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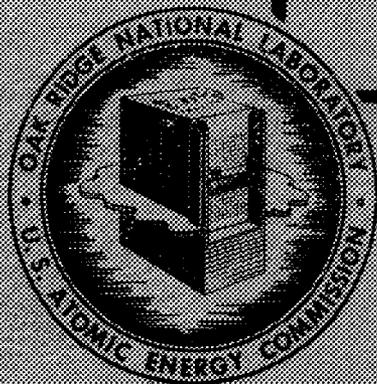
NEUTRON ACTIVATION ANALYSIS:
A USEFUL ANALYTICAL METHOD FOR
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NEUTRON ACTIVATION ANALYSIS:

A USEFUL ANALYTICAL METHOD FOR DETERMINATION OF TRACE ELEMENTS

by

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ABSTRACT

The application of neutron activation analysis to the determination of trace elements as impurities in many substances is reviewed. The method of activation analysis is prefaced by a generalization of the types of nuclear interactions occurring in the ORNL reactor. The utility of this type of analysis, its detection limits and other pertinent facts are appraised.

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I. Introduction

The concept of utilizing radioactivation analysis as an analytical technique for the detection of trace elements arose soon after the discovery of artificial radioactivity. In 1936, Hevesy and Levi⁽¹⁾ applied the neutron activation method to the detection of a small amount of dysprosium in a sample of yttrium. Using the same method, they were also able to detect traces of europium in gadolinium⁽²⁾. Since that time, modifications of the method have been used. These methods of analysis by induced radioactivity have been aptly reviewed in the literature^(3,4). The principles outlined in these reviews now serve as the basis of a convenient method for the quantitative analysis for trace amounts of impurities in various materials by means of the Oak Ridge graphite reactor (Oak Ridge National Laboratory).

Most elements when irradiated by slow neutrons* give rise to a radioactive species of the same atomic number. These artificial isotopes have their own characteristic radiations and modes of decay, thus making activation analysis an unusual technique in that a means of checking purity is furnished. Also, once the irradiation has been completed, there is no danger of contamination with the element being determined. These constitute unique features of the method.

* Radioactivation analysis is not necessarily limited to neutron reactions, but can also be accomplished by the use of charged particles, e.g. deuterons. However, charged particle activation is somewhat complicated by competing reactions and "self-shadowing" effects.

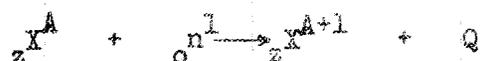
II. Types of Nuclear Reactions Encountered in Neutron Radioactivation Analysis

Basically neutron activation is a process in which a radioactive tracer of the element sought is produced by a type of nuclear reaction. These processes for tracer production are interactions between fundamental particles (neutrons, in this case) and the stable nuclei of the elements. These nuclear reactions are analogous to ordinary chemical reactions in that they have a mass change (heat of reaction), an energy of activation (sometimes), and exhibit varying reaction rates depending on experimental conditions.

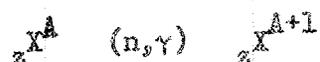
Bohr's idea⁽⁵⁾ of a compound nucleus has been generally accepted as an explanation of nuclear interactions. His theory pictures a neutron coming close enough to a nucleus to unite with it to form a "compound nucleus." This new nucleus may then break up and change into a more stable state, since the kinetic energy of the neutron and the additional binding energy are rapidly distributed among all the particles of the new nucleus.

Since the chain reacting pile is an important neutron source, it is proper to afford some idea of its use in neutron production. In this reaction, U^{235} is split, on absorption of a slow neutron, to give two more nuclei. During this splitting (or fission) a number of neutrons are emitted, some of which may be used to build up and maintain the fission chain reaction, others becoming available for activation of materials. Most of the neutrons produced in this process appear as slow or thermal neutrons, after passing through a reasonable thickness of the graphite moderator, and the neutron yield is such that large quantities of radioisotopes can be prepared by this means. Similarly, this high neutron density affords a sensitivity of detection of elements in amounts, in some cases, of as little as 10^{-5} microgram.

For slow neutrons, the most common interaction is the (n,γ) reaction. The capture of the slow neutron results in γ -ray emission from the compound nucleus shortly after it is formed. The atomic number of the nucleus does not change in the reaction but the mass number increases by one unit. The general equation for the reaction is



or in "shorthand" notation



where

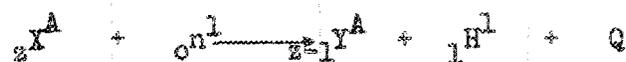
${}_Z X^A$ = the target nucleus with mass A
and charge Z

${}_Z X^{A+1}$ = the product nucleus

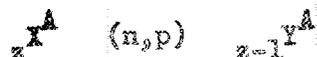
Q = the energy of the reaction appearing
mainly as γ -ray energy.

Since the ratio of neutrons to protons is increased in this reaction, one may expect that in many cases the resultant nucleus will be a negative β -ray emitter.

Certain fast-neutron interactions are also encountered. These include (n,p) , (n,α) and $(n,2n)$ reactions. The general equation for the (n,p) reaction is

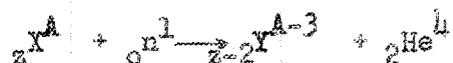


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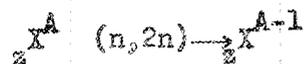
where ${}_{z-1}Y^A$ is the product nucleus. The mass number of the newly formed nucleus will be the same, but the atomic number will decrease by one. The product will often be a positron emitter, since a neutron is added and a proton subtracted.

The general equation for the neutron-alpha reaction (n, α) is expressed in the usual nuclear terminology.



