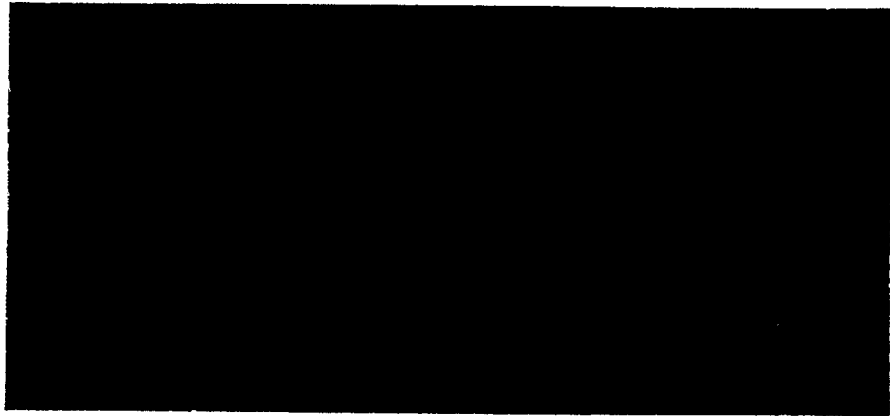


WATER RESEARCH

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DETERMINATION OF ORGANOTINS IN NATURAL WATERS BY
TOLUENE EXTRACTION GRAPHITE FURNACE AAS

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SUMMARY

The determination of tributyltin in natural waters by toluene extraction and graphite furnace AAS detection of tin has been investigated. The effect of pH on the extraction of mono-, di-, tributyltin and triphenyltin, has been examined and the optimum conditions for the estimation of tributyltin assessed. Investigation of graphite furnace conditions has shown that AAS performance is greatly improved by using furnace tubes pretreated by soaking in sodium tungstate solution. Such pretreatment is essential if low detection limits are to be attained. On extraction from aqueous media, a marked signal enhancement effect was observed (irrespective of furnace tube type) which varied according to the nature of the aqueous solution. The enhancement effect is believed to result from water in the toluene extract activating the tube surface.

Methods for the estimation of tributyltin in waters that are appropriate for screening samples as part of routine monitoring programmes, are described. Using a 1 litre sample, limits of detection (based on 4.65 times the within-batch standard deviation of the blank) of below 4 ng Sn/l were attained. Relative standard deviation on six replicate analyses of seawater containing 170 ng Sn/l of tributyltin was 1.5%.

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1. INTRODUCTION

Tributyltin (TBT) is the active biocidal constituent of many current antifouling preparations used to coat the hulls of boats. Much concern has recently been expressed over the potentially harmful effects of tributyltin to many forms of marine life, even when this substance is present at low ng/l concentrations. For instance, lethal concentrations as low as 100 ng TBT/l for shellfish (DoE 1986) have been reported, and damaging sub-lethal effects, such as shell thickening in the oyster (DoE 1986, Cleary and Stebbing 1987) and imposex in the dog whelk (Bryan et al 1986), are known to occur as a result of exposure to TBT concentrations in the 2 to 50 ng TBT/l range. Concern is highest in areas having a high density of pleasure craft, where the boats are moored for long periods of time and the circulation of water is often restricted. As a result, the UK Government has proposed a provisional ambient water target concentration of 20 ng TBT/l for waters (DoE 1986). Lower water quality standards are being considered. Sensitive and accurate analytical methods are therefore required for routine monitoring purposes that have detection limits in the low ng/l range.

Several analytical methods have been developed for the determination of butyltin compounds in waters that involve extraction, derivatisation, gas chromatographic separation and selective detection using flame photometry (Maguire and Huneault 1981, Mathias et al 1986, Muller 1987), atomic absorption spectrometry (Andreae and Byrd 1984, Randall et al 1986, Hodge et al 1979) or mass spectrometry (Muller 1984, Meinema et al 1978). Whilst these techniques are capable of attaining the required low ng/l detection limits, a considerable drawback to their use is their complexity and low sample

throughput. For routine monitoring purposes, much simpler and more rapid methods are desirable.

Several studies have made use of a relatively simple method for the determination of organotins, involving liquid extraction into a neutral organic solvent such as toluene and determination of the extract's tin content by graphite furnace AAS (M&T Chemicals 1976). Despite its reported use in TBT monitoring studies (Cleary and Stebbing 1985, 1987, Waldock and Miller 1983, Bryan et al 1986) there is little information available in the open literature regarding optimisation of analytical conditions and data on typical analytical performance.

