Because of the short half life of $^{28}$Al, the determination of aluminum by neutron activation is subject to many inaccuracies: variation of irradiation conditions between sample and standard, uncertainties in timing, and the effects of high and varying count rate, in addition to other sources of error that must be controlled even in work with long-lived nuclides. These errors can all be made smaller than the fundamental limit set by counting statistics, even when that limit is below 0.5 percent. The transfer function from the observed number of net counts to the counting rate at the end of irradiation is modeled as a product of three processes: radioactive decay and extending and nonextending dead time.

The procedure has been applied to the analysis of NBS SRM 1633a Fly Ash. The mean concentration measured was 14.085% Al, with a standard deviation of the mean 0.023% Al for four determinations. The final results showed no significant imprecision beyond counting statistics. The accuracy of the method is shown by the analysis of high-purity single-crystal sapphire.

**Introduction**

Aluminum is the second most abundant metal in the earth's crust, and activates so well with thermal neutrons that $^{28}$Al often greatly predominates in the gamma-ray spectrum of neutron-irradiated materials of geological or botanical origin for the first few minutes after irradiation. For both of these reasons, aluminum is often useful as a normalizing element, or a marker for the crustal component of the sample. Although determination of Al by neutron activation analysis to $^{28}$Al is subject to many systematic errors, the aggregate of these errors can be made comparable to counting statistics, even when that error is below 0.5 percent. No specialized equipment is required.

**Accuracy requirements**

If an overall accuracy of better than 0.5% is to be attained, each source of systematic error must be controlled to be substantially less than this. Allowing for some cancellation of high bias by low, a reasonable goal is that each individual source of error be less than 0.1%. Most biases may be made sufficiently small by the choice of experimental conditions; others can be compensated by the use of a suitable standard which is treated in the same way as the sample.
Sample Preparation

For the analysis of SRM 1633a Fly Ash, errors in sampling due to heterogeneity of the material are assumed to be negligible for a major element in 200-mg samples /1,2,3/. This assumption is testable after the analysis is completed by comparing the observed scatter among replicates with that expected from counting statistics. The low moisture affinity of this material and the large sample size taken ensure that weighing errors are negligible.

Irradiation

There are in general variations in the neutron flux with space and time, there may be neutron shielding by the sample container or self-shielding by the sample itself, and there may be interfering nuclear reactions which produce the radionuclide measured or another nuclide which may be confounded with the desired species. In the RT4 pneumatic facility of the NBS Reactor, for example, there is a longitudinal flux gradient of 5%/cm /4/, which requires positioning accuracy of $(0.1%)/(5%/cm) = 0.02$ cm. This is clearly difficult to attain directly; the alternative employed is to irradiate each sample sandwiched between two standards (or vice versa). The fact that the flux gradient is closely linear for this facility /5/ ensures that the compensation is excellent. Simultaneous irradiation of the sample and standard also removes all error from temporal variations in the flux and uncertainties in the irradiation timing.

Shielding of the sample from neutrons by the irradiation container may not be negligible at this level of precision. (The hydrogen in a snap-top polyethylene vial with a wall thickness of 0.09 cm absorbs 0.3% of the neutrons passing through the wall.) This external absorption is the same for sample and standard. Neutron shielding of the sample-standard sandwich by itself is calculable for simple geometries /6/ if the contents of the major absorbing elements are known. The calculations may be checked by varying the sample size. For the size and composition of the samples used in this work, self-shielding is small. Fast-neutron reactions on P and Si produce Al-28 which can interfere with the slow-neutron capture in Al, but are entirely negligible in this matrix in the RT4 facility of the NBSR /7/.

Counting

The unknown sample and the standard must be counted with the same efficiency. A solid-angle calculation shows that for 0.1% error, reproducibility to 0.2 mm (at 40 cm) is required in the distance between the detector and the counting specimen. Because Al-28 decays so rapidly, for 0.1% accuracy the time difference between the start of the sample count and that of the standard be known to within 0.2 sec.

The most troublesome sources of error in counting arise from the necessity of collecting more than 1,000,000 counts in the photopeak within 5 minutes in order to attain 0.1% precision. Depending on the peak/Compton ratio of the detector and