CHAPTER 7

Beryllium Analysis by Non-Plasma Based Methods*,‡

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Abstract

This chapter provides an overview of beryllium analysis methods other than plasma spectrometry (inductively coupled plasma atomic emission spectrometry or mass spectrometry). The basic methods, detection limits and interferences are described. Specific applications from the literature are also presented.

7.1 Introduction

The most common method of analysis for beryllium is inductively coupled plasma atomic emission spectrometry (ICP-AES). This method, along with inductively coupled plasma mass spectrometry (ICP-MS), is discussed in Chapter 6. However, other methods exist and have been used for different

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applications. These methods include spectroscopic, chromatographic, colorimetric, and electrochemical.

7.2 Fluorescence

7.2.1 Background

Fluorescence spectroscopy (also known as fluorometry or spectrofluorimetry) is a type of electromagnetic spectroscopy which analyzes fluorescence from a sample. It involves using a beam of light, usually ultraviolet light, that excites the electrons in molecules of certain compounds and causes them to emit light of a lower energy, typically, but not necessarily, visible light. Generally, the species being examined will have a ground electronic state (a low energy state) of interest, and an excited electronic state of higher energy. Within each of these electronic states are various vibrational states.

Fluorescence is an ideal method of detection because it is extremely sensitive and non-destructive, and can be performed quickly using inexpensive instrumentation. Further, the fluorescence method is routinely employed in biological industries using high throughput methods. Thus, a fluorescence test to analyze beryllium offers an opportunity to significantly increase the test throughput and reduce cost for high volume users.

Fluorescent detection of beryllium has been reported since the 1950s, with literature reports on a variety of fluorescent indicators including morin,[1–3] chromotropic acid,[4] and Schiff bases.[5] Despite the many reports of fluorescent indicators for beryllium, a complete system for its fluorescence detection was not approved by a regulatory agency. However, recent developments to change the situation targeted three goals simultaneously.[6] These goals were: (a) a dissolution method that is able to dissolve beryllium and beryllium oxide (BeO), and remains compatible with the fluorescence indicator; (b) tolerance to a wide variety of interferences; and (c) a minimal number of simple steps from dissolution to detection. This has resulted in standardized tests approved by both ASTM International[7] and the US National Institute of Occupational Safety and Health (NIOSH) for wipes and air filters,[8] while a test method for soil analysis has also been approved by ASTM International.[9] The discussion below focuses on the development and the efficacy of the standardized tests. Dissolution is described in greater detail in Chapter 4 and a summary is provided below that pertains to the use of dissolution methods adopted for the standardized test procedures.

7.2.2 Applications

7.2.2.1 Dissolution

Typical dissolution methods for the dissolution of BeO from a swipe involve concentrated inorganic acid and heating; in addition, some methods use

Table 7.1 Summary of results for filters spiked with BeO (μg Be/filter).

Expected	*Rotated*	*75 °C*	*90 °C*
0.2	0.19	0.23	0.21
1–1.2	0.65	1.11	1.20
4–5	2.68	4.54	4.20

hydrogen peroxide (see Chapter 4). Such conditions are not compatible with any known fluorescent indicator, so the solution must be evaporated to dryness and further treated before it can be added to the fluorescent indicator. In order to eliminate the time-consuming digestion steps of current standard methods, the use of a fluoride-based medium to dissolve beryllium was investigated. The efficacy of fluoride as a dissolution ligand is described in Chapter 5. It was found that Be metal was dissolved within seconds in 1% ammonium bifluoride (NH_4HF_2; ABF).[6] Results obtained from the dissolution of high-fired BeO are shown in Table 7.1.[10] These data were generated by preparing a slurry of UOX (high fired BeO available from Brush Wellman, Cleveland, OH), spiking Whatman 541 filter papers with this slurry, and analyzing these filters by dissolution in 1% aqueous ABF solution. The solution volume was 5 mL, and the dissolution time was 30 min. Rotation was conducted at room temperature and at elevated temperature; the samples were not rotated or stirred. All of these experiments were conducted using standard 15 mL conical centrifuge tubes.

