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lake waters by laser-excited atomic fluorescence
spectrometry

By:

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**DIRECT DETERMINATION OF DISSOLVED AND TOTAL THALLIUM
IN LAKE WATERS BY LASER-EXCITED ATOMIC
FLUORESCENCE SPECTROMETRY**

by

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MANAGEMENT PERSPECTIVE

The determination of ultratrace metals in environmental samples by conventional methods requires resource-consuming and contamination-prone preconcentration and separation steps. Under the "Great Lakes Prevention Initiative", Canada's Green Plan calls for the development of "New Technologies" and an increase in "Analytical Capabilities". To meet this challenge, we have developed an ultrasensitive instrument, the Laser-Excited Atomic Fluorescence Spectrometer (LEAFS) which enables direct, accurate trace metals determination, Lead being a prime example. Thallium (Tl) is even a better example. Even though Tl is a very toxic priority pollutant (more toxic than Pb), there has been no reported Tl data for Great Lakes waters. A plausible explanation is that there has been no method sensitive enough to detect it, thus no data nor interest in it. Indeed, Thallium has been a neglected element in environmental studies.

This paper describes the first LEAFS method for direct determination of dissolved and total of Tl in Great Lakes waters. Sub-ppt level detection has been achieved and is more than adequate to analyse the waters as is. Laborious tasks for sample preconcentration and acid digestion are no longer necessary. In lake waters Thallium appears to be predominantly in dissolved form.

ABSTRACT

Thallium is a highly toxic, under-studied priority element. However, it has recently created much interest due to afresh and rapid improvements in detection limit. It appears that there are no published Tl data for Great Lakes waters, likely due to the poor sensitivity of classical methods. An electrothermal Laser-Excited Atomic Fluorescence Spectrometer has been optimized to detect sub-femtogram of thallium and used to develop a method for direct determination of dissolved and total thallium in lake waters. The method voids the labor-intensive, contamination-prone tasks of filtration, centrifugation and acid digestion of collected particulates. Adequate precision and recoveries were achieved using several lake waters (undigested and digested) and a certified reference material. The concentration of Thallium in the acidified (0.2 % HNO₃) Milli-Q-Water was monitored over a period of four months and averaged 0.02 ± 0.01 ng/l. The concentration in Hamilton Harbor ranged from 3 to 48 ng /l. The mean of dissolved / total fraction of Tl in the Harbor water was 80 %.

Keywords: Laser-Excited Atomic Fluorescence Spectrometry, LEAFS, Thallium, *in situ* known addition, Great Lakes, Hamilton Harbor, direct determination, clean room.

INTRODUCTION

Thallium and compounds is one of the thirteen priority pollutant metals listed along with lead, cadmium, and mercury by the United States Environmental Protection Agency [1]. Chemically, it is an interesting element as it exhibits both the properties of alkali metals and those of heavy metals. This dual character has earned it a nickname of paradoxical, enigmatic metal. Although the average Tl content in the earth's crust is only 6×10^{-5} weight %, it is higher than some of the more commonly studied elements such as Pb, Cd or Bi [2].

The element and its compounds are very toxic, its toxicity being reported soon after its discovery in 1861 [2-8]. Tl-based rodenticides were extensively used but had to be discontinued due to its high toxicity. An annual average of 13 thallium poisoning cases were reported between 1958 and 1964 in Canada [3]. Twenty four percent of bald eagles found sick or dead in 18 states during 1971-1972 were poisoned by Tl [9]. "Thallium is neither essential nor stimulatory in either man or animals. It is the most highly toxic cumulative cation" [6]. Thus it is important to determine thallium accurately.

Thallium is used to manufacture alloys, electrical devices, dyes, fireworks, special optical glass, as a dopant for nuclear spectrometer crystals, depilatory agents, fungicide, ant bait or rodenticide. One of the most important alloys is Pb alloy (20-65% Tl) which is harder and more corrosion-resistant than pure lead [2]. However, when compared to Pb, Hg, Cr, Cu, Ni or Zn for example, Thallium has limited industrial uses. So economically, Thallium is hardly important thus scarcely recovered from metal-based mining, ore-processings or smelting operations. It is readily disposed into the environment. High levels of Tl relative to the two commonly studied toxic elements Pb and Cd were recently found in several water samples from a region of abandoned mine tailings at Wells, B. C. [10]. In mining industry, the usual wastewater treatment to remove heavy metals cannot remove Thallium (I). Thus Tl usually ends up in the tailings and abounds in the disposal sites. Beside these point sources, the air emissions from coal-burning power plants form the largest collective source of Thallium discharge into the environment [11].

Thus, since Tl is a very toxic element and has received minimal attention relative to other trace metals, it is obvious that Thallium has been badly neglected in environmental studies.

