

REPORT NO. 1861

**THE DETERMINATION OF SOME IMPURITIES IN
ZIRCONIUM METAL BY INSTRUMENTAL
NEUTRON-ACTIVATION ANALYSIS**

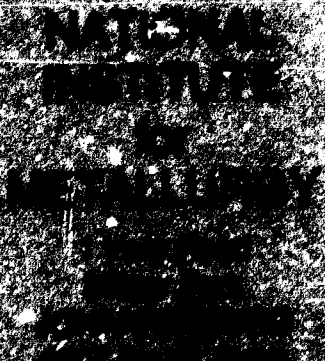
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Date 20th September, 1976

Project 61075

Project Report No. 1





NATIONAL INSTITUTE FOR METALLURGY

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SYNOPSIS

This report describes the work done on the development of an instrumental neutron-activation method for the analysis of impurities in reactor-grade zirconium. Nine samples were analysed, and the results were compared with those obtained by other techniques. No statistically significant differences were observed for ten of the twelve elements that could possibly be determined by instrumental neutron-activation analysis. Cadmium cannot be determined at the 0,5 p.p.m. level, and there is doubt about the comparative values recorded for aluminium. The precision of measurement by direct instrumental neutron-activation analysis ranges from 1.4 per cent for tungsten to 17 per cent for chromium.

SAMEVATTING

Hierdie verslag beskryf die werk wat in verband met die ontwikkeling van 'n instrumentele neutronaktiveringsmetode vir die ontleding van onsuierhede in reaktorgraadsirkonium gedoen is. Daar is nege monsters ontleed en die resultate is vergelyk met die wat met ander tegnieke verkry is. Daar is geen statisties beduidende verskille waargeneem vir tien van die twaalf elemente wat deur instrumentele neutronaktiveringsanalise waargeneem kan word nie. Kadmium kan nie op die peil van 0,5 d.p.m. bepaal word nie, en daar bestaan twyfel oor die vergelykende waardes wat vir aluminium geregistreer is. Die meetpresisie met direkte instrumentele neutronaktiveringsanalise wissel van 1,4 persent vir wolfram tot 17 persent vir chroom.

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1. INTRODUCTION

The accurate analysis of pure zirconium has become increasingly important over the last few years with the rapid growth of modern nuclear technology. Because of its low neutron-absorption cross-section and its strength and resistance to corrosion in the environment of a nuclear reactor, zirconium and its alloys are used extensively as cladding for nuclear fuel and in the manufacture of different components of the reactor. The specifications¹ for the allowable impurities and their concentrations in reactor-grade zirconium are given in Table 1. Many of the elements have high neutron-absorption cross-sections that would counteract the advantages resulting from the use of zirconium, but that should favour their determination by instrumental neutron-activation analysis (INAA).

The analysis of zirconium by neutron activation is well reported in the literature, many authors describing the determination of hafnium², while others have determined various elements by making use of different chemical separations³. The nuclear data given in Table 2 for 18 elements listed in the specifications for nuclear-grade zirconium indicate that it should be possible to determine 12 of these elements by INAA on shavings of the metal without the necessity for any pretreatment, which would probably be required with alternative techniques⁴.

2. NUCLEAR DATA

Spectrographically pure zirconium oxide was irradiated and counted so that the zirconium isotopes produced under the expected analytical conditions could be determined (Table 3). The isotopes⁴ of interest for the elements given in the specification are listed in Table 2. Only the major lines, or the lines of interest in the determination of mutual interferences, are listed. Table 4 lists data for the elements that were found to cause significant interferences but were not determined.

3. EXPERIMENTAL METHOD

3.1. Preparation of Standards and Samples

A series of four standards was prepared for calibration purposes, each standard containing a certain group of elements that would provide interference-free spectra (Table 5). Then, 100 μ l of the standard solutions was pipetted into quartz or polyethylene vials containing pure silica, and was dried for 48 hours in a heated desiccator (80 to 110 °C) containing a silica-gel desiccant. Quartz vials were used for the preparation of standards 1, 2, and 3, and polyethylene vials for standard 4 (Table 5) in accordance with reactor regulations relating to long irradiations. Although cadmium was included, it is unlikely that it could be determined by INAA at the required concentration level of 0.5 p.p.m.