The atomic number of the product nucleus (${}_{z-2}Y^{A-3}$) is lower than the target by two units, while the mass number is lower by three units.

In an (n,2n) reaction two neutrons are ejected from the nucleus by the entrance of one neutron. The reaction may be written



The atomic number of the nucleus does not change in the reaction, but the mass number decreases by one unit.

Only the (n, γ) reaction is of major importance in activation analysis, because of the high cross sections usually encountered. However, (n, α) and (n,p) reactions are known to occur in the region of low atomic number while a (n,2n) reaction can be responsible for radioisotope products in quite a number of instances. So, with this in mind, Table A is set forth to indicate the products from the above four nuclear reactions. Only those products as induced radioactivities having half-lives of at least ten minutes are given. The half-life of the radioactivity produced and the type of radiation emitted are shown.

TABLE A

Radioisotopes Produced By Reactor Neutron Bombardment

Isotope Produced	Atomic No.	T-1/2	Radiation	n, γ	n, α	n,p	n,2n
H(3)	1	12 y	β	H ²	Li	He ³	
Be(10)	4	2.5 x 10 ⁶ y	β	Be	C	B	
C(14)	6	~5700 y	β	C	O	N	
F(18)	9	1.9 h	β^+				F
Na(22)	11	~3 y	β^+, γ				Na
Na(24)	11	15 h	β, γ	Na	Al	Mg	
Mg(27)	12	10 m	β, γ	Mg		Al	
Si(31)	14	170 m	β	Si	S	P	
P(32)	15	14.3 d	β	P	Cl	S	
S(35)	16	87 d	β	S		Cl	
Cl(34)	17	33 m	β^+, γ				Cl
Cl(36)	17	~3 x 10 ⁵ y	β	Cl			
Cl(38)	17	38 m	β, γ	Cl	K		
A(37)	18	34.1 d	K	A	Ca		
A(41)	18	110 m	β, γ	A		K	
K(42)	19	12.4 h	β, γ	K	Sc	Ca	
Ca(45)	20	152 d	β	Ca		Sc	
Sc(44m)	21	2.4 d	γ, e				Sc
Sc(44)	21	3.9 h	β^+, γ, K				Sc
Sc(46)	21	85 d	β, γ	Sc		Ti	
Sc(47)	21	3.4 d	β, γ			Ti	
Sc(48)	21	44 h	β, γ		V	Ti	
Sc(49)	21	57 m	β			Ti	
Ti(45)	22	3.1 h	β^+, γ				Ti
Ti(51)	22	6 m	β, γ	Ti			
Cr(49)	24	~42 m	β^+, γ				Cr
Cr(51)	24	26.5 d	K, γ	Cr			Cr
Cr(55)	24	~2 h	β	Cr			
Mn(56)	25	2.59 h	β, γ	Mn	Co	Fe	
Fe(55)	26	3 y	K	Fe			
Fe(59)	26	46 d	β, γ	Fe		Co	
Co(58m)	27	9 h	γ			Ni	
Co(58)	27	72 d	β^+, K, γ			Ni	
Co(60)	27	5 y	β, γ	Co	Cu	Ni	
Co(60m)	27	10.7 m	β, γ, e	Co		Ni	
Co(61)	27	1.8 h	β			Ni	
Co(62)	27	14 m	β, γ		Cu	Ni	
Ni(59)	28	~105 y	K	Ni			
Ni(63)	28	85 y	β	Ni			
Ni(65)	28	2.6 h	β, γ	Ni	Zn	Cu	
Cu(62)	29	10 m	β^+, γ				Cu
Cu(64)	29	12.8 h	K, β, β^+, γ	Cu		Zn	Cu
Zn(63)	30	38 m	β^+, K, γ				Zn
Zn(65)	30	250 d	β^+, K, γ	Zn			
Zn(69m)	30	14 h	γ	Zn		Ga	
Zn(69)	30	52 m	β	Zn		Ga	

TABLE A (Continued)

Isotope Produced	Atomic No.	T-1/2	Radiation	n, γ	n, α	n, p	n, $2n$
Ga(68)	31	68 m	β^+				Ga
Ga(70)	31	20 m	β, γ	Ga		Ge	Ga
Ga(72)	31	14 h	β, γ	Ga		Ge	
Ga(73)	31	5.0 h	β			Ge	
Ge(69)	32	40 h	K, β^+, γ				Ge
Ge(71)	32	11 d	K	Ge			
Ge(75)	32	85 m	β, γ	Ge	Se	As	Ge
Ge(77)	32	12 h	β, γ	Ge	Se		
As(74)	33	~17 d	$\beta, \beta^+, K, \gamma$				As
As(76)	33	26.8 h	β, γ	As	Br	Se	
As(77)	33	40 h	β	Ge			
As(78)	33	~80 m	β, γ		Br	Se	
Se(75)	34	127 d	K, γ, e	Se			
Se(81m)	34	59 m	γ, e	Se		Br	
Se(81)	34	17 m	β	Se		Br	
Se(83)	34	25 m	β, γ	Se			
Br(80m)	35	4.4 h	γ, e	Br			Br
Br(80)	35	18 m	γ, β^+	Br			Br
Br(82)	35	~35 h	β, γ	Br	Rb		
Br(83)	35	2.4 h	β	Se			
Br(84)	35	30 m	β, γ		Rb		
Kr(79)	36	34 h	K, β^+, γ	Kr			
Kr(83m)	36	1.9 h	γ, e	Kr			
Kr(85)	36	4.5 h	β, γ	Kr	Sr	Rb	
Kr(85)	36	~10 y	β	Kr			
Kr(87)	36	74 m	β	Kr		Rb	
Rb(84)	37	~40 d	β^+, K				Rb
Rb(86)	37	19.5 d	β, γ	Rb			
Rb(88)	37	17.5 m	β, γ	Rb			
Sr(87m)	38	2.7 h	γ, e	Sr	Zr		
Sr(89)	38	53 d	β	Sr	Zr	Y	
Sr(91)	38	9-10 h	β, γ		Zr		
Y(88)	39	2.0 h	β^+				Y
Y(88)	39	105 d	K, γ, β^+				Y
Y(90)	39	62 h	β	Y	Nb	Zr	
Y(91m)	39	51 m	γ, e			Zr	
Y(91)	39	57 d	β			Zr	
Y(92)	39	3.5 h	β, γ			Zr	
Y(94)	39	~20 m	β, γ			Zr	
Zr(89)	40	80 h	β^+		Mo		Zr
Zr(95)	40	65 d	β, γ	Zr	Mo		Zr
Zr(97)	40	17 h	β, γ	Zr	Mo		
Nb(92)	41	10 d	β, γ			Mo	Nb
Nb(94m)	41	6.6 m	γ, e, β	Nb			
Nb(95m)	41	90 h	γ, e	Zr			
Nb(95)	41	35 d	β, γ	Zr			
Nb(97)	41	76 m	β, γ	Zr		Mo	
Mo(93)	42	6.7 h	γ, e	Mo			Mo
Mo(99)	42	67 h	β, γ	Mo			Mo
Mo(101)	42	14 m	β, γ	Mo			
Tc(96)	43	4.3 d	K, γ, e			Ru	

