An Overview of Neutron Activation Analysis

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Introduction

Neutron Activation Analysis (NAA) is a sensitive analytical technique useful for performing both qualitative and quantitative multielement analysis of major, minor, and trace elements in samples from almost every conceivable field of scientific or technical interest. For many elements and applications, NAA offers sensitivities that are superior to those attainable by other methods, on the order of parts per billion or better. In addition, because of its accuracy and reliability, NAA is generally recognized as the "referee method" of choice when new procedures are being developed or when other methods yield results that do not agree. Worldwide application of NAA is so widespread it is estimated that approximately 100,000 samples undergo analysis each year. (For more information about all analytical services at MURR)

Neutron activation analysis was discovered in 1936 when Hevesy and Levi found that samples containing certain rare earth elements became highly radioactive after exposure to a source of neutrons. From this observation, they quickly recognized the potential of employing nuclear reactions on samples followed by measurement of the induced radioactivity to facilitate both qualitative and quantitative identification of the elements present in the samples.

The basic essentials required to carry out an analysis of samples by NAA are a source of neutrons, instrumentation suitable for detecting gamma rays, and a detailed knowledge of the reactions that occur when neutrons interact with target nuclei. Brief descriptions of the NAA method, reactor neutron sources, and gamma-ray detection are given below.

The NAA Method

The sequence of events occurring during the most common type of nuclear reaction used for NAA, namely the neutron capture or (n,gamma) reaction, is illustrated in Figure 1. When a neutron interacts with the target nucleus via a non-elastic collision, a compound nucleus forms in an excited state. The excitation energy of the compound nucleus is due to the binding energy of the neutron with the nucleus. The compound nucleus will almost instantaneously de-excite into a more stable configuration through emission of one or more characteristic prompt gamma rays. In many cases, this new configuration yields a radioactive nucleus which also de-excites (or decays) by emission of one or more characteristic delayed gamma rays, but at a much slower rate according to the unique half-life of the radioactive nucleus. Depending upon the particular radioactive species, half-lives can range from fractions of a second to several years.



Fig. 1. Diagram illustrating the process of neutron capture by a target nucleus followed by the emission of gamma rays.

In principle, therefore, with respect to the time of measurement, NAA falls into two categories: (1) prompt gamma-ray neutron activation analysis (PGNAA), where measurements take place during irradiation, or (2) delayed gamma-ray neutron activation analysis (DGNAA), where the measurements follow radioactive decay. The latter operational mode is more common; thus, when one mentions NAA it is generally assumed that measurement of the delayed gamma rays is intended. About 70% of the elements have properties suitable for measurement by NAA.

Neutrons

Although there are several types of neutron sources (reactors, accelerators, and radioisotopic neutron emitters) one can use for NAA, nuclear reactors with their high fluxes of neutrons from uranium fission offer the highest available sensitivities for most elements. Different types of reactors and different positions within a reactor can vary considerably with regard to their neutron energy distributions and fluxes due to the materials used to moderate (or reduce the energies of) the primary fission neutrons. However, as shown in Figure 2, most neutron energy distributions are quite broad and consist of three principal components (thermal, epithermal, and fast).



Fig. 2. A typical reactor neutron energy spectrum showing the various components used to describe the neutron energy regions.

The thermal neutron component consists of low-energy neutrons (energies below 0.5 eV) in thermal equilibrium with atoms in the reactor's moderator. At room temperature, the energy spectrum of thermal neutrons is best described by a Maxwell-Boltzmann distribution with a mean energy of 0.025 eV and a most probable velocity of 2200 m/s. In most reactor irradiation positions, 90-95% of the neutrons that bombard a sample are thermal neutrons. In general, a one-megawatt reactor has a peak thermal neutron flux of approximately 1E13 neutrons per square centimeter per second.

The epithermal neutron component consists of neutrons (energies from 0.5 eV to about 0.5 MeV) which have been only partially moderated. A cadmium foil 1 mm thick absorbs all thermal neutrons but will allow epithermal and fast neutrons above 0.5 eV in energy to pass through. In a typical unshielded reactor irradiation position, the epithermal neutron flux represents about 2% the total neutron flux. Both thermal and epithermal neutrons induce (n,gamma) reactions on target nuclei. An NAA technique that employs only epithermal neutrons to induce (n,gamma) reactions by irradiating the samples being analyzed inside either cadmium or boron shields is called epithermal neutron activation analysis (ENAA).

