U.S. Department of Commerce
Juanita M. Kreps
Secretary
National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards Report of Investigation

Research Material 50

Albacore Tuna

P. D. LaFleur and W. P. Reed

A lyophilized (freeze-dried) marine biological tissue sample has been prepared in an attempt to satisfy many of the analytical requirements for a base line marine reference material.

Tuna fish muscle tissue was chosen for this purpose because of availability, use as a fish foodstuff, and its oceanographic interest.

The tuna is available as a Research Material (RM), in sets of two cans. Each can contains approximately 35 grams of lyophilized tuna tissue in a polyethylene bag, inside the hermetically-sealed, nitrogen filled can.

Material Application

This particular RM is intended to be used in the measurement of elements present at trace concentrations. In addition, some measurements of trace hydrocarbons have been made. The material represents a typical marine tissue that has been lyophilized. It should prove useful to those scientists who may wish to evaluate analytical methods, or to use a generally available "real" sample matrix in interlaboratory comparisons. It is important to note, however, that this is not a Standard Reference Material and none of the data presented here are certified. For the convenience of the analyst using this material, we have included a discussion for each component reported. It is apparent that there is significant heterogeneity for some elements, specifically the "bone-seeking" elements. The homogeneity of other elements appears to be acceptable. In most instances the heterogeneity of the material is observed between samples from the same can. There is some evidence that this heterogeneity is due primarily to fine particles of cartilage or bone present with the tissue in this RM.

Material Preparation

The tuna tissue used in this research material is from albacore tuna caught in the San Diego area in July of 1971. The tuna was cleaned, filleted, frozen and transported to a lyophilization facility. It was thawed, ground, and mixed using stainless steel equipment. For the final mixing, the entire lot was held in a single stainless steel container. The tuna was then lyophilized in aluminum trays lined with polyethylene. After lyophilization, the material was again ground in a stainless-steel mill, carefully transferred to new, individual polyethylene bags, and canned under nitrogen for storage.

Preliminary studies performed on these canned samples raised serious problems about the homogeneity of the material. In an effort to improve this condition the material was reground, reblended and recanned under the same conditions, as before. Except where stated, all measurements provided in this report were made on the reprocessed material.. During the second regrinding, fibrous material tended to "float" to the top due to the action of the mixer. This material was discarded.

(over)

Material Use

The lyophilized tuna tissue, sealed in metal cans, should have an indefinite storage life under normal room conditions. Thus far, no evidence exists to indicate deterioration of the material with time as long as the can remains sealed. Once opened, the possibility exists that the raw material will turn rancid. Researchers using only a portion of the material have successfully stored the remainder by placing the polyethylene bag in a glass jar with lid and storing at or below 0°C. A shelf life of 6 months is not unusual under these circumstances and a 2 year stability has been reported. The freeze-dried material represents only about 30% of the original weight of the tuna tissue. Consequently, for direct comparisons with fresh tissue the researcher may wish to adjust the sample weight for this difference. Please note, however, that all values in this "Report of Investigation" are based on the lyophilized weight of the tuna tissue.

The dissolution of this material is not difficult and researchers have reported using a variety of techniques. These techniques include nitric and perchloric acid digestion, nitric-sulfuric-perchloric (4:1:1) acid digestion, low temperature ashing, and oxygen combustion. Because of the apparent presence of small bits of cartilage and/or bone in the sample, the sample size for analysis should be 250 mg or greater to obtain reproducible results. For "bone-seeking" elements (e.g., Pb, U, Ca, Sr) the sample may be nonhomogeneous even with much larger sample sizes.

For the elements listed below, sufficient analytical work has been performed to permit some evaluation of the data. These brief evaluations are not certified values, but only judgments as to the amount of the element present. Problems encountered by analysts making these measurements are also described.

Mercury

The question of the mercury content of various foodstuffs has been studied by many investigators. It is hoped that this material will provide a base line RM for environmental studies of mercury in food. A value of 0.95 ± 0.1 ppm encompasses the means of all of the reported values for mercury with one exception and extensive studies have indicated good homogeneity for this element.

