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A BRIEF REVIEW OF THE DETERMINATION OF CADMIUM BY PROMPT GAMMA-RAY NEUTRON ACTIVATION ANALYSIS

B. L. GRAZMAN, E. A. SCHWEIKERT

Texas A&M University, Department of Chemistry, Center for Chemical Characterization and Analysis, College Station, TX 77843-3144 (USA)

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An examination of the literature concerning the determination of cadmium by Prompt Gamma-Ray Neutron Activation Analysis (PGNAA) has been conducted. In-vivo activation analysis of the liver and kidney is the most common application reported and is briefly reviewed here. This review will concentrate on the determination of cadmium in in-vitro systems. These include a number of different complex matrices such as geological, environmental and biological materials, as well as water, sediments, foods and construction material. Nuclear reactors, accelerators, and radioisotopes have all been used as neutron sources with varying degrees of sensitivity.

Introduction

As the concern about the presence of toxic metals in the biosphere increases, the need for the determination of such metals in real samples grows. Typical of these anthropogenic metals is cadmium which appears in batteries, plastics, and consumer electronics and much of which will ultimately end up in the waste stream. Complex materials such as environmental samples have properties that make them very difficult to characterize by many analytical techniques. The samples can be extremely complex and inhomogeneous, requiring the analysis of large subsamples in order to ensure that the analytical result is truly representative of the material from which the analytical portions were taken. One of the few suites of techniques capable of addressing these concerns are the nuclear techniques.

An advantage that a number of the nuclear techniques possess is the wide range of choices for both excitation source and detected radiation, and in the cases where nuclear parameters are favorable, a host of reactions are possible. One technique which uses very penetrating radiations is prompt gamma-ray neutron activation analysis (PGNAA) which uses only neutrons and gamma-rays.[1] In the case of Cd, the isotopic abundance of ^{113}Cd is 12.22% and the cross section for absorption of a thermal neutron by ^{113}Cd is 19896 barns giving elemental cadmium an effective cross section of 2430 barns. The most abundant prompt gamma-ray is at 558.3 keV.

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Collections of detection limits for numerous elements by PGNAA have been published by many authors including ISENHOUR and MORRISON[2] who present a collection of calculated limits of detection for 63 elements including cadmium by PGNAA, traditional neutron activation analysis (NAA) and a modulated activation analysis technique.

Since the product of neutron capture on ^{113}Cd is stable, Instrumental Neutron Activation Analysis (INAA) cannot be used to take advantage of the large capture cross section. PGNAA, on the other hand, measures the gamma-rays emitted when the cadmium nuclei capture a neutron and is not dependent on the nature of the neutron capture product. Thus, PGNAA can be used to measure cadmium content quite sensitively.

PGNAA can be performed using a number of different sources of neutrons. With a nuclear reactor as the neutron source, the samples are usually irradiated outside the core (external geometry)[3] but there is an example of an in-core irradiation (internal geometry) in the literature[4] and both have been used for the determination of cadmium. Cadmium has also been measured by irradiating samples with isotopic neutron sources such as ^{252}Cf and ^{238}PuBe[5] as well as accelerator produced neutrons.[6]

General examples of PGNAA for the analysis of cadmium

A number of reactions besides the absorption of thermal neutrons have been examined for the determination of cadmium by prompt gamma-rays. YATES et al.[7] explored the possibility of using the gamma-rays emitted following inelastic neutron scattering for the determination of cadmium and report a detection limit of 2.13 g using 2.5 MeV accelerator produced neutrons. The determination of cadmium via the production of the metastable product of inelastic neutron scattering, ^{111m}Cd, has been reported, but is not included in this review because of the long halflife of the metastable state. SOWERBY[8] has considered nuclear resonance scattering of gamma-rays by Cd but reported no limit of detection for the determination. The nuclear parameters for determination of cadmium by this technique compare well with the other elements determined, but the technique is most likely only applicable at the percent level.

The use of a neutron guide and an irradiation position far from the reactor core allowed HENKELMANN and BORN[9] to significantly lower the detection limits for cadmium by thermal neutron capture prompt gamma-ray activation analysis as compared to the results of Isenhour and Morrison mentioned above. Also included in this publication, is a comparison of the limits of detection for the low (0–1.5 MeV) and high (greater than 3 MeV) energy regions of the gamma-ray spectrum.

ALIMARIN et al.[10] have studied the decrease in the specific count rate (count rate per unit mass) for cadmium as a function of sample self-shielding. They report a decrease in the specific count rate of cadmium with increasing concentrations of elements with high neutron capture cross sections such as cadmium and boron due to neutron flux depression within the samples. In this work, the concept of a screening coefficient was both derived and experimentally determined.

