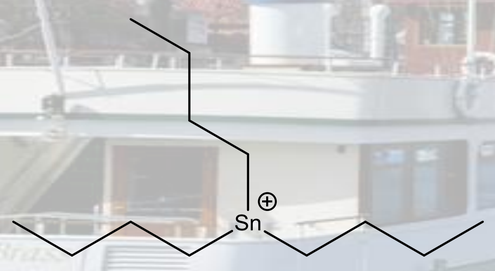
Behavior of tributyltin under the influence of suspended particulate matter

Janine Richter[1], I. Fettig[1], R. Philipp[1], C. Piechotta[1], N. Jakubowski[1], U. Panne[1] and M. Ricking[2]

1 janine.richter@bam.de • +49 30 8104 5755 • www.bam.de
Federal Institute for Materials Research and Testing
Richard-Willstätter-Straße 11, 12489 Berlin, Germany
2 Freie Universität Berlin
Malteserstrasse 74-100, 12249 Berlin, Germany

Introduction

- Tributyltin (TBT) is listed as one of the priority substances in the European Water Framework Directive (WFD) with an Environmental Quality Standard (EQS) of 0.2 ng L^{-1}.
- Despite its decreasing application high concentrations of TBT still occur in surface water, biota, sludge, and sediments.
- TBT shows a high potential to interact with organic compounds like humic substances (HS) and solids like suspended particulate matter (SPM). Sediments even act as natural sinks. Therefore TBT will be continuously emitted into the environment from sediments for the next decades.
- Since WFD claims the analysis of whole water samples the quantification of TBT is challenging at the low EQS level. Especially the strong interaction with organic and solid matter is a problematic factor for the recovery of TBT.



Objective

Development of a traceable measurement method to quantify TBT at EQS in whole water samples.

Experimental

Sample
1L water

Internal standard
^{119}Sn-enriched Butyltin Mix

Buffering and derivatization
pH-adjustment with acidic acid/acetate buffer to pH=5 and
ethylation with 1% $NaBEt_4$

Extraction

Analysis
GC-ICP-MS

Quantification
species-specific isotope dilution analysis

Method parameter

GC
Agilent 7890A
Detection
Agilent 7500cx ICP-MS
Column
Agilent DB-5MS UI
(30m x 0.25mm x 0.25µm)
Oven program
60°C, heating rate 30K/min to 300°C (8min)
Carrier gas
Helium, 1.05mL/min
Plasma power
500W
Detected isotopes
119 Sn, 120 Sn
Integration time
0.18s

Results and Discussion

- Evaluation of different extraction techniques: solid phase extraction, solid phase microextraction, and liquid-liquid extraction
  → Liquid-liquid extraction with 1-2 mL n-hexane showed lowest blanks, best recovery rates and good reproducibility
  → Simple handling, low amount of extraction agent used, no further preconcentration steps necessary
- For quantification of TBT in whole water samples the release of TBT from HS and SPM is essential
- Different chemical and mechanical sample treatment procedures based on sediment analysis were evaluated
  → Best recovery rates with addition of 3:1 acidic acid-methanol-mixture and one-step shaking procedure
- Developed method for analysis of whole water samples containing HS and SPM:

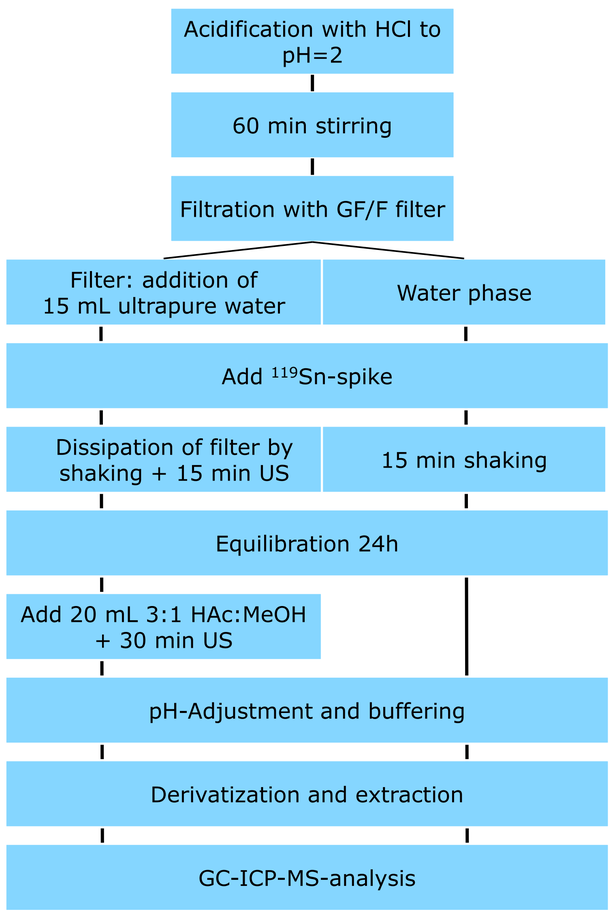


Fig 1: Sample preparation for whole water samples with filtration step

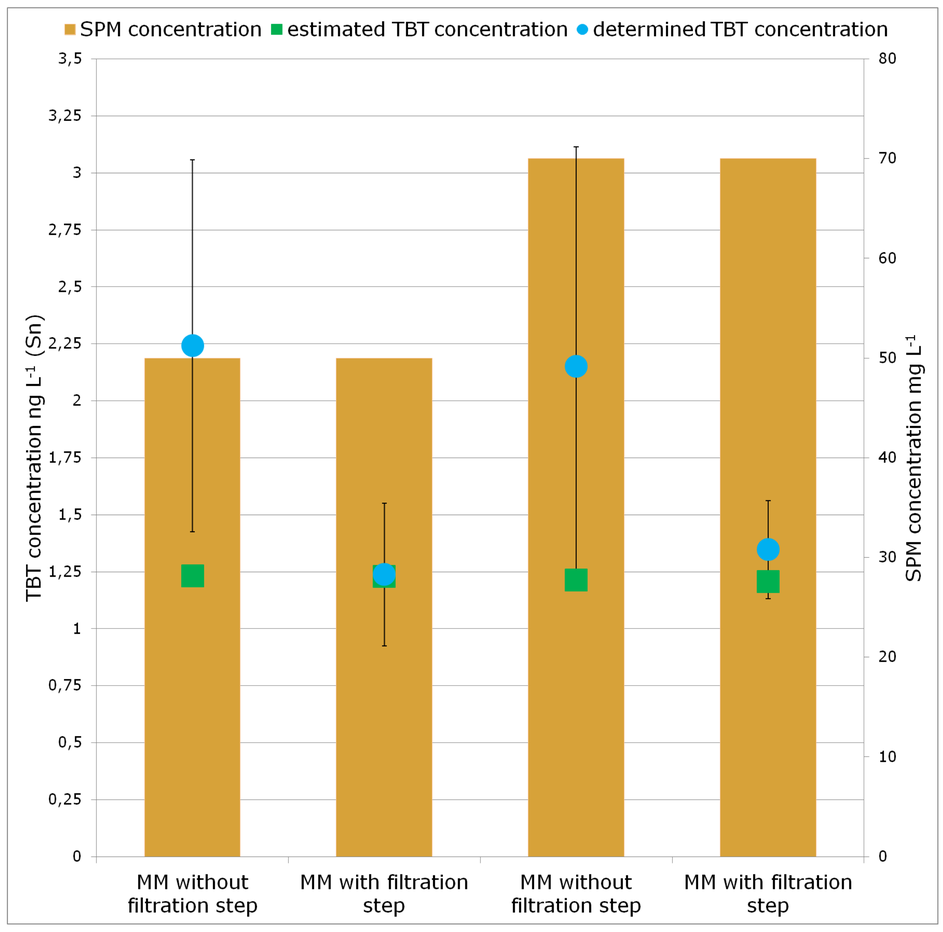


Fig 2: Determined TBT concentration in water model matrix (MM) with spiked TBT polluted SPM (milled CRM sediment) and HS (10 mg L^{-1})

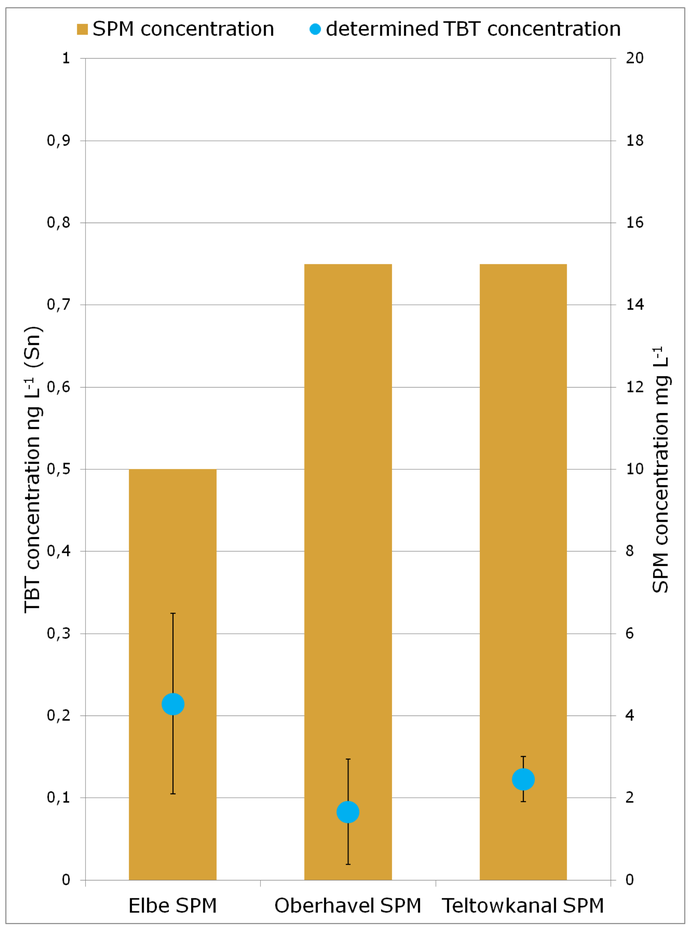


Fig 3: Determined TBT concentration in mineral water spiked with natural SPM

Conclusion

- A method to determine TBT at EQS level in whole water samples was developed.
- Analysis of whole water body is important for a correct mass balance and a filtration step leads to better recovery rates and precision.

References

[1] J. Richter et al., Environ Sci Pollut Res, 2015, (DOI) 10.1007/s11356-015-4614-4
[2] E. Alasonati et al., Talanta, 2015 (134), 576-586
[3] S. Elordui-Zapatarietxe, Anal Bioanal Chem, 2015 (407), 3055-3067

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