In this report are the results of our evaluation of solvent extraction GFAAS methods for tributyltin analysis. The work had the aim of developing a simple method appropriate for routine monitoring purposes with low ng/l detection limits. This included an assessment of interferences arising from the co-extraction of other organotins that may be present in waters: triphenyltin and mono- and dibutyltin. The latter two are known breakdown products of tributyltin in waters (Seligman et al 1986) which are considerably less toxic. Recommended analytical conditions and analytical performance data are presented.

2. EXPERIMENTAL

A Perkin Elmer 4000 AAS equipped a tin hollow cathode lamp, deuterium lamp background correction and a HGA 400 Furnace programmer was used for the determination of tin. Initially, a Perkin Elmer AS-40 Autosampler was used for sample introduction into the furnace, however, high blank concentrations were observed (presumably due to leaching of inorganic tin from worn pump components) and so its use was abandoned in favour of manual sample injection with a micropipette.

The following instrumental conditions were employed: wavelength 286.3 nm, slit width 0.2 nm. Furnace programme: dry, 120 °C, 10 second ramp, 10 second hold; ash, 900 °C, 10 second ramp, 10 second hold; atomise, 2500 °C (maximum heating), 3 second hold; clean out, 2700 °C, 3 second hold. Injection volume was 20 µl. To maximise sensitivity, argon gas flow was stopped during atomisation.

Graphite tubes were pretreated by soaking for at least 24 hours in sodium tungstate solution (5% m/v). Prior to use, the tubes were dried in an oven at 110 °C for about 30 minutes and conditioned in the furnace itself by progressively increasing heating temperature to 2700 °C. For prolonged storage, tubes were simply kept in the tungstate solution until required. Tube life was greater than 300 firings.

Stock standard solutions were prepared from tributyltin chloride (BDH Chemicals), dibutyltin chloride (DBT), monobutyltin chloride (MBT) and triphenyltin hydroxide (TPT) (Aldrich Chemicals) at a concentration of 1000 ppm as tin in methanol. These were found stable for several months when stored in the dark. Working standards at 1 and 10 mg/l were prepared in methanol and were kept for a few days. Aristar grade toluene (BDH Chemicals, Poole) and HPLC grade pentane (Rathburn Chemicals) were used for solvent extractions.

All glassware was cleaned by soaking in 10% (v/v) nitric acid solution, rinsing with deionised water, then methanol and drying before use. Extraction funnels were rinsed with methanol then copious quantities of deionised water between extractions.

During investigation of furnace conditions and extraction procedures, extracted standards were

prepared by spiking 10 ml of aqueous sample with the organotin compound followed by extraction in a 25 ml pyrex glass bottle for 5 minutes with the same volume of toluene. The phases were not separated prior to analysis. Care was taken to pipette from the upper toluene layer only. Extracts could be stored in this manner for at least a week with no observed change in concentrations.

Seawater for performance tests was collected from the Solent, UK and was filtered (0.45 μ m membrane) on return to the laboratory. In preliminary experiments, such as the investigation of pH on extraction, artificial seawater (Grasshof et al 1983) was used.

3. ANALYTICAL PROCEDURES

3.1

Toluene extraction

Samples were filtered (0.45 μ m membrane filter), and one litre of water extracted in a glass separating funnel with 2 ml or 5 ml of toluene (depending on the desired detection limit). After vigorous shaking for 10 minutes, the solutions were allowed to settle for 10 minutes, prior to separation of the toluene layer. Extracts were stored in glass sample vials having PTFE inserts in the caps. No effort was made to completely separate the two liquid phases (care was taken to pipette from the toluene phase only). In some cases an emulsion formed on extraction. This was transferred to a glass centrifuge tube (by washing walls of the funnel with clean seawater if necessary) and broken down by centrifugation at 2000 rpm for 5 minutes prior to transfer to a clean sample vial. Calibration of the method was achieved by spiking a river or seawater sample with tributyltin and extracting in the same manner as samples. The maximum volume of methanol spiking

solution (TBT standard) added to one litre of aqueous solution was kept below 200 µl. Over the salinity range 3 to 35 parts per thousand, calibration should be against standards prepared in clean seawater. When analysing freshwaters, samples should be arranged into groups having similar bulk compositions and calibrated against standards prepared in a chemically similar freshwater.

3.2

Pentane extraction

One litre of water was extracted with 10 ml of pentane in the same manner as described above. After separation, the funnel was washed with a further 10 ml of pentane and the aliquots combined. The extract was dried by freezing at -15 °C in a freezer for greater than 3 hours and pipetting the pentane phase into a dry vial. The pentane phase was then evaporated to dryness under a gentle stream of nitrogen and the residue redissolved in 0.5 ml of toluene.