LOMBARD and ISENHOUR[11] described an instrument combining the neutron flux available from reactors with the beam pulsing ability of accelerators by using a chopped neutron beam. With this apparatus they were able to achieve detection limits of 1 g/l cadmium in an aqueous solution and 100 μg of cadmium blotted onto a filter paper in ten minute irradiations. They found that the peak at 511 keV from hydrogen (pair production from the full energy peak at 2223 keV) limited the sensitivity of their analysis. They report that the background near the 559 keV line of cadmium was increased by a factor of 1.7 by the presence of a piece of filter paper and was increased by a factor of 2 by the presence of a polyvial containing one milliliter of water.

In-vivo prompt gamma neutron activation analysis

The most common application of the determination of cadmium by PGNAA has been the in-vivo activation of the cadmium in the human liver and kidney which was first mentioned in the literature by BIGGIN et al.[6] In this work, the level of organ cadmium in normal patients was determined to be very near the detection limit as determined by calculation, analysis of body phantoms and the examination of a human cadaver. Abnormal levels of cadmium, reported to be some 2 orders of magnitude higher than normals as determined by atomic absorption, were predicted to be detectable by PGNAA. Proton bombarded lithium was used as the neutron source and a pulsed beam/pulsed analysis mode of detection was used in order to maximize the signal to noise ratio in these experiments. Shortly thereafter, HARVEY et al.[12] reported the actual measurement of cadmium in-vivo. This study included the determination of cadmium in phantoms, actual livers obtained by post-mortem necropsy, and two groups of patients. Patients with and without exposure histories exhibited organ concentrations of 0–2 and 35–200 ppm, respectively. The efficacy of the neutron source/detector pulsing schemes used in these experiments has been shown by ZAMENHOF[13] using both monte carlo simulations and experiments.

There has been a great deal of effort put into two aspects of in-vivo activation analysis–understanding the neutron field inside the body and the minimization of the dose to the body. In addressing these issues ELLIOT et al.[14] mention the influence of 4 factors on the uniformity of the neutron flux within the body. These include the

energy spectrum of the neutrons impinging on the body, neutron scattering by the physical facility, the degree of thermalization of the neutrons as they pass through the body, and the sizes, shapes, and densities of various organs and body parts. Cohn, in an extensive review of activation analysis in diagnosis and therapy[5] cites 10 studies of neutron yield and flux density measurements using whole or partial body phantoms before 1980. Since that time, numerous other investigators have continued studying the effect of this parameter on measurements in their various facilities.[15-21]

As already noted, a continuing concern has been minimizing the dose rate. SCOTT et al.[22] were able to increase the usable neutron fluence by 14% for the same patient dose rate by the use of a graphite source holder and collimating assembly. McLELLAN et al.[23] reported a sensitivity of 0.5 mg/kg cadmium in a liver sized phantom with a neutron dose of 0.004 Gy, improving both the detection limit and decreasing the patient dose reported in Biggin's earlier work.

RYDE and BERGMAN[24] have shown that by the use of a filtered 24 keV reactor neutron beam, the dose to the patient can be reduced by a factor of 4 relative to the dose from the large PuBe source, used by the group active at Brookhaven National Laboratory,[25] without loss of sensitivity. This work also reported the low level of biological damage caused by neutrons of energies less than 25 keV.

FRANKLIN et al.[26] have presented conclusive data that in-vivo activation results agree with post mortem analyses of liver and kidney cadmium. FLETCHER et al.[27] report the use of in-vivo PGNAA to begin to elucidate the relationships between organ cadmium concentrations and other biological parameters such as mechanisms of toxicity and biological halflife.

It has been shown by AL-HADDAD et al.[28] that inter-organ interferences can affect the determination of cadmium in-vivo. AL-HADDAD reported the interference of cadmium in the liver on the determination of cadmium in the kidney. This is in apparent disagreement with an earlier paper by McLELLAN[29] in which it was reported that kidney cadmium does not interfere with liver cadmium determination. The discrepancy may be explained by the fact that the two works examined complementary situations.

In-vitro prompt gamma neutron activation analysis

Activation analysis of inorganic and mineral materials

GLADNEY et al.[30] measured the concentration of cadmium in NBS SRM-1633 (Coal Fly Ash) and SRM-1632 (Coal) with a high degree of precision and accuracy. They felt that the irradiation times necessary to determine cadmium at concentrations "normally found in natural matrices" were too long compared to atomic absorption

spectrometry for PGNAA of cadmium to become routine. They predicted that the use of a ^{252}Cf isotopic neutron source and standard Ge(Li) gamma-ray detector would give results comparable to those they achieved at the Los Alamos Omega West Reactor with a flux at the sample position of approximately $2 \cdot 10^{11}$ n $\cdot$ cm^{-2} $\cdot$ s^{-1}. The reduced neutron flux available from an isotopic neutron source should be compensated for by the increased detection efficiency available by moving the detector close to the sample.