TABLE A (Continued)

Isotope Produced	Atomic No.	T-1/2	Radiation	n, γ	n, α	n, p	$n, 2n$
Tc(97)	43	90 d	γ, e	Ru			
Tc(98)	43	2.7 d	β, γ			Ru	
Tc(99m)	43	6 h	γ, e			Ru	
Ru(95)	44	1.6 h	β^+, K, γ				Ru
Ru(97)	44	2.8 d	K, γ, e	Ru			Ru
Ru(103)	44	42 d	β, γ	Ru			Ru
Ru(105)	44	4.5 h	β, γ	Ru			
Rh(102)	45	210 d	β, β^+, γ				Rh
Rh(103)	45	57 m	γ, e	Pd			
Rh(105)	45	37 h	β, γ	Ru			
Pd(103)	46	17 d	K	Pd			
Pd(109)	46	13 h	β	Pd		Ag	
Pd(111)	46	26 m	β	Pd			
Ag(106)	47	~24 m	β^+			Cd	Ag
Ag(106)	47	~8 d	K, γ, e			Cd	Ag
Ag(110)	47	~270 d	β, γ, e	Ag		Cd	
Ag(111)	47	7.5 d	β	Pd		Cd	
Ag(112)	47	3.2 h	β, γ		In	Cd	
Cd(107)	48	6.7 h	K, γ, β^+	Cd			
Cd(109)	48	~330 d	K	Cd			
Cd(111m)	48	48.7 m	γ, e	Cd			Cd
Cd(115)	48	2.3 d	β, γ	Cd		In	Cd
Cd(115m)	48	43 d	β, γ	Cd	Sn	In	Cd
Cd(117)	48	2.8 h	β	Cd			
In(112m)	49	~20 m	γ, e, β^+				In
In(113m)	49	1.7 h	γ, e	Sn			
In(114m)	49	50 d	γ, e, β	In			In
In(116)	49	54.3 m	β, γ	In			
Sn(113)	50	~112 d	K	Sn			
Sn(117)	50	14 d	γ, e	Sn			Sn
Sn(121)	50	1.1 d	β	Sn		Sb	Sn
Sn(123)	50	40 m	β, γ	Sn		Sb	Sn
Sn(123)	50	130 d	β, γ, e	Sn		Sb	Sn
Sn(125)	50	9.5 m	β, γ	Sn			
Sn(125)	50	10 d	β	Sn			
Sb(120)	51	17 m	β^+				Sb
Sb(122)	51	2.8 d	β, γ	Sb			
Sb(124)	51	60 d	β, γ	Sb	I		
Sb(124m)	51	21 m	γ, e	Sb			
Sb(125)	51	2.7 y	β, γ	Sn			
Te(123m)	52	~100 d	γ, e	Te			
Te(125m)	52	58 d	γ, e	Sn			
Te(127m)	52	90 d	γ, e	Te		I	
Te(127)	52	9.3 h	β	Te		I	Te
Te(129m)	52	32 d	γ, e	Te			Te
Te(129)	52	72 m	β, γ	Te			Te
Te(131m)	52	30 h	γ, e	Te			
Te(131)	52	25 m	β	Te			
I(126)	53	13 d	β, γ				I
I(128)	53	25 m	β, γ	I			
I(130)	53	12.6 h	β, γ		Cs		
Xe(127)	54	34 d	β	Xe			
Xe(133)	54	5.3 d	β, γ, e	Xe	Ba	Cs	Xe

TABLE A (Continued)