The fast neutron component of the neutron spectrum (energies above 0.5 MeV) consists of the primary fission neutrons which still have much of their original energy following fission. Fast neutrons contribute very little to the (n,gamma) reaction, but instead induce nuclear reactions where the ejection of one or more nuclear particles - (n,p), (n,n'), and (n,2n) - are prevalent. In a typical reactor irradiation position, about 5% of the total flux consists of fast neutrons. An NAA technique that employs nuclear reactions induced by fast neutrons is called fast neutron activation analysis (FNAA).

Prompt vs. Delayed NAA

As mentioned earlier, the NAA technique can be categorized according to whether gamma rays are measured during neutron irradiation (PGNAA) or at some time after the end of the irradiation (DGNAA). The PGNAA technique is generally performed by using a beam of neutrons extracted through a reactor beam port. Fluxes on samples irradiated in beams are on the order of one million times lower than on samples inside a reactor but detectors can be placed very close to the sample compensating for much of the loss in sensitivity due to flux. The PGNAA technique is most applicable to elements with extremely high neutron capture cross-sections (B, Cd, Sm, and Gd); elements which decay too rapidly to be measured by DGNAA; elements that produce only stable isotopes; or elements with weak decay gamma-ray intensities.

DGNAA (sometimes called conventional NAA) is useful for the vast majority of elements that produce radioactive nuclides. The technique is flexible with respect to time such that the sensitivity for a long-lived radionuclide that suffers from an interference by a shorter-lived radionuclide can be improved by waiting for the short-lived radionuclide to decay. This selectivity is a key advantage of DGNAA over other analytical methods.

Instrumental vs. Radiochemical NAA

With the use of automated sample handling, gamma-ray measurement with solid-state detectors, and computerized data processing it is generally possible to simultaneously measure more than thirty elements in most sample types without chemical processing. The application of purely instrumental procedures is commonly called instrumental neutron activation analysis (INAA) and is one of NAA's most important advantages over other analytical techniques. If chemical separations are done to samples after irradiation to remove interferences or to concentrate the radioisotope of interest, the technique is called radiochemical neutron activation analysis (RNAA). The latter technique is performed infrequently due to its high labor cost.

Measurement of Gamma Rays

The instrumentation used to measure gamma rays from radioactive samples generally consists of a semiconductor detector, associated electronics, and a computer-based, multi-channel analyzer (MCA/computer). Most NAA labs operate one or more hyperpure or intrinsic germanium (HPGe) detectors which operate at liquid nitrogen temperatures (77 degrees K) by mounting the germanium crystal in a vacuum cryostat, thermally connected to a copper rod or "cold finger". Although HPGe detectors come in many different designs and sizes, the most common type of detector is the coaxial detector which in NAA is useful for measurement of gamma-rays with energies over the range from about 60 keV to 3.0 MeV.

The two most important performance characteristics requiring consideration when purchasing a new HPGe detector are resolution and efficiency. Other characteristics to consider are peak shape, peak-to-Compton ratio, crystal dimensions or shape, and price.

The detector's resolution is a measure of its ability to separate closely spaced peaks in a spectrum. In general, detector resolution is specified in terms of the full width at half maximum (FWHM) of the 122-keV photopeak of Co-57 and the 1332-keV photopeak of Co-60. For most NAA applications, a detector with 1.0-keV resolution or below at 122 keV and 1.8 keV or below at 1332 keV is sufficient.

Detector efficiency depends on the energy of the measured radiation, the solid angle between sample and detector crystal, and the active volume of the crystal. A larger volume detector will have a higher efficiency. In general, detector efficiency is measured relative to a 3-inch by 3-inch sodium iodide detector using a Co-60 source (1332-keV gamma ray) at a distance of 25 cm from the crystal face. A general rule of thumb for germanium detectors is 1 percent efficiency per each 5 cc of active volume. As detector volume increases, the detector resolution gradually decreases. For most NAA applications, an HPGe detector of 15-30 percent efficiency is adequate.

Typical gamma-ray spectra from an irradiated pottery specimen are shown in Figures 3-5 using two different irradiation and measurement procedures.





