93.3±6.08 and 98.5±5.49% for the chlorite spikes of 3.0 and 0.6 mg/kg, respectively, and 98.3±3.83 and 99.0±3.03%, respectively, for the two concentrations of chlorate. The repeatability of the triplicate measurements was also very high, with the RSD values calculated to be 3.09 and 4.14%, respectively, for chlorite and 1.57 and 1.73%, respectively, for chlorate, demonstrating high precision (Table 1).

### Method detection limits and linearity evaluation

The MDLs and the LOQs were determined to be 0.48 and 1.6 mg/kg, respectively for chlorite and 0.50 and 1.7 mg/kg, respectively for chlorate. The linearity of the chlorite and chlorate response was very high, with the coefficient of determination ($r^2$) calculated to be 0.9973 for chlorite and 0.9987 for chlorate (Table 1).

### Uncertainty of standard reagent purity

Given that the purities of sodium chlorite and sodium chlorate (80 and 99%, respectively) did not include the uncertainties, the uncertainty of the standard reagent purity was calculated by dividing half of the error by $\sqrt{3}$ under the assumption of a

**Table 1. QA/QC data for the determination of chlorite and chlorate in a 1 g sample of homogenized fresh-cut vegetable using IC-CD**

| Oxyanion | Concentration (mg/kg) (number of samples) | QA/QC | | | | |
|---|---|---|---|---|---|---|
| | | Average recovery (%) | RSD[a] (%) | MDL[b] (mg/kg) | LOQ[c] (mg/kg) | $r^2$ |
| Chlorite | 3.0 (7) | 93.3 | 3.09 | 0.48 | 1.6 | 0.9973 |
| | 6.0 (7) | 98.5 | 4.14 | | | |
| Chlorate | 3.0 (7) | 98.3 | 1.57 | 0.50 | 1.7 | 0.9987 |
| | 6.0 (7) | 99.0 | 1.73 | | | |

[a]Relative standard deviation.
[b]Method detection limit.
[c]Limit of quantitation.

rectangular distribution, which yielded the values of 0.057735% for chlorite and 0.002887% for chlorate.

## Uncertainty involved in the sample pre-concentration process

The uncertainty stated in the calibration certificate of the balance is ±0.00030 g at the 95% confidence level. By dividing this value by 2, a standard uncertainty of 0.00015 g was obtained. The standard uncertainty of the repeatability of the measurements was calculated to be 0.000016 g, half of the standard deviation obtained from ten repeated measurements using a 1 g weight approximating the weight of the standard and the homogenized samples. The readability of the analytical balance (AP250D, OHAUS, Parsippany, NJ, USA) was 0.0001 g and the standard uncertainty for readability was 0.0000289 g. The standard uncertainty of the volumetric flask was stated as 0.01 mL in its calibration certificate and the standard uncertainty of its measurement repeatability was calculated to be 0.001107 mL, based on the standard deviation of 0.003502 mL obtained from ten measurements by converting the weight of the water-filled flask into the volume. The temperature-dependent standard uncertainty of the volumetric flask was calculated as 0.001455 mL using a temperature difference of 1.2°C and a water expansion coefficient of 0.00021/°C. The relative standard uncertainty of the analytical balance was calculated to be 0.0001536, that of the volumetric flask was 0.0021375, and the combined relative standard uncertainty was 0.002143.

## Uncertainties related to the standard stock solution preparation process

The combined relative standard uncertainty obtained by combining the relative standard uncertainties of the analytical balance and the volumetric flask was 0.05776066 for the chlorite determination and 0.00336066 for chlorate.

## Uncertainty of the calibration curves

Spiked samples with standard concentrations of 0.3, 3.0, 4.5, 6.0, and 7.5 mg/kg were used to determine the uncertainty of the calibration curves. The concentration-dependent combined relative standard uncertainty of the food samples was 0.0057897, 0.0578966, 0.0578963, 0.0578966, and 0.1155618 for chlorite, and 0.0051965, 0.0051977, 0.0051948, 0.0051978, and 0.1001475

for chlorate. The standard uncertainty of the calibration curve, calculated using Eq. (1) after determining the combined relative standard uncertainty of all standard solution concentrations and the calibration curve sample concentrations, was 0.164 mg/kg for chlorite and 0.101 mg/kg for chlorate, and the relative standard uncertainty was 0.0818 and 0.0534, respectively (Table 2).

## Uncertainty of repeated measurements

To determine the uncertainty of the repeated measurements, triplicate analysis was performed after injecting a standard solution to a final concentration of 3 mg/kg in a 1 g homogenized sample. From this, the mean concentrations of chlorite and chlorate were calculated to be 3.18 and 3.10 mg/kg, respectively. The standard deviations associated with these measurements were calculated to be 0.11584 and 0.09808 mg/kg, respectively, the respective standard uncertainties were 0.0669 and 0.0566 at the degree of freedom of 2, and the respective relative standard uncertainties were 0.0334444 and 0.0283152.