Table 7.1 shows that recovery from dissolution is dependent on the amount of BeO present and the temperature of dissolution. There is another study where larger BeO particles were processed using ABF solutions at 80 °C.[11] Lower recoveries were reported; however, no experimental details were given in terms of the amount of BeO to ABF solution, or whether the reaction vessel shape allowed good interaction between the solid and the liquid phases. Since a dissolution process is dependent on the particle size, the protocol needs to be optimized for the largest particle size depending on the need of the end user. In another study the dissolution method was extended to soils, sediments and fly ash.[12] These experiments showed that the dissolution rate of these materials could be increased by increasing the concentration of ABF, and increasing both temperature and time. A study on the dissolution of high-fired BeO particles about 200 μm in size was completed recently.[13] The results showed that, by using 3% ABF at 90 °C, almost 100% recovery could be achieved in a time period of 4 h when 1 mg of BeO was used in 5 mL dissolution solution and particles had good access to the liquid medium. However, it is important to remember that to reach the US Department of Energy (DOE) action level[14] only 0.2 μg of BeO needs to be solubilized in the dissolution medium. One spherical particle of BeO, which is 200 μm in diameter, has a beryllium content of 4.5 μg, which means a recovery of only 5% is needed to draw attention; however, if the intention is a true analysis, then close to 100% recovery will be needed.

7.2.2.2 Fluorescence Indicator

When the dissolution process using ABF was adopted for test development, it was clear that a fluorescent indicator that would work with this procedure must be capable of tolerating large concentrations of fluoride. The indicator, 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS) had previously been reported to tolerate up to 20 000 000 equivalents of fluoride; in addition, this is a water-soluble dye and chelates well with Be^{+2} ion[15] (Figure 7.1).

A tightly bound hydrogen bonded proton leads to weak triplet emission at 580 nm. When the proton is displaced by a metal such as beryllium, fluorescence emission is observed at 475 nm (Figure 7.2).

To keep the test process simple and to avoid titration, the dye solution was buffered with lysine monohydrochloride. This ensured that when the acidic ABF solution was mixed with the dye solution, the pH always stayed in excess of 12 – a necessary condition to bind beryllium to the 10-HBQS dye. Further, the high pH also ensured low interference from the other metals, as most of these precipitate and the binding constant of the dye to beryllium is high. Further specificity for beryllium was also achieved by adding EDTA (disodium dihydrate ethylenediamine tetraacetic acid) to the dye solution in order to bind and mask the effects from the other metals.[16,17] Iron and titanium impart a yellow color to the final solution; these tend to precipitate in a few hours and can be removed by filtration once the solutions become clear.[6,18]

Figure 7.3 shows the excitation spectrum of a measurement solution comprising one part of 1% ABF solution with different concentrations of beryllium when mixed with 19 parts of the dye solution as outlined in the standard NIOSH and ASTM International methods. The dye solution was prepared by the addition of 12.5 mL of 10.7 mM EDTA and 25 mL of 107 mM L-lysine monohydrochloride to 3 mL of 1.1 mM 10-HBQS. The pH was adjusted to 12.85 with the careful addition of 10 M NaOH, and water added to a total of 50 mL. This excitation was measured based on the emission at 475 nm and a bandwidth of ±5 nm. The sample can be excited at any of the three peaks shown in Figure 7.2. However inexpensive instruments with excitation capabilities at 365 and 385 nm are readily available; Figure 7.3 shows the emission spectrum of the same solutions when excited by a source at 365±10 nm.

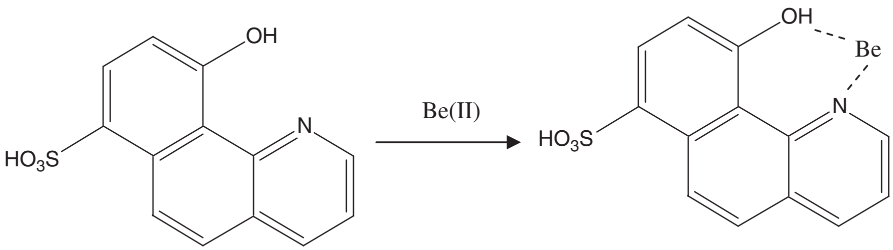

Figure 7.1 Binding of beryllium ion to 10-HBQS.