Fig. 3. Gamma-ray spectrum showing several short-lived elements measured in a sample of pottery irradiated for 5 seconds, decayed for 25 minutes, and counted for 12 minutes with an HPGe detector.



Fig. 4. Gamma-ray spectrum from 0 to 800 keV showing medium- and long-lived elements measured in a sample of pottery irradiated for 24 hours, decayed for 9 days, and counted for 30 minutes on a HPGe detector.



Fig. 5. Gamma-ray spectrum from 800 to 1600 keV showing medium- and long-lived elements measured in a sample of pottery irradiated for 24 hours, decayed for 9 days, and counted for 30 minutes on a HPGe dectector.

Using Gamma-ray Counts to Calculate Element Concentration

The procedure generally used to calculate concentration (i.e., ppm of element) in the unknown sample is to irradiate the unknown sample and a comparator standard containing a known amount of the element of interest together in the reactor. If the unknown sample and the comparator standard are both measured on the same detector, then one needs to correct the difference in decay between the two. One usually decay corrects the measured counts (or activity) for both samples back to the end of irradiation using the half-life of the measured isotope. The equation used to calculate the mass of an element in the unknown sample relative to the comparator standard is

$$\frac{A_{sam}}{A_{sad}} = \frac{m_{sam}}{m_{sad}} \frac{(e^{-\lambda T_d})_{sam}}{(e^{-\lambda T_d})_{sad}}$$

where A = activity of the sample (sam) and standard (std), M = mass of the element, $\lambda = decay$ constant for the isotope and $T_d = decay$ time. When performing short irradiations, the irradiation, decay and counting times are normally fixed the same for all samples and standards such that the time dependent factors cancel. Thus the above equation simplifies into

$$C_{sam} = C_{sai} \frac{W_{sai}}{W_{sam}} \frac{A_{sam}}{A_{sai}}$$

where C = concentration of the element and W = weight of the sample and standard.

Sensitivities Available by NAA

The sensitivities for NAA are dependent upon the irradiation parameters (i.e., neutron flux, irradiation and decay times), measurement conditions (i.e., measurement time, detector efficiency), nuclear parameters of the elements being measured (i.e., isotope abundance, neutron cross-section, half-life, and gamma-ray abundance). The accuracy of an individual NAA determination usually ranges between 1 to 10 percent of the reported value. Table I lists the approximate sensitivities for determination of elements assuming interference free spectra.

Table I. Estimated detection limits for INAA using decay gamma rays. Assuming irradiation in a reactor neutron flux of 1×10^{13} n cm⁻² s⁻¹.

| Sensitivity (picograms) | Elements |
|----------------------------|---|
| 1 | Dy, Eu |
| 1 - 10 | In, Lu, Mn |
| 10 - 100 | Au, Ho, Ir, Re, Sm, W |
| 100 - 1E3 | Ag, Ar, As, Br, Cl, Co, Cs, Cu, Er, Ga, Hf, I, La, Sb, Sc, Se, Ta, Tb, Th, Tm, U, V, Yb |
| 1E3 - 1E4 | Al, Ba, Cd, Ce, Cr, Hg, Kr, Gd, Ge, Mo, Na, Nd, Ni, Os, Pd, Rb, Rh, Ru, Sr, Te, Zn, Zr |
| 1E4 - 1E5 | Bi, Ca, K, Mg, P, Pt, Si, Sn, Ti, Tl, Xe, Y |
| 1E5 - 1E6 | F, Fe, Nb, Ne |
| 1E7 | Pb, S |

Related Books

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Possible Applications for NAA

1. Archaeology

The use of neutron activation analysis to characterize archaeological specimens (e.g., pottery, obsidian, chert, basalt and limestone) and to relate the artifacts to sources through their chemical signatures is a well-established application of the method. Over the past decade, large databases of chemical fingerprints for clays, obsidian, chert and basalt have been accumulated through analysis of approximately thirty elements in each of more than 42,000 specimens. The combination of these databases with powerful multivariate statistical methods (i.e., principal components analysis, factor analysis, discriminant analysis, and Mahalanobis distance probabilities) allows many archaeological materials to be sourced with a high degree of confidence. The sourcing information can help archaeologists reconstruct the habits of prehistoric peoples. For example, the "fingerprinting" of obsidian artifacts by NAA is a nearly 100 percent successful method for determining prehistoric trade routes since sources of obsidian are easily differentiated from one another through their chemical compositions. For more information concerning this application of NAA, see the Archaeometry Laboratory at MURR. A recommended charge schedule for instrumental neutron activation analysis of archaeological and geological specimens is available (See Charge Schedule).