Isotope Produced	Atomic No.	T-1/2	Radiation	n, γ	n, α	n,p	n,2n
Xe(135)	54	9.2 h	β, γ, e	Xe	Ba		Xe
Xe(135m)	54	15.6 m	γ, e	Xe	Ba		Xe
Cs(131)	55	10 d	K	Ba			
Cs(132)	55	7.1 d	K, γ, e				Cs
Cs(134m)	55	3.15 h	β, γ, e	Cs			
Cs(134)	55	2.3 y	β, γ, e	Cs			
Cs(136)	55	13.7 d	β, γ		La	Ba	
Cs(137)	55	37 y	β	Xe			
Cs(138)	55	33 m	β, γ			Ba	
Ba(131)	56	12 d	K, γ, e	Ba			
Ba(133m)	56	38.8 h	γ, e				Ba
Ba(135m)	56	28.7 h	γ, e	Ba			
Ba(139)	56	84 m	β, γ	Ba	Ce	La	
La(140)	57	40 h	β, γ	La		Ce	
Ce(141)	58	28 d	β, γ	Ce		Pr	Ce
Ce(143)	58	33 h	β, γ	Ce			
Pr(142)	59	19 h	β, γ	Pr		Nd	
Pr(143)	59	13.8 d	β	Ce			
Nd(141)	60	2.5 h	β^+, K, γ				Nd
Nd(147)	60	11 d	β, γ, e	Nd			
Nd(149)	60	~2 h	β, γ	Nd			Nd
Pm(147)	61	3.7 y	β	Nd			
Pm(149)	61	47 h	β, γ	Nd			
Pm(151)	61	12 m	β	Nd			
Sm(151)	62	20 y (?)	β	Sm			
Sm(153)	62	47 h	β, γ, e	Sm			Sm
Sm(155)	62	25 m	β, γ	Sm			
Eu(150)	63	27 h	β^+				Eu
Eu(152)	63	9.2 h	β, γ, e	Eu			Eu
Eu(152)	63	5 y	K, β, γ	Eu			
Eu(154)	63	5 y	β, γ	Eu			
Eu(155)	63	2 y	β, γ	Sm			
Gd(153)	64	~155 d	K, γ, e	Gd			
Gd(159)	64	18 h	β, γ	Gd			
Tb(160)	65	73 d	β, γ	Tb			
Tb(161)	65	7 d	β, γ	Gd			
Dy(159)	66	14.6 d	K	Dy			
Dy(165)	66	14.5 m	β, γ	Dy			
Ho(164)	67	38 m	β				Ho
Ho(166)	67	27 h	β, γ	Ho			
Er(169)	68	9.4 d	β	Er			
Er(171)	68	~7 h	β, γ, e	Er			
Tm(168)	69	85 d	K, γ, e				Tm
Tm(170)	69	127 d	β, γ	Tm			
Yb(169)	70	33 d	K, γ, e	Yb			
Yb(175)	70	99 h	β, γ	Yb			
Yb(177)	70	2 h	β	Yb			
Lu(176m)	71	3.7 h	β	Lu			
Lu(177)	71	6.8 d	β, γ	Lu			
Hf(181)	72	4.6 d	β, γ	Hf			
Ta(180)	73	8 h	K, γ, e				Ta
Ta(182)	73	117 d	β, γ, e	Ta			
Ta(182)	73	16 m	β, γ	Ta			
W(185)	74	73 d	β, γ	W			W

TABLE A (Continued)

Isotope Produced	Atomic No.	T-1/2	Radiation	n, γ	n, α	n, p	n, $2n$
W(187)	74	24 h	β, γ, e	W			
Re(184)	75	50 d	$\gamma, e, K(?)$				Re
Re(184)	75	2.2 d	$\gamma, e, K(?)$				Re
Re(186)	75	92 h	β, γ	Re			Re
Re(188)	75	18.9 h	β, γ, e	Re			
Os(185)	76	97 d	K, γ	Os			
Os(191)	76	15 d	β, γ, e	Os			Os
Os(193)	76	32 h	β, γ	Os			
Ir(190)	77	10.7 d	$\beta(?), \gamma$				Ir
Ir(192)	77	70 d	β, γ, e	Ir			Ir
Ir(194)	77	19 h	β, γ	Ir			
Pt(191)	78	3.0 d	K, γ				Pt
Pt(193)	78	4 d	K, γ, e	Pt			Pt
Pt(195m)	78	80 m	γ, e		Hg		Pt
Pt(197)	78	18 h	β	Pt	Hg		Pt
Pt(199)	78	31 m	β	Pt	Hg		
Au(196)	79	14 h	γ, e				Au
Au(196)	79	5.6 d	K, β, γ, e				Au
Au(198)	79	2.7 d	β, γ	Au		Hg	
Au(199)	79	3.3 d	β, γ	Pt		Hg	
Au(200)	79	48 m	β		Tl	Hg	
Hg(197)	80	25 h	K, γ, e	Hg			Hg
Hg(197)	80	64 h	K, γ, e	Hg			Hg
Hg(203)	80	44 d	β, γ, e	Hg	Tl		Hg
Tl(202)	81	~12 d	$K(?), \gamma, e$				Tl
Tl(204)	81	~2.7 y	β	Tl			
Pb(203)	82	52 h	K, γ, e				Pb
Pb(209)	82	3.3 h	β	Pb		Bi	
Bi(210)	83	5 d	β	Bi			

The last four columns give the element or elements producing the radionuclide and the type of reaction by which production is achieved.

Since neutrons above 0.4 ev are responsible for most reactions other than (n,γ) , and some pile neutrons have such high energies, it is obvious from Table A that in some cases these reactions would produce an extra amount of the element sought. Freedom from the extra activities produced is possible by using a cadmium filter in the bombardment equipment ("rabbits" or stringer cans). Cadmium has a strong absorption resonance at 0.18 ev, so that a small thickness of the metal will remove practically all neutrons with energies below 0.4 ev (slow neutrons). Thus, by bombarding a sample unshielded as well as in a cadmium shield, it is possible to correct for the fast-neutron reactions above, since the flux of fast neutrons is virtually unaffected by cadmium.