Samples were submitted for analysis in thin strips cut from the bulk sample with a tungsten carbide cutting-tool. The strips were broken into small pieces (approximately 2 mm by 2 mm) and washed in nitric acid, then in distilled water, and finally in acetone for the removal of surface contaminants. Approximately 200 mg of sample was accurately measured into each quartz vial, and 100 mg into each polyethylene vial. All the quartz vials (standards and samples) were heat-sealed in preparation for irradiations of one or more hours (Section 3.2), and the polyethylene vials were packed into polyethylene rabbits for the pneumatic irradiations (Section 3.2). Iron foil was wrapped round each quartz vial for monitoring of the neutron flux. After irradiation, the foil was removed and the quartz vials were washed in hydrochloric acid, water, and acetone for the removal of any surface contamination. The quartz and polyethylene vials were then positioned and packed in polyethylene rabbits (polyethylene spacers being used) in preparation for the counting in the automatic system.

3.2. Irradiation and Counting Conditions

The irradiation and counting conditions are listed in Table 6. The walls of the cadmium cans used had a thickness of 1.5 mm, which gave an effective neutron 'cut-off' at a neutron energy of about 0.6 eV. All the samples were counted at a distance from the detector face that was adjusted so that the dead-time of the analyser did not exceed 15 per cent and 25 per cent for short- and long-lived isotopes respectively when the most active sample of the batch was being counted. Analysing system I (Table 6) consisted of a Ge(Li) 45 cm³ detector having a resolution of 2.39 keV for the 1332 keV cobalt-60 peak. This detector was connected to a 4000-channel Packard 900 Series analyser having data output to a magnetic-tape unit for later processing. Analysing system II (Table 6) consisted of a 60 cm³ Ge(Li) detector having a resolution of 2.27 keV for the 1332 keV cobalt-60 peak and coupled to a 4000-channel Intertechnique Didac analyser having data output to a magnetic-tape unit for later processing. An automatic sample-changer^{7,8} was employed so that the counting system could be used to its full capacity. Counting losses on both systems were corrected for by the use of a system proposed by Strauss *et al.*⁹. Spectral data were reduced on an IBM 370/145 computer by the use of a modified version¹⁰ of the computer programme HEVESY¹¹. Uranium was determined by use of a delayed-neutron method described elsewhere¹².

3.3. Flux Monitoring

Flux monitoring was not used in the pneumatic irradiation since the reactor power was constant to better than 0.5 per cent during the period in which the irradiations were carried out. In addition, irradiation times were determined to better than 0.5 per cent, and flux-gradient variations during the irradiation of up to three vials was no greater than 1.3 per cent, corresponding to an error of 0.7 per cent and an overall possible error for all three factors of 1 per cent, which is small compared with other sources of error, e.g., counting errors.

Because there is a variation in flux between the different positions used in the hydraulic and poolside facilities, these irradiations were monitored. This monitoring was done by the use of an iron-foil wrapping round each quartz vial¹⁴. The iron-59 isotope was used for measurement of the thermal flux, manganese-54 being used for measurement of the fast flux.

3.4. Interferences and Choice of Analytical Lines

The analytical lines used in this work, which are shown in Table 2, were found to be free of interferences under the conditions used for the analyses, except for those samples having high concentrations of uranium, when peaks from neptunium-239 at 316 and 334 keV interfered with the determination of titanium-51 and cadmium-115 at 320 keV and 335 keV respectively. Tantalum-182 at 1289 keV would interfere with iron-59 at 1292 keV. Neodymium-147 would interfere with cadmium-115 at 528 keV. There is a less-important mutual interference between the alternative tungsten-187 lines at 479 and 134 keV, and the lines of hafnium-181 at 482 and 133 keV. However, hafnium-181 can be determined at 482 keV after tungsten-187 has decayed away. It is difficult to resolve the hafnium-181 lines at 133 and 136 keV when the concentration of hafnium is high, and this difficulty is best avoided by selection of the 482 keV line of higher intensity.