An explanation for fewer Tl studies is that Tl is often undetected by classical analytical methods, which normally have poorer sensitivity towards Tl than other trace elements. Being a hard-to-detect, "unwanted", paradoxical and very toxic element, Thallium is an interesting and important element to study. In fact there is an increasing number of publications dealing with Tl determination in environmental samples going hand in hand with improved instrument sensitivities in for example Potentiometric Stripping Analysis [12], Inductively Coupled Plasma Mass Spectrometry (ICPMS) [13-15], or Laser-Excited Atomic Fluorescence Spectrometry (LEAFS) [15-17]. Axner et. al. [17] used an excimer laser - based LEAFS for water analysis achieving a detection limit of 1 ng Tl/l. There are other LEAFS papers dealing with Tl determination in substrates beside natural water samples [18-22].

The lack of any historical Tl data in the Great Lakes waters compared to the large amount of data for other trace metals is a prime example of very few Tl studies. Up to now, we have no method for Tl determination in Great Lakes waters. No method means no data, no interests. In this paper we adapt a recently developed *in situ* known addition technique [23] to develop a simple method for direct determination of dissolved and total Tl in Great Lakes waters using a Copper Vapor Laser - based LEAFS. Thallium concentration lower than 0.03 ng/l (0.6 fg absolute) can be directly detected. The direct determination of total Tl in unfiltered samples is especially beneficial to analysts as it voids the conventional painstaking tasks of filtration / centrifugation followed by acid digestion of collected particulates. Also, a discussion on the dissolved Tl / total Tl ratio and the distribution of Tl found in Hamilton Harbor water are presented.

EXPERIMENTAL

Laser-Excited Atomic Fluorescence Spectrometry

The details of the spectrometer have been described elsewhere [24-25]. Some essential features are given here. The 511 nm line of a Copper Vapor Laser (Metalaser Technologies MLT20) was used to optically pump a Rhodamine 575 dye laser (Laser Photonics). The dye laser output (554 nm) was then frequency-doubled by a second harmonic generator (Autotracker II, Inrad Inc.) to give the 276.79 nm UV light. This light, directed through a pierced mirror into a graphite furnace (Perkin-Elmer HGA 2100), was used to excite Tl atoms generated in the furnace. The Stokes direct-line fluorescence light (352.94 nm) emitted by the excited atoms was collected and measured via a narrow bandpass filter (1 nm) - monochromator- photomultiplier-boxcar system. A six kilohertz repetition rate was used. Dye laser tuning was achieved using a Thallium EDL lamp as was detailed earlier [26].

Chemicals and sample handling

Ultrapure chemicals were used. Milli-Q Water acidified to 0.2 % with ultrapure (Seastar) nitric acid, simply referred to as MQW, was used as standards matrix and carrier. Sample handling was carried out in a class 100 clean room and in a class 100 laminar flowhood (Microzone Corporation). Filtration was done using membrane filter with 0.45 μ m pore size. Details of labware, cleaning procedure and sample collection have been described earlier [27]. In spite of very careful sample handling during sample injection into the furnace, some contamination from the surrounding air is expected since the LEAF spectrometer is located in an ordinary laboratory. However, this contamination effect was found to be minimal.

In situ known addition analysis

A programmable micropipette (Rainin Instrument Co.) was used to carry out *in situ* known addition pick-ups and injections into the graphite furnace [23]. The sequence 20/5/2/5 (20 μ l of MQW carrier followed by 5 μ l of sample, 2 μ l of air spacer, and 5 μ l of MQW or standard) was found optimal, but the sequence 20/10/2/10 was sometimes used for extremely low

concentration samples (low and sub-ng/l). Standard concentrations used varied from 0.1 to 50 ng/l and all computations utilized peak height readings.

Digestion for total metals

The National Laboratory for Environmental Testing 's digestion procedure for determination of total metals was used [28] and is briefly summarized here. Evaporate 100 ml of unfiltered water sample in a quartz volumetric flask to near dryness. Add 2 ml of concentrated HNO₃ and evaporate to near dryness. Add 1 ml of 50 % of concentrated HCl and evaporate to almost dryness. Fill the flask back to 100 ml with ultrapure water. For LEAFS work, 20 ml (not 100 ml) of sample was used and all the reagents were proportionally adjusted.

RESULTS AND DISCUSSION

LEAFS and furnace optimization

LEAFS optimization has been described elsewhere [24-25]. Uncoated graphite tubes had to be used to provide adequate rise time, the coated ones or platforms giving too slow a rise time for the Perkin-Elmer 2100 furnace. For this purpose also, the atomization temperature was set at 2400 °C for 3 s unramped instead of the recommended 2100 °C. The drying and ashing temperatures were set at 120 °C for 40 s ramped and 400 °C for 40 s ramped, respectively. Argon was found to be a better purge gas than Ar-H₂ mixture giving a superior fluorescence intensity by a factor of about 3. The gas flow was interrupted during atomization. A standard calibration curve starting with 0.1 ng/l is shown in Figure 1. The linearity extends to 100 µg/l, which is a six orders of magnitude of linear dynamic range. The curvature starts at about 200 µg/l.

