APPLICATION OF SELECTIVE EVAPORATION
IN THE DETERMINATION OF ARSENIC AND BROMINE
IN DRY BIOLOGICAL MATERIAL
BY THERMAL NEUTRON ACTIVATION ANALYSIS

C. A. WEERS, D. HOEDE, H. A. DAS

Research Centre, 3. Westerdrijn Weg Petten (NH) (The Netherlands)

(Received March 10, 1977)

The application of selective evaporation in an air-flow to the determination of arsenic and bromine in neutron-activated freeze-dried biological material is reported. Bromine is evaporated as Br₂ during oxidative mineralisation. After addition of 48% HBr, arsenic is removed as AsBr₃. The evaporated elements are collected in 7M NaOH; ²¹²Br (T = 35.3 h) and ⁷⁶As (T = 26.4 h) are used for quantitative assay. The chemical yield is (98 ± 2)% for arsenic, irrespective of the amount of arsenic carrier. For bromine it varies from (70 ± 10)% for carrier-free evaporation to (98 ± 2)% if sufficient carrier is added. This addition is not permitted if arsenic has to be determined. The limits of determination are 5 ng · g⁻¹ and 2 ng · g⁻¹ respectively. The precision is < 10% for both elements. The procedure is performed in a standardised apparatus. It was applied to the analysis of standard kale, orchard leaves and animal blood. Bromine was determined instrumentally for purpose of comparison.

Introduction

In an earlier investigation,¹ the evaporation of µg-amounts of a volatile compound from concentrated mineral acid was discussed in terms of the half-volume, \( V_\frac{1}{2} \). This is the gas-volume necessary to evaporate half of the volatile compound. This quantity varies somewhat over the process. The initial value, \( (V_\frac{1}{2})_0 \), can be used to calculate the gas-volume necessary for quantitative evaporation; it is equal to 6.6 \( (V_\frac{1}{2})_0 \). The separation factor for a pair of elements can be expressed in terms of the ratio of the two half-volumes.*

Two practical conclusions on the separation of arsenic from biological samples emerged from earlier work:

(a) Separation should be performed by evaporation at 90–115 °C from a 96% H₂SO₄ – 48% HBr solution.

*In Ref.¹ the symbol for initial value of the half-volume is \( (V_\frac{1}{2})_0 \).
(b) The necessary decontamination from bromine cannot be achieved by this evaporation.

The following procedure proved to be convenient for the determination of arsenic by thermal neutron activation:

- The sample is dissolved and mineralized in 96% H$_2$SO$_4$ under dropwise addition of 50% H$_2$O$_2$. Bromine is evaporated as Br$_2$ at 250 °C in an air-stream of ~2 l·h$^{-1}$ and adsorbed in 10 ml 7M NaOH.

- Sufficient 48% HBr is added to the solution to eliminate the excess of oxidant. Arsenic is then evaporated at 90 °C and adsorbed in 7M NaOH. The resulting solution does not contain any other radionuclide than $^{76}$As (T = 26.4 h).

- The process is performed in one apparatus of standard dimensions.

The influence of temperature, amount of sample, amount of 50% H$_2$O$_2$ and the rate of addition were studied, using $^{74}$As (T = 17.8 d) and $^{82}$Br, to optimize the procedure.

It was then applied to NBS orchard leaves, standard kale, IAEA dried animal blood and a homogenised grass sample.

Experimental

**Chemicals and apparatus**

Demineralised water.

H$_2$O$_2$, 50%; H$_2$SO$_4$, 96%; HBr, 48%; NaOH, 7.5M; As$_2$O$_3$; KBrO$_3$. All reagents are of analytical purity.