FAILEY et al.[31] examined the capabilities of PGNAA as a means of increasing the utility of activation analysis by adding the ability to analyze isotopes whose capture products are stable. This work reported the analysis of various coal and fly ash reference materials and a tabulation of the most effective gamma-rays to use for the determination of various elements were presented. Cadmium was detected in SRM-1632 (the original coal standard) and in 1632a (Bituminous Coal) at the hundreds of μg/kg level and in good agreement with the certified values. Cadmium was not oberved in SRM-1635 (Sub-bituminous Coal) which has a certified value of 30 μg/kg. The analysis of fly ashes (SRM-1633 and SRM-1633a) showed good agreement with the certified values of 1–1.5 mg/kg. They suggest, in their conclusions, that detection limits could be lowered by the use of a NaI shield detector to reduce Comptons and single and double escape peaks, by filtration of the neutron beam with single crystal Bi to minimize the gamma-ray and fast neutron component of the beam or by bending the beam away from other components near the facility with a beam guide.

Using this same facility, ZOLLER and ANDERSON[32] reported the measurement of cadmium in environmental samples. They found cadmium present at approximately 1 mg/kg in Illinois soil and at approximately 0.5 mg/kg in Oil Shale but did not observe it in fall ash from the Mount St. Helens eruption in contrast to previous work by VOSSLER et al.[33] which had demonstrated the effectiveness of PGNAA for the determination of cadmium in ash samples taken by U-2 aircraft from the smoke plume high above Mount St. Helens which were found to contain 99 μg/l to 150 ng/l of air.

More recent work by ANDERSON et al.[34] has continued to improve detection limits for cadmium. Using long (5–15 hour) irradiation/counting times, they report a detection limit for cadmium in the 10–100 μg/kg range in crustal samples.

In further work, ANDERSON et al.[35] have continued their analysis of crustal materials and provide a table of neutron flux reduction in these materials due to the presence of various elements with high cross sections. They used Compton suppression to reduce both the single escape and double escape peaks in their spectra and were able to lower the Compton background in the high energy region of the gamma-ray spectrum (4–11 MeV) by a factor of 3–5. The improvement is less dramatic at low energies because of the small fraction of gamma-rays that escape the detector, but

for cadmium using the 559 keV peak, they report an improvement in the limit of detection in coal from 70 to 35 μg/kg.

In continuing work in this vein, ANDERSON et al.[36] have published the results of PGNAA on twenty two multielement geochemical reference standards including soils, obsidian, oil shale and lake sediments. ANDERSON's results agree well with literature and certified values for cadmium demonstrating the applicability of PGNAA to nondestructively analyze geological materials with very little sample preparation.

River sediment from the Elbe River has been analyzed by SPYCHALA and FANGER[37] using an "internal geometry" arrangement. In their experiments, samples were irradiated in the core of the FRG-1 reactor at the GKSS Research Center (Geesthacht, FRG) and the prompt gamma-rays were observed outside the reactor shield with a pair spectrometer. Using this arrangement they reported a 2.5 mg/kg detection limit for cadmium in sediment. The change of cadmium concentration from 5 to 17 mg/kg with depth within the core sample was taken to indicate the anthropogenic nature of the cadmium in contrast to iron which is constant with depth and taken to be "natural".

River sediments have been analyzed in-situ using a ^{252}Cf neutron source by WOGMAN et al.[38] In this work, the detection limit for cadmium measured in marine sediments and laboratory models of those sediments was reported to be at the percent level demonstrating the use of an isotopic neutron source and Ge(Li) detector for in-situ analysis.

An in-situ probe for cadmium in water was reported by HANDLEY and DECARLO.[39] In this work, the analysis of the solution filling a 208 liter tank using a ^{252}Cf source is described. They report a detection limit of 20 mg/l using almost no neutron source/detector shielding and with the source at one side of the tank.

A more sensitive probe using a ^{252}Cf source was reported by CHUNG and TSENG.[40] Their probe was specifically designed for the prompt gamma-analysis of sea water. Laboratory tests could detect 7 mg/l cadmium in simulated sea water, with a detection limit reported to be in the sub-mg/l range for 1000 second counts. The effective irradiated sample used for analysis defined as the material irradiated from the probe to where the average neutron flux is 1 n · cm^{-2} · s^{-1} was a sphere of radius.70 cm which for sea water is a sample mass of approximately 1400 kg. The range irradiated by 90% of the flux is limited to about 30 cm, a sample of approximately 100 kg. This neutron source was one fourth the size of that used by HANDLEY and DECARLO, yet CHUNG and TSENG achieved a detection limit which was one third of HANDLEY and DECARLO's demonstrating the importance of signal to noise ratio to the analysis. It should be noted that both of the above studies report limits of detection between three and five orders of magnitude higher than cadmium concentrations found in unpolluted waters.