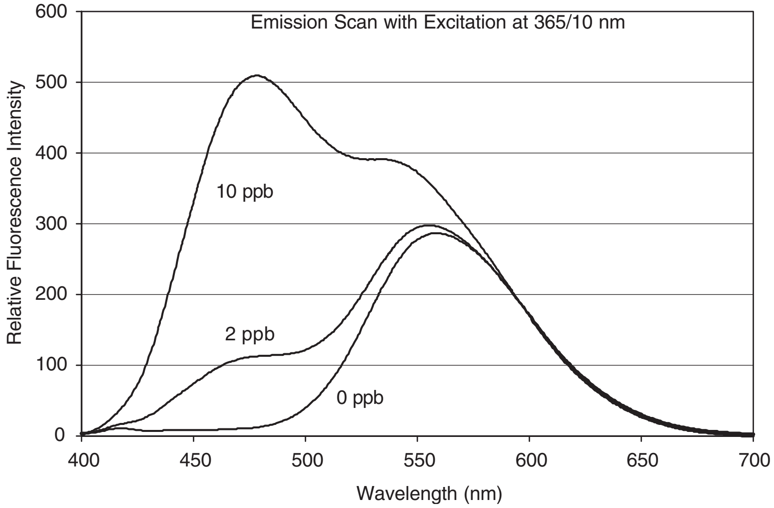

Figure 7.2 Emission spectrum of samples with various amounts of beryllium when mixed with 10-HBQS dye solution.

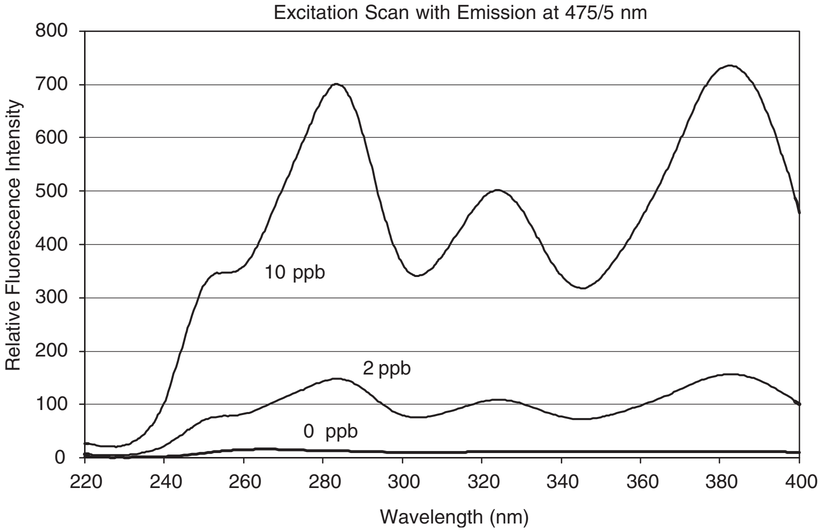

Figure 7.3 Excitation spectrum of samples with various amounts of beryllium when mixed with 10-HBQS dye solution.

In developing the standard test method, it has been demonstrated that the procedure results in good agreement when the data from different labs are compared, and, as expected, other metals do not interfere with the analysis.[6,18] To check on non-interference, some of the metals were added in concentrations in excess of 50 000 fold as compared to beryllium.

The sensitivity of the system is dependent on many factors including the ratio of the dissolution solution (with beryllium) and the dye solution. This ratio has been varied from 1 : 19 (dilution factor of 20×) to 1 : 4 (dilution factor of 5×) for 1% ABF dissolution solution. It is important to maintain pH higher than 12 to ensure good binding of the dye and beryllium.

It has been shown that beryllium in wipe or air filter samples can be quantified at 0.002 μg (2 ng), with the detection limit being even lower.[19] An amount of 0.002 μg beryllium in the wipe or air filter is equivalent to quantifying 0.08 parts per billion (ppb) of beryllium in the final measurement solution at 5× dilution.

7.2.2.3 Adaptation for Soils, Sediments, and Fly Ash

To analyze soils, sediments, and fly ash, the method described above has been slightly modified by changing the dissolution conditions.[12] The main change in dissolution condition for soils has been use of 3% ABF solution with a dissolution temperature of 90 °C and a time period of 40 h. This procedure has also been approved as an ASTM test method.[9]

Important results are shown in Figure 7.4, where they are compared with an analysis performed using ICP-MS. In these data, the Standard Reference Materials are from the Geological Survey of Japan (GSJ), the Canadian Certified Reference Materials Project (CCRMP), and the US National Institute of Standards and Technology (NIST). Samples JR-2, JA-2 and SY2 are crushed rocks, sample JB2 is volcanic ash, sample Till-1 and 2702 are soils, samples 1944 and 2710 are sediments, and sample 1633a is a fly ash.

