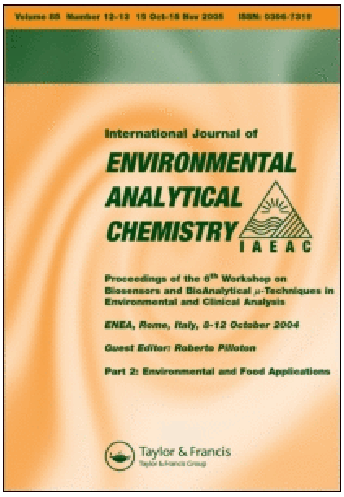

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Continuous flow hydride generation laser induced fluorescence spectrometry for trace determination of lead in water and sediment samples

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Continuous flow hydride generation laser induced fluorescence spectrometry for trace determination of lead in water and sediment samples

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Continuous flow hydride generation coupled with laser induced fluorescence spectrometry (HG-LIF) has been used for the determination of Pb in aqueous samples. Lead hydride is generated in $K_3Fe(CN)_6$-HCl medium using $NaBH_4$ as the reducing agent. Parameters such as acidity and the concentrations of oxidising and reducing agents have been studied in order to obtain the highest sensitivity. Laser excitation of Pb has been performed at 283.306 nm and fluorescence has been detected at 365 nm and 405.8 nm. The limit of detection calculated for the optimised conditions (1% $K_3Fe(CN)_6$ in 0.3% Oxalic Acid, 0.055 mol L^{-1} HCl, 1% $NaBH_4$ in 0.1% NaOH) is 0.3 ng mL^{-1}. However, a significant Pb background was observed for blank measurements, which degraded the limit of detection. The limit of detection that is estimated for a Pb free blank was determined by detuning the laser to a wavelength of 282.806 nm and was found to be 0.45 pg mL^{-1}. Method repeatability is 3.5% RSD at the 10 ng mL^{-1} level. The accuracy of this method has been evaluated by analysis of a water reference material. The results obtained for a multielement standard determined using the HG-LIF approach have been found to be in agreement with those obtained by using a comparison ICP-AES approach. The HG-LIF approach has been applied to the measurement of Pb in a contaminated sediment sample material. The results of this study demonstrate that a small tunable laser system combined with hydride generation sample introduction can be used to measure Pb with high sensitivity, precision and selectivity in different sample matrices.

Keywords: lead; hydride generation; laser induced fluorescence; sediment analysis

1. Introduction

Elemental lead and its compounds are generally considered to be toxic. The primary routes of human exposure to Pb are inhalation and ingestion. The primary health effects of Pb are to the hematological system, the central nervous system and the renal system [1]. The health effects include reduced haem synthesis and anemia, cognitive difficulties, fine motor dysfunction and encephalopathy, mitochondrial damage and irreversible nephropathy. The measurement of Pb concentrations in the environment using atomic spectrometry methods is critical for studying the environmental cycling of Pb and monitoring human exposures to this element.

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Hydride generation (HG) is a sample introduction technique used in atomic spectrometry that is useful for enhancing sensitivity and in some cases providing speciation information for metal and metalloid elements [2,3]. Hydride generation has been combined with several atomic spectrometry detection techniques [4,5], including Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) [6], Inductively Coupled Plasma Mass Spectrometry (ICP-MS) [7–9], Atomic Absorption Spectrometry (AAS) [10] and Laser Induced Fluorescence (LIF) Spectrometry [11,12].

From previous studies it has been demonstrated that Pb hydride generation requires an oxidant to convert Pb to plumbane (PbH_4) [13,14]. A number of oxidising agents have been used to increase the efficiency of the hydride generation of Pb, including potassium dichromate, potassium permanganate, ammonium cerium (IV) nitrate, hydrogen peroxide, peroxodisulfate and potassium hexacyanoferrate (III) [15–18]. The highest sensitivity has been reported when potassium hexacyanoferrate (III) is used as the oxidising agent [19,20].