3.5. Limits of Detection

The limits of detection for the method were estimated from the counts obtained from sample Ezs 17¹¹ (see Figures 1 to 3) since many of the impurities are roughly at the concentration levels quoted for the specifications (Table 1). The limits of detection (L_D) were calculated by use of the formula proposed by Currie¹⁵ for the minimum detectable number of counts, i.e.,

$$L_D = 2.71 + 4.65 \sqrt{\text{Background counts}}$$

and are listed in Table 7. Relevant irradiation and counting conditions are given in Table 6. The limits of detection can be regarded as only approximate since they will be appreciably lower for a sample containing less impurities, and higher for samples having more impurities, e.g., copper-64 was detected at 6 p.p.m. in very pure zirconium sulphate, whereas, under identical conditions, it was not detected in sample Ezs 17 at 102 p.p.m. In the latter determination, an alternative isotope was used (copper-66).

4. RESULTS

Tests of precision and accuracy were carried out on three samples of zirconium metal obtained from the American Industrial Co. Ltd (Ezs 15, 16, and 17) and six obtained in the course of some laboratory experiments (2A, 2B, 3, 4, 5, and 6).

4.1. Precision

As the precision of measurement for the short-lived isotope of tungsten without flux monitoring was high (1.4 per cent, confirming earlier estimates, Section 3.3.), flux monitoring was ignored in the determination of the other short-lived isotopes, i.e., aluminium, copper, manganese, and titanium.

The coefficient of variation for the overall method for the different elements (Table 7) ranged from 1.4 per cent for tungsten to 17 per cent for chromium at concentrations ranging from 20 to 200 p.p.m. The lowest precision was obtained for titanium, iron, and chromium, the coefficients of variation being 17, 12, and 12 per cent respectively.

4.2. Accuracy

Table 8 gives the results obtained by INAA on the nine samples and compares them with results obtained by emission spectrography and values reported by an independent laboratory for three of the samples.

For aluminium, the comparison covers the determination of all nine samples by the INAA, spectrographic, and spectrophotometric methods. The figure in parentheses after each INAA result (Table 8) indicates the expected error in the last reported figure, the reported value being the mean of at least three determinations (separate portions of the sample were used for each determination).

For the three samples Ezs 15, 16, and 17, there are no statistically significant differences, at the 95 per cent confidence limits, between the three sets of values reported for copper, titanium,

manganese, iron, nickel, cobalt, and chromium when the coefficients of variation used are those given in Table 7 for INAA and an average value of 10 per cent for the other two procedures. (This is a degree of precision that can be reasonably assumed at least for the emission-spectrographic method.) The comparison of INAA and spectrographic values for tungsten and hafnium on samples Ezs 15 to 17, and between INAA and chemical values for uranium on the remaining samples, is acceptable within the precision of the method. No values were reported for cadmium by INAA since the limit of detection (about 5 p.p.m.) is well above the specification limit of 0.5 p.p.m. For aluminium, the INAA values at the higher concentrations are lower than those reported by a wet-chemical procedure¹⁶ and by emission spectrography, the agreement between these last two being good. The values for samples Ezs 15 to 17 compare more favourably with the values reported by the independent laboratory for the three samples, these values being at the lower concentrations of near-grade zirconium are well below the specification limits (Table 7).

5. DISCUSSION

The lack of agreement in the results for aluminium has been observed previously in the determination of this element in sulphide concentrates, where low values were also reported at relatively high concentrations of aluminium (greater than 600 p.p.m.). No explanation for these differences has yet been found.