AQ45

In order to capitalize on the success of this method and the promise of being able to analyze beryllium contamination in a variety of samples, this method is being developed for high throughput analysis.

In one analytical technique, use of a flow cell has been suggested.[20] In another, tools from the biological industry are being adopted, whereby fluorescence plate readers are being used to analyze multiple samples.[21] For the latter, the analytical procedure is being integrated with an automated sample preparation method using liquid handling systems. This will allow several hundred samples to be analyzed per day and significantly reduce labor, instrumentation needs, consumables, and chemical waste.

7.3 Atomic Absorption

7.3.1 Background

Atomic absorption (AA) spectroscopy uses the absorption of light to measure the concentration of gas phase atoms. Since samples are usually liquids or solids, the analyte atoms or ions must be vaporized in a flame or graphite furnace. The atoms absorb ultraviolet or visible light, and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption. Concentration measurements are usually determined

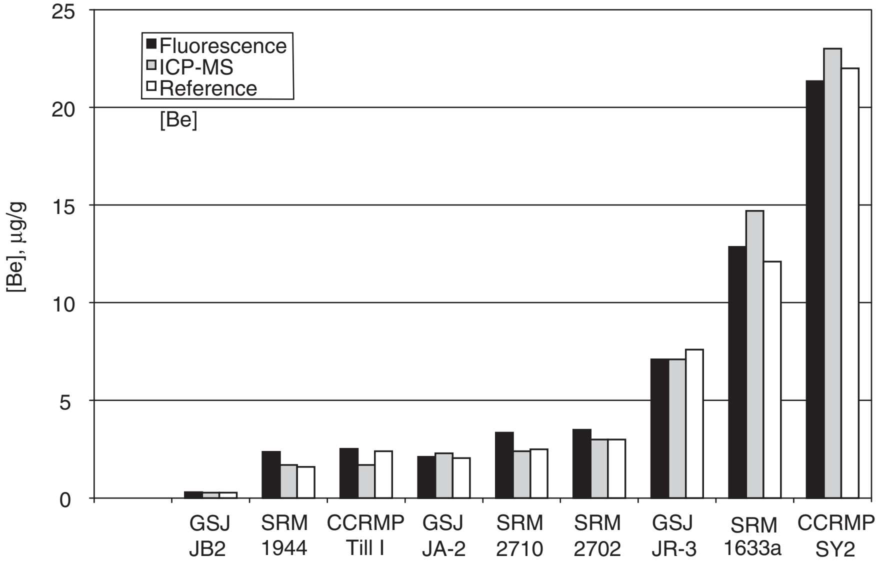

Figure 7.4 Fluorescence analysis of various standard materials and comparison with published values and ICP-MS analytical method.

from a working curve, after calibrating the instrument with standards of known concentration.

Several methods have also been reported for the atomic absorption spectrometric determination of beryllium after solid phase extraction. Okutani *et al.*[22] determined beryllium at $\mu g\,mL^{-1}$ levels in water samples by graphite furnace atomic absorption spectrometry (GFAAS) after preconcentration and separation as the beryllium acetylacetone complex on activated carbon. Szczepaniak and Szymanski[23] determined trace beryllium by GFAAS after preconcentration on silica gel with immobilized morin. Beryllium in the range 0.07–0.184 $\mu g\,mL^{-1}$ could be determined by this method. Kubova *et al.*[24] determined trace beryllium in mineral waters by GFAAS after preconcentration on a salicylate chelating resin.