4. RESULTS AND DISCUSSION

4.1

Optimisation of furnace conditions

Furnace conditions were optimised using TBT standards prepared directly in toluene. The type of furnace tube used was found to have a profound effect on sensitivity (Figure 1). Pyrolytic tubes gave very poor sensitivity and non-pyrolytic tubes were only slightly better. A marked increase of sensitivity of about 3 fold, was obtained by soaking tubes in sodium tungstate solution (Vickrey *et al* 1980, 1981a, 1981b). This treatment had the additional advantages of increasing tube life and improving precision. Tungsten coated pyrolytic tubes were slightly more sensitive than non-pyrolytic ones and were therefore used in subsequent work. Pretreatment of furnace tubes with other transition metal salts (eg

salts of zirconium, vanadium and molybdenum) (Vickrey et al 1980, 1981a, 1981b), was not evaluated, but these may be equally as effective.

Irrespective of furnace tube type, TBT was found stable on ashing up to 600 °C (900 °C in a tungsten-treated furnace tube). This indicates stabilisation of tributyltin compounds in the furnace, as the boiling points of these compounds (tributyltin chloride 171-173 °C, bis-tributyltin oxide 180 °C) would suggest losses by volatilisation. Signal response was constant for atomisation temperatures over the range 2300-2600 °C. The optimum furnace conditions are given in the section 2.

Using the optimised furnace conditions derived for TBT, standards of TPT and DBT in toluene gave the same signal response as TBT. The signal response obtained for MBT however, was approximately a third of the other organotins.

4.2

Effect of extraction on signal response

It was apparent that extraction of TBT from aqueous solution caused a marked signal enhancement relative to that obtained for standards prepared directly in toluene (Figure 2). Experiments indicated that differing responses were obtained on extraction of tributyltin from deionised water, seawater and 3% saline solution (see Figure 2). Typically, extraction from seawater resulted in a 40% signal enhancement, but the degree of enhancement was variable between days (enhancement effects were studied on at least 10 separate days). In addition, it was observed that the response obtained for standards prepared directly in toluene reduced over the course of the working day (see Figure 2), whereas, the response obtained from extracted samples remained the same throughout the day. The enhancement effect was found to be

independent of tube type (non-pyro, pyro, L'vov platform - with or without tungsten pretreatment). To see if the enhancement effect was related to the solvent used, the experiments were repeated with a different solvent - 1,1,1 trichloroethane. Results were similar, but sensitivity was lower.

The above set of experiments demonstrated the importance of preparing standards by extraction from a similar matrix to that of the sample. Calibration of the extraction method with standards prepared directly in toluene, which, in any respect, is a questionable analytical practice, can lead to an overestimation of organotin concentration.

The effect of variation of salt content, as would be found in estuarine samples, was checked by comparing calibrations obtained in full strength seawater with the same seawater diluted ten-fold. Results (Figure 3) indicated that bias caused by variations in salinity over this range was not important.

It was thought that differences in the degree of enhancement observed on extraction from different aqueous solutions was due to variable water content in the toluene extracts. The difference in response between 3% saline solution and seawater (Figure 2) led us to think a surfactant mechanism may increase transport of water into the toluene layer from seawater samples; however, repeat experiments with ultraviolet irradiated aliquots of the same seawater showed no difference in response. Perhaps the most interesting experiment involved pretreatment of the furnace tube by drying of deionised water in the tube prior to analysis of a TBT standard prepared directly in toluene (Table 1). This experiment clearly demonstrated an enhancement effect that was related to the presence

of water in the tube. Observation of absorbance-time profiles during the atomisation step indicated no observable difference in peak shape between extracted samples and those made up direct in toluene. We can only conclude from our limited experiments, that water present in the toluene extract in some way activates the tube surface, possibly affecting the efficiency of the ashing step.

4.3

Effect of pH on extraction

The effect of pH on the extraction behaviour of tributyltin, dibutyltin, monobutyltin and triphenyltin from artificial seawater solutions was investigated. The pH of each solution was adjusted to values within the range of 0 to 10 by addition of dilute acid or base. Results (Figure 4) indicated some interesting differences in behaviour. Similar results were obtained for TBT and TPT, which both showed little variation in extraction behaviour over the pH range investigated. By contrast, DBT gave a similar response over the pH range 0 to 4 and fell off to only 19% of this response at pH 10. At pH 8.0 (the approximate pH of seawater), only 30% of DBT was extracted from solution. MBT extraction was uniformly poor over the pH range investigated, the signal responses obtained for extracted MBT standards were typically 8 to 10% of that obtained for extracted TBT standards.

