NEUTRON ACTIVATION ANALYSIS OF TIN IN BIOLOGICAL MATERIALS AND THEIR ASH USING $^{123}$Sn AND $^{125}$Sn

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Tin has been determined in biological materials by NAA of the $\gamma$-emitting 40-min $^{123}$Sn and 9.7-min $^{125}$Sn isotopes at the sub-ppm level. For $^{123}$Sn, samples are wet-ashed after irradiation, whereas to allow fast radiochemistry for $^{125}$Sn, the samples are dry-ashed prior to the irradiation. Both separation techniques rely on selective solvent extraction of tin(IV) iodide, and NaI(Tl) counting. Comparative analyses of several materials by both methods gave good agreement, indicating that tin is not lost on dry-ashing and that simple dissolution of the ash in an HCl - HI mixture is complete. Results by both techniques are presented for the standard materials Bowen's Kale and NBS Orchard Leaves, and for some other materials.

Introduction

Tin has until recently been a rather neglected trace element, partly due to difficulties in its analysis at the natural levels (sub-ppm) in biological and environmental materials. The discovery of its essentiality by Schwarz and coworkers, its toxicity to algae and plants, and what Bowen has termed its high "pollution potential", taken in conjunction with the uses of organo-tin compounds as biocides, make it desirable to increase our knowledge of the role of this element in nature.

The aims of the present work were two-fold: firstly to improve a previous activation method based on 40-min $^{123}$Sn, especially to allow its application to the standard reference material NBS Orchard Leaves (SRM 1571), which has troublesome high levels of arsenic, antimony and other elements present, and to similarly contaminated materials; and secondly and mainly, to develop a rapid method based on 9.7-min $^{125}$Sn using pre-ashing of the sample to allow a rapid post-irradiation separation. The concurrent aim here was naturally to investigate how significant possible losses of tin are on dry-ashing of typical samples, by comparative analyses using the two methods. If losses are negligible, this fact could prove of value in other techniques, and would also allow long high-flux ash irradiations, enabling the $^{113}$Sn and $^{117}$Sn isotopes to be sensitively employed.
Methods and materials

Activation of Sn isotopes

A variety of neutron-produced tin isotopes have been used in analysis of biological materials, but mainly the 27-hr pure β⁺-emitter $^{121}$Sn. Of the γ-emitters, 40-min $^{123}$Sn has been used, but as indicated above, 14-day $^{117}$Sn and 115-day $^{113}$Sn/$^{113}$In are less suitable owing to decomposition problems on irradiation, quite apart from the long time required for analysis. Rather than tabulate the nuclear data for the various γ-emitting isotopes, Table 1 shows experimentally measured specific counting rates, as measured under the conditions described below, for typical irradiation and cooling periods. It is clear that, provided a rapid radiochemical separation can be achieved, $^{125}$Sn offers a sensitivity comparable to those of the other isotopes, with the advantage of shorter irradiations (and necessarily shorter separation and counting periods). It appears that this isotope has not previously been used for tin analysis.

Solvent extraction of tin iodide

Solvent extraction would seem to offer the best possibilities for rapid and specific separation of the induced $^{123}$Sn and $^{125}$Sn activities. Of the various reagents available, most lack selectivity. However, earlier work on the extraction of tin(IV) iodide with non-polar solvents showed it to be a selective system, the main impurities being arsenic, antimony and, at high acidities, germanium. The be-

Table 1
Experimental specific count rates from neutron-produced γ-emitting Sn isotopes for typical irradiation and decay periods (flux $4.5 \times 10^{12}$ n cm$^{-2}$ sec$^{-1}$)

<table>
<thead>
<tr>
<th>Isotope produced</th>
<th>Half-life</th>
<th>Irradiation time</th>
<th>Decay time</th>
<th>γ-Energy, MeV</th>
<th>Peak area, cpm/µg Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{113}$Sn($^{113}$In)</td>
<td>115 d (100 m)</td>
<td>14 d*</td>
<td>14 d</td>
<td>0.392</td>
<td>355*</td>
</tr>
<tr>
<td>$^{117}$Sn</td>
<td>14 d</td>
<td>14 d*</td>
<td>14 d</td>
<td>0.158</td>
<td>1,530*</td>
</tr>
<tr>
<td>$^{123}$Sn</td>
<td>40 m</td>
<td>40 m</td>
<td>40 m</td>
<td>0.160</td>
<td>1,050</td>
</tr>
<tr>
<td>$^{125}$Sn</td>
<td>9.7 m</td>
<td>10 m</td>
<td>10 m</td>
<td>0.332</td>
<td>3,100</td>
</tr>
</tbody>
</table>

*Extrapolated from shorter irradiations.
**Counted in 3" x 3" well-type NaI(Tl) detector.