Effect of sample matrix

Thallium signals are suppressed by the matrices of natural samples so that analysis by standard calibration curve is likely unreliable. Since the use of a matrix modifier or other mean of sample pretreatment was deemed undesirable as it may increase background and blank signals,

a recently developed *in situ* known addition technique was relied upon [23]. Efforts to find an optimum sample volume to be used for lake waters failed to show any common denominator, such as a maximum observed in case of seawaters, except a steady increase (with slight suppression) in signals with increase of sample volume used. Each water has its own slope. A normalized behavior of this increase for lake waters is shown in Figure 2 along with that for standard and for seawaters showing a common maximum response [23]. It can be seen that any volume of lake water may be optimally used in this known addition technique. A 5 μ l of sample was found suitable for most waters and used in the sequence of 20/5/2/5 described above.

The sample acid content also affects Tl responses as seen in Figure 3, showing signal dependence on % HNO₃ using 10 ng /l of Thallium. Without acidity-matching, the traditional standard calibration curve will bias the results even at low acid content (Fig. 3) whereas the *in situ* known addition technique will effectively compensate for this acid effect.

Background signals produced by sample matrices may interfere with analyte fluorescence responses [21] and were determined as follows. Signals for standards and filtered or unfiltered natural samples were obtained at ± 0.05 nm away from the analytical line and were found insignificantly different from one another. Also they are statistically the same as that of MQW, which is extremely small.

Figures of merit

More than forty different analyses of 20 μ l of blank (MQW acidified to 0.2% HNO₃) were made during a period of 4 months. The mean value was found to be 0.02 ng Tl/l with a standard deviation of 0.01 ng/l, which according to IUPAC definition corresponds to a detection limit of 0.03 ng/l (0.6 fg absolute). Other outstanding detection limits have been reported earlier: 0.7 fg by Falk et. al. [18], 0.3 fg by Michel et. al.[29] and 0.1 fg by Smith et. al.[30]. Further improvement to our system can be achieved by for example using a pierced ellipsoidal mirror instead of a pierced first surface mirror coupled with the usual focusing lenses, thus minimizing the fluorescent light losses caused by aberrations and optical alignment [31]. (Vera et. al. [32], however, obtained a slightly poorer detection limit when they used an off-axis ellipsoidal mirror). Also, according to Wei et. al. [29], if only the narrow bandpass filter is used without the

spectrometer the detection limit will further improve. But presently there is no requirement for us to further improve the detection limit. A practical detection limit was determined as we usually do by making several replicate analyses of a natural sample containing a concentration 5-10 times greater than the lowest concentration that can be detected [33]. In our case this concentration can be easily 0.01 ng/l. We used a sample containing 0.25 ng Tl/l and made 12 replicate analyses giving a standard deviation of 0.05 ng/l, which results in an upper, working detection limit for the method of 0.1 ng/l.

Figure 4 shows actual data of sixteen groups of replicate analyses of various samples, indicating that very precise results can be achieved. The relative standard deviation ranges from 1 to 8 % for these groups of analyses, with an average of 4% for the whole set of data.

The accuracy of the method was demonstrated by several recovery tests using a NIST certified reference material (the only one available with a Tl value) and six different natural samples related to lake waters. Table 1 summarizes the test results, which show that the values obtained by direct analysis agree well with the certified value and those derived from the multiple standard addition technique (MSA). Three levels of concentrations overlapping the concentration originally present in each sample were used in the MSA. The percent recoveries were also calculated and given in Table 2 showing acceptable recoveries, which are well within $100 \pm 10\%$.

Total and dissolved Thallium

To further ensure that the direct determination of Tl in unfiltered samples represents total amount, a proven digestion procedure [28] was used to digest unfiltered samples followed by LEAFS analysis. Table 3 shows good agreement between the results for undigested and digested samples, which indicates that the direct analysis of unfiltered samples gives total Tl values. The blank values were negligible. As additional tests, the unfiltered samples were also spiked, digested and analysed. The 4th column of Table 3 give these results, which indicate complete recoveries of original total and spiked amounts. The analytical results for filtered samples refer to dissolved concentrations of Thallium. The knowledge on the dissolved and total fraction of a metal in natural waters is very useful for bioavailability / toxicity studies.

Thallium distribution in Hamilton Harbor water

Figure 5 shows the location of Hamilton Harbor with respect to some key sites such as Stelco and Dofasco steel companies and our laboratory (CCIW). Van Dorn bottle was used to collect water samples from ten different stations selected to give a fair representation of the whole Harbor. Field blanks were obtained as follows. Right before and after sample collection, one liter of doubly deionized distilled water was used to rinse the Van Dorn bottle, and the rinsing solution was saved and used as blank. Four such blanks were collected and analysed giving an average of 0.22 ± 0.06 ng Tl/l. (The concentration of doubly deionized distilled water was $\ll 0.2$ ng Tl/l). Filtration was done in class 100 clean hood, and the blank concentration was as low as the concentration of the ultrapure water itself.