Since some workers (Bryan et al 1986, M&T 1976) describe the separation of DBT from other organotins in organic extracts by back extraction into an aqueous alkaline solution, this method was investigated. A 10 ml toluene extract containing 50 µg/l DBT was shaken with 10 ml of 0.1 M sodium hydroxide solution for 5 minutes. A reduction in signal response of about 99% was observed. The same test on a TBT standard showed no reduction in

signal response. This step may therefore be included to improve the accuracy of the TBT determination, or employed to give an estimation of DBT content. Alternatively, dibutyltin and tributyltin may be determined together, if the pH of the sample solution is adjusted to below 4 prior to extraction. An alkaline back extraction step was not included in our final method on the grounds of simplicity. Instead, it was deemed acceptable to extract samples at pH 8 and accept the possible inclusion of a small fraction of DBT.

TPT was found to be potentially the most serious source of bias amongst organotin compounds tested. The co-extraction of TPT with butyltin compounds is not likely to present serious problems in most natural water samples as this compound is a relatively minor constituent (at the present time) of antifouling preparations.

4.4

Performance characteristics

Within-batch performance data for the described toluene extraction methods is presented in Table 2. Detection limits of below 5 ng Sn/l were easily attainable (Table 2). Precision at 100 ng Sn/l was excellent. Recovery was evaluated by comparison with a standard solution obtained from by a 1:1 ratio extraction of 10 ml of spiked seawater. This approach was used to overcome the difficulties created by signal enhancement on extraction. At a concentration of 250 ng Sn/l, recovery was estimated as $91 \pm 3\%$ (4 replicates).

In this work, all performance data was obtained on filtered samples. Recoveries from unfiltered samples may well be lower, owing to the incomplete recovery of TBT adsorbed to particulates. This is a problem common to all analytical methods for organotins that do not make a distinction between dissolved and particulate forms.

Interferences apart from those arising from the co-extraction of other organotin compounds were not investigated. The presence of substances such as surfactants, oil or grease in contaminated samples may well affect extraction efficiency and/or GFAAS detection. If interferences are suspected, these should be checked by standard additions.

4.5

Sample storage

A brief experiment was carried out on sample storage. Pyrex glass and polycarbonate bottles were evaluated. Polyethylene bottles were not tested as these have been shown to be unsuitable in a previous study (Blair et al 1986). Pyrex and polycarbonate bottles (250 ml volume) were rinsed in warm detergent solution, thoroughly washed with deionised water and soaked for at least 24 hours in 5% (v/v) hydrochloric acid solution. After rinsing with deionised water and drying, each bottle was filled with clean seawater (settled, but not filtered) and spiked with 1 µg Sn/l of tributyltin. All solutions were prepared in triplicate and stored for five days in the dark at room temperature. On analysis, the final concentrations were 0.76 ± 0.01 µg Sn/l for the polycarbonate bottles and 1.01 ± 0.11 µg Sn/l for the pyrex glass bottles. Despite recommendations in the literature (Seligman et al 1986, Valkirs et al 1987), polycarbonate containers were found unsuitable for sample storage, pyrex glass bottles appear more suitable. In the absence of any other information, it is advised that samples are stored in pyrex glass and analysed as soon as possible.

4.6

Pentane extraction

To obtain detection limits of below 2 ng Sn/l, it was found impractical to reduce the volume of toluene used to much below 2 ml, owing to losses on the funnel walls and problems with emulsion formation. Emulsions could be broken down to some

extent by centrifugation, but turbid samples presented serious problems in recovering the organic phase. As a result, an alternative low detection limit method was therefore evaluated which involved preconcentration of the organic phase by blowdown. The initial extraction was carried out with pentane. This solvent was preferred to toluene owing to its low boiling point and as it is less prone to emulsion formation. As pentane was not suitable for introduction into the furnace (high volatility and poor pipetting behaviour), the pentane extract was evaporated to dryness and the residue redissolved in toluene.