The presence of significant amounts of uranium (more than 100 p.p.m.) interferes seriously with the determination of titanium and cadmium. The presence of large amounts of hafnium (about 300 p.p.m.) seriously affects the precision with which measurements for chromium can be made, since the chromium-51 line at 320 keV is superimposed on a high background from the Compton peak from the major hafnium line, hafnium-181. For samples of low hafnium content, both the precision and limits of detection for chromium will be better. The analysis time for a sample (analysed in triplicate) would be about 2 to 3 hours for a complete determination of 11 elements. Aluminium, copper, titanium, manganese, and tungsten could be determined within a few hours, but the rest would require a decay time of more than about 20 days for optimum determination.

6. CONCLUSIONS

For 11 of the 18 elements listed in the specification for nuclear-grade zirconium, INAA gives detection limits that are well below the specification limits. The precision of measurement varies from 1.4 to 17 per cent over the concentration range 20 to 200 p.p.m. With the exception of aluminium and uranium, there is no statistically significant difference between the sets of results tabled for 10 elements, and the values obtained can serve to give only a mean value of greater confidence to the three samples used as standards, namely, samples Ezs 15 to 17. The determinations of aluminium and uranium are under further consideration. The limits of detection and precision for titanium and chromium would improve in the absence of significant amounts of hafnium and uranium. Even in the absence of uranium, a detection limit of less than 0.5 p.p.m. cannot be achieved by INAA.

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TABLE I

Specifications for nuclear-grade zirconium¹

Element	Al	B	Cd	C	Cr	Co	Cu	Hf	Fe	H	O	Mn	Ni	N	Si	Ti	W	U
Concn. p.p.m.	75	0.5	0.5	270	200	20	50	200	1500	25	*	50	70	65	120	50	100	3.5

*To individual specifications.

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TABLE 2

Isotopes[†] of elements for which INAA is feasible

Element	Reaction	Cross-section barn	Half-life	Gamma-ray energy [‡] keV*
Al	²⁷ Al(n,γ) ²⁸ Al	23.2	2.3 min	1779‡ (100)
Cu	⁶³ Cu(n,γ) ⁶⁴ Cu	4.5	12.8 h	1345‡ (0.5)
Cu	⁶⁵ Cu(n,γ) ⁶⁶ Cu	2.3	5.1 min	1039‡ (9.2) 834 (0.25)
Mn	⁵⁵ Mn(n,γ) ⁵⁶ Mn	13.3	2.58 h	847‡ (99.0) 1811‡ (29.7) and others
Ti	⁵⁰ Ti(n,γ) ⁵¹ Ti	0.14	5.8 min	320‡ (95.5) 929 (4.5) 608 (1.5)
Hf	¹⁷⁸ Hf(n,γ) ^{179m} Hf	52	5.5 h	332 (94.9) 443 (83.5) 215 (81.8) 501 (14.2) and others
Hf	¹⁸⁰ Hf(n,γ) ¹⁸¹ Hf	12.6	42.5 d	482‡ (81.0) 133 (40.0) 346 (13.0) and others
W	¹⁸⁶ W(n,γ) ¹⁸⁷ W	38	23.8 h	686‡ (33.0) 479 (26.5) 134 (9.7) 618 (7.7) 773 (4.9) and others
Cd	¹¹³ Cd(n,γ) ^{113m} Cd	0.3	54 h	335‡ (52.8) 528 (26.4) 492 (10.1) and others
Cr	⁵⁰ Cr(n,γ) ⁵¹ Cr	16	27.8 d	320‡ (9.0)
Co	⁵⁹ Co(n,γ) ⁶⁰ Co	37	5.25 y	1332‡ (100) 1173‡ (99.9)
Fe	⁵⁸ Fe(n,γ) ⁵⁹ Fe	1.23	45 d	1099‡ (56.0) 1292 (44.0) 192 (2.5) and others
Ni	⁵⁷ Ni(n,p) ⁵⁶ Co	0.237*	71.3 d	811‡ (100) and others
U	U(n,f)	582.2	—	—

*The figures in parentheses denote the relative intensities in per cent.