The analytical figures of merit of hydride generation-based approaches may be improved through the use of high-sensitivity laser-based detection techniques such as laser induced fluorescence (LIF) spectrometry. This HG-LIF technique has been applied previously for trace determinations of As, Se, and Sb hydrides in a flame atomiser [11]. In this paper, trace level measurements of Pb using continuous hydride generation followed by LIF spectrometry detection is described. Optimisation of the hydride generation reagents and the fluorescence detection for Pb has been performed. The HG-LIF technique has been applied to the measurement of Pb in a certified reference material (1643 e Trace Elements in Water by NIST), an ICP mix standard (LPC standard 1 SPEX CertiPrep P), and river water and sediment samples. The determination of Pb in river water and sediments is important because Pb is a useful indicator of river water pollution caused by human activities [21]. The results obtained using the HG-LIF approach for one of the multielement standards have been compared to those obtained using an ICP-AES approach.

2. Experimental

2.1 *Reagents*

The stock standard solution of Pb was an atomic absorption standard (Acros Organics-1 $mg\,mL^{-1}$ in 2% HNO_3). The reagents used were as follows: hydrochloric acid (Fisher-trace metal grade and Pharmaco-AAPER-ACS reagent grade), sodium tetrahydroborate (III) (Sigma-Aldrich-AF granules 10–40 mesh 98%), sodium hydroxide (FLUKA-ACS grade), nitric acid (EMD-Omni trace 69.0–70%), potassium hexacyanoferrate (III) (Fisher Scientific-Certified ACS), oxalic acid (Allied Chemical General Chemical Division-anhydrous powder, reagent grade), DL-lactic acid (Sigma-85%(w/w)), ICP mix standard; LPC Standard 1(Spex Certi Prep P-20 $mg\,L^{-1}$ Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Tl, V, and Zn).

2.2 *Experimental setup*

The experimental set up for continuous hydride generation has been described previously [11]. The continuous flow system consists of a four channel peristaltic pump

(Model RP-1, Rainin Instrument, Co. Inc. Energyville, CA, USA) with tygon tubing, a gas liquid separator (GLS) made of pyrex glass, a nafion tube dryer (MD Series, Permapure Inc., Toms River, NJ, USA) and reaction coils of various lengths (1–2 m, 1 mm i.d.) of Teflon tubing. Two channels of the four channel peristaltic pump were used to pump the sodium tetrahydroborate (III) and acidified samples at a rate of $5.4\,\mathrm{mL\,min^{-1}}$ and $10\,\mathrm{mL\,min^{-1}}$, respectively. The flows were combined in a mixing block, and sent through a reaction coil to the GLS. The volatile hydrides and excess H_2 were carried from the GLS in a flow of He ($90\,\mathrm{mL\,min^{-1}}$). To minimise the amount of liquid aerosol transferred to the drier and atomiser a PTFE membrane filter (0.45 µm pores, 47 mm diameter, Gelman Sciences, Ann Arbor, MI, USA) was placed in the GLS. A reaction coil of about 4 m was also used between the mixing block and gas liquid separator.

During this study, a Q-switched Nd : YAG laser (Surelite II-10, Continuum, Santa Clara, CA, USA) operating at a repetition rate of 10 Hz, in the 3rd harmonic (wavelength equal to 354.7 nm) was used to pump a single-stage dye laser (Northern Lights, Dakota Technologies Inc.). The dye laser produced radiation near 283–283.5 nm by frequency doubling of 566–567 nm radiation produced using Coumarin 540A laser dye (Lambda Physik) in a BBO I crystal. The laser had a pulse width of approximately 5 ns and is reported to have a linewidth of $<1\,\mathrm{cm^{-1}}$, which corresponds to a spectral resolution of <0.013 nm at the frequency doubled wavelength. Typical laser pulse energies were approximately 8–10 µJ at 283 nm. This dye laser is relatively compact (30 cm × 6.25 cm × 11.25 cm) and yet provides moderately high power and spectral resolution and can serve as an effective excitation source for trace level laser induced fluorescence spectrometry measurements of atomic species.