The efficiency of the blowdown step was investigated by evaporating 5 ml of pentane containing 25 ng Sn/l of TBT and redissolving in 500 μ l of toluene. Mean recovery (five replicates) was $98 \pm 6.7\%$. Initially, it was thought that the main source of imprecision was loss on evaporating completely to dryness. But, when 25 μ l aliquots of methanol containing 25 ng of TBT were blown to dryness and maintained under the nitrogen stream for a further minute before redissolution of TBT in toluene, results indicated $98 \pm 1.5\%$ recovery (three replicates), confirming that this step was satisfactory. When combined with extraction, an overall detection limit of 0.75 ng Sn/l (based on 4.65 times the within-batch standard deviation on the blank, with six replicates) was obtained. Recovery was evaluated on seawater samples spiked with 25 ng Sn/l (7 replicates) and was 83.4% with a relative standard deviation of 9.0%. Further work is required to improve precision. It was anticipated improvements may be obtained by using a more sophisticated preconcentration step involving use of a Kuderna-Danish apparatus. However, time did not allow us to pursue this any further. In our experiments, a maximum sample volume of 1 litre was used with a single extraction - giving a

preconcentration factor of 2000. It would, however, be possible to increase sample volume and extract samples with several portions of solvent, if improved detection limits are desired.

5. CONCLUSIONS

1. Using toluene extraction and determination of tin by graphite furnace AAS, limits of detection for tributyltin of below 4 ng Sn/l were attained. Enhancement of GFAAS sensitivity by tube pretreatment is an essential step if low detection limits are required. The relative standard deviation on six replicate analyses of seawater containing 170 ng Sn/l tributyltin was 1.5%.
2. A significant GFAAS signal enhancement effect is observed on extraction of tributyltin from aqueous samples. It is therefore essential to prepare standards from matrices similar to those of the samples. In the case of estuarine samples, there was little observed difference in signal response between samples of salinity 35 parts per thousand and 3.5 parts per thousand. Estuarine samples may therefore be calibrated against standards prepared in a full strength seawater matrix, low in TBT.
3. The extraction studies indicated that when determining TBT, the co-extraction of MBT is a relatively unimportant source of bias. The co-extraction of DBT may be reduced by adjustment of sample pH to greater than 8. Alternatively, DBT may be quantitatively removed from the extract by washing with sodium hydroxide solution. TPT appears potentially to be the most important source of bias, as it has a similar extraction behaviour to TBT.

4. The methods described in this paper may be used to screen aqueous samples for TBT. Samples identified as containing high levels of organotins may then be submitted to a more detailed, but time-consuming determination of the specific organotin species present.
5. Preliminary investigations suggest extraction into pentane, preconcentration by evaporation and redissolution in toluene, may allow the attainment of sub ng/l detection limits.

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Table 1. Effect of tube pretreatment with deionised water on signal response

Run	Signal response (absorbance)	
1	0.200	water added
2	0.102	no water
3	0.231	water added
4	0.091	no water

Sample: 50 µg Sn/l tributyltin chloride dissolved in toluene. Water pretreatment involved pipetting 20 µl of deionised water into the furnace, followed by heating at 100 °C until dry. The sample was then analysed as soon as the tube had cooled to room temperature. The samples were analysed consecutively, with a maximum delay time of 2 minutes.

Table 2. Within-batch performance data

Precision (5 ml toluene extraction):

Seawater, 170 Sn ng/l (6 replicates), 1.5% relative
standard deviation

Seawater, 55 Sn ng/l (7 replicates), 5% relative
standard deviation

Detection limits:

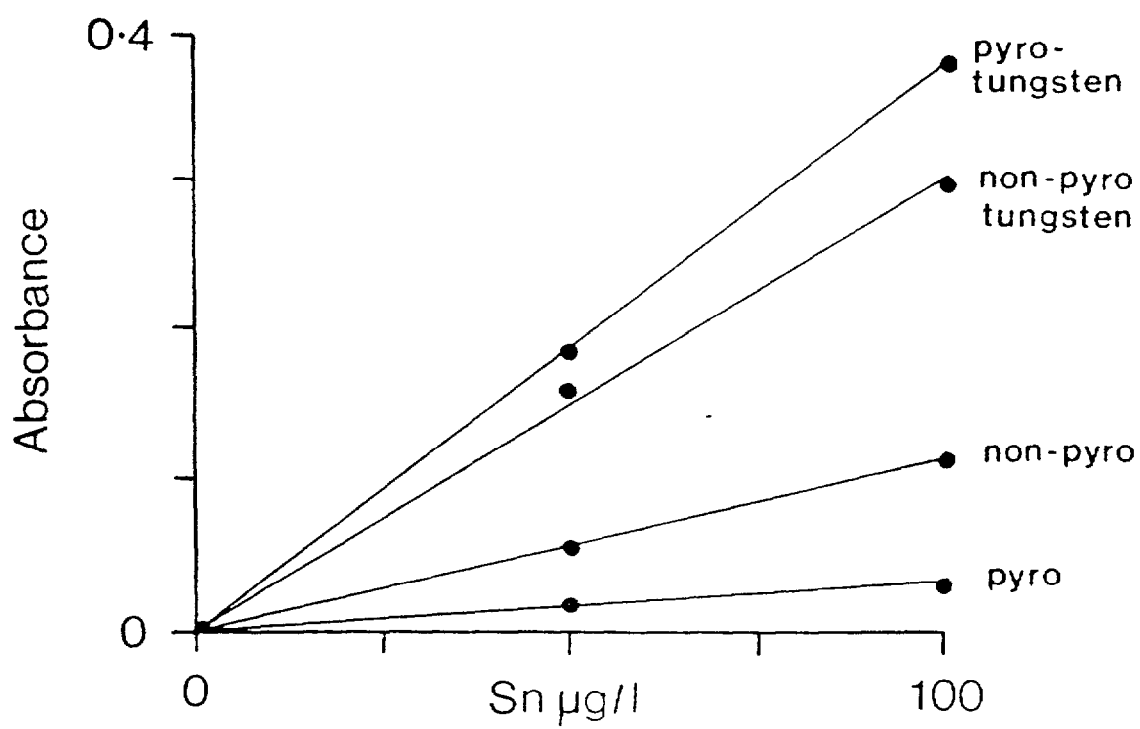
5 ml toluene extraction: 3.96 ng Sn/l
(6 replicates)

