



Chlorate ion standard solution established by multipath titration techniques

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ABSTRACT

Analyses of chlorate ion are essential in evaluating water quality; however, there is no standard solution of chlorate ion characterized by a metrologically valid procedure. This paper describes the measurement procedures and assays of chlorate ions with the linkage to the International System of Units (SI) by multipath gravimetric titration techniques. Three different titration techniques were employed to assay chlorate ions: oxidimetric-reductometric back titration with an iron(II) solution standardized with standard potassium dichromate, precipitation titration with a silver nitrate solution standardized with standard sodium chloride, and oxidimetric-reductometric titration with a thiosulfate solution standardized with standard potassium iodate. The results obtained through three different reactions were in good agreement with each other within their uncertainties. A certified reference material of chlorate ions with the linkage to the SI was first established by evaluating the storage stability of the solution and estimating measurement uncertainties. The assay (mass fraction) of sodium chlorate was characterized by using a weighted mean of the results of three methods; $99.829\% \pm 0.029\%$ (the value following \pm gives a level of confidence of approximately 95%, coverage factor $k = 2$). Providing with the standard solution through Japan Calibration Service System (JCSS) under Japanese Measurement Act widely allows all analysts to obtain their reliable analytical results.

1. Introduction

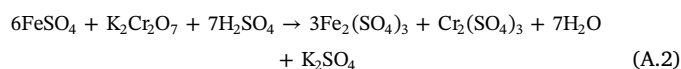
Chlorate is one of the analytes of interest, especially from the viewpoint of evaluating water quality. Several institutions such as the World Health Organization and the American Public Health Association have issued documents to examine chlorate contents in water [1, 2] due to its toxicity for not only human being but also other living organisms [3–5]. The Ministry of Health, Labour and Welfare has posed chlorate measurements in Japanese Water Supply Act [6].

Chlorate ions are measured by analytical instruments such as ion-selective electrodes [7], spectrophotometers [8, 9], flow injection analyzers [10–13] and chromatography [14–19]. These instruments require a standard solution of chlorate to quantify; however, no standard solution of chlorate ion characterized by a metrologically valid procedure has existed [20, 21].

In the present study, chlorate ions were assayed by multipath gravimetric titration techniques with the linkage to the International System of Units (SI). Titrimetry is one of the most classic and precise methods for measurements of both major and minor constituents, and is highly superior in terms of the following two points in comparison to other instrumental methods: measurements of major constituents and its repeatability. Titrimetry is applicable to determine major constituents up to 100%, and has a higher repeatability below 0.01% relative. It is difficult using instrumental analyses mentioned above to

obtain repeatabilities below 0.5% relative.

The all assays for chlorate ions given in the present paper were traceable to the SI (mass, time and ampere) because the roots of them reached to coulometric assays which yield the amount of substances on an absolute basis based on Faraday's laws of electrolysis [22]. Gravimetric titrations performed in the present study were based on the following reactions (Fig. 1):



Chlorate ions were assayed by oxidimetric-reductometric titration based on standard potassium dichromate traceable to the SI which was determined by oxidimetric-reductometric coulometric titration with electrogenerated iron(II) [23, 24]. Excess iron(II) which was standardized with potassium dichromate was added to chlorate, and then the excess iron(II) was back-titrated with potassium dichromate.



Chlorate ions were assayed by argentometric titration based on standard sodium chloride traceable to the SI which was determined by