An H_2-Ar flame sustained on a quartz tube (1/4 inch o.d.) was used as the atomiser for LIF spectrometry. The laser beam intersected the flame at a height of 2–3 mm above the tip of the quartz tube and was adjusted to optimise the fluorescence signal for Pb. Fluorescence emissions of Pb were collected at 90° to the laser beam direction and were transmitted to the entrance of a 0.27 m monochromator (1000 µm vertical slits, Spectra Pro-275, f/3.5, Acton Research Corp., Acton, MA, USA). The slit width of the monochromator was adjusted to provide the best signal to noise ratio. The fluorescence emissions were isolated using the monochromator and detected using a photomultiplier tube (PMT, R955, Hamamatsu Corp.). The PMT output was sampled by a boxcar averager (SR250, Stanford Research Systems, Sunnyvale, CA, USA).

An ARL Plasma Vision 3410 model ICP-AES was used for Pb determinations in selected samples for comparison with HG-LIF measurements. Sample introduction for the ICP-AES measurements was performed using solution nebulisation. The instrument conditions used for the analysis forward power, coolant gas flow, plasma gas flow, and carrier gas flow are 650 W, $7.5\,\mathrm{L\,min^{-1}}$, $0.8\,\mathrm{L\,min^{-1}}$, and $0.7\,\mathrm{L\,min^{-1}}$, respectively.

2.3 *Sample preparation*

A sediment sample was provided by Dr Carl Johnston of the Youngstown State University, Department of Biological Sciences. The sediment sample was collected from a site along the Mahoning River near Youngstown, Ohio. Each sediment subsample was transferred to a watch glass and dried at 110°C for 2 h. After drying to remove water, approximately 1 g mass of the dried sediment was weighed out and 20 mL of concentrated

HNO_3 was added and the mixture was heated until a few mL of sample digest solution remained. The digest solution was transferred to a 50 mL centrifuge tube and centrifuged at 5500 rpm for 15 min at 4°C. The clear volume from the centrifuged sample was transferred to a 100 mL volumetric flask and diluted to volume with deionised water. A method blank was run with the sediment samples under the same conditions. Volumes of 0.5 mL from each acid digested sediment solution were combined with standard additions of 10, 20 or 30 ng mL^{-1} Pb and diluted to a final volume of 100 mL. The diluted solutions were measured using the HG-LIF approach for determination of the Pb content.

The LPC Standard 1 (Spex Certi Prep P-20 mg L^{-1} Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Tl, V, and Zn) was diluted so that the final Pb concentration was 10 ng mL^{-1}. Standard additions of 10, 20, and 30 ng mL^{-1} Pb were added prior to final dilution of the LPC Standard 1 solution. During the Pb measurements using the ICP-AES method, the LPC Standard 1 was diluted so that the final Pb concentration was 0.25 μg mL^{-1}.

3. Results and discussion

Laser excitation of Pb was performed at 283.306 nm with direct line fluorescence detection at either 365 nm or 405 nm. The laser excitation of Pb at 283.306 nm has been reported previously for trace level fluorescence measurements in other atomisers [22–28]. In the optimisation of the HG-LIF approach, the relative performance of the two fluorescence emission wavelengths was evaluated in the flame atomiser. The most sensitive fluorescence line for Pb was determined by scanning the monochromator in the range of 340–440 nm. Figure 1 shows the scan of a 10 ng mL^{-1} Pb fluorescence signal trace using the HG-LIF system, which shows the characteristic emissions of Pb near 365 nm and 405.8 nm. From this scan and individual calibration plots, it was determined that 405.8 nm was the most sensitive wavelength for Pb measurements by HG-LIF spectrometry.