III. Procedure in Radioactivation Analysis

Activation analysis by slow neutrons is accomplished by placing the sample to be analyzed in an intense neutron flux for a given length of time to produce a measurable amount of a radioisotope of the element to be determined. The activity, A , present at a time t after the start of the irradiation, is

$$A = Nf\sigma S$$

where A = activity in disintegrations per second, N = number of nuclei of the element present in the sample, f = slow neutron flux in neutrons per square centimeter per second, σ = "atomic" cross section in square centimeters for the slow neutron reaction leading to the formation of the radioisotope of the element, and S = saturation factor, $1 - e^{-\lambda t}$, or the ratio of the amount of activity produced in time t to that produced in infinite time. Here, λ is the decay constant for the radioisotope of interest ($\lambda = 0.693/\text{half-life}$).

In terms of the weight, W , of the element, assuming that the rate of production of radioactivity, given by $Nf\sigma$, is constant during irradiation, the above equation may be written as

$$W = \frac{AM}{6.02 \times 10^{23} f \sigma S} \quad \text{or}$$

$$W = \frac{AM}{0.602 f \sigma S}$$

with σ in barns (units of 10^{-24} cm^2)

In this equation, M = the chemical atomic weight of the element sought.

To make a precise determination, the unknown sample and a standard consisting of a known weight of the element being determined are irradiated under the same reactor conditions. The radioactivity of the element of interest is isolated and then the standard and the unknown are counted under identical conditions. A comparison of the radioactivity of the element in the unknown is made with the radioactivity of the comparative standard. The purity of the radioactivity may be checked by measurements of half-lives and radiation energies.

Whenever a chemical separation is necessary to remove bulk constituents or interfering radioactive elements, both the unknown and the standard are treated in the same way. A known weight of the element being determined is added as inactive isotopic carrier to the solutions of the unknown and standard after irradiation, and the solution carried through a chemical separation. Chemical separation using carriers serves as a means for correcting for losses during the analysis, since the amounts added and recovered are known.

IV. The Sensitivity of the Method

Table B outlines graphically the calculated sensitivity for most of the elements using the Oak Ridge graphite reactor. The term sensitivity is used to indicate the weight of an element that would produce a readily measurable amount of activity after it had been exposed to the usual reactor flux for a given period of irradiation.

It can be seen that the sensitivities obtainable are in general quite satisfactory, and in some cases rather remarkable. The method, using conventional measurement technique, is not easily applied to hydrogen, lithium, beryllium, boron, carbon, nitrogen, oxygen, fluorine, magnesium, aluminum, columbium, titanium, and rhodium, because these elements have very low cross-sections, or a very short half-life for the radioactivity produced.

The sensitivity with which the small mass of the element sought may be detected is dependent upon the following factors: (1) the "atomic" cross section, i.e., the product of the abundance of the stable isotope producing the desired radioactivity multiplied by its activation cross-section; (2) neutron flux (since the activity produced is proportional to the flux); (3) half-life of the radioisotope produced; (4) the nature of the radiation emitted (must be favorable for measurement).

Radioisotopes with half-lives greater than 10 minutes and not longer than several days are best to use in these measurements. Radioisotopes emitting beta rays or positrons of sufficient energy can be determined with reasonable accuracy by ordinary counting equipment. If the energy of the particles emitted is low or if the radioisotope emits only gamma rays or x-rays, special measurement techniques are necessary. These techniques are now being developed. If the sample contains large amounts of high cross section material or interfering substances, difficulty arises.

TABLE B

Unclassified
DWG. 10923

ACTIVATION SENSITIVITIES

Na	Mg											Al	Si	P	S	Cl
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I
Cs	Ba	R.E.	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At
Fr	Ra	Ac	Th	Pa	U											

FAIR (0.1-10 μ g)

GOOD (10⁻⁵-0.1)

Table C shows only those activities of most elements from 1 to 83 (half-life \geq 10 minutes) which can be applied to activation analysis. The first four columns indicate the target element, the radioisotope produced by neutron bombardment, its half-life, and radiations emitted. The fifth column gives the cross section of the total element for production of the isotope by neutron bombardment. Column six indicates the amount of activity produced at "saturation" (ten half-lives) or in 30 days, whichever is shorter. Column seven gives the sensitivity in micrograms of the element, assuming the usually available flux in the graphite reactor.

From the table, it is possible to indicate the weight of an element necessary to give a predetermined amount of radioactivity (40 disintegrations per second of beta, or the equivalent). For example, Na^{24} , produced by the reaction, $\text{Na}^{23} (n, \gamma) \text{Na}^{24}$ decays with a half-life of 15 hours. Column seven (sensitivity in μg) shows that in a bombardment time equivalent to the saturation value of Na^{24} , enough activity to be measured by ordinary techniques would be produced by 0.007 μg of Na^{23} . Thus, it can be said that 0.007 μg is a lower limit for measurement of sodium in any sample submitted for analysis.

Activation analysis permits the use of larger sized samples than the amount used in some conventional methods of analysis, e.g. spectroscopy. However, as stated above, an element may be determined with ease only if it has a high cross section, suitable radiation characteristics, etc. Thus, the desires of the investigator influence the size of the sample irradiated. For example, samples in the order of 50 grams should be submitted for the analysis of traces of metals in organic materials, while analysis for major constituents might require only 1 mg of sample.