The distribution of total Tl in the Harbor's water is shown in Figure 5. Average concentrations of 38 ± 7 ng/l and 30 ± 4 ng/l were found respectively at 1 m from the surface and at 1 m from the bottom of the main water body (7 deep stations in the middle). This amounts to a 27% concentration difference between the top and bottom water. Also the stations with the highest surface concentrations are near the steel companies and La Salle Park, where numerous recreation activities take place daily.

Furthermore eight different water samples were subdivided then filtered, analysed and the results shown in Fig. 5 along with the unfiltered ones. An average of 30 ± 5 ng Tl/l for the top water and 23 ± 2 ng Tl/l for the bottom water was observed, giving a concentration difference similar to that for the unfiltered samples. Thus the dissolved fraction with respect to total Tl ranges from 71% to 92% with an overall mean of $80 \pm 8\%$, which is much higher than Pb dissolved fraction (work in progress).

Conclusion

A LEAFS method has been developed for direct determination of dissolved and total thallium in lake waters. It is a superior alternative to the conventional indirect method for determination of total metal which involves filtration / centrifugation and acid digestion. The LEAFS method is also ideal for small-volume samples as it is ultrasensitive. It appears that the particulate fraction of Tl in lake waters is small compared to Pb's.

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Table 1. Comparison of analytical results (ng/l) determined by direct and MSA analyses of SRM and lake-related waters (n > = 6).

Sample*	Values by direct analysis	MSA Values ^a	Certified value
NIST SRM 1643c	4.18 ± 0.36	N. A.	(3.95) ^b
Hamilton Harbor nearshore, uf	32.71 ± 1.74	33.46	N. A.
Macfarlane Lake - 4m, f	7.48 ± 0.60	7.39	N. A.
Macfarlane Lake - 4m, uf	7.62 ± 0.65	7.59	N. A.
Lake Ontario nearshore, uf	7.47 ± 0.42	7.73	N. A.
Burlington Tapwater, uf	3.98 ± 0.29	4.32	N. A.
Lake Ontario - 33m, f	6.42 ± 0.35	6.25	N. A.

* Hamilton Harbor is a harbor of the Great Lakes; Macfarlane is a lake in Sudbury mining area in Northern Ontario; Lake Ontario is one of the Great Lakes; uf = unfiltered; f = filtered.

^a MSA = Multiple Standard Addition; N.A. = not applicable.

^b Value calculated after a 2000-fold dilution.

Table 2. Summary of % recoveries for natural samples related to lake waters

Sample*	Average % recovery	No. of determinations
Lake Ontario nearshore, uf	100 ± 5	10
Burlington tapwater, uf	102 ± 5	9
Lake Ontario - 33m, f	102 ± 5	9
Hamilton Harbor nearshore, uf	101 ± 3	8
Macfarlane Lake - 4m, f	100 ± 4	7
Macfarlane Lake - 4m, uf	100 ± 4	6

* Same footnote as in Table 1

Table 3. Comparison of total Tl, ng/l, determined from direct injection of undigested and digested unfiltered samples, and recovery of digested spiked-samples (mean \pm sd, n > = 6)