3.1 *Effect of potassium hexacyanoferrate (III) and oxalic acid concentrations on Pb laser induced fluorescence signal*

The measurement capabilities of the HG-LIF approach were optimised by evaluating the effects of each of the reagents used in the HG sample introduction process. The dependence of the fluorescence signal on the concentration of the potassium hexacyanoferrate (III) solution shows that the fluorescence signal increased and reached a maximum with increasing concentration of the potassium hexacyanoferrate (III).

A similar effect was observed when the concentration of oxalic acid was increased. Lactic acid was also investigated as an alternative to oxalic acid, but it was observed to cause a four times higher background and smaller Pb fluorescence signal than that of oxalic acid. At higher concentrations of potassium hexacyanoferrate (III), the Pb fluorescence signal increased but the signal intensity and standard deviation of the blank values were also increased. The optimum conditions for the oxidising solution were 1.0% potassium hexacyanoferrate (III) in 0.3% oxalic acid. Without the use of potassium hexacyanoferrate (III) in oxalic acid solution as the oxidising agent, no Pb fluorescence signal was observed using hydride generation sample introduction.

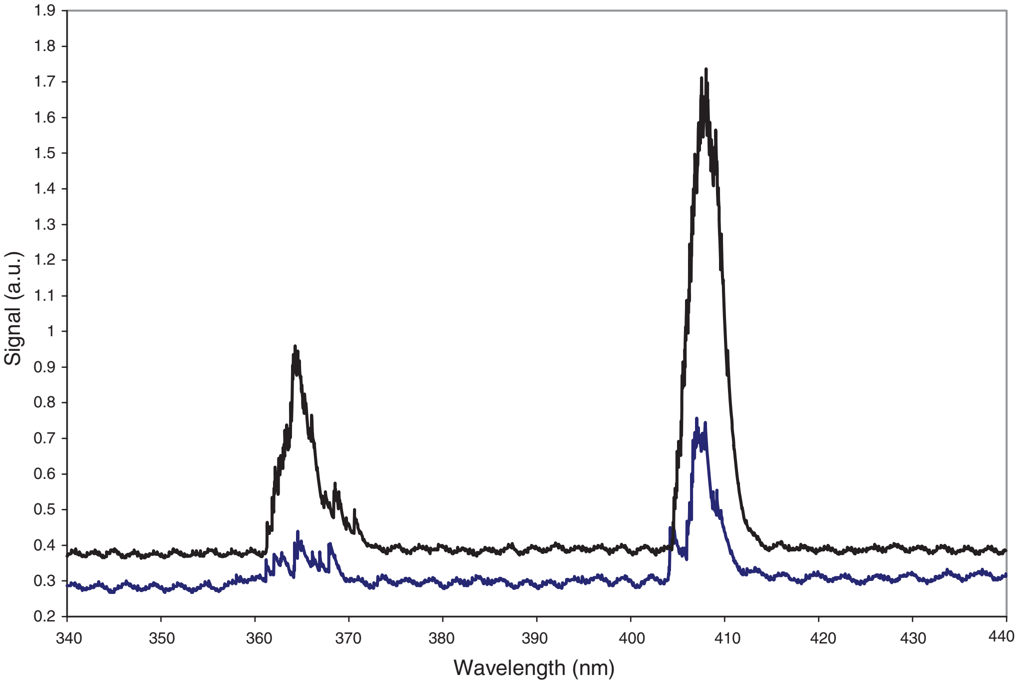

Figure 1. Fluorescence emission spectra of 10 ng mL^{-1} Pb signal (upper trace) and blank signal (lower trace) using the HG-LIF approach (laser excitation at 282.806 nm with No. 2 NDF).

3.2 *Effect of hydrochloric acid concentration on Pb laser induced fluorescence signal*

After evaluating the optimum concentrations of potassium hexacyanoferrate (III) and oxalic acid solutions for Pb hydride generation, the effect of hydrochloric acid concentration was investigated. During this part of the study, 1.0% potassium hexacyanoferrate (III) in 0.3% oxalic acid and 1.0% sodium tetrahydroborate (III) in 0.1% sodium hydroxide solutions were used for hydride generation of a 20 ng mL^{-1} Pb standard. The fluorescence signal increased and reached a maximum with increased concentration of hydrochloric acid, but after 0.055 M hydrochloric acid concentration the Pb fluorescence signal was observed to decrease sharply. These results demonstrate that the hydrochloric acid concentration is a key factor for hydride generation of Pb. A concentration of 0.055 M hydrochloric acid was used during subsequent Pb hydride generation measurements.