2 ml toluene extraction: 1.90 ng Sn/l
(6 replicates)

(based on 4.65 times the standard deviation on the
blank)

Linear range:

Up to at least 100 µg Sn/l in the organic extract,
ie 0-500 ng/l for 5 ml toluene extraction
0-200 ng Sn/l for 2 ml toluene extraction



Tributyltin standards prepared directly in toluene

Figure 1 Effect of furnace tube type on sensitivity.

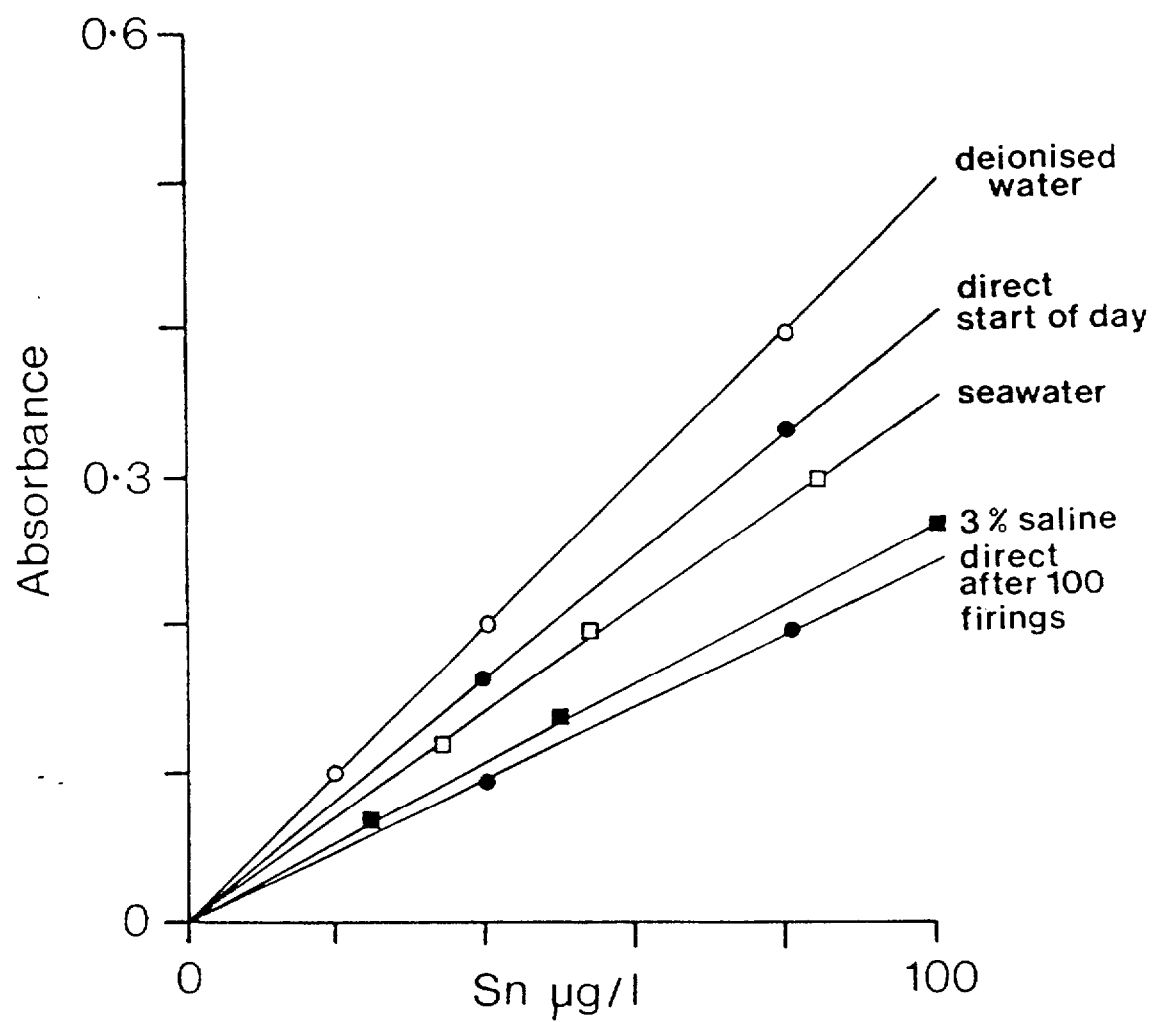
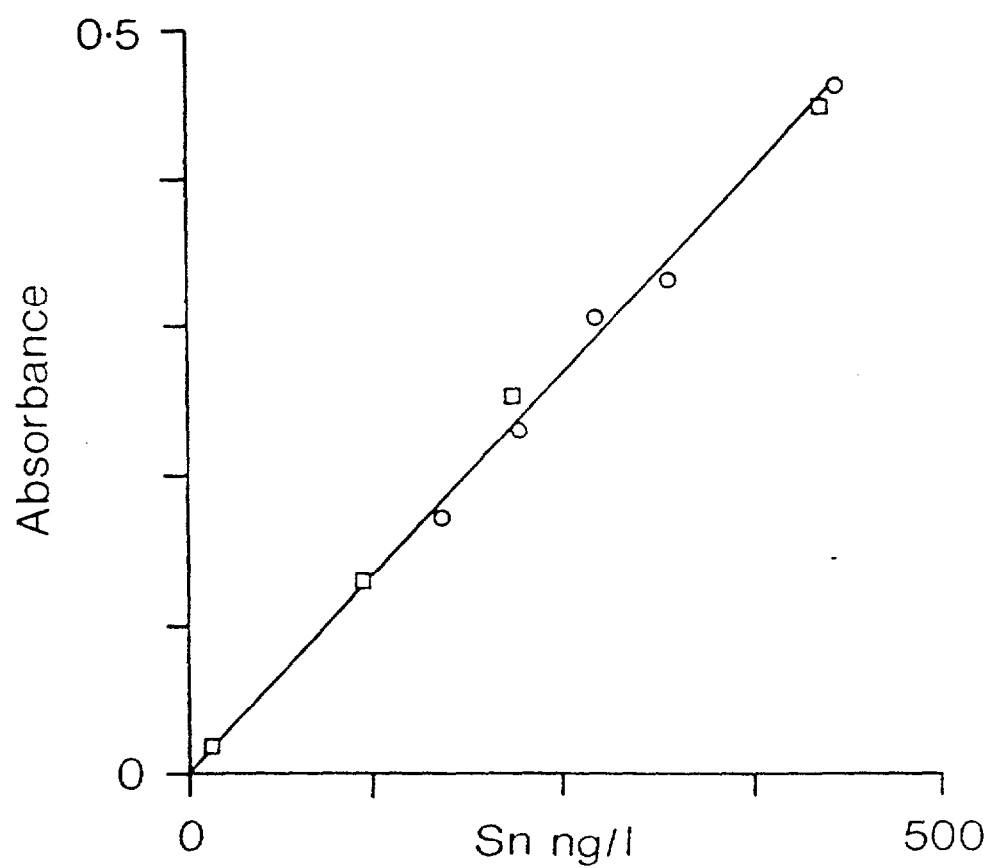