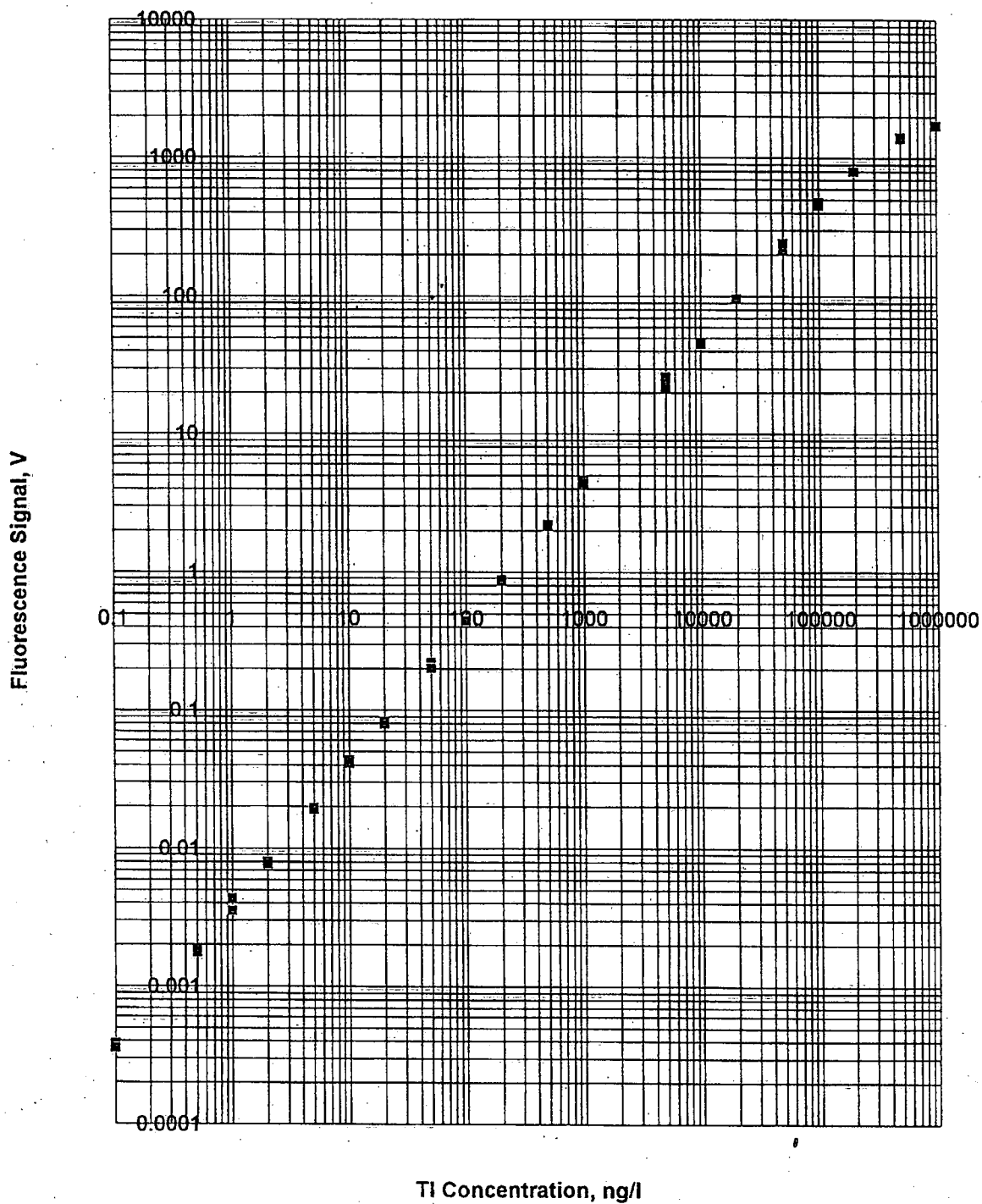
Sample	Undigested	Digested	*Total recovery of Digested Spiked-sample
Lake Ontario nearshore, uf	7.47 \pm 0.42	7.57 \pm 0.68	^a 17.66 \pm 1.15
Hamilton Harbor nearshore, uf	32.71 \pm 1.74	32.32 \pm 4.08	^b 52.41 \pm 3.86
Lake Erie - 22-10 m, uf	9.18 \pm 0.42	8.92 \pm 0.64	Not sufficient sample

* Unfiltered sample was spiked^{a,b} and then digested

^a 10 ng/l was spiked to Lake Ontario nearshore, uf, resulting in total % recovery of 101%

^b 20 ng/l was spiked to Hamilton Harbor nearshore, uf, resulting in total % recovery of 99%