3.3 *Effect of sodium tetrahydroborate (III) concentration on Pb laser induced fluorescence signal*

The effect of sodium tetrahydroborate (III) concentration on the Pb fluorescence signals was investigated. Sodium tetrahydroborate (III) solutions were prepared in 0.1% (w/v) sodium hydroxide to improve stability. The results of experiments at different sodium tetrahydroborate (III) concentrations are shown in Figure 2. From these results, it was determined that 1.0% potassium hexacyanoferrate (III) in 0.3% oxalic acid with

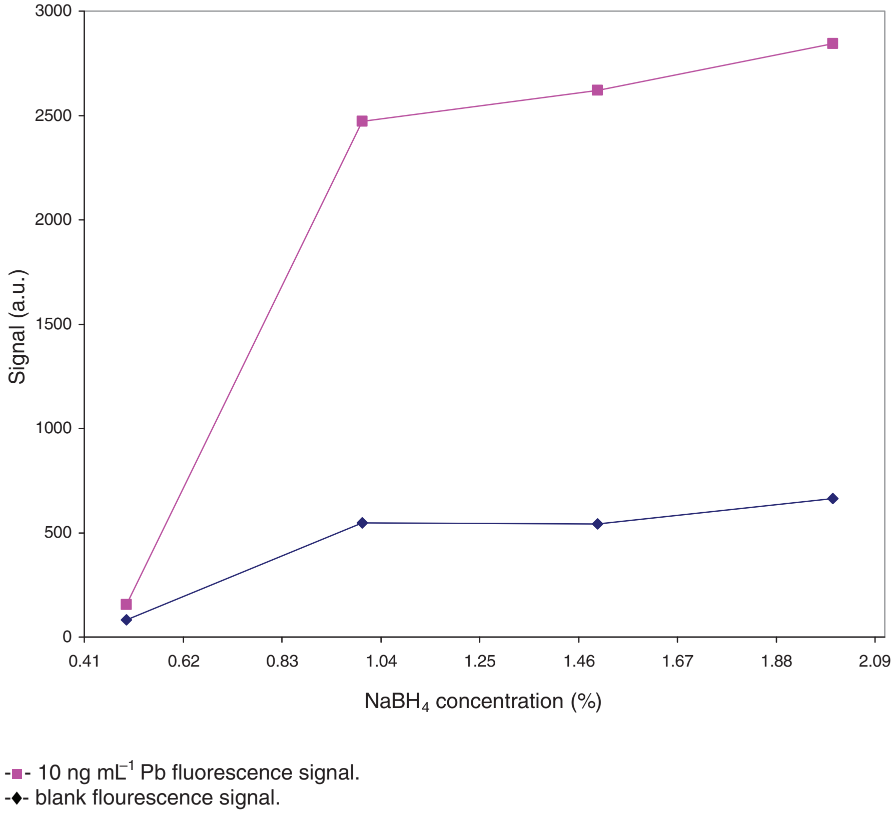

Figure 2. Effect of sodium tetrahydroborate (III) concentration on the 10 ng mL^{-1} Pb fluorescence signal and blank signal at 405.8 nm.

1.0% (w/v) sodium tetrahydroborate (III) in 0.1% (w/v) sodium hydroxide and 0.055 M HCl provided the best results for Pb HG-LIF spectrometry measurements. These results are in general agreement with those reported previously for Pb measurements by HG-based approaches [20,29].