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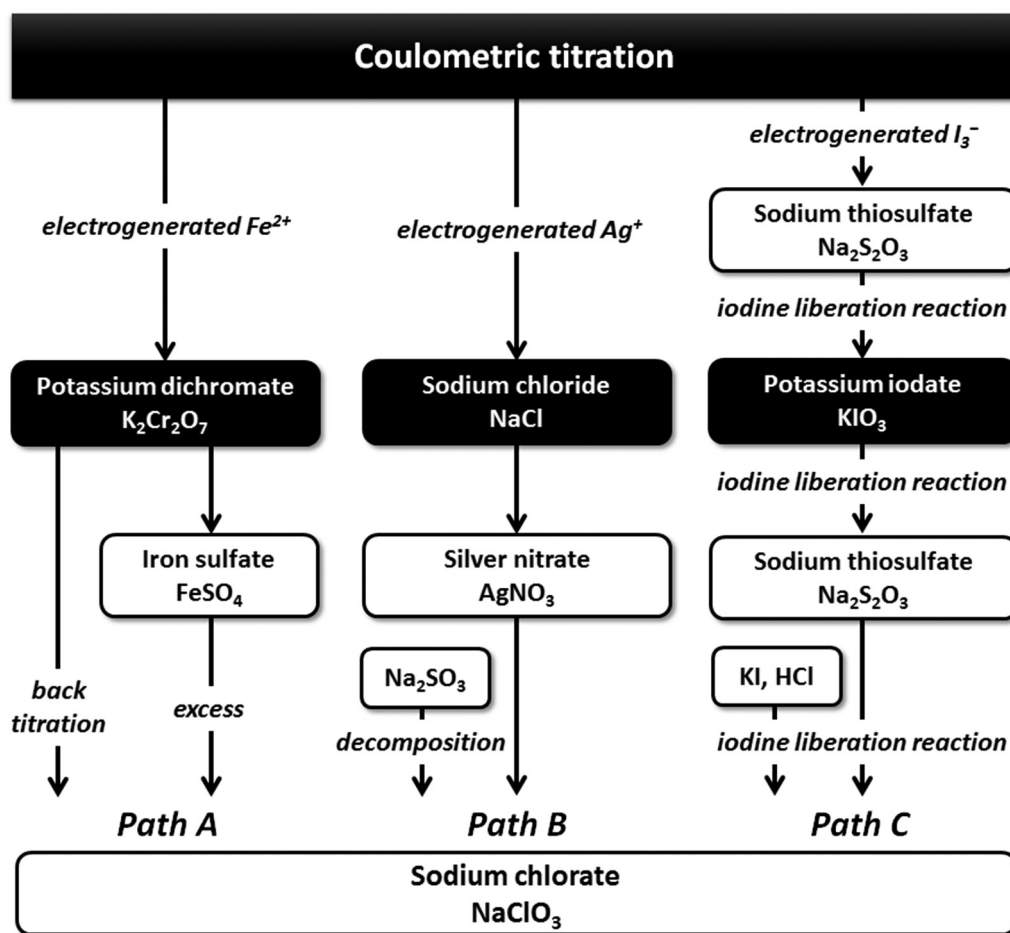
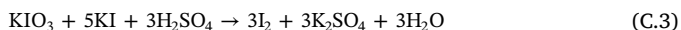
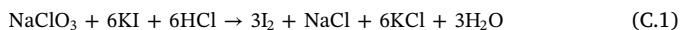


Fig. 1. Experimental design for the assay of chlorate ions through three different titration techniques.

argentometric coulometric titration with electrogenerated silver(I) [25]. The decomposition reaction of chlorate ions by sodium sulfite could be proceeded according to the reaction (B.1) [26–29]. Chloride ions generated through the decomposition of chlorate ions by excess sodium sulfite were titrated with a silver nitrate solution which was standardized with sodium chloride in precipitation reaction.



Chlorate ions were assayed by oxidimetric-reductometric titration based on standard potassium iodate traceable to the SI. Iodine (triiodide) liberated by chlorate ions in an acidic potassium iodide solution is titrated with a sodium thiosulfate solution [30, 31]. This sodium thiosulfate solution was standardized with potassium iodate which was determined based on the oxidimetric-reductometric coulometric titration and was traceable to the SI [32].

Chlorate ions were assayed by metrologically valid multipath titration techniques with the linkage to the SI. Multipath titrations based on different reactions eliminated unknown biases and gave further superior results than those based on a conventional single titration method. The reactions of oxidation of iron(II) and iodide ions are known in volumetric analysis; however, the present study has several advantages and new information: first establishment of a certified reference material (CRM) of chlorate ions by multipath titration techniques eliminated unknown biases, a reliable back titration between chlorate ions and iron(II) with examining appropriate titration conditions, an application of precipitation titration for chlorate ions through

the decomposition of them with sodium sulfite, an examination of iodine liberation reactions by chlorate ions with iodide ions and acids, and stability data of sodium chlorate and chlorate ion solutions. Titrimetry traceable to the SI on an absolute basis could give the stability as new information because the examinations for stability need an absolute method not a relative one. Estimating related uncertainties, a CRM of chlorate ions was first established with the linkage to the SI on an absolute basis. This CRM has provided through Japan Calibration Service System (JCSS) under Japanese Measurement Act [33], in which it widely allows all analysts to obtain reliable CRMs in solution form and their reliable analytical results.

2. Material and methods

Sodium chlorate was of analytical reagent grade, Wako first grade, obtained from Wako Pure Chemical Industries, Ltd., Osaka, Japan. Three CRMs and the coulometric titrations for their determinations were previously described (NMIJ CRM 3002-a potassium dichromate [23, 24], NMIJ CRM 3008-a sodium chloride [25] and NMIJ CRM 3006-a potassium iodate [32]). Analytical reagent grade chemicals were used unless otherwise stated.

Gravimetric titrations were performed with a plastic syringe with a polyetheretherketone (PEEK) or PFA needle. Solid materials were weighed with resolution of 1 μg by a type XP26 balance. Solution-form samples were weighed with resolution of 10 μg by a type XP205 balance. The balances used were of Mettler Toledo, Tokyo, Japan. Buoyancy corrections were always applied.

Chlorate ion solutions were kept in a high density, narrow-neck polyethylene bottle with inner lid (100 mL) purchased from Sanplatec

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