Figure 1. Thallium Standard Calibration Curve



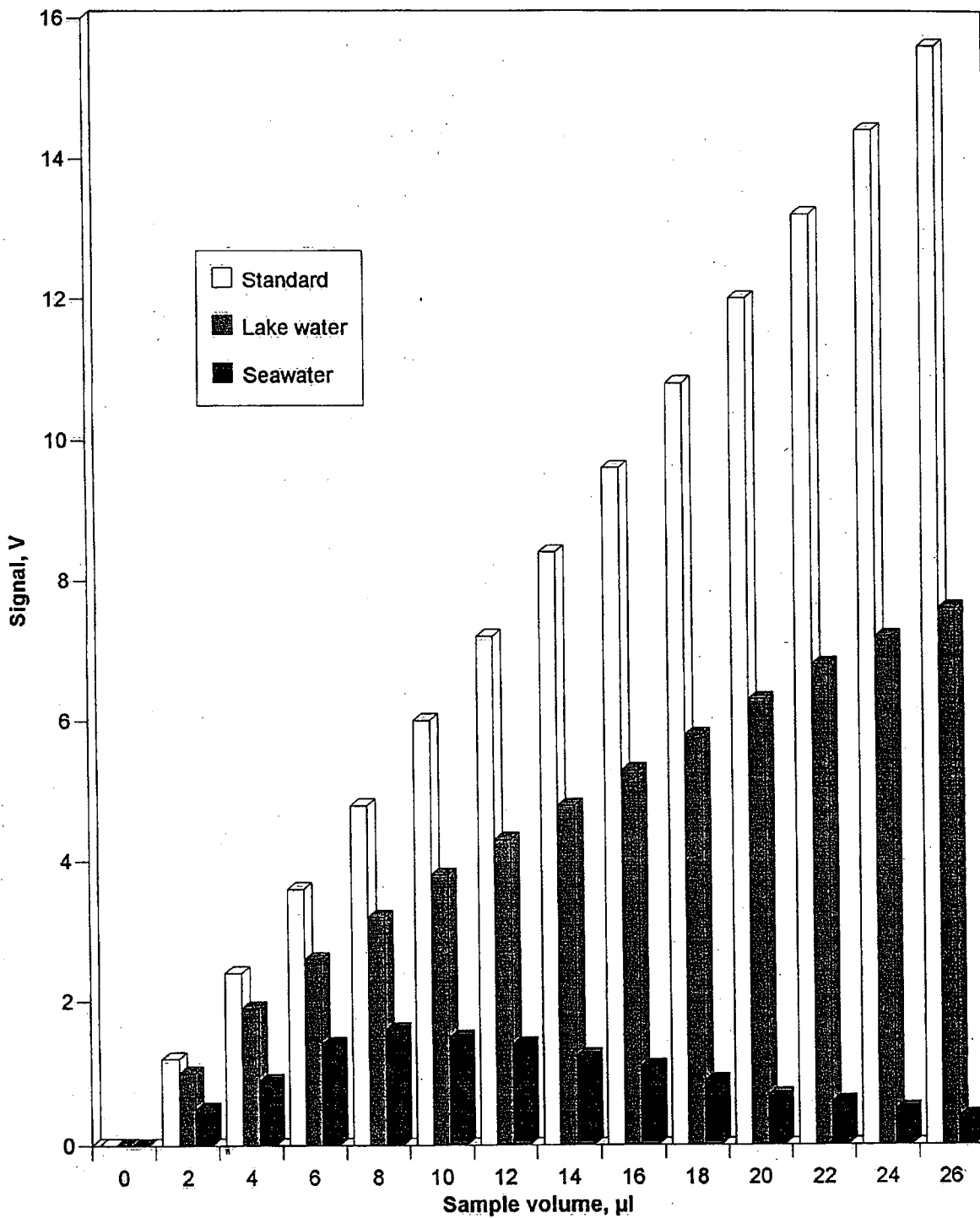


Figure 2. Signal dependence on sample volume in a constant injection volume of 26 μl (sample+MQW=26 μL)

