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**A Literature Survey of Chemical Analysis by
Thermal Neutron Induced Capture
Gamma Ray Spectrometry**

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A LITERATURE SURVEY OF CHEMICAL ANALYSIS BY THERMAL NEUTRON INDUCED CAPTURE GAMMA RAY SPECTROMETRY

by

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ABSTRACT

A brief discussion of the principles and techniques of chemical analysis by neutron capture gamma radiation is presented and the widely scattered literature is collected into a single table arranged by element measured.

I. INTRODUCTION

The application of nuclear techniques to chemical analysis is widely practiced. Almost every research reactor has some facility for irradiating small amounts of material in neutron fluxes, extracting the sample, and counting the gamma-radiation as the sample decays. Few reactors, however, are set up so that gamma rays from the sample may be observed during irradiation. Although less versatile than conventional neutron activation analysis, capture gamma-rays have unique analytical applications complementary to other nuclear techniques. This survey will provide an introduction to the equipment and techniques of capture gamma ray analysis and a compilation of the recent literature in the area. It does not present a critical review of the published work, but rather collects the widely scattered references into a single table keyed by element determined.

The emission of gamma radiation during the capture of thermal neutrons was first observed almost simultaneously in two laboratories in 1934.^{1,2} These observations occurred during experiments designed to study neutron scattering from metal foils irradiated with neutrons from a point source. When an ionization chamber alone was employed, only ionization induced by scattered neutrons was observed. Intense hard gamma radiation was observed whenever paraffin or liquid hydrogen were used in the experiments. The experimentors correctly interpreted the phenomenon to be the reaction ${}^1\text{H}(n,\gamma){}^2\text{H}$ and estimated the energy of the gamma-ray as 2 to 4 MeV (2.223 MeV is the presently accepted value). They also observed the newly formed ${}^2\text{H}$ nuclei as short recoil tracks in their detectors.

Analytical utility did not immediately follow discovery. Before the advent of nuclear reactors in the 1940s, Ra-Be and Po-Be sources placed in hydrogenous moderators such as paraffin were the only thermal neutron generators available. Inhibited by low neutron fluxes, these sources were little used in elemental analysis, although they are seeing some use today in bulk materials measurement and quality control.³⁻⁷ With the higher neutron fluxes available from reactors and the evolution of high resolution semiconductor detector systems,⁸⁻¹¹ the use of capture gamma rays in a variety of analytical applications has been increasing. Details of the nuclear physics of the capture process may be found in texts on nuclear chemistry and physics.¹²⁻¹⁵

A. Nuclear Activation and Quantitative Analysis

In general, nuclear activation involves an impinging particle (n, p, ^2H , ^3H , ^3He , ^4He , and heavier ions), or radiation (γ , X), a target nucleus, and emitted radiation and/or particles. Not all these processes result in a radioactive product with a characteristic half-life. The short range of some of these incident particles in matter limits their utility to surface layer analyses only. Although it is possible to do bulk compositional analysis with nonpenetrating particles through the use of special thin-layer sample preparation techniques, only neutrons and gamma rays (photons) have sufficient penetrating power to be used for bulk analysis with minimal sample preparation (typically only grinding to achieve homogeneity).

The basic analytical principles of capture gamma-ray analysis differ little from more conventional instrumental thermal neutron activation techniques (ITNA). The major difference in data accumulation is that capture gamma spectra must be taken *during* the neutron irradiation. Capture gamma-rays have no measurable half-life, with the neutron capture and gamma-ray emission taking place in $\leq 10^{-14}$ seconds. The sample cannot be removed from the reactor and counted at another location, as in conventional thermal neutron activation applications. Neutron fluence during irradiation is determined by measuring the neutron flux near the point of irradiation using a fission ion counter,¹⁶ or by simultaneous or periodic observation of capture γ -rays from a flux monitor (e.g., H in the polyethylene vial or Cr foil) placed in the container with the sample.¹⁶⁻¹⁸