7.3.2 Applications

7.3.2.1 Determination of a Trace Amount of Beryllium in Water Samples by Graphite Furnace Atomic Absorption Spectrometry after Preconcentration and Separation as a Beryllium-Acetylacetonate Complex on Activated Carbon

This is a simple preconcentration method which involves selective adsorption using activated carbon as an adsorbent and acetylacetone as a complexing agent.[25] It is used for the determination of trace amounts of beryllium by GFAAS. The beryllium–acetylacetonate complex is adsorbed onto activated carbon at pH 8–10. The activated carbon which adsorbed the beryllium–acetylacetonate complex is separated and dispersed in pure water, and the resulting suspension introduced directly into the graphite furnace atomizer. The detection limit is 0.6 ng L^{-1} (S/N = 3), and the relative standard deviation at 0.25 pg L^{-1} was 3.0–4.070 ($n = 6$). No interference is seen from the major ions AQ46 such as Na(I), K(I), Mg(II), Ca(II), Cl^{-}, and SO$_4^{2-}$ in seawater, or from other minor ions. The method was applied to the determination of nanogram per milliliter levels of beryllium in seawater and rainwater.

7.3.2.2 Solid Phase Extraction Flame Atomic Absorption Spectrometric Determination of Ultra-Trace Beryllium

AQ47 This method is based on the preconcentration of Be^{+2} on an OH-form strong base anion exchange resin.[26] The retained beryllium is eluted with 1.5 M HCl and measured by flame atomic absorption spectrometry (AAS) at 234.9 nm. Be^{+2} in the range 0.05–15 μg could be determined. The relative standard deviation (RSD) for 10 replicate measurements of 20 ng mL^{-1} beryllium was 1.2% and the 3σ limit of detection of the method was 0.045 ng mL^{-1}. The method was applied to the determination of beryllium in several water samples. In solutions with ionic strengths >0.2, the adsorption of beryllium on the resin fell significantly, because passing solutions with high ionic strength causes elution of the OH^{-} from the column. Therefore, the method is less suitable for determination of beryllium in solutions with ionic strength >0.2.

7.4 UV–Visible Spectroscopy

Ultraviolet–visible spectroscopy or ultraviolet–visible spectrophotometry (UV/VIS) involves the spectroscopy of photons in the UV–visible region. It uses light in the visible and adjacent near ultraviolet (UV) and near infrared (NIR) ranges. In this region of the electromagnetic spectrum, molecules undergo electronic transitions. This technique is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state. This section also addresses those methods that have been developed to look at visual color changes to estimate the quantity of beryllium.

A variety of chemistries have been adopted to look at color change or optical properties in presence of beryllium. Table 7.2 summarizes a number of these methods.

None of these tests have been developed to a point where they can be adopted by regulatory agencies or approved as standardized quantitative test methods. In addition, these test procedures have not been shown to be effective for difficult to solubilze beryllium compounds such as high-fired beryllium oxide. Recently, wipes for semi-quantitative analysis have been commercialized

Table 7.2 Colorimetric methods for beryllium determination[a]

Beryllium Form/Medium Evaluated	*Optical Compound*	*Reference*
Beryllium in solution	Disodium salt of *o*-carboxyphenylazo chromotropic acid (infra-red region)	27
Beryllium in water	Chrome Azurol S	28
Beryllium in water	Eriochrome cyanine R	29
Inorganic salts in solution	LI-complex (reaction product of 2-trichloro-methylbenzimidazole (TCMB)pyridine)	30
Beryllium in drinking water	Aluminon	31
Beryllium determination on human skin (skin treated with H_2SO_4)	Chrome Azurol S and NH_3 buffered solution; color compared to standard	32
Beryllium determination in water and biological samples	Precipitate of beryllium ammonium phosphate with ammonium molybdate, then treated with succinyldihydroxamic acid	33
Beryllium in coal fly ash	Chrome Azurol S in the presence of Zephiramine (ZCl)	34
Beryllium(II) determination in water and BeO in atmosphere	Anion exchange resin Amberlite IRA-400 and Chrome Azurol S in aqueous solution	35
Beryllium determination in water and waste water	Chromazurine S and hexadecylpyridine chloride (pH 5–5.3)	36
Absorption spectra of complexes with alkali and alkaline earth metal ions, including beryllium	4-(2,4-Dinitrophenylazo)-phenol(I), (II), and (III)	37
Beryllium determination in water	Ion exchange colorimetry with Eriochrome R	38
Beryllium determination in ore samples	Beryllon(III)	39
Beryllium determination in water	8-Hydroxynaphthalene-3,6-disulfonic acid (1-azo-1)-2,4-resorcinol	40
Analysis of various metals in solution	Isticin-9-imine, alizarin-9-imine, 3-sulfoalizarin-9-imine	41
Spectrophotometric determination of fluoride	Beryllium–carboxylate dye complex	42
Beryllium determination in air	Chrome Azurol S	43
Be(II) determination in solution	$K_3Cr(CN)_6$	44
Beryllium surface spot test	Chrome Azurol S	45
Beryllium chloride in solution	Ammonium aurintricarboxylate (aluminon)	46
Beryllium determination in water	Sample extracted three times with CCl_4 (pH adjusted to 5–9), $Na_2H_2[(O_2CCH_2)2CH_2]_2$(I) and Ac_2CH_2 and beryllon added	47
Beryllium determination in waste waters	Beryllon(II)	48