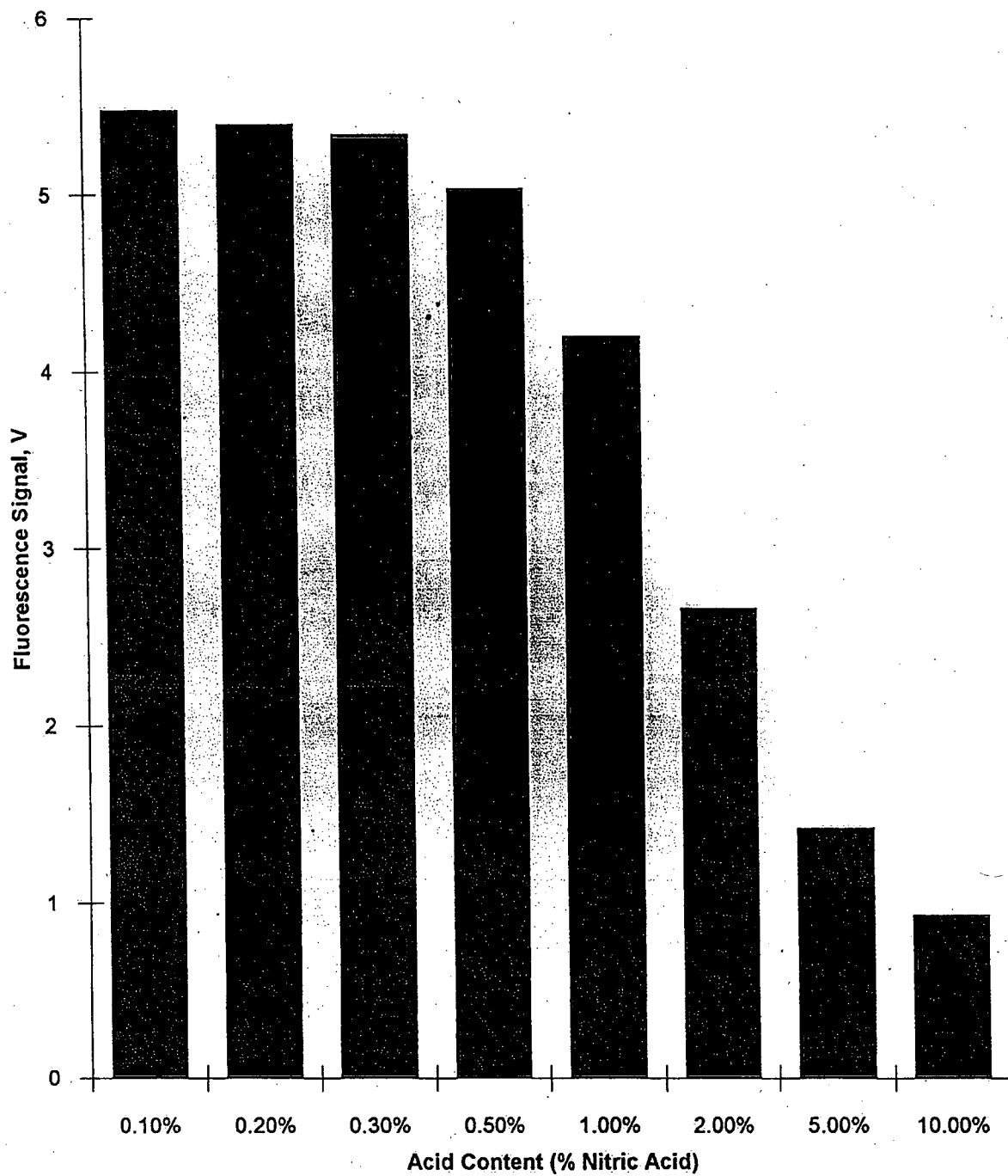


Figure 3. TI Signal Dependence on Acid Content

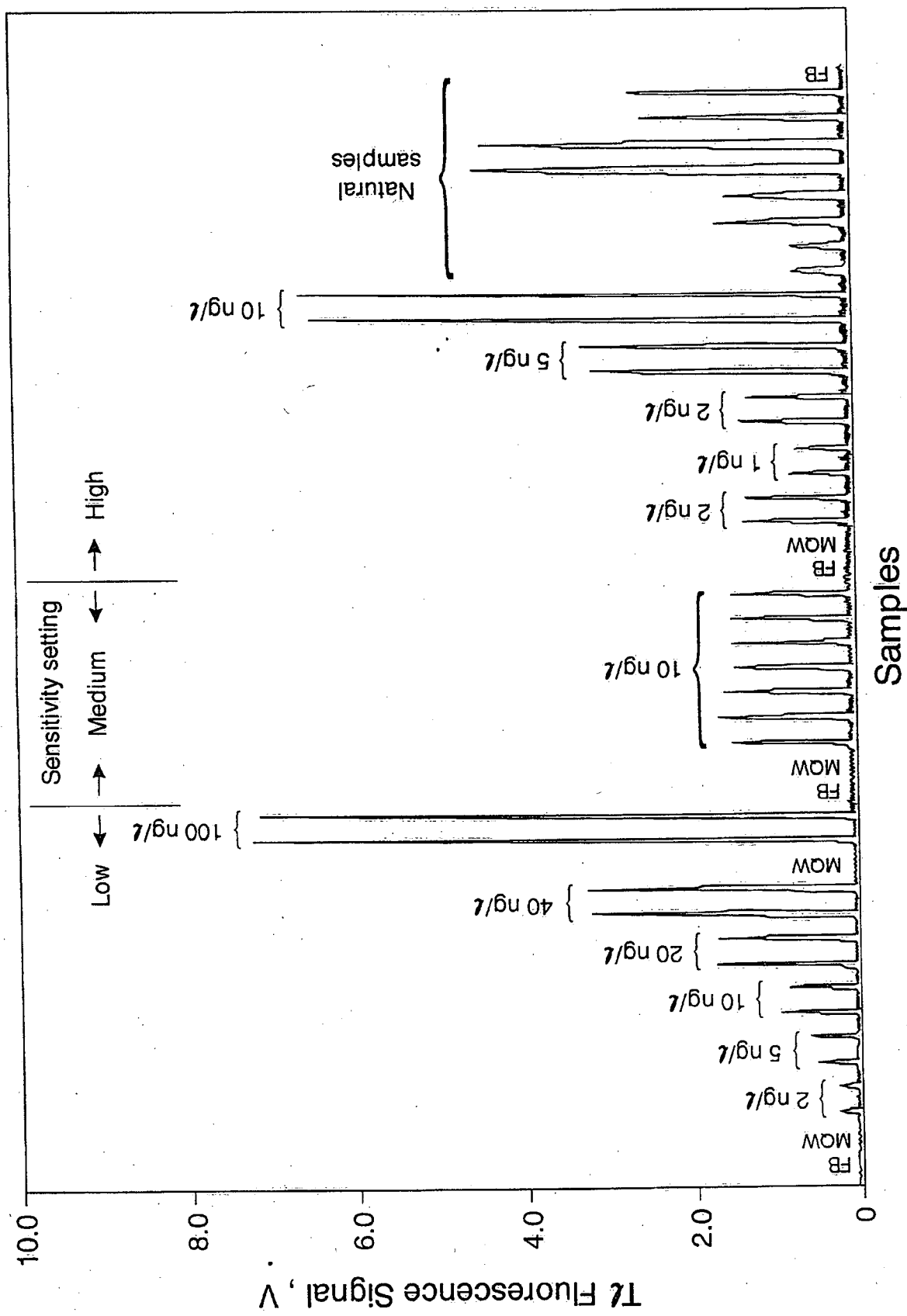


Figure 4. Replicate fluorescence responses of various samples.
 (FB= Furnace blank; MQW= 0.2% HNO₃ Milli-Q water blank)