The time required for analyses depends upon factors of pile scheduling, load of other work, the separation process and the nature of the radioisotope

TABLE C
Sensitivities of Radioactivation Analysis^a
(ORNL Graphite Reactor)

Element	Isotope Produced ^b	Half-life ^b	Radiation ^b	*Atomic* ^c (barns) ^c	Sat'n (1 mo.) ^d %	Sens. (μ g of element)	Refer- ences
Na	Na(24)	15 h	β, γ	0.4		0.007	9, 10
Mg	Mg(27)	10 m	β, γ	0.0056		0.6	6
Si	Si(31)	2.7 h	β	0.0037		1	6
P	P(32)	14.3 d	β	0.029	76.7	0.02	6
S	S(35)	87 d	β	0.0052	21.2	4	7
Cl	Cl(38)	38 m	β, γ	0.14		0.03	10
K	K(42)	12.4 h	β, γ	0.067		0.08	6
Ca	Ca(45)	152 d	β	0.013	11.9	3.8	6
Sc	Sc(46)	85 d	β, γ	14.4	21.5	0.002	7
Cr	Cr(51)	26.5 d	K, γ	0.732	54.3	0.2	13
Mn	Mn(56)	2.59 h	β, γ	13		0.0006	6
Fe	Fe(59)	46 d	β, γ	0.0024	33.2	9	7
Co	Co(60)	5 y	β, γ	34	1.08	0.02	11
Ni	Ni(65)	2.6 h	β, γ	0.03		0.03	13
Cu	Cu(64)	12.8 h	$K, \beta, \beta^+, \gamma$	2.97		0.007	14
Zn	Zn(69)	57 m	β	0.20		0.04	6
Ga	Ga(72)	14 h	β, γ	1.35		0.007	6
Ge	Ge(75)	85 m	β, γ	0.22		0.04	11
As	As(76)	26.8 h	β, γ	4.3		0.002	6
Se	Se(81)	17 m	β	0.23		0.05	8
Br	Br(80)	4.4 h	β, β^+, γ	4.09		0.003	6
Br	Br(82)	35 h	β, γ	1.11		0.01	6
Rb	Rb(86)	19.5 d	β, γ	0.52	65.6	0.03	6
Sr	Sr(87m) ^e	2.7 h	γ, e	0.13		0.6	6
Y	Y(90)	62 h	β	1.24		0.01	6
Zr	Zr(95)	65 d	β, γ	0.06	27.3	0.7	12
Zr	Zr(97)	17 h	β, γ	0.045		0.3	12
Nb	Nb(94m)	6.6 m	γ, e (β)	1.20		~10	6
Mo	Mo(99)	67 h	β, γ	0.099		0.1	6
Ru	Ru(103)	42 d	β, γ	0.38	39.0	0.1	6
Ru	Ru(105)	4.5 h	β, γ	0.12		0.1	6
Pd	Pd(109)	13 h	β	3.1		0.005	6
Ag	Ag(110)	~270 d	β, γ, e	1.1	7.0	0.11	6, 16
Cd	Cd(115)	2.3 d	β, γ	0.317		0.05	6
In	In(116)	54 m	β, γ	139		0.0001	6
Sn	Sn(121)	1.1 d	β	0.072		0.2	15
Sb	Sb(122)	2.8 d	β, γ	3.89		0.004	6
Te	Te(127)	9.3 h	β	0.15		0.1	10
I	I(128)	25 m	β, γ	7.0		0.002	10

- a - This is a revision of the table of sensitivities published in Nucleonics(17)..
- b - Unless otherwise indicated, this information appears in "Nuclear Data," National Bureau of Standards Circular 499. (Sept. 1, 1950).
- c - "Best" values, as indicated in the literature, have been used.
- d - Unless indicated, saturation is to be assumed.
- e - The superscript m following the mass number denotes a metastable isomer of measured half-life of either a stable or unstable ground state.

TABLE C (Continued)

Element	Isotope Produced ^b	Half-life ^b	Radiation ^b	*Atomic* ^o (barns) ^c	Sat'n (1 mo.) ^d %	Sens. (μ g of element)	Refer- ences
Cs	Cs(134m)	3.15 h	β, γ, e	0.016		~1	6
Cs	Cs(134)	2.3 y	β, γ, e	26	2.4	0.03	6
Ba	Ba(139)	84 m	β, γ	0.36		0.05	6
La	La(140)	40 h	β, γ	8		0.002	10
Ce	Ce(141)	28 d	β, γ	0.35	52.3	0.1	10
Ce	Ce(143)	33 h	β, γ	0.122		0.2	6
Pr	Pr(142)	19 h	β, γ	10		0.002	6
Nd	Nd(147)	11 d	β, γ, e	0.26		0.1	10
Nd	Nd(149)	~2 h	β, γ	0.14		0.1	10
Sm	Sm(153)	47 h	β, γ, e	36		0.0006	6
Eu	Eu(152)	9.2 h	β, γ, e	659		0.00003	6
Gd	Gd(159)	18 h	β, γ	0.9		0.02	6
Tb	Tb(160)	73 d	β, γ	22	26	0.004	10
Dy	Dy(165)	145 m	β, γ	738		0.00003	6
Ho	Ho(166)	27 h	β, γ	60		0.0004	6
Er	Er(171)	~7 h	β, γ, e	1.0		0.02	10
Tm	Tm(170)	127 d	β, γ	100	15.0	0.002	10
Yb	Yb(175)	99 h	β, γ	15	99.3	0.002	10
Lu	Lu(176m)	3.7 h	β	19.5		0.001	10
Lu	Lu(177)	6.8 d	β, γ	91	95.2	0.0003	6
Hf	Hf(181)	46 d	β, γ	3.5	34.2	0.02	6
Ta	Ta(182)	117 d	β, γ, e	21	16.2	0.007	6
W	W(187)	24 h	β, γ, e	9.9		0.003	6
Re	Re(186)	92 h	β, γ	37	99.5	0.0007	6
Re	Re(188)	18.9 h	β, γ, e	47.2		0.0006	6
Os	Os(191)	15 d	β, γ, e	1.4	75	0.02	6
Os	Os(193)	32 h	β, γ	1.0		0.03	6
Ir	Ir(192)	70 d	β, γ, e	285	25	0.0004	6
Ir	Ir(194)	19 h	β, γ	80		0.0003	6
Pt	Pt(197)	18 h	β	0.28		0.1	6
Au	Au(198)	2.69 d	β, γ	96		0.003	6
Hg	Hg(203)	44 d	β, γ, e	0.71	38	0.13	6
Tl	Tl(204)	~2.7 y	β	2.25	2.0	0.6	6
Bi	Bi(210)	5 d	β	0.015	98.4	2	6

chosen to characterize the desired element. Analyses for such elements as sodium can be accomplished within a few days, whereas elements whose radioactive isotopes are of a longer half-life will require a correspondingly longer time.