The most common source of thermal neutrons is the nuclear reactor. Two different equipment configurations for capture gamma ray spectrometry have emerged. The sample may be inserted into the thermal neutron flux inside the reactor and the capture gamma rays observed from outside the reactor by a detector viewing a highly collimated gamma-ray beam.^{19,20} The more common approach involves removing a beam of neutrons from the reactor and irradiating the sample outside the reactor.^{17,18,21-38} In the former case, the neutron flux is typically 10^{11} n/cm²/sec¹⁹ and in the latter 10^8 n/cm²/sec.¹⁷ Solid angle considerations result in approximately the same counting rates for these two geometries.¹⁹⁰

The transuranic element Californium also provides a relatively portable source of neutrons. The isotope is ^{252}Cf , which emits fast neutrons during spontaneous fission ($t_{1/2} = 2.55$ y). The wide availability of μg -mg quantities of this isotope has revolutionized the applications of small neutron sources. For thermal neutron work, the fission neutrons must be moderated before use. Usually a hydrogenous or carboniferous matrix is used for moderation - plastics, waxes, or graphite. The advantages of small size and reduced cost relative to reactor neutrons are offset by the much lower neutron fluxes that are obtained.³⁹⁻⁶⁸

Particle accelerators may also be used to excite capture gamma-rays. If thermal neutrons are desired, the charged particles must hit a primary reaction target and the neutrons produced must be thermalized before impingement upon the sample.^{43,69-72} Direct charged particle induction of capture gammas from a sample is an entirely separate field from thermal neutron capture. It permits analysis of a different set of elements. The literature for this technique is not reviewed here, but several papers are suggested as a starting point.⁷³⁻⁸¹

The modern activation facility consists of Ge(Li) and Si(Li) detectors, whose signals are routed through a preamplifier, a linear amplifier, and analog-to-digital converter to digitize the analog energy pulse and then stored in the appropriate channel of a pulse height analyzer or data acquisition minicomputer. Conventional neutron activation consists of the irradiation of unknown samples in a flux of thermal neutrons to produce radioactive isotopes with fixed half-lives for radioactive decay. For the purpose of quantitative analysis, a known amount of the element of interest is usually irradiated simultaneously with the unknown. Complex materials of known composition can also be used [e.g., National Bureau of Standards Standard Reference Materials (see Ref. 82)]. The sample and "standard" are then counted with the same detector for measured periods of time in the same or calibrated "geometries" relative to the detector. During the

counting period, the gamma-ray spectra are accumulated in the pulse height analyzer and transferred to an output medium for computer data reduction. When all counts are complete, the net area under the gamma-ray peak of interest is determined. The method may be as simple as determining the number of channels in the peak, taking $1/2$ this number of channels on each side of the peak and subtracting the contents of these two sets of "background" channels from the sum of the contents of the channels within the peak. Numerous computer programs have been written for "fitting" the spectrum with, for example, Gaussian functions, to smooth discontinuities in the digitized contents of the channels.⁸³⁻⁸⁷

Capture-gamma techniques offer greater precision than ITNA for the determination of elements with short radioactive decay half lives (e.g., ²⁸Al). The counting data from ITNA must have special corrections to compensate for the response time of the measurement electronics to the changing dead times of rapidly decaying sources.⁸⁸⁻⁹⁰ However, the problems of proper elemental identities and overlapping gamma-ray lines are greater in capture gamma analysis, because there is no half-life associated with the line, only relative intensity and energy. The absence of decay also prohibits simplification of spectra by waiting for shorter lived species to decay out as in ITNA.

Two methods of standardization were experimentally explored by Gladney, *et al.*¹⁶ If the neutron cross section of the element of interest is accurately known, then elemental measurements may be accomplished by comparison of gamma ray peak areas with another element of accurately known cross section, specifically either gold or hydrogen. One drawback of this approach is that the relative efficiency of the detector for gamma-rays of widely different energy must have been measured. This comparison of gamma-ray intensities can work well where stoichiometric compounds of the element are unavailable. Gladney, *et al.*¹⁶ found no quantitative difference between using elemental standards or cross section reference standards. Polyethylene irradiation containers provide a built-in H reference, but this convenience is effective only with samples containing little or no H. For example, the H cross section standard was ineffective for NBS Orchard Leaves because it contains some 6% H by weight.