2. Study the Redistribution of Uranium and Thorium due to Ore Processing

In the 10 years between 1948 and 1958 uranium ore mining in the United States expanded from a cumulative total of 38,000 tons to 5.2 million tons involving more than 400 mines. Ores from these mines were chemically processed at an estimated 50 to 100 sites. These activities have resulted in the contamination of hundreds of square miles of surface and subsurface soils, and their corresponding ground waters, with uranium-238, thorium-232 and their radioactive daughters. In most cases these sites have various radioactive waste materials such as ore tailings, processing residues and leachates mixed with soil and generally covered with an uncontaminated soil layer several feet in thickness. In other cases the tailings or wastes have remained exposed and hence have been further distributed by wind and erosion. EBNAA has been developed as a methodology suitable for automation by which contaminated ore-processing sites can be characterized and their restoration monitored.

3. The Use of Radiotracers to Study the Fate of Hazardous Elements in Waste Material/Coal-Char Admixtures under Gasification-an Emerging Waste Management Technology

With an increasing emphasis being placed on the cleanup of hazardous waste sites from past technological operations, science has had to come up with ideas on how to simultaneously decompose and stabilize a variety of mixed wastes which include radioactive materials. The most difficult of all wastes to cleanup is mixed waste. These may contain organic materials (dichlorobenzene, naphthalene, etc) along with hazardous elements (cadmium, mercury, plutonium, thorium, uranium and other transuranics). Current waste destruction technology relies on oxidation of the waste (incineration); an alternative is the storage of this waste. Both have drawbacks. Incineration requires high temperatures and the resulting high fuel costs and volatilization of some hazardous components. Storage costs are proportional to the amount of space taken up by the waste and the regulations surrounding the waste, therefore, long term storage of some existing radioactive waste is unacceptable without volume reduction and stabilization. A process developed at MU, ChemChar gasification, helps minimize these problems. ChemChar gasification using a coal char (a triple reverse burn coal product which is very porous) acts as both a surface for chemical reactions and a sequestering agent of hazardous elements. Initially the process uses the char as an absorber of the waste stream which is mixed on a 2:1 weight ratio (char: waste). A flame front is established which moves opposite the direction of oxygen flow and in the flame front organics are decomposed and other species tend to be chemically reduced. The volatile organics are carried off and trapped later while the immobile metals, metalloids and some non-metals are sequestered on the char for final disposal or other disposition. The gasification process also results in volume reduction, partitioning of potentially usable organic solvents and the production of a gas that could be used as an energy source. The objective of the MURR component of this project is to develop a procedure by which radiotracers can be used to determine the fates of hazardous elements during gasification in support of the use of gasification as a means of decomposing, stabilizing and reducing the volume of hazardous mixed wastes including radioactive wastes resulting from the nuclear weapons program. To date the fates of arsenic, strontium, cadmium, cesium, mercury, uranium, thorium, neptunium and protactinium, chlorine (as an organochloride) and phosphorus (as organophosphorus surrogates for military wastes) have been studied in this reducing atmosphere using MURR produced radiotracers

4. Selenium Distribution in Aquatic Species in Selenium-contaminated Fresh-water Impoundments

This work is being done in collaboration with both federal and state agencies. Fresh-water ecosystems in California have been grossly contaminated with selenium as a result of irrigation run-off from heavily used agricultural areas. The effect has been observed throughout most of the food chain. The objective of these studies is to evaluate the extent of the contamination and evaluate methodologies that might be efficacious in the reduction of selenium in these ecosystems.

5. In-situ Radiotracers for Dosage-Form Testing

Over the last several years there has been a growing interest in the use of *in-situ* radiotracers to test new pharmaceuticals and dosage forms being developed for commercial distribution. This work has been done in collaboration with several pharmaceutical companies and university research centers. The *in-situ* radiotracers are produced through carefully designed irradiations done at the MURR and used in laboratory, animal, and in a few cases, human experiments. These methodologies offer significant advantages in the evaluation of encapsulations, time release, clearance and the distribution of the pharmaceutical in animal and human models.