3.4 *Analytical performance*

After evaluating the optimum conditions for hydride generation, the HG-LIF approach was used for the determination of Pb in a reference material, a commercial multielement standard solution, and surface water and sediment samples. Using the optimised conditions, the analytical figures of merit including the detection limit and precision of replicate measurements were evaluated.

The limit of detection (LOD = 3 * s/m) was determined from the sensitivity (slope of the calibration curve; m) and the noise (s), which was taken to be the standard deviation of 16 measurements of the blank. Limits of detection for Pb at 405.8 nm and 363.9 nm were 0.3 ng mL^{-1} and 0.6 ng mL^{-1}, respectively (Table 1). The limit of quantification (LOQ = 10 * s/m) was determined the same way as LOD. LOQs for Pb at 405.8 nm and 363.9 nm were 1.0 ng mL^{-1} and 2.0 ng mL^{-1}, respectively. The linear calibration curve

Table 1. Limits of detection and Linear Dynamic Ranges for Pb determinations by continuous flow Hydride Generation Atomic Spectroscopy Techniques.

Technique	LOD (ng mL^{-1})	Linear Dynamic Range (ng mL^{-1})
HG-LIF	0.3 (0.5 pg mL^{-1})[a]	0.5–50
	0.6 (1 pg mL^{-1})[a]	0.5–50
HG-ICP-AES [34,35]	6.0	6–600
HG-AFS [36]	0.014	0.02–20
HG-ICP-MS [37]	0.002	0.002–50
HG-AAS [38]	0.7	0.7–70

[a] Blank measurements with laser induced off-resonance wavelength.

ranged from 0.5 ng mL^{-1} to 50 ng mL^{-1}. Due to a considerable background signal observed for Pb in the blanks, the blank solutions were also measured with the laser tuned away (off wavelength) from the analyte wavelength at 282.806 nm which allowed a better estimate of the true blank value when no Pb was present. Although the source of the Pb background remains to be determined, it may be due to one or more of the reagents. The limits of detection obtained by HG-LIF determined off-resonance wavelength (282.806 nm) were 0.5 pg mL^{-1} and 1 pg mL^{-1} for 405.8 nm and 363.9 nm, respectively. The precision was evaluated using a solution standard containing 10 ng mL^{-1} of Pb and % RSD values obtained were in the range of 3.5% for 8 replicate measurements.

3.5 *Analysis of a multielement standard, river water and sediment samples*

Laser induced fluorescence has high spectral selectivity resulting from two dimensions of selectivity provided separately by the laser excitation source and the fluorescence monochromator. The high selectivity of the HG-LIF approach is expected to result in high measurement accuracy for Pb, even in complex sample matrices. To check the accuracy of the optimised HG-LIF method, the Pb concentration was determined in a water reference material (1643 e Trace Elements in Water by NIST) that contains low concentrations of several elements. Three sub-samples were diluted 1 : 1 and after that were analysed by the method of calibration curves. The Pb content was also determined using the method of standard additions. The results for the calibration curve method (at 405.8 nm) and the method of standard additions for Pb content in diluted water reference material were 20.44 ± 0.56 ng mL^{-1} and 20.34 ± 1.30 ng mL^{-1}, respectively. The close agreement of these two results is an indication of the reliability of the HG-LIF approach.

Determinations of the Pb concentration were also performed with the calibration curve method at two different fluorescence emission wavelengths of 364 nm and 405.8 nm and were observed to be 20.62 ± 0.60 ng mL^{-1} and 20.44 ± 0.56 ng mL^{-1}, respectively. The certified Pb concentration in the reference material is 19.63 ± 0.21 µg L^{-1}, which is in good agreement with the concentration of 20.44 ± 0.56 ng mL^{-1} as determined by the HG-LIF approach using the calibration curve method.

To further demonstrate the accuracy of the optimised HG-LIF method, the Pb concentration was determined in a multielement standard (LPC Standard 1, Spex Certi Prep P: 20 µg mL^{-1} Pb) using the HG-LIF method and a comparison ICP-AES method (Table 2). The determination of Pb in the multielement standard by the ICP-AES method was performed at 216.993 nm and 220.353 nm Pb emission lines. The limits of detection for

Table 2. Analytical results of Pb (mean $\pm$ SD, $n = 8$) in various samples determined by HG-LIF and ICP-AES methods.