Neutron self-shielding is occasionally a problem. Self-shielding occurs in materials that have a high neutron cross section (e.g., Cd, Cu, Au, B). When placed in a neutron flux, the outside layers of the sample absorb so many neutrons that the inside receives a much lower neutron flux. Thus the inside portion is not activated to the same extent as the outer layers and a low estimation of elemental concentrations results. This problem is not common, but the analyst must be alert to its possible occurrence. A detailed consideration of self shielding in ITNA, including graphs and equations for determining the severity of the effect for various geometries, has been published.⁹¹

B. Guide to the Literature

Thermal neutron capture gamma-ray analysis is a multielement technique. As such, a narrative review of the accumulated literature would be both awkward and lengthy. The available literature on analyses has been summarized on an elemental basis in Table I. Elements are in order of increasing atomic number.

In general, capture gamma-ray analysis is not a trace element technique. Two primary factors govern the detection limit for elemental analysis using the (n,γ) reaction: the partial capture cross section for producing a useful transition and the elemental composition of the matrix material being examined. Limitations of the first type are fundamental and can be assessed from known nuclear properties of the elements. Figure 1 illustrates these considerations.¹⁵⁷⁻¹⁵⁹ Plotted is the partial thermal (n,γ) cross section per unit mass of the most intense transition (regardless of energy) emitted by most of the elements. In most cases the intensity of the characteristic gammas vary over about 2.5 orders of magnitude. It is those cases where elements have a substantial part of the capture cross section concentrated into one gamma that prompt

TABLE I

ELEMENTS MEASURED BY CAPTURE GAMMA-RAY SPECTROMETRY

Element	Reference	Element	Reference
H	18, 25, 29, 36, 41, 45, 49, 53, 56, 92-112	Fe	7, 18, 22, 24, 28, 34, 37, 41, 45, 46, 48, 49, 53, 57, 59, 61, 95, 96, 106, 107, 109, 112, 118, 129, 131-133, 139, 148, 150, 151
Li	113	Co	5, 22, 29, 37, 48, 106, 107, 152
Be	109	Ni	22, 28, 29, 34, 37, 41, 48, 64, 106, 107, 139, 152
B	5, 16, 18, 21, 25, 31, 34, 35, 99, 101, 106, 108, 109, 112-117	Cu	22, 47-49, 61, 69, 106, 131, 133
C	18, 45, 53, 61, 93, 95, 96, 101, 106, 109, 112, 118-121	Zn	22, 28, 48, 61, 133
N	6, 18, 72, 93, 96, 99, 101, 103, 106, 110, 112, 121-128	As	48
O	101, 121	Se	106
Na	18, 48, 49, 72, 99, 101, 103, 104, 106, 107, 112, 118, 126, 129, 130	Sr	61
Mg	18, 61, 107, 120, 129	Zr	42
Al	18, 22, 24, 45, 48, 59, 61, 69, 94, 96, 106, 107, 112, 129, 131	Nb	42, 109
Si	18, 24, 41, 45, 46, 48, 49, 57, 61, 95, 96, 106, 107, 112, 118, 120, 121, 129, 131, 132	Mo	29, 152
P	18, 61, 72, 99, 103, 104, 106, 109, 129, 130,	Rh	21
S	18, 45, 46, 48, 49, 53, 58, 59, 61, 95, 96, 99, 106, 107, 111, 112, 118, 120, 121, 133-141	Ag	21, 28, 48
Cl	5, 18, 22, 45, 46, 48, 49, 72, 96, 99, 103, 104, 106, 112, 118, 130, 142	Cd	5, 16, 18, 21, 22, 54, 61, 103, 106, 109, 112, 115, 124, 126
K	18, 24, 45, 49, 50, 57, 61, 72, 99, 101, 103, 104, 106, 112, 118, 129, 130, 143-145	In	21
Ca	18, 112, 129	Rare Earths	5, 18, 21, 25, 27, 31-33, 48, 107-109, 112, 115, 116, 153
Sc	21, 23	Hf	21
Ti	18, 22, 28, 45, 48, 61, 96, 106, 109, 112, 118, 129, 143, 146	Ta	42
V	18, 22, 28, 48	W	29, 37
Cr	22, 28, 34, 37, 48, 106, 139, 147	Ir	21
Mn	18, 22, 28, 34, 37, 48, 61, 67, 69, 106, 112, 147, 148	Au	21, 22, 24, 28, 48, 51, 61, 150
		Hg	5, 21, 22, 48, 54, 154, 155
		Th	27
		U	55, 156

