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NIES Certified Reference Material  
No.11 "Fish Tissue"

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No.11, Fish Tissue. This reference material is intended primarily for use in developing and validating analytical methods for the determination of total tin, tributyltin and triphenyltin in fish tissue or in other similar matrices.

Certified value ( $\mu\text{g/g}$ )

Total tin 2.4  $\pm$  0.1  
Tributyltin 1.3  $\pm$  0.1 (as chloride)

Reference value ( $\mu\text{g/g}$ )

Triphenyltin 6.3 (as chloride)

Coastal water pollution with organotin compounds has recently become one of the most serious environmental problems in Japan. Organotin compounds such as tributyltin (TBT) and triphenyltin (TPT) have been used as antifouling paints for fish nets and ship's hulls. The Environment Agency of Japan has been conducting surveys for four years on the concentrations of organotins in seawater, marine sediments and marine organisms. Although the use of TBT on fish nets in fish farms was prohibited about 3 years ago, a relatively high level of TBT has still been detected in fish samples. The survey in 1988 on organotin concentrations has revealed that coastal water pollution by TPT is a more serious problem than pollution by TBT. The TPT concentrations in fish tissues are high due to the accumulation of lipophilic TPT by fish.

The two most common techniques for TBT determination are gas chromatography with flame photometric detection (GC-FPD) and hydride generation-atomic absorption spectrometry (HG-AAS). Both methods require fairly extensive sample workup including extraction, cleanup and derivatization. These analytical procedures invariably need skill and experience, and therefore it is difficult to obtain accurate analytical results for TBT in natural matrix materials. The determination of TPT is more difficult than that of TBT, because TPT is considered easily decomposed during the sample preparation procedure. For this reason, large discrepancies in analytical results for TBT and TPT have been reported. The most practical way to maintain the accuracy and precision of analytical results is through the use of appropriate certified reference materials. Therefore, NIES has recently undertaken the preparation of Fish Tissue reference material for the determination of total tin, TBT and TPT.

Sea bass (Leteolabrax japonicus) was selected because sea bass and mussel (Mytilus edulis) have been routinely used in the biomonitoring surveys of organotins being conducted by the Environment Agency of Japan. About 150 kg of sea bass collected in early July 1988 in Tokyo Bay was used. The fish samples (body length 50-60 cm, individual weight 1-2 kg) were stored in a liquid nitrogen jar until use. After thawing, the fish samples were dissected with a stainless-steel knife and only the fish fillets (50 kg) were used for further processing. These were cut into small pieces and a batch of about 2 kg were minced at 3,000 rpm for 3 min with a Tecator 1094 homogenizer. After repeating this mincing procedure for the remaining batches, the fish tissue homogenate was combined and freeze-dried in one lot. The dry fish tissue (about 12 kg) was alumina ball-milled for 1h and then mixed in a V-blender for 2 h. Twenty gram samples of the homogenized fish tissue were packaged into polyethylene bags, which were subsequently vacuum-packed doubly into polyethylene laminate bags together with an oxygen absorber. The prepared NIES Fish Tissue reference material (600 samples, 20 g each) are currently stored at -20 °C.

A collaborative study on the analysis of the fish tissue reference material for TBT and TPT concentrations was carried out by 9 qualified participating laboratories. The TBT was determined by GC-FPD, GC-ECD and GC-MS as its chloride, hydride or propyl derivative. The certified value for TBT in the fish tissue reference material was determined, based on the results of determinations by 3 different analytical methods. For the determination of TPT, however, there were analytical problems related to extraction, cleanup and measurement, etc, and, presently, the reference value is given for the TPT concentration in the fish tissue reference material.

Tin is considered essential for animal growth and is present usually at ppb levels in biological materials. Among essential trace elements, tin is classified as one of the most difficult to accurately determine. In order to promote nutritional and environmental research on tin, the certified value for total tin in NIES Fish Tissue reference material is provided, based on the results of determinations by graphite furnace AAS, HG-AAS, isotope dilution/ICP-MS, spectrophotometry and neutron activation analysis.

#### Instructions for Use

- ① A minimum sample weight of 1 g of the material should be used.
- ② The material should be stored in a freezer at -20°C.

For more information, please contact Dr.M.Morita at the address shown above.