6. Nutritional Epidemiology -- Nutritional and Biochemical/Genetic Markers of Cancer

This project is being done in collaboration with the Harvard Medical School stemming from the rather striking findings reported in JAMA (December, 1996) showing a significant protective effect due to a 200 microgram/day supplement of selenium against cancers of the colon, lung and prostate. One specific aim of the project is to evaluate the relationship between selenium status and cancer of the colon and prostate in the Physicians= Health Study. As proposed, this is a five-year study using prospectively collected blood samples as the biologic monitor of selenium status. If fully funded, this would be the largest prospective case-control study to date of selenium status and incidence of prostate cancer. Over the five years 2470 prostate cancer cases and 850 colon cancer cases are anticipated.

7. Nutritional Epidemiology -- A Cohort Study of the Relationship Between Diet, Molecular Markers, and Cancer Risk: the Canadian Study of Diet, Lifestyle and Health

A pilot study to characterize the nail as a biologic monitor for the dietary intake for selenium in Canada has been undertaken to extend our work to the major Canadian provinces. Suspected selenium determinants such as smoking, use of dietary supplements, age and gender will be statistically evaluated in a population of over 700 subjects. Results will be compared with our earlier study of a

female population drawn from 11 states in the U.S. The objective of this work is to expand the collaborative MURR nutritional epidemiology program, that has enjoyed good success in the U.S., to Canada. This pilot will serve as a basis for two grant applications that will be submitted to the National Cancer Institute of Canada and the Canadian Research Council. The work is being done in collaboration with the Faculty of Medicine, University of Toronto.

8. Nutritional Epidemiology -- Thyroid Cancer Study

This study is being done in collaboration with the Northern California Cancer Center, Stanford University and the University of California, San Francisco. Asian women who have immigrated to the U.S. west coast experience a significantly increased incidence of thyroid cancer which is hypothesized to be a consequence of their substantial change in diet. Specifically, iodine intake is substantially increased and may stress a hypersensitive thyroid --a condition observed in many of these subjects presumably due to chronically low iodine intakes prior to immigration. MURR is responsible for two specific aims: characterizing the nail as a biologic monitor for iodine; and to measure iodine in nails obtained in a nested case-control study of thyroid cancer. In these subjects, the use of iodine-containing contrast agents can confound analytical interpretations of any biologic monitor. From our work we have found that biologic monitors from subjects exposed to iodine-containing contrast agents will be influenced by the exposure for over a year. Consequently, these subjects should be screened out of nested case-control studies having an iodine hypothesis. The case-control comparison is blinded to our laboratory and will be evaluated beginning in 1999.

9. Nutritional Epidemiology -- Non-Melanoma Skin Cancer Study

This study is being done in collaboration with Dartmouth Medical School. There is some evidence that higher than normal intakes of arsenic may increase the risk of non-melanoma skin cancer. This hypothesis is being studied in a New Hampshire population routinely ingesting comparatively high levels of arsenic in their drinking water supplies which are typically wells serving a single residence or just a few residences. We have shown that the arsenic concentration in nails is directly correlated with drinking-water arsenic levels. An NAA procedure to measure arsenic in nails has been developed and applied in this nested pseudo-prospective case-control study. Over 1000 samples have been analyzed over the last 2 years which is approximately the midpoint of the project. The case-control status will remain blind to our laboratory until the project is concluded.

10. Nutritional Epidemiology -- Molecular Epidemiology of Prostate Cancer

In this study, being conducted in collaboration with Johns Hopkins University, School of Hygiene and Public Health, we are investigating the relationship of dietary and occupational exposures to selenium, cadmium and zinc and the incidence of prostate cancer. This is the most extensive study to date in which we have the opportunity to compare two biologic monitors, nails and blood sera or plasma, in a nested case-control study having a specific disease outcome.

11. Knock-Out Gene Mouse Model for Cystic Fibrosis

Mineral characterization studies with the knockout gene cystic fibrosis (CF) mouse model have been done in collaboration with the MU Department of Veterinary Biomedical Science. Bone and tooth mineralization differences between CF and normal mice were studied using NAA to measure Ca, Mg, P, Na, K, F, Cl, Br and Mn. NAA was also used to measure mineralization differences in the whole-body of the CF and normal mouse.