†Fast neutron cross-section.

‡Energies used in analysis.

TABLE 3

Isotopes produced from the irradiation of zirconium

Reaction	Cross-section barn	Half-life	Gamma-ray energies keV*
⁹⁰ Zr(n,γ) ⁹⁰ Zr	0.075	65 d	757(54.9), 724(43.1)
⁹⁰ Zr(n,γ,β ⁻) ⁹⁰ Nb	—	65 d and 35.2 d	766(10 0.0)
⁹⁶ Zr(n,γ) ⁹⁷ Zr	0.05	17 h	743(94.2), 1148(3.1), 356(3.0) 1363(2.7), 603(1.7), 704(1.3) 1852(0.3)
⁹⁶ Zr(n,γβ ⁻) ⁹⁶ Nb	—	17 h and 1.23 h	658(98.0), 743(2.0)

* The figures in parentheses denote the relative intensities in per cent.

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TABLE 4

Isotopes causing significant interferences

Isotope	Cross-section barn	Half-life [†]	Gamma-ray energies [†] keV*
¹⁸² Ta	21	115 d	1121(35), 1221 (27.8), 1189(16.6) 100(14.2), 1231(11.7), 222(8.0) 152(7.2), and others
²³⁸ Np	2.71	56.4 h	106(21.9), 278(13.5), 228(11.3) 209(3.3), 334(2.0), 315(1.4), and others

*The figures in parentheses denote the relative intensities in per cent.

TABLE 5

Composition and combination of elements in synthetic standards

Standard no.	Medium	Element	Amount of element in standard
1	HF 20% (v/v)	Hf	400
	HCl 20% (v/v)	Ni	4 000
2	HCl 5% (v/v)	Cr	500
	H ₂ O	W	100
3	HNO ₃ 10% (v/v)	Cd	200
		Co	200
		Fe*	25 000
4	HCl	Ti [†]	2 000
	H ₂ O	Al [†]	100
	H ₂ O	Mn [†]	10
	H ₂ O	Cu [†]	750
	H ₂ O	W	100

*Iron added as a strip of foil.

†These elements added from standard solutions of 'Titrilsols'.

TABLE 6
Irradiation and counting conditions

Method	Facility used	Length of irradiation	Approximate thermal flux $\times 10^{13}$ n cm ⁻² s ⁻¹	Approximate Cd ratio for Co ⁶⁰ Co(n,γ) ⁶⁰ Co	Decay time	Counting time	Isotopes determined	Counting system
A	Pneumatic	6 min	3	33	5 min	300	²⁶ Al, ⁶⁴ Cu, ⁶⁰ Ti ⁵⁴ Mn ¹⁸⁷ W, ⁶⁴ Cu	I
					5 h	720		II
					12 to 18 h	3600		II
B	Pneumatic	30 min	3	33	0.5 to 0.8 d	900	⁵⁴ Mn ¹⁸⁷ W, ⁶⁴ Cu ¹⁸⁷ W, ⁶⁴ Cu	II
					1 to 2 d	1800		II
					2 to 3 d	1800		II
C	Hydraulic	1 h	0	7	3 to 4 d	1800	¹⁸⁷ W ¹⁰⁹ Cd ⁶⁰ Fe, ⁶⁰ Co* ⁶⁰ Fe, ⁶⁰ Co* ⁵⁹ Fe, ⁶⁰ Co, ¹⁰⁹ Cd, ⁶⁰ Cr	II
					7 to 8 d	1800		II
					14 to 16 d	1800		II
					24 to 27 d	1800		II
					34 to 36 d	3600		II
D	Poolside in cadmium cans	24 h	5	40	3 to 4 d	1800	¹⁸⁷ W ¹⁰⁹ Cd ⁶⁰ Co ⁶⁰ Co	II
					7 to 8 d	1800		II
					34 to 36 d	1800		II
					57 to 59 d	7200		II

*These isotopes were determined only when the concentration was high enough to be determined with reasonable precision at the decay time used.