Table 7.2 (*Continued*)

Beryllium Form/Medium Evaluated	*Optical Compound*	*Reference*
Beryllium determination in bronze sample	Eriochrome cyanine	49
Beryllium determination in coal ash	Beryllon(II)	50
Beryllium determination in solution	Chrome Azurol S	51
Beryllium determination in solution	Beryllon(II), thoron, and arsenazo(I) compared	52
Beryllium determination in bronze sample	Chrome Azurol S	53
Beryllium on surfaces and air	Chrome Azurol S	54
Beryllium on surfaces	Chrome Azurol S	55
Beryllium in water, beryl ore, and beryllium alloy	Anthralin at pH 11.3	56

[a] This table has largely been adopted from Taylor and Sauer[54] and updated.

using Chrome Azurol S which changes color based on the beryllium content.[56] These wipes have been shown to change color at 0.2 µg of beryllium in order to meet the US DOE action limit.

7.5 Electrochemistry

There are a few electrochemical methods available in the literature for the determination of beryllium, but these are not widely used. There are only limited reports in the literature on Be^{+2} selective electrodes to directly determine the beryllium concentration.

7.5.1 Adsorptive Stripping Voltammetric Measurements of Trace Beryllium at the Mercury Film Electrode

This method involves adsorptive stripping voltammetric measurement of trace beryllium using preconcentration by adsorption of a beryllium–arsenazo-I complex at a preplated mercury-coated carbon fiber electrode.[57] It is a sensitive cathodic stripping protocol for detecting trace beryllium based on the adsorptive accumulation of the Be–arsenazo-I complex at a mercury film electrode. Optimal conditions were found to be a 0.05 M ammonium buffer AQ48 (pH 9.7) containing 5 υM arsenazo-I, an accumulation potential of 0.0V (*vs.* Ag/AgCl) and a square-wave voltammetric scan. A linear response is observed over the 10–60 µg L^{-1} concentration range (90 s accumulation), along with a detection limit of 0.25 µg L^{-1} beryllium with a 10 min accumulation. The same mercury film can be used for a prolonged operation with proper electrochemical cleaning. High stability is seen from the reproducible response of a 100 µg L^{-1} beryllium solution over 2.5 h operation. Examples of data using different stripping modes are shown in Figure 7.5. The new sensor shows

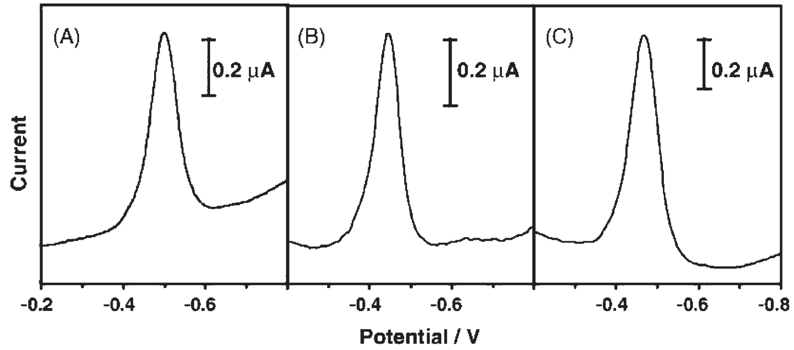

Figure 7.5 Comparison of different stripping modes for a mercury-coated carbon-fiber electrode; supporting electrolyte, 0.05M ammonium buffer (pH 9.7) containing 100 _g l–1 beryllium and 5 _M arsenazo-I: (A) Linear scan voltammetry; (B) differential pulse voltammetry; (C) square-wave voltammetry. From ref. 57.

promise for on-site environmental and industrial monitoring of beryllium. This procedure obviates the need for the large mercury-drop electrode and related mercury disposal issues. The same preplated mercury film can be employed for multiple measurements of beryllium.