Sample	HG-LIF Measured value	ICP-AES Measured value	Certified value
1643 e Trace Elements in Water SRM	$20.44 \pm 0.56\,\text{ng mL}^{-1}$		$19.63 \pm 0.21\,\text{ng mL}^{-1}$
LPC standard 1	$20.44 \pm 0.67\,\mu\text{g mL}^{-1}$ $19.88 \pm 0.70\,\mu\text{g mL}^{-1}$	$20.55 \pm 0.86\,\mu\text{g mL}^{-1}$ $20.50 \pm 0.65\,\mu\text{g mL}^{-1}$	$20\,\mu\text{g mL}^{-1}$
Sediment	$6.37 \pm 0.41\,\text{ng mL}^{-1}$ $316.60 \pm 20.37\,\mu\text{g Pb/g}$	$5.04 \pm 0.16\,\mu\text{g mL}^{-1}$ $500.59\ 15.89\,\mu\text{g Pb/g}$	Not certified

Pb at these two emission wavelengths were $0.04\,\mu\text{g mL}^{-1}$ and $0.02\,\mu\text{g mL}^{-1}$, respectively. The determination of Pb in the multielement solution using the ICP-AES approach at 216.993 nm and 220.353 nm resulted in concentration values of $20.55 \pm 0.86\,\mu\text{g mL}^{-1}$ and $20.50 \pm 0.65\,\mu\text{g mL}^{-1}$, respectively.

The Pb amount determined in the multielement standard by the HG-LIF calibration curve method at 363.9 nm and 405.8 nm was $20.44 \pm 0.67\,\mu\text{g mL}^{-1}$ and $19.88 \pm 0.70\,\mu\text{g mL}^{-1}$, respectively. The HG-LIF results show fair agreement at both fluorescence wavelengths and with the amount determined by the ICP-AES method. In addition to showing consistent results with the ICP-AES method, the HG-LIF method for Pb shows no interference from other hydride forming elements such as As, Sb, Se, and Sn that are present in the ICP mix standard. This suggests that the method has good feasibility for Pb determinations in samples containing these and other elements.

In order to demonstrate an application of HG-LIF technique to more complex samples, the Pb contents of surface waters and a sediment sample were measured. The sediment sample is from the Mahoning River near Youngstown, Ohio that has been contaminated in the past by high levels of metals, polyaromatic hydrocarbons and volatile organic compounds [30]. Due to the high levels of contamination present, these sediments are under investigation for remediation and any such activities will require accurate chemical monitoring of the contaminant levels in order to demonstrate remediation [31].

The Pb concentration in the sediment sample was determined by HG-LIF at a fluorescence wavelength of 405.8 nm using the calibration curve and standard addition methods. The results were $6.37 \pm 0.41\,\text{ng mL}^{-1}$ Pb and $6.18 \pm 0.60\,\text{ng mL}^{-1}$ Pb in the diluted sample solution using the calibration plot and standard addition methods, respectively, and showed excellent agreement to each other. The final concentration of Pb in the undiluted sample digest solution was determined by the HG-LIF calibration curve method to be $3.166 \pm 0.020\,\mu\text{g mL}^{-1}$, which corresponded to a mass concentration of $316.60 \pm 20.37\,\mu\text{g Pb/g}$ in the dry sediment. This is consistent with the range of Pb concentrations in the sediment that have been reported previously [30,32].

