DESIGN AND PROGRESS OF A COMPREHENSIVE INTERNATIONAL LABORATORY SPECIATION
INTERCALIBRATION STUDY OF A MULTISPECIES BUTYLTIN REFERENCE MATERIAL:
SIGNIFICANCE FOR NEW ANALYTICAL METHODS AND BIOFILM TESTING

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During 1984 the U. S. National Bureau of Standards (NBS), with support from the U. S. Navy, distributed a chromatographically prepared aqueous solution of tributyltin (ca. 1 $\mu\text{g/mL}$ as elemental tin) to an international group of laboratories interested in participating in a tin measurement methods intercomparison. Most intercomparison participants returned data on the total tin concentration of the sample. Statistical analysis of the data at NBS indicated there was no inherent bias in any of the seven different tin measurement methods employed.

In the time since the above methods intercomparison was conducted, new methods of organotin analysis have improved the detection levels for state-of-art tin determinations by several orders of magnitude. The sensitivity improvements of the new methods allow routine detection of organotin compounds at environmental concentrations in the ng/L level for individual organotin species. Because of the large differences in toxicity between the individual organotin compounds, qualified speciation data is an absolute requirement for accurate assessment of the environmental effects of organotins and evaluation of their persistence. Moreover, in view of increasing international concern with the environmental fate and effect of organotins, an evaluation of organotin speciation methods is both desirable and feasible.

As a continuation of the tin measurement methods intercomparison program, NBS has organized a definitive intercomparison designed to evaluate organotin speciation methodology by use of a multi-species organotin research material. Chromatographic synthesis employing a generator column technique (1) was used to produce solutions of di-, tri-, and tetrabutyltin species which were blended into a single homogenous sample of 25 liter volume. For distribution to intercomparison participants, individual solution samples in pyrex glass bottles of approximately 100 mL volume were filled from the large 25 liter volume of solution.

Production of a multi-species butyltin research material required that NBS additionally certify, by independent non-destructive means, the total tin concentration along with the individual species concentrations in the multi-species

research material, thereby insuring total mass balance agreement in solution. Concern about biologically induced redistribution in the multi-species research sample was eliminated by the production of a sterile sample. Additionally, since the samples were prepared aseptically, laboratories interested in biological or toxicological applications of the research material can be assured of noncontamination by butyltin-resistant microorganisms.

Over 120 letters inviting participation in the organotin speciation methods intercomparison were mailed to an international group of laboratories. As this was written, requests for samples of the multi-species butyltin research material were being received, with over 25 samples having been mailed.

Current organotin research at NBS seeks to determine the role of microbial biofilms that develop on surfaces painted with tributyltin (TBT) containing antifouling coatings in mediating TBT release from the antifouling coating. The rate of TBT accumulation in the biofilm, as well as the TBT concentration levels reached in the biofilm will be measured. Surface analysis by epifluorescence microscopy of biofilms charged with TBT holds promise of allowing direct imaging of TBT binding locations on the biofilm by use of tin-complexing fluorescent ligands.

REFERENCES

- (1) Wasik, S.P., Tewari, Y.B., Miller, M.M. and Martire, D.E. 1981. "Octanol - Water Partition Coefficients and Aqueous Solubilities of Organic Compounds," NBSIR 81-2406, National Bureau of Standards, Gaithersburg, MD.