7.5.2 Beryllium-Selective Membrane Electrode Based on Benzo-9-crown-3

A polyvinylchloride (PVC) membrane electrode for Be^{+2} ions based on benzo-9-crown-3 as membrane carrier was prepared and tested.[58] The sensor exhibited a Nernstian response for Be^{+2} ions over a wide concentration range (4.0×10^{-3}–2.5×10^{-6} M) with a limit of detection of 1.0×10^{-6} M (9.0×10^{-3} parts per million). The sensor has a response time of 50 s and can be used for several months without any divergence in potential. The electrode displayed good selectivity for Be^{+2} over a wide variety of other cations including alkali, alkaline earth, transition, and heavy metal ions, and could be used in a pH range of 2.0–6.0. It was successfully applied to the determination of beryllium in a mineral sample.

Benzo-9-crown-3 (B9C3) was found to be an excellent neutral carrier in construction of a Be^{+2} PVC membrane electrode. The small size of the B9C3 cavity increased both the stability and the selectivity of its beryllium complex over those of other metal ions. In addition, the existence of a benzo ring on the crown's ring results in its diminished solubility in aqueous solutions.

The Be^{+2} ion-selective electrode was found to work well under the laboratory conditions. It was successfully applied to the determination of beryllium in mineral samples such as beryl. The beryllium content in a prepared solution was determined using the proposed membrane sensor and atomic absorption spectrometry. The results obtained by the ion selective electrode are in satisfactory agreement with those obtained by AA.

7.5.3 New Diamino Compound as Neutral Ionophore for Highly Selective and Sensitive PVC Membrane Electrode for Be^{+2} Ion

This method uses diamine 2,6-bis[2-(*o*-aminophenoxy)methyl]-4-bromo-1-methoxybenzene compound as a beryllium ion carrier in a PVC membrane electrode for potentiometric determination of Be^{+2}.[59] The electrode exhibited a Nernstian response to Be^{+2} ion over a wide concentration range from 3.0×10^{-6} to 7.0×10^{-2} M, and a detection limit of 2.0×10^{-6} M. It had an appropriate response time and suitable reproducibility, and could be used for a period of at least one month without degradation of performance. The potentiometric response is independent of the pH of the test solution in the range of 4.0–7.0. The sensor revealed good selectivity toward Be^{+2} ion with respect to many alkali, alkaline earth, transition, and heavy metal ions. It was applied successfully to the determination of beryllium in tap water samples and also samples containing interfering ions.

7.5.4 Beryllium-Selective Membrane Sensor Based on 3,4-Di[2-(2-Tetrahydro-2H-Pyranoxy)]Ethoxy Styrene–Styrene Copolymer

3,4-Di[2-(2-tetrahydro-2H-pyranoxy)]ethoxy styrene–styrene copolymer was used to prepare a beryllium-selective PVC-based membrane electrode.[60] The resulting sensor works well over a wide concentration range (1.0×10^{-6} to 1.0×10^{-3} M) with a Nernstian slope of 29 mV per decade of Be^{+2} activity over a pH range 4.0–8.0. The detection limit of the electrode is 8.0×10^{-7} M (7.6 ng mL^{-1}). The electrode showed excellent selectivity toward Be^{+2} ion with regard to alkali, alkaline earth, transition, and heavy metal ions. It was successfully applied to the determination of beryllium in a mineral sample.

The Be^{+2} ion-selective electrode worked well under laboratory conditions. It was applied to the direct measurement of Be^{+2} in beryl samples after suitable treatment of the mineral. The results obtained by the ion-selective electrode are in satisfactory agreement with those obtained by AA.