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TABLE 7

Precision and calculated limits of detection for sample Ezs 17

Method	Isotope	Coefficient of variation %*	Limit of detection p.p.m.
A	²⁶ Al	5,6 (100)	2
	⁶⁴ Cu	8,2 (100)	2
	⁹⁰ Ti	17,0 (80)	48
	⁵⁵ Mn	3,3 (70)	0,2
	¹⁸⁷ W	1,4 (200)	2
C	⁵⁹ Fe	12,0 (1000)	400
	⁵⁷ Co	3,5 (20)	0,5
	⁵¹ Cr	12,0 (100)	28
	²⁰³ Hg	3,5 (300)	3
	¹⁰⁹ Cd	—	5
D	⁶⁰ Co (Ni)	8,6 (80)	35
E†	U (summation of isotopes)	14,0 (10)	1

*The figures in parentheses denote the concentration in p.p.m.

†Irradiation 40s

Decay time 25s

Counting time 25s

Pneumatic facility.

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TABLE 8
Results for nine samples

Element (ppm)	ES 15	ES 16	ES 17	2A	2B	3	4	5	6
Aluminum									
INAA	22(2)	27(2)	47(5)	396(20)	370(20)	8500(200)	122(12)	15(5)	8(2)
W.C.M.	16	45	117	536	469	9150	148	10	19
E.S.	19	43	100	500	440	1000	140	6	ND
Recommended	22	27	69						
Barium									
INAA	50(10)	64(6)	80(10)	ND	ND	ND	ND	ND	ND
E.S.	45	63	105	< 3	< 3	< 3	< 3	< 3	< 3
Recommended	38	87	102						
Bismuth									
INAA	34(3)	41(7)	84(6)	Interference from ²¹⁰ Pb (from U)					
E.S.	30	46	64	38	36	180	18	90	28
Recommended	29	53	70						
Calcium									
INAA	22(2)	31(3)	73(5)	10(2)	10(2)	10(2)	0.5(2)	0.4(2)	10(2)
E.S.	21	30	70	3	3	3	3	3	3
Recommended	22	31	87						
Chromium									
INAA	68(2)	104(5)	200(10)	D	D	D	D	D	D
E.S.	55	95	250	< 30	< 30	< 30	< 30	< 30	< 30
Recommended									
Cobalt									
INAA	D	600(100)	1400(500)	ND	ND	ND	ND	ND	ND
E.S.	320	670	1100	100	80	800	30	< 30	< 30
Recommended	17	904	1200						
Copper									
INAA	77(6)	86(4)	93(10)	ND	ND	ND	ND	ND	ND
E.S.	7	44	72	< 10	< 10	< 10	< 10	< 10	< 10
Recommended	26	46	86						
Iron									
INAA	8(2)	11(2)	22(3)	ND	ND	D	D	ND	ND
E.S.	6	9	19	< 10	< 10	< 10	< 10	< 10	< 10
Recommended	8	10	22						
Lead									
INAA	ND	44(10)	86(15)	ND	ND	ND	ND	ND	ND
E.S.	26	48	102	< 30	< 30	< 30	< 30	< 30	< 30
Recommended	8	68	109						
Nickel									
INAA	194(6)	185(4)	130(11)	154(90)	165(50)	123(70)	115(50)	123(60)	112(50)
E.S.	160	180	270	1000	< 1000	1000	1000	1000	1000
Strontium									
INAA	ND	ND	ND	Interference from ²¹⁰ Pb					
Recommended	< 0.25	< 0.25	< 0.25						
Titanium									
INAA	10(5)	1(3)	9(2)	5(0)	8(0)	3(2)	2(8)	2(9)	2(3)
W.C.M.				473		373	239	208	216

W.C.M. Wet chemical method
 E.S. Emission spectrography
 () Estimated error
 ND Not detected
 D Detected but not quantitatively determined because of poor counting statistics
 N figures are available