Fig. 1.

Plot of the single most intense capture gamma-ray emitted by elements following thermal neutron capture. Points for the noble gases and for radioactive elements, except Th and U, are not included.

(n, γ) spectroscopy can be considered a practical tool for trace analysis. With these exceptions, the main analytical usefulness of prompt γ -ray spectroscopy lies in its ability to give fairly rapid, nondestructive determinations of many elements having moderately high concentrations, generally without chemical separations or other complicated sample preparation.

The best elements are usually abundant in the matrices being analyzed, e.g., Si, Al, Fe, C, N, H, or S. Elements with minor abundance in rocks (Na, Mg, Ti, Ca, K) usually require relatively long periods of data accumulation in order to achieve good precision and accuracy.¹²⁹ Only H, B, Cd, and several rare earths exhibit trace to ultra-trace sensitivities.^{16,18,129} The boron peak is especially characteristic in shape. The 478 keV transition is the de-excitation radiation from $^7\text{Li}^*$ produced by the ^{10}B (n, α) ^7Li reaction. Because the $^7\text{Li}^*$ is in recoil flight when the 478 keV γ -ray is emitted, the line is severely doppler broadened (Fig. 2). Many other elements shown in Table I have usually been studied after chemical separation or where they are present in higher than normal crustal abundance (e.g., ores).

A potentially unique feature of capture γ -ray analysis is the ability to study volatile elements in environmental matrices without having to take special precautions. The determination of Hg by conventional thermal neutron activation requires that samples be sealed in quartz irradiation containers. Study revealed that the activated Hg nuclei were escaping from polyethylene containers conveniently used for irradiation of samples.^{160,161} When a neutron is captured by a Hg nucleus the de-exciting prompt gamma rays in the product nucleus causes a recoil energy of 30 to 100 eV, which is sufficient to remove the Hg atom from whatever compound it is in. These "free" atoms readily escape during long neutron irradiations. Although the same thing happens during capture gamma-ray irradiations, the capture gamma is emitted before the volatile element can be lost, so accuracy is preserved. The references under Hg in Table I provide further discussion of this point.

Irradiation containers frequently provide the greatest source of background radiation. Plastic or graphite "rabbits" have strong H and C lines. Most common metals also have strong capture radiations. Beryllium offers some potential,⁹³ but has an intense capture gamma-ray at 853 keV. The Compton scattering at energies below this line effectively eliminates the measurement of any capture radiation in this part of the spectrum. However, for determination of C, H, and N in biologicals, this Be "rabbit" eliminates the interference from capture radiation of the more common "rabbit" construction materials.

C. Data Reduction

One of the cornerstones of any form of gamma or x-ray analysis is a good table of energies, relative intensities, and, where appropriate, half lives. Extensive tabulations of capture gamma rays exist for both NaI(Tl)¹⁶²⁻¹⁶⁵ and Ge(Li)^{157-159,166,167} detectors.