The accuracy for determining trace constituents by radioactivation is usually of the order of 10%. However, wherever practical, probable errors are included in data for individual analyses.

V. Applications

The method of neutron activation analysis can be applied to the analysis of any of the elements listed in Table C which may be present in any of the material listed in Table D.

TABLE D

Activation Analysis Can Be Applied To The Determination Of Trace Elements In

Biological Material
 Drugs, Pharmaceuticals and Related Materials
 Fertilizers and Feeding Stuffs
 Fine Chemicals
 Foods and Food Additives
 Fuels
 Glass and Ceramic Materials
 Insecticides, Disinfectants, etc.
 Lubricants
 Metals and Alloys
 Minerals and Ores
 Paints, Pigments and Related Materials
 Petroleum Products
 Plastics and Resinous Materials
 Soils, Dusts and Water
 Toxicants

In order to afford an additional appraisal of this technique of analysis, some quantitative results are given. The final results were obtained as indicated in sections II and III of this report. Comparative standards were used and measurement of the radionuclide produced was done in either of the following ways:

(1) Following irradiation, the sample and standard were discharged from the "rabbit" directly into a small glass tube and the radioactivity was measured in a high-pressure ionization chamber (applicable to determination of high cross section material in a matrix of low cross-section).

(2) Following irradiation, the sample and standard were put into solution, isotopic carrier added and the desired elements separated radiochemically before measurement by means of either a Geiger-Mueller counter or a high pressure ionization chamber.

The determination of tantalum in stainless steel, manganese in metal, and cobalt and manganese in aluminum alloy, are typical examples of technique (1) above, are given below. The slow neutron reactions which produce radioactive Ta, Mn, and Co have reasonably high activation cross sections and have gamma radiation of high energy that can be efficiently measured by a high pressure ionization chamber. A review of some pertinent features of these radioactive isotopes and the actual "in-pile time" for these analyses are shown in Table E.

TABLE E

*Nuclear Data for Tantalum, Manganese, and Cobalt

<u>Stable Isotope</u>	<u>Activation Cross Section (barns)</u>	<u>(n,γ) Product</u>	<u>T_{1/2}</u>	<u>Radiation</u>	<u>"In-pile Time"</u>
Ta181	21	Ta182	117 d	β, γ, e ⁻	64 h
Mn55	13	Mn56	2.59 h	β ⁻ , γ	3.0 h
Co59	34	Co60	5 y	β, γ	1-4 weeks

The results obtained by neutron activation analysis are shown in Tables F, G, and H, respectively, and are compared with those results obtained by conventional methods. The accuracy is well within the limits of 10% assigned to this technique.

* From NBS Circular 499(18).

TABLE F

Tantalum in Stainless Steel

<u>Sample</u>	<u>% Tantalum Activation</u>	<u>Spectrographic</u>
1	0.08	0.08
2	0.12	0.11
3	0.23	0.23
4	0.11	0.13
5	0.20	0.20
6	0.16	0.17
7	0.31	0.29
8	0.29	0.29
9	0.19	0.18

TABLE G

Manganese in Metal Samples

<u>Sample</u>	<u>Mn Concentration (ppm)</u>	
	<u>Activation</u>	<u>Spectrographic*</u>
1	242	292
2	193	236-150
3	170	205-180
4	237	220
5	114	88-100
6	152	164-120
7	166	184-130
8	131	157
9	111	152-100
10	163	180-100
11	120	160-96
12	125	192-110
13	121	152
14	101	128-110
15	128	
16	116	136-99
17	100	96-83
18	98	
19	102	126-100
20	103	100

* Spectrographic results not from ORNL. Two different laboratories gave these results.

TABLE H

Manganese and Cobalt in Aluminum Alloy

<u>Analysis For</u>	<u>Activation</u>	<u>Spectrographic</u>	<u>Colorimetric</u>
% Mn	0.70	0.74	0.75
% Co	0.70	0.68	---

As examples of technique (2), above, the results obtained for the determination of (a) cadmium in vinylite resins, (b) sodium in magnesium, (c) gallium in aluminum, and (d) barium in aqueous solutions, are given below. In all instances, radiochemical separations were made by either precipitation methods or by a combination of precipitation and solvent extraction methods. The separated radionuclide was counted on a G-M counter. Table I shows the pertinent nuclear data used in these analyses.