12. Calcium Metabolism Study

In support of the MU Department of Child Health we have studied calcium absorption in pig and lamb models using Ca-47 produced and processed at the MURR from an enriched Ca-46 target. In support of human studies with low birth-weight infants, juvenile rheumatoid arthritics and cystic fibrotics, a dual-enriched-isotope methodology, based on stable isotopes measured via NAA, has been developed to measure true absorption of calcium from experimental diets.

13. Geological science

Analysis of rock specimens by neutron activation analysis assists geochemists in research on the processes involved in the formation of different rocks through the analysis of the rare earth elements (REEs) and other trace elements. About thirty elements can be measured routinely in almost any geological sample. An additional 15-20 elements can be measured by applying specialized procedures. In addition to modeling geochemical processes, other applications include location of ore deposits and tracking elements of environmental importance. For example, the discovery of anomalously high iridium concentrations in 65-million-year old limestone deposits from Italy and Denmark could only have been accomplished by NAA. The NAA findings support the theory that

extinction of the dinosaurs occurred soon after the impact of a large meteorite with the earth. For more information about this application for NAA, see the Archaeometry Laboratory at MURR. A recommended charge schedule for instrumental neutron activation analysis of archaeological and geological specimens is available (See Charge Schedule).

14. Semiconductor materials and other high-purity materials

Neutron Activation Analysis (NAA) is used to measure trace- and ultra trace-element concentrations of impurities and/or dopants in semiconductors and other high-purity materials. The behavior of semiconductor devices is strongly influenced by the presence of impurity elements either added intentionally (doping with B, P, As, Au, etc.) or contaminants remaining due to incomplete purification of the semiconductor material during device manufacture. Small quantities of impurities present at concentrations below 1 ppb can have a significant effect on the quality of semiconductor devices. The objective of the client is to demonstrate that a chemical or material meets or exceeds purity requirements required by the end user. In some instances the MURR Nuclear Analysis Group conducts a multi-element qualitative and quantitative analysis which then is used as the certificate of analysis for that substance. In other cases the NA staff work with clients to establish purification factors at various stages in the production process of high-purity materials such as silicon or, the efficacy of cleaning and leaching procedures. In still other cases we are ask to demonstrate that a specific impurity or set of impurities is below the level of technical or regulatory concern. NAA is the elemental analysis method of choice for these projects because of the limited sample handling required and the high sensitivity for many elements of interest. For more information about this application of NAA, see NAA of Semiconductor Materials.

15. Soil Science

Many agricultural processes and their consequences, such as fertilization and herbicidal and pesticidal control, are influenced by surface and sub-surface movement, percolation and infiltration of water. Stable activatable tracers, such as bromide, analyzed by NAA, have allowed the soil scientist to quantify the distribution of agricultural chemicals under a wide variety of environmental and land use influences. In soil science (ca. 1970), the use of bromide ion (Br⁻) in various forms (e.g. KBr, NaBr, SrBr₂) was introduced as a non-reactive stable tracer in solute transport studies normally moving freely with the flux of water without substantial chemical or physical interactions with the soil. Typically, Br⁻ is extracted from soil and quantified using either a bromide selective electrode (sensitivity is ~10 mg/mL) or by high-performance liquid chromatography (sensitivity is ~0.010 mg/mL). Where the sensitivity is adequate, the selective conductivity method, which is simple, affordable and fast, is preferred. More recently (ca. 1990), workers have reported that 20% of Br⁻ tracers, at low groundwater pH, may be absorbed by iron oxides and kaolinite when present in the alluvial aquifer. We investigated the use of epithermal neutron activation analysis (ENAA) as a means of measuring Br⁻ directly in soil samples without an extraction. ENAA was chosen because of its high theoretical advantage factor over aluminum (i.e. ~20), the principal interfering soil constituent, calculated for the ⁷⁹Br(n,g)⁸⁰Br reaction compared to ²⁷Al(n,g)²⁸Al Br⁻ was measured (sensitivity is ~0.050 mg/g) in one gram soil samples from a five-second irradiation (f_{th} = 2.5 x 10¹² n/cm²/sec) using a BN capsule.

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