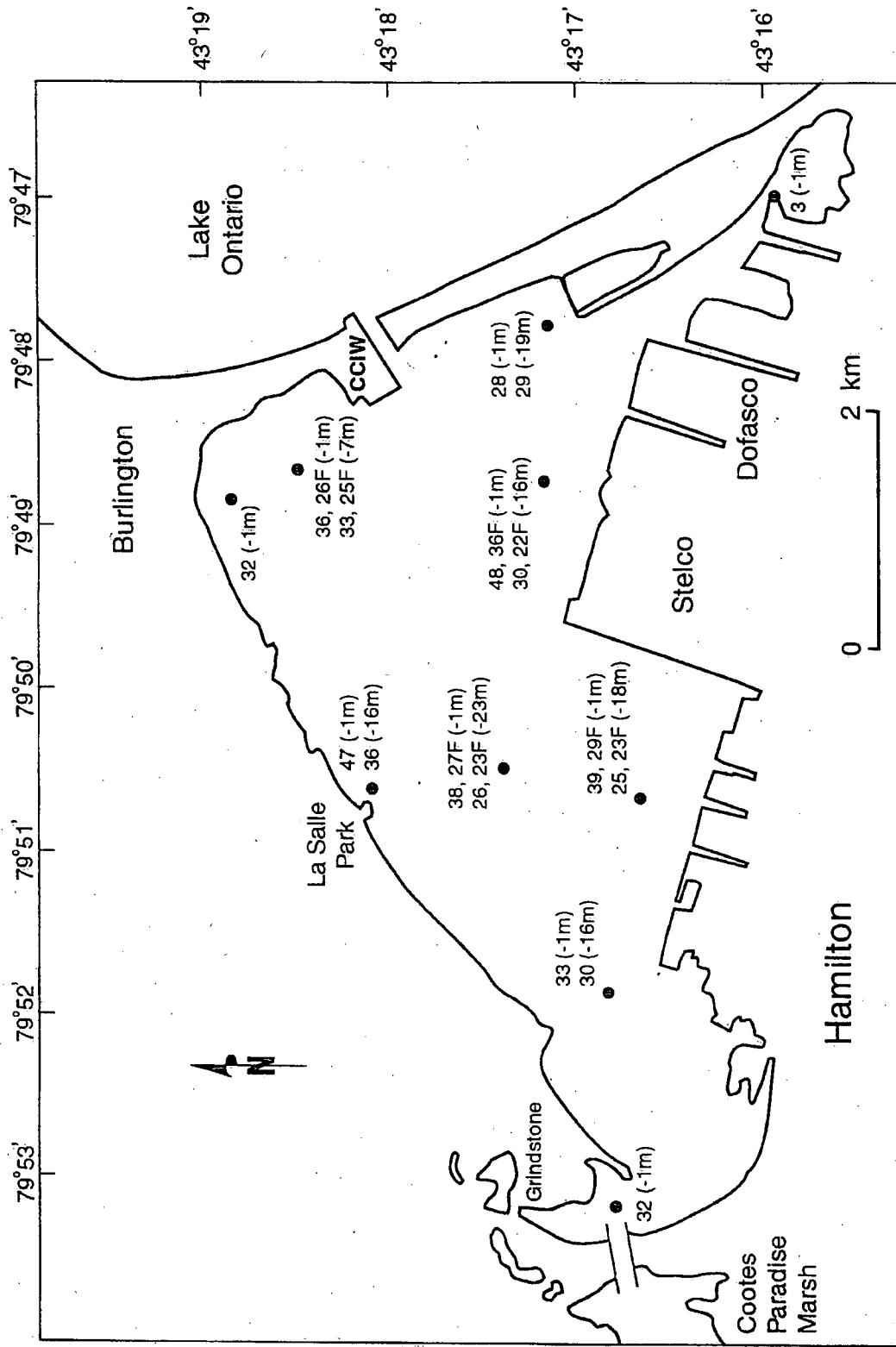


Figure 5. Thallium distribution in Hamilton Harbour water.
 38, 27F (-1m) = 38ng/L total Tl, 27ng/L dissolved Tl, (1m deep)

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