Data reduction is normally accomplished using standard gamma-ray analysis programs from ITNA. The complexity of the capture spectrum has prompted several attempts to tailor a computer program specifically for these analyses.^{143,168,169} On occasion, reducing spectral data by hand has certain advantages. Computer programs will generally not fit the flat topped, non-Gaussian 478 keV boron γ -ray (Fig. 2). When there is another gamma-ray nearby, such as the 473 keV Na capture gamma illustrated in Fig. 2, computer programs have a great deal of difficulty. What can be done is select a portion (10 channels) where the boron peak is still flat, and integrate just this region in both sample and standards. Curtis, *et al.*¹⁷⁰ have demonstrated that this approach is quantitative; in fact, Fig. 2 is drawn from work on boron abundance in meteorites where spectral interference on one or both flanks of the peak was a common problem.

D. Applications of Capture Gamma-Ray Analysis

The applications discussed in the literature studied are as varied as the number of elements measured. The primary one in the past has been nuclear spectroscopy (e.g., Refs. 190, 94, 171.

Fig. 2.

Doppler broadened capture gamma ray from $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction with severe Na interference on left flank.

and 172). The construction and evaluation of nuclear level schemes has long employed capture gamma-rays, but only a few examples are cited because a review of that material is outside the scope of this paper. The establishment of correct decay schemes is a prerequisite for analytical use of activation analysis.

E. Industrial

The bulk analysis and quality control potentiality of capture gammas is being exploited.^{3,48,119,173} Capture gamma-rays induced by rugged neutron-emitting isotopic sources have proven to be a good monitoring technique for elemental concentrations in various process streams and slurries.^{47,48,59,95,114,133,174} On-line determination of sulfur contents of oil^{58,134} and coal^{45,46,53,59,60,95,96,111,112,121,135,138} as well as other major elements permits rapid evaluation of product quality. Gross compositions and specifications of steels, alloys, glasses, and concrete can be checked at the time of production and again at delivery.^{28,34,36,37,50,94,138,175} Chlorine and nitrogen contents of industrial organic compounds may also be evaluated.^{5,6} Shielding and materials for reactor construction have been examined.^{65,94,115,176,177} Metals and water content in catalysts are being measured.^{29,56,97,152} It may also be possible to use capture gamma techniques to search for buried explosives.¹²²

F. Biological—Agricultural—Medical

Major element analysis of biological materials can be easily performed with capture gamma-rays.^{23,76,93,98,99} Medical diagnosis based on elemental data has been examined¹⁷⁸ and *in-vivo*, *in-situ* measurements of whole body concentrations of N, H, Na, Ca, Cl, P, and Cd, as well as exchange rates in various tissues, have been studied.^{23,71,72,99-104,110,123-126,130,144,180} Neutron radiography in the medical environment has also been examined.¹⁷⁸ The determination of N and B in agricultural fertilizers has been reported⁸⁰ and *in-situ* soils analyses have been attempted.¹⁰⁵ Rapid assessment of protein content (through nitrogen content) and chloride content of wheat and seed grains has been demonstrated.^{22,127,128,142}

G. Geological

Rock and water analysis is a common application of capture gammas.^{27,35,54,116,139} Analysis of ores permits determination of many elements that are otherwise below detection limits.^{7,22,24,32,33,66,94,115,131,148-151,153,154} Minerals exploration through borehole logging is well suited to capture gamma technology.^{41,51,64,106,120,132,145,147,151,154,181-183} Remote exploration of the ocean floor^{52,57,61-63,66,67,132,138,143,158-160,164,167,170,184,185} and lunar and planetary surfaces^{39,94,107,186-189} are made possible by the rugged nature of ²⁵²Cf sources and NaI(Tl) detectors. In the field, determinations of U isotope ratios in natural fission reactors using similar techniques has been demonstrated.⁵⁵ *In-situ* or laboratory analysis of geothermal waters has also been reported.⁴⁹ As a result of its non-destructive nature, rare and valuable samples may be examined, e.g., the redetermination of the cosmic abundance of B through its measurement in meteorites.¹⁷⁰

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