Agreement among methods appears to be good. The data show very little bias among the three methods used. Sample sizes for these measurements have been 0.25 grams and larger.

The question of volatile mercury was explored by one investigator who reported the mercury content decreasing after opening the container. This decrease amounted to approximately 0.1 ppm over a period of 3 weeks after opening even though stored at −25 °C. This work has not been confirmed. Further work by several investigators has suggested that 80-90% of the mercury content is present as methylmercury.

Selenium

This element was determined by four laboratories using both neutron activation analysis and atomic absorption spectroscopy. The range of reported values ($\bar{x}$) is from 3.27 to 4.01 ppm. A most probable value is 3.6 ± 0.4. There has been very little indication of heterogeneity for this element.

Zinc

The analysis for zinc was performed by three laboratories using two analytical methods, neutron activation analysis and atomic absorption analysis. The range of mean values ($\bar{x}$) is 11.4 to 14.6 ppm. Our estimate of the probable value is 13.6 ± 1 ppm. No random behavior has been noted in the analysis of this element.

Arsenic

The analysis for arsenic was performed by 4 laboratories using both neutron activation and atomic absorption techniques. Data obtained within each laboratory appears to be consistent, although there appears to be some disagreement among laboratories analyzing for this element. The range of average values ($\bar{x}$) between laboratories is 2.74 to 4.6 ppm. The recommended value for the arsenic content is 3.3 ± 0.4 ppm.

Lead

There is a limited amount of data available for lead. The average value is 0.46 ppm. The homogeneity of the material for this element is questionable and the range of individual values is quite large. The use of this Research Material as a control, or for the development of methods for lead cannot be recommended. The isotopic composition, however, has been determined and is: ^{208}Pb 52.2%; ^{207}Pb 21.5%; ^{206}Pb 24.9%; and ^{204}Pb 1.38%.

Other Elements

Manganese was found to be distributed homogeneously and the measured value in the original lot of tuna (not the reblended material) was 1.3 ppm. Sodium also was found to be homogeneous in the original lot of material and the reported value was 0.11%.

Potassium was found to be distributed homogeneously and the measured value on the original lot of tuna (not the reblended material) was 1.22%. The following elements have been found to be heterogeneously distributed in the tuna tissue: uranium, thorium, calcium, and strontium.

Organic Materials

The tuna research material was subjected to headspace sampling and analysis. The concentrations given in the following table are to be considered only as order-of-magnitude estimates of the hydrocarbon compounds present, as they were calculated relative to the amount of naphthalene added to the sample and most of the compounds identified were not aromatic hydrocarbons.

The value for the largest constituent, 2,6-di-t-butyl-p-cresol, is an especially poor estimate. However, this compound is interesting because it is a common antioxidant used in food packaging. It probably has its origins in one of the handling steps between tuna collection and packaging in plastic. An indication of petroleum pollution in the tuna sample comes from the aliphatic hydrocarbons present.

The identification of the monoterpene, limonene, is reasonably certain. As with pristane this compound is composed of isoprene units, but limonene is generally considered to be a product of plant biosynthesis. Its origin in this sample is uncertain, but it may arise from plant material ingested by tuna.

Identification and Approximate Quantitation of Major Isolated Organic Constituents

Compound Identification*	Amount Present (ppm)
Heptadiene (?)	0.6
Toluene	0.7
Limonene	0.4
2-nonanone (?)	0.7
2-undecanone (?)	0.1
2,6-di-t-butyl-p-cresol	1.0
Hexadecane	trace
Heptadecane	trace
Pristane	0.03

*Identification followed by a (?) is probable but not definite.

Summary

The analytical values presented in this report represent the authors' evaluation of a considerable amount of data. For many elements where the reports were inconclusive, probable values are not given. If more information becomes available in the future, this report will be updated. Your help in reporting data will be appreciated.

Send any reports to:

Office of Standard Reference Materials (R-701)
B316, Chemistry
National Bureau of Standards
Washington, D.C. 20234