TABLE I

*Nuclear Data For Cadmium, Sodium, Gallium, and Barium

<u>Stable Isotope</u>	<u>Activation Cross Section</u>	<u>Product of (n,γ)</u>	<u>T_{1/2}</u>	<u>Radiation</u>	<u>"In-pile" Time</u>	<u>Final Precipitate</u>
Cd ¹¹⁴	0.317	Cd ¹¹⁵	2.3 days	β^- , γ	64 hrs.	CdS
Na ²³	0.41	Na ²⁴	15 hrs.	β^- , γ	16 hrs.	NaCl
Ga ⁷¹	1.35	Ga ⁷²	14.3 hrs.	β^- , γ	16 hrs.	Oxine Salt
Ba ¹³⁸	0.36	Ba ¹³⁹	84 min.	β^- , γ	1.5 hrs.	BaCl ₂ ·H ₂ O

The procedures that have been developed for quantitative isolation and measurements of the radionuclides produced by slow neutron activation follow a general pattern similar to one usually applied to the separation and isolation of Ba¹³⁹ activity: To a solution of the bombarded sample, a known amount of stable barium carrier is added and precipitated as barium nitrate with fuming HNO₃. The precipitate is redissolved and reformed several times in the presence of "holdback" carriers or complexing agents in order to prevent

* From NBS Circular 499(18).

contamination of the product by increasing the solubility of unwanted radio-species. Further purification may be achieved by precipitation of a small amount of iron hydroxide, which carries readily hydrolyzable elements forming insoluble hydroxides. Such a procedure is known as "scavenging." The barium is finally precipitated as $\text{BaCl}_2 \cdot \text{H}_2\text{O}$ for weighing and counting. The procedures need not be quantitative in the usual sense, since the activity of the sample may be corrected for "chemical yield" by multiplying by the ratio of carrier added to carrier isolated.

The results shown in Tables J, K, L, and M are some that were obtained by precipitation methods. They are compared with results on the same sample by conventional methods.

TABLE J

Cadmium in Vinylite Resins

<u>Sample</u>	<u>Cd, ppm</u>		<u>Activation</u>
	<u>Spectrographic</u>	<u>Polarographic</u>	
1	---	228	220
2	980	452	449
3	225	---	214
4	45	15	38
5	175	142	128

TABLE K

Sodium in Magnesium

<u>Sample</u>	<u>Activation</u>	<u>% Na</u>
		<u>Spectrographic</u>
1	0.0008	0.0007
2	0.0006	0.0006
3	0.0008	0.0009
4	0.0015	0.0013
5	0.0010	0.0009

TABLE L
Gallium in Aluminum

<u>Sample</u>	<u>Activation</u>	<u>% Ga</u>	<u>Spectrographic</u>
1	0.014		0.013
2	0.012		0.013
3	0.010		0.012
4	0.012		0.014
5	0.013		0.013

TABLE M
Barium in Aqueous Solutions

<u>Sample</u>	<u>Activation</u>	<u>Ba-mg/ml</u>	<u>Spectrographic</u>
1	0.33		0.32
2	0.98		1.05
3	0.24		0.21
4	0.25		0.20
5	3.8		3.3

In addition to the methods given above, it is now possible to use a routine ion exchange procedure for the determination of trace amounts of the alkali metals in the presence of each other⁽¹⁹⁾. After irradiation, the activated sample is dissolved in a suitable medium, adsorbed on an ion exchange resin column and elution begun. The order of elution is (1) Na (2) K (3) Rb and (4) Cs. The collection of the elutriant is usually made by an automatic device and the desorption of the radioactive ions is monitored by the use of automatic counting techniques. Each activity of interest in the unknown sample is compared with a known amount of the same element bombarded under the same pile conditions.

Duplicate analysis for sodium and potassium in the same sample can be completed within 48 hours after submission of the unknown sample. Analysis for

Rb and Cs in the same sample requires about twice that time. The results on the determination of Na, K, and Cs in two samples of Rb_2CO_3 are given in Table N.

TABLE N
Sodium, Potassium, and Cesium in Rb_2CO_3
by Activation and Ion-Exchange

<u>Sample</u>	<u>Metal in Samples, Per Cent</u>		
	<u>Na</u>	<u>K</u>	<u>Cs</u>
1	Present	0.055	0.10
2	0.007	0.050	0.40

In addition to the above examples, (Tables E-N) neutron radioactivation methods have been applied to the determination of

- | | |
|--|----------------------------------|
| (1) Sb, As, Cd, Ca, Cl,
Co, Cu, Ga, Au, Mg,
K, Nb, Ag, Na, Sr, W,
and the Rare Earths | In metals and alloys |
| (2) As, Cd, Cl, Cu, Fe,
Pb, and U | In organic compounds |
| (3) Cs, K, Na, and the
Rare Earths | In fine chemicals |
| (4) Fe, P, and S | In clays, shales, and limestones |
| (5) Mo | In ores |

VI. Conclusions

The results obtained in the development of this technique of analysis indicate that neutron activation analysis is extremely sensitive for many elements, and allows quantitative determination of trace quantities of many of these elements far below the range of other chemical and physical testing methods. It is also a method which is generally more specific than other techniques because the elements to be determined, when irradiated by a neutron

source, produce artificially-radioactive isotopes that have their own characteristic modes of decay. These types of radiation and decay rates are never exactly duplicated in any other radioisotope produced by neutron irradiation.

Contamination, often encountered in conventional methods of analysis, is negligible in neutron activation analysis, once the irradiation has been completed. Interferences are encountered only when samples contain gross amounts of materials that strongly absorb neutrons.

There is usually no limit on sample size in activation analysis and most determinations can be made with an accuracy of 10% or better.

Although the type radiation emitted and the half-life of the radioisotope are specific, these same characteristics can also limit the possible uses of activation analysis. Radioisotopes with half-lives of ten (10) minutes to several days are best; those radioisotopes with longer half-lives usually do not produce sufficient activity in a reasonable length of irradiation time. The radiations emitted must have sufficient energy for detection. Those radioisotopes emitting beta or beta and gamma rays can be detected easily with ordinary counting equipment, while those radioisotopes which emit only x-rays or low-energy conversion electrons require special counting techniques.

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