Construction materials have been examined by PGNAA by ANDERSON et al.[41] In addition to the analysis of some of the IAEA and USGS soils mentioned previously, other terrestrial building components were examined including steels and various concretes, sands and aggregates. Cadmium was incidentally measured and found to be present in the soils at concentrations in good agreement with earlier work[42] and was also found in some of the concretes in the 200–350 μg/kg range.

Analysis of organic and biological materials

Many of the studies mentioned above, References 9, 30, 31, 34 and others, have included the incidental analysis of such organic matrices as SRM Bovine Liver or Orchard Leaves as part of the reported work but there are a number of papers specifically on the analysis of biological materials and foodstuffs.

ANDERSON et al.[35] have reported elemental concentrations of freeze-dried human tissues including values for cadmium in the kidney, liver, stomach and spleen; in most cases, these agreed well with values previously in the literature.

ZEISLER et al.[43] have studied the pollutant content of marine bivalves as part of a trio of non-destructive analyses useful for irreplaceable samples (such as banked specimens). They report the use of the techniques of X-ray fluorescence, PGNAA and traditional (instrumental) neutron activation analysis for the sequential determination of 45 elements including cadmium. For both NBS SRM-1566 (Oyster) and National Institute of Environmental Sciences (NIES) CRM-6 (Mussel Tissue Powder) cadmium was determined by PGNAA and INAA and was found to be in close agreement with certified values in the low mg/kg range.

ANDERSON and CUNNINGHAM[44] have reported multi-elemental analyses of a large number of SRM's including biological and mixed and total diet materials, as well as 40 food and mineral supplements. Cadmium was occasionally found in the food and mineral supplements (capsules and tablets), as well as in some of the liquid supplements.

A recent paper by ANDERSON, CUNNINGHAM and MACKEY[45] reported the concentrations of cadmium in various food and mineral supplements which ranged from 120 μg/kg to 1.8 mg/kg (Cd was observed in twelve of the forty supplements analyzed). The sensitivity enhancement for the analysis of various elements including cadmium as a function of the amount of hydrogen in the sample (the so called "hydrogen effect") is also described in the paper. These enhancements can range from nearly zero to almost 20% in the range of standards examined which contained up to twelve weight percent hydrogen in D_2O/H_2O solutions.

ANDERSON et al.[34] reported the analysis of a novel sample–Dissostichus mawsoni, a fish which is indigenous to the very cold waters of the Antarctic region. These fish and similar species are suspected to live as long as a century or more, and because of their distance from anthropogenic sources of pollution and their life span, ANDERSON suggests that they may provide a way to monitor natural levels of heavy metals in the environment. Two of these fish were analyzed, and cadmium was detected in the liver, kidney and brain but not in muscle. The smaller and younger fish had a lower level of liver cadmium, which ANDERSON postulated was due to the age of the fish.

Concluding comments

As shown by the number of papers in the field, in-vivo analysis is the most common application of PGNAA to the determination of cadmium. The accuracy and non-invasive nature of this method of analysis make it a unique tool for in-vivo analysis and ensure its continued use.

Table 1
Materials for which cadmium has been determined by PGNAA

Material	Reference
In-vivo (liver and kidney)	5, 6, 12, 14–29
Aq. solutions	11, 39, 40
Coal and coal fly ash	30, 31
Construction materials	41
Crustal samples	32, 34, 35
Food and dietary supplements	44, 45
Human tissues (in-vitro)	12, 26, 35
Lake and river sediments	36–38
Lava	34
Marine animal tissues	34, 43
Obsidian	36
Oil shale	32, 36
Organic matrix SRM's	9, 30, 31, 34, 43–45
Soil	32, 36, 41, 42
Steel	42
Volcanic ash	32, 33

The cadmium content of a wide range of materials has been determined by PGNAA using numerous irradiation and detection schemes. These materials are listed in Table 1, along with references. The analysis of cadmium in other materials is still

relatively undeveloped and optimum irradiation and counting conditions are still being investigated. The range of samples that can be analyzed and the locations at which they can be analyzed, combined with the ability of this technique to accurately measure cadmium non-invasively in large, inhomogeneous samples is its greatest asset and the key to future applications.

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