For comparison purposes, the acid digested sediment and method blank were analysed for Pb content by the ICP-AES method. The final concentration of Pb in the undiluted sample solution as determined by the ICP-AES approach was $5.04 \pm 0.16\,\mu\text{g mL}^{-1}$ and $7.39 \pm 0.11\,\mu\text{g mL}^{-1}$ at emission wavelengths of 220.353 nm and 216.993 nm, respectively. Concentration of Pb in the sediment was determined by the ICP-AES method (at 220.353 nm) to be $5.04 \pm 0.16\,\mu\text{g mL}^{-1}$ corresponding to $500.59 \pm 15.89\,\mu\text{g Pb/g}$ in the dry sediment. Sediment Pb content was determined by the ICP-AES method (at 216.993 nm) to be $7.39 \pm 0.11\,\mu g\,mL^{-1}$ corresponding to $734.99 \pm 11.00\,\mu g$ Pb/g in the dry sediment. As these results indicate, the Pb level in the sediment analysed by the ICP-AES method is not consistent at the two different wavelengths and is almost two times greater than that determined by the HG-LIF method.

The higher levels determined by the ICP-AES method are believed to be the result of elevated concentrations of spectrally interfering elements in the sediment samples, particularly Fe [33]. According to previous chemical assessments of the river sediment from nearby locations, several elements are present at high levels including Fe, Al, Cu and Cr [30]. The ranges of sediment concentrations for Fe, Al, Cu and Cr are 110,000–245,000 mg/kg, 7550–15,200 mg/kg, 91–210 mg/kg, and 84.4–142 mg/kg, respectively [30]. The two Pb emission wavelengths and the potentially interfering emission lines from other elements such as Fe, Cu(I), Al(I), and Cr(II). It is noteworthy that the highest Pb concentration is observed for measurements performed at 216.993 nm, which is almost directly overlapped by two Fe emission lines at 216.95 nm and 217.03 nm. Due to the very high levels of Fe present, it is reasonable to expect that spectral interferences may occur in these measurements resulting in higher observed values.

In this case where the samples are contaminated sediments that have high metal content, the high selectivity and high sensitivity of LIF detection provides important advantages over the ICP-AES approach. The high selectivity of the HG-LIF approach is suggested by the close agreement of the two determinations of the sediment by the calibration curve and standard addition methods, which also suggests high accuracy for the HG-LIF method. This is in contrast to the two different results obtained using the ICP-AES approach that is known to suffer from spectral interferences from elements like Fe, which is present at high levels in the sediment sample. Furthermore, the high sensitivity of the LIF detection allows a relatively simple procedure to be used where the digested samples can be diluted so that the effects of potential spectral or chemical interferences are reduced.

River and surface water samples collected from the Mahoning River (samples from 2 locations), and Mill Creek and Yellow Creek near Youngstown, Ohio, were also analysed by the HG-LIF and ICP-AES methods. The Pb levels in all of the river water samples were below the LOQ for both the ICP-AES and HG-LIF methods. These results are in agreement with previous chemical measurements of the river water where the Pb concentrations were reported to be below the detection limit [30].

4. Conclusions

Hydride generation sample introduction combined with laser induced fluorescence detection can be used to determine Pb at trace levels in different sample solutions. This is the first report of the HG sample introduction technique combined with LIF detection for trace determinations of Pb. The effects of potassium hexacyanoferrate (III), hydrochloric acid and sodium tetrahydroborate (III) concentrations on the efficiency of Pb hydride generation have been evaluated. Interferences from other hydride forming elements have not been specifically investigated. However, no interference effects were observed in any of the sample solutions that were known to contain multiple elements, including other hydride forming elements. The close agreement of the determinations of the concentrations of Pb in a contaminated sediment sample by the standard addition and calibration curve methods demonstrates the consistency and reliability of the HG-LIF approach for trace level Pb determinations. The HG-LIF results have been compared to those obtained using an ICP-AES approach and are believed to demonstrate the advantages of the HG-LIF approach in the analysis of a complex sample material where the potential for interferences is high. The results of these studies indicate that a relatively modest laser system combined with hydride generation sample introduction provides good analytical performance in terms of sensitivity, precision and selectivity and can be used to determine Pb reliably at ng mL^{-1} levels.

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