ACTIVATION ANALYSIS OF HIGH-PURITY SUBSTANCES
BY MEANS OF SHORT-LIVED ISOTOPES

E. M. LOBANOV, A. G. DUTOV, G. V. LEUSHKINA

Atomic Energy Commission, Moscow (USSR)

A non-destructive activation analysis method intended for industrial utilization is described, using isotopes with half-lives from 2 sec to 20 min. Expected sensitivity values for 33 elements are listed. The effects on the results of neutron flux variation and other errors of measurement are discussed. Requirements to be met by the electronic spectrometer apparatus are stated.

Introduction

The objective of the present work was to develop rapid instrumental activation analysis methods based on the use of short-lived isotopes. Isomers and isotopes of various elements with half-lives from 2 sec to 20 min were used.

Experimental

Equipment for analyses consisted of a pneumatic transfer apparatus mounted on the horizontal channel of a VVR-S reactor, a scintillation spectrometer and electronic apparatus.

The two-channel pneumatic transfer apparatus consisted of aluminium tubes 31 mm in diameter. To allow irradiation with epithermal neutrons, one of the irradiation channels was coated with a 1 mm layer of cadmium and located at a distance from the active zone of the reactor exceeding that of the second irradiation channel by 5 cm. This layout eliminates the effect of the large amount of cadmium on the neutron spectrum in the second irradiation channel.

Compressed nitrogen was used for pneumatic transfer, because it fully satisfies the main requirement of low initial activity. A pressure reducer and a receiver having a volume ten times the total volume of the two pneumatic channels provided for constant operating pressure.
High-precision calibration of the inner diameter of the tubes and high polish of their internal surface ensured constant transfer times. The operating time of the valves was less than one tenth of the transfer time.

To reduce friction and thereby transfer time, the diameter of the containers was 1.5-2 mm less than the inner diameter of the tubes. The design of the polyethylene containers guaranteed automatic ejection of the sample in a period shorter than the transfer time. Polyethylene was chosen as the material for the containers owing to its low friction, good abrasive properties and relatively high resistance to radiation.

The activity of the container, when in the measuring position, was shielded from the detector by a lead sheath fitted with profiled tubes, conveying the sample ejected from the container onto the detector.

The transfer time of the sample for a channel length of ~10 m, at a pressure of 6 atm. in the reversing bulb, was 0.5 sec. Variations in transfer time (affecting the accuracy of analysis) did not exceed 1.3% at a fixed pressure. The position of the container (and sample) in the active zone of the reactor was fixed by means of the nitrogen pressure with an accuracy better than 1%.

Particular attention was paid to the effect of neutron flux variations with time.

The use of flux monitors serves no purpose, since not only the flux, but also the energy spectrum of the neutrons changes during reactor operation. The measured variation of the flux integrated over 7.2 sec (i.e. the half-life of $^{197m}$Au) and over 17.5 sec (i.e. the half-life of $^{77m}$Se) did not exceed 11% during 4 hrs of reactor operation. In the course of shorter periods (5-10 min), flux variations were substantially lower. Such periods were therefore accepted as the maximum time during which the sample and the standard must be irradiated in the case of separate irradiation. Since the time of measurement of one sample was low (~2T), it appeared reasonable to irradiate one standard for 4-5 specimens.

The number of short-lived isotopes whose nuclear characteristics required for sensitivity calculations are fully known is relatively small: only about 30% of all isotopes. Common calculation procedures do not fully reflect all experimental conditions, and their results usually differ by one or two orders of magnitude from the experimental values. For this reason, it appears preferable to determine sensitivity values experimentally.

These determination were carried out by irradiating of the most frequently occurring elements and measuring the activities of their short-lived isotopes formed in $(n, \gamma)$ or $(n, n')$ reactions with thermal and resonance neutrons. A flux of $3.8 \times 10^{11}$ n.cm$^{-2}$sec$^{-1}$ was used for activation. The ratio between the counting rate in the photopeak and the absolute activity of the isotope was measured as a function of the energy of the gamma-radiation, at a fixed position of the sample of the detector. The content of the respective element in the standard was $10^{-6}$-$10^{-3}$ g. The absolute activity of the isotope was calculated from the measured area of the photopeak, taking photoefficiency values into account. The obtained values were then reduced to the value corresponding to an irradiation time of T, a decay time of...