7.5.5 New Diamino Compound as Neutral Ionophore for Highly Selective and Sensitive PVC Membrane Electrode for Be^{+2} Ion

In this work, five macrocyclic diamides were investigated to characterize their ability as beryllium ion carriers in potentiometric PVC membrane electrodes.[61] The electrodes based on 1,15-diaza-3,4;12,13-dibenzo-5,8,11-trioxabicyclo[13,2,2] heptadecane-2,14-dione exhibited a Nernstian response for Be^{+2} ion over wide concentration ranges [from 3.0×10^{-6} to 3.0×10^{-2} M for polymeric membrane electrode (PME), and from 5.0×10^{-7} to 2.0×10^{-2} M for coated glassy carbon electrode (CGCE), and very low detection limits (2.0×10^{-6} M for PME and 4.0×10^{-7} M for CGCE]. The electrodes had low resistances, fast responses, satisfactory reproducibilities and, good selectivities for Be^{+2}. The potentiometric response of the electrodes is independent of the pH of the test solution in the pH range 4.0–7.5. The sensors were used to determine beryllium ion in water samples.

7.6 Other Methods

7.6.1 Utilization of Solid Phase Spectrophotometry for Determination of Trace Amounts of Beryllium in Natural Water

Solid-phase spectrophotometry (SPS) is a technique based on the pre-concentration of the substance in question on a solid using complexing or other reagents, followed by measurement of the absorbance of the species in the solid phase.[62] This provides SPS with an increase in selectivity and sensitivity over other methods. Detection limits as low as $0.09\,\mu g\,L^{-1}$ have been reported. EDTA is used as the complexing agent in solution for the spectrophotometric method of beryllium determination.

In this work, a dextrane-type exchanger (*i.e.* a mixed ligand complex) is used as the basis of a method to determine beryllium with aurintricarboxylic acid (ATCA) as complexing agent. Beryllium reacts with ATCA in the presence of EDTA to give a complex with a high molar absorptivity ($1.50 \times 10^7\,L\,mol^{-1}\,cm^{-1}$), which is fixed on a dextran-type anion-exchange gel (Sephadex DEAE A-25). The absorbance of the gel, at 575 and 750 nm, packed in a 1.0 mm cell, is measured directly. Calibration is linear over the range $0.03–1.0\,\mu g\,L^{-1}$ with RSD of $<2.4\%$ ($n = 8.0$). The detection and quantification limits of the 500 mL sample method are 6.0 and $20\,ng\,L^{-1}$, respectively, using a 90 mg exchanger. For 1000 mL samples, the detection and quantification limits are 5.0 and $17\,ng\,L^{-1}$, respectively, using a 45 mg exchanger. Increasing the sample volume can enhance the sensitivity. The methods were applied to the determination of beryllium in tap, mineral and well water using the standard addition technique with recoveries of close to 100% at concentration levels of 0.032, 0.221 and $10.92\,g\,L^{-1}$, respectively.

7.6.2 Selective Determination of Beryllium(II) Ion at Picomole per Decimeter Cubed Levels by Kinetic Differentiation Mode Reversed-Phase High-Performance Liquid Chromatography with Fluorometric Detection Using 2-(2¢-Hydroxyphenyl)-10-hydroxybenzo[h]quinoline as Precolumn Chelating Reagent

This method for the determination of the Be^{+2} ion uses reversed-phase high-performance liquid chromatography (HPLC) with fluorometric detection using 2-(2¢-hydroxyphenyl)-10-hydroxybenzo[h]quinoline (HPHBQ) as a precolumn (off-line) chelating reagent.[63] The reagent HPHBQ forms the kinetically inert

Be chelate compatible with high fluorescence yield, which is appropriate to the HPLC-fluorometric detection system. The Be-HPHBQ chelate is efficiently separated on a LiChrospher 100 RP-18(e) column with a methanol (58.3 wt%)-water eluent containing 20 mmol kg^{-1} of tartaric acid and is fluorometrically detected at 520 nm with the excitation at 420 nm. Under the conditions used, the concentration range of 20–8000 pmol dm^{-3} of Be^{+2} ion can be determined without interferences from 10 mmol dm^{-3} each of common metal ions, typically Al(III), Cu(II), Fe(III), and Zn(II), and still more coexistence of Ca(II) and Mg(II) ions at 0.50 mmol dm^{-3} and 5.0 mmol dm^{-3}, respectively, is tolerated. The detection limit is 4.3 pmol dm^{-3} (39 fg cm^{-3}).

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