A SIMPLE METHOD FOR THE ACTIVATION ANALYSIS OF MERCURY IN FISH BY USING IODINATED RESINS

F. Montoloy, M. Heurtebise, J. Lubkowitz

Departamento de Tecnología Nuclear, Instituto Venezolano de Investigaciones Científicas, Caracas (Venezuela)

(Received September 28, 1971)

After irradiation and decay periods, samples are dissolved in 20 min in a sulfuric-nitric acid mixture containing a metavanadate salt. The aqueous phase is neutralized and passed through an iodinated resin which selectively isolates mercury from all other interfering radionuclides. The activity of \(^{197}\)Hg is determined by \(\gamma\)-spectrometry using a thin NaI(Tl) detector. A sensitivity limit of 2.5 ppb* is obtained and the relative standard deviation of the method is 6.7\% at a level of 85 ppb.

Introduction

Established limits of mercury concentrations in fresh fish, canned fish and other products have created in our laboratories the necessity to analyze mercury routinely in large numbers of samples. These analyses are important in establishing a check of the quantity of mercury ingested by human consumption of these products.

Mercury can be analyzed by atomic absorption\(^1\)–\(^3\) and also by fluorescence measurements.\(^4\) Neutron activation analysis (NAA) has also been used since it possesses the inherent quality of high sensitivity.

The possibility has been indicated of determining mercury solely by instrumental means with a sensitivity limit of 30 ppb.\(^5\) This value seems somewhat optimistic. Other methods possess high decontamination factors and permit mercury determination below the 1 ppb level by means of several time-consuming separation steps.\(^6\)–\(^8\) The yields of these separation steps are not generally constant, and the time of manipulation is increased by the determination of the yield factor for every analysis. The precision of these methods is influenced by the necessity of determining the yields. On the other hand, other authors\(^9,10\) indicate the possibility of isolating mercury in one step after dissolution of the sample, but the sensitivities obtained are above the 10 ppb level. An ingenious procedure was recently devised by Kosta and Byrne,\(^11\) which consists in selectively trapping the mercury after combusting the sample at 400 °C. The method is rapid and ensures a sensitivity of 1 ppb. Nevertheless it was not shown whether the method is applicable to

* Throughout the paper the American (10^9) billion is meant.
matrices such as whole fish material. The principal limitation of this method lies in the material needed. The method requires disassembly of the equipment after each manipulation and consequently it does not lend itself to routine application. Previous work in this laboratory by Heurtebise and Ross has shown that iodine is specifically retained on iodinated resins and that it can be separated from the principal constituents of biological fluids. Further studies have been made to establish whether the resin specifically retains other ions. It has been shown that mercury is essentially quantitatively retained on iodinated resins. This work specifically presents the application of such a resin to the analysis of mercury in fresh fish or canned fish. The technique is rapid and a one-step separation after sample dissolution ensures a sensitivity of 2.5 ppb. The efficiency of the procedure is constant at a level of about 97%, and thus there is no need to measure yields.

**Experimental**

*Sample preparation, irradiation and dissolution*

A sample of 1 – 2 g is homogenized and sealed in a quartz ampoule. The standards are prepared by placing 100 – 200 mg NH₄Cl in a quartz ampoule. 5 – 7 µl of a solution of Hg(CH₃COO)₂ (content 100 – 500 ng) at pH 2 is added with a Hamilton 701 NWG microsyringe.

The samples are irradiated for periods of 7 hrs at a flux of approximately \(6 \times 10^{12} \text{n cm}^{-2} \text{ sec}^{-1}\). Each batch of 2 or 3 samples is irradiated with a standard. After irradiation the samples are withdrawn and the isotopes of short half-life are allowed to decay during two or three days.

The quartz ampoules are washed externally with concentrated nitric acid, water and acetone and placed in a liquid nitrogen bath for 3 – 5 min. They are then removed from the bath and wrapped in thin polyethylene sheets. The ampoules are broken, and quartz, plastic sheet and fish material are transferred to a 100-ml round-bottomed flask provided with a 24/40 taper. About 500 µg Hg(CH₃COO)₂ is added to the flask as carrier. A dissolving mixture consisting of 100 ml 14N HNO₃, 20 ml 36N H₂SO₄ and 800 mg ammonium metavanadate is prepared, and 3 ml is added to each flask. A 12-cm Vigreux column is then placed on each flask. Eight samples are processed simultaneously. The round-bottomed flasks are heated to 90 – 96 °C in a water bath for 20 min. Subsequently, 0.5 ml 30% hydrogen peroxide is added to each flask, and the flasks are heated for an additional period of 5 – 10 min. The final solution should be light green and clear; the fats and oils appear as a supernatant phase. The sample mixture is neutralized with 25% NH₄OH solution. The solution changes color from green to brown when the pH is between 5 and 7. The flasks are cooled in an ice bath so as to harden the fats and oils, which are separated by decantation after thorough rinsing with distilled water. The nearly neutral aqueous phase and the organic phase washings are passed through an iodinated resin. The resins are washed with four or five 1-ml washings.

*J. Radioanal. Chem. 11 (1972)*