Figure 2 Effect of extraction on GFAAS signal response

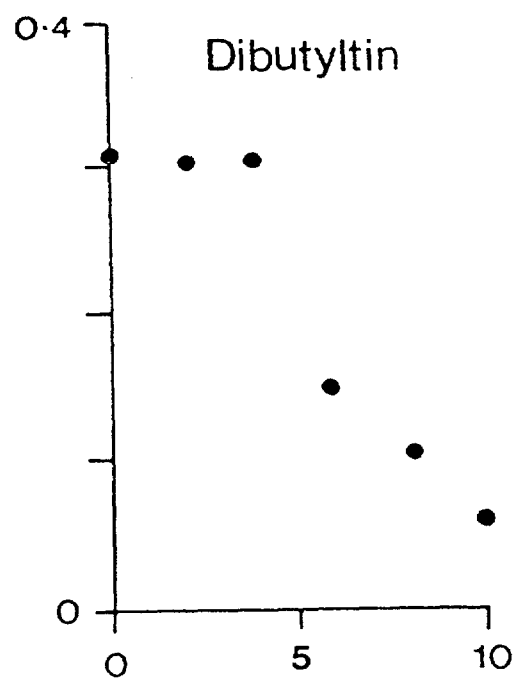
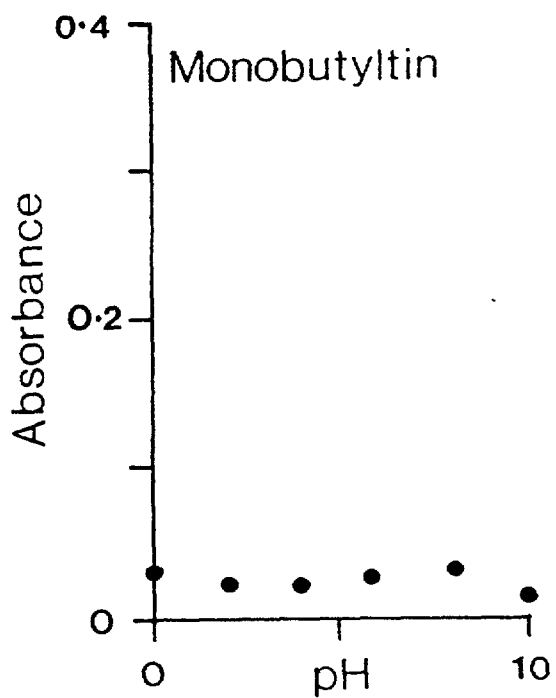


1 litre sample extracted with 5 ml of toluene

○ full strength seawater

□ 10% seawater solution

Figure 3 Comparison of calibrations obtained in seawater and 10% seawater solution



10 ml of artificial seawater spiked with 50 ug Sn/l organotin standard and extracted with the same volume of toluene

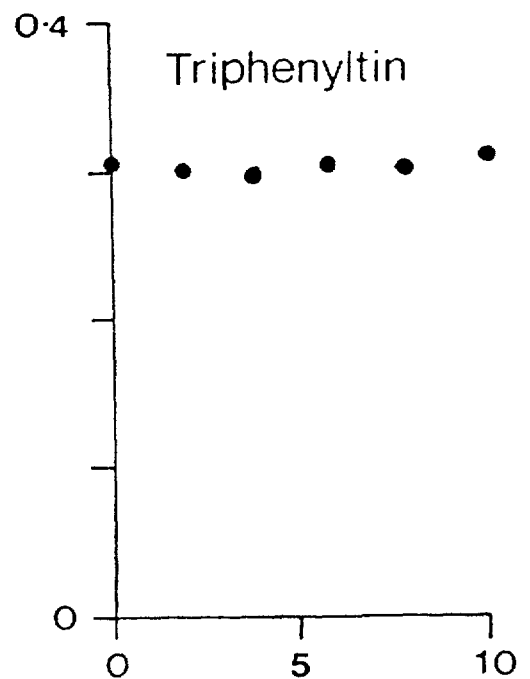
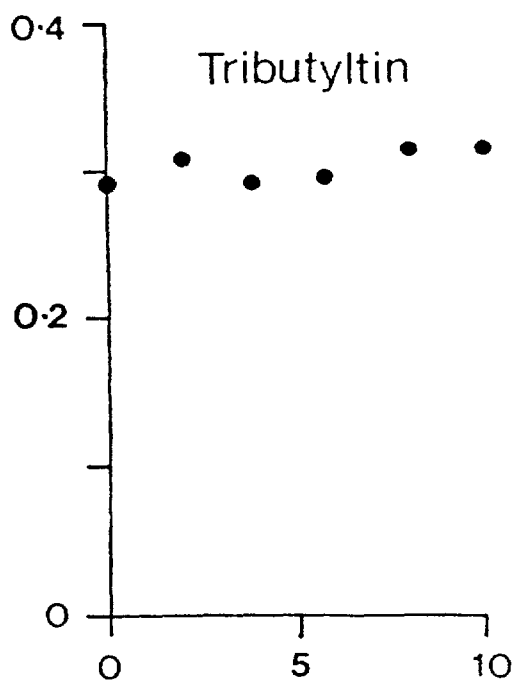


Figure 4 Effect of extraction pH on signal response

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