## Combined standard uncertainty and expanded uncertainty

The combined standard uncertainty was calculated using the chlorite and chlorate concentrations measured in the samples combined with all the components of standard and relative standard uncertainties. The expanded uncertainty was calculated by multiplying the combined standard uncertainty by the coverage factor, $k$, as per Eq. (11). The coverage factor was set to 2 if the degree of freedom was 11 or higher and was calculated according to the Student's $t$-distribution table if the degree of freedom did not exceed 10. The combined standard uncertainties of the chlorite and chlorate measurements for the 3 mg/kg spike samples were 0.16 and 0.21 mg/kg, respectively, and the expanded uncertainties were 0.32 and 0.42 mg/kg, respectively (Table 3).

## Reliability of the analytical method

The expanded uncertainties of the quantified chlorite and chlorate were calculated as 10.1 and 13.5% at the measured concentrations of 3.18 and 3.10 mg/kg, respectively. These values are far below the reference value of 22% applicable to concentrations lower than 10 mg/kg according to a method used in the European Union (20), and thus confirms the reliability of the analytical method developed in this study for quantifying chlorite and

**Table 2. Uncertainty values for the determination of standard solutions**

| Uncertainty factor | Description | Uncertainty value |
|---|---|---|
| Weight of sample (1 g) | $U_{weight} = \sqrt{U^2_{calibration} + U^2_{readability} + U^2_{stability}}$ | 0.000153612 |
| Volume of sample (10 mL) | $U_{volume} = \sqrt{U^2_{calibration} + U^2_{readability} + U^2_{temperature}}$ | 0.002137592 |
| Pre-concentration of sample | $U_{preconcentration} = \sqrt{U^2_{weight} + U^2_{volume}}$ | 0.002143105 |
| Standard solution (1,000 mg/L) | $U_{STD\ sol.} = \sqrt{U^2_{purity} + U^2_{weight} + U^2_{volume}}$ | 0.013700480 |
| STD 1 working solution (3 mg/L) | | 0.014100355 |
| STD 2 working solution (30 mg/L) | | 0.014100784 |
| STD 3 working solution (45 mg/L) | $U_{working\ sol.}$ $= \sqrt{U^2_{STD\ stock\ sol.(1,000\ mg/L)} + U^2_{pipet\ volume} + U^2_{flask\ volume}}$ | 0.014099711 |
| STD 4 working solution (60 mg/L) | | 0.014100844 |
| STD 5 working solution (75 mg/L) | | 0.101001704 |
| STD 1 sample (0.3 mg/kg) | $U_{STD1\ sample}$ $= \sqrt{U^2_{STD1\ working\ sol.} + U^2_{sample\ weight\ (1\ g)} + U^2_{pipet\ volume\ (100\ \mu g)}}$ | 0.014262290 |
| STD 2 sample (3.0 mg/kg) | $U_{STD2\ sample}$ $= \sqrt{U^2_{STD2\ working\ sol.} + U^2_{sample\ weight\ (1\ g)} + U^2_{pipet\ volume\ (100\ \mu g)}}$ | 0.014262714 |
| STD 3 sample (4.5 mg/kg) | $U_{STD3\ sample}$ $= \sqrt{U^2_{STD3\ working\ sol.} + U^2_{sample\ weight\ (1\ g)} + U^2_{pipet\ volume\ (100\ \mu g)}}$ | 0.014261653 |
| STD 4 sample (6.0 mg/kg) | $U_{STD4\ sample}$ $= \sqrt{U^2_{STD4\ working\ sol.} + U^2_{sample\ weight\ (1\ g)} + U^2_{pipet\ volume\ (100\ \mu g)}}$ | 0.014262773 |
| STD 5 sample (7.5 mg/kg) | $U_{STD5\ sample}$ $= \sqrt{U^2_{STD5\ working\ sol.} + U^2_{sample\ weight\ (1\ g)} + U^2_{pipet\ volume\ (100\ \mu g)}}$ | 0.101024438 |

chlorate in ready-to-eat fresh-cut vegetables.

## Conclusion

This study evaluated the measurement uncertainty for a newly developed method to quantify chlorite and chlorate in ready-to-eat fresh-cut vegetables. The method used IC-CD, with a solution containing hydroxide ion as the mobile phase and a Dionex AS27 column as the stationary phase. The mean chlorite and chlorate concentrations measured in the standard samples at spiked with a concentration of 3 mg/kg were 3.18 and 3.10 mg/kg, respectively. The respective expanded uncertainties (95% CI, $k=2$), determined according to the procedures specified in the EURACHEM and JCGM guidelines for determining potential errors, were 0.32 and 0.42 mg/kg, respectively. The expanded uncertainties have lower rates (10.1 and 13.5%) with respect to the measured concentrations, thus ensuring the reliability of the developed analytical method.

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**Table 3. Uncertainty values for the determination of chlorite and chlorate in fresh-cut vegetables using IC-CD**

| Parameters | Fresh-cut vegetable samples ($n$=3) | |
|---|---|---|
| | Chlorite (3 mg/kg) | Chlorate (3 mg/kg) |
| Measured concentrations (A) | 3.18 | 3.10 |
| Combined standard uncertainty | 0.16 | 0.21 |
| Expanded uncertainty (B) ($k$ =2, 95% confidence level) | 0.32 | 0.42 |
| $\frac{B}{A} \times 100$  (%) | 10.0 | 13.4 |

## Acknowledgment

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