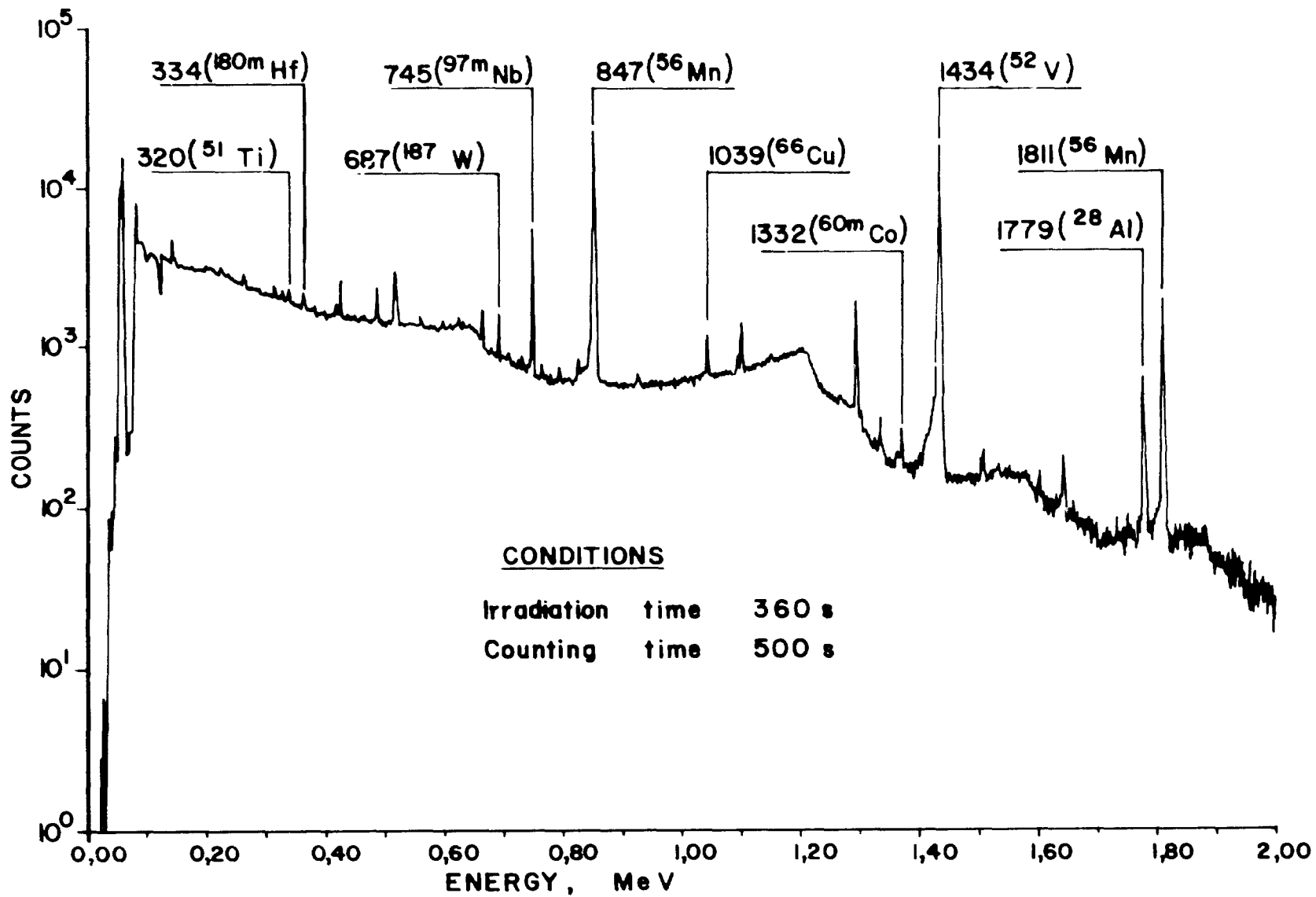


FIGURE 1 Spectrum of sample Ezs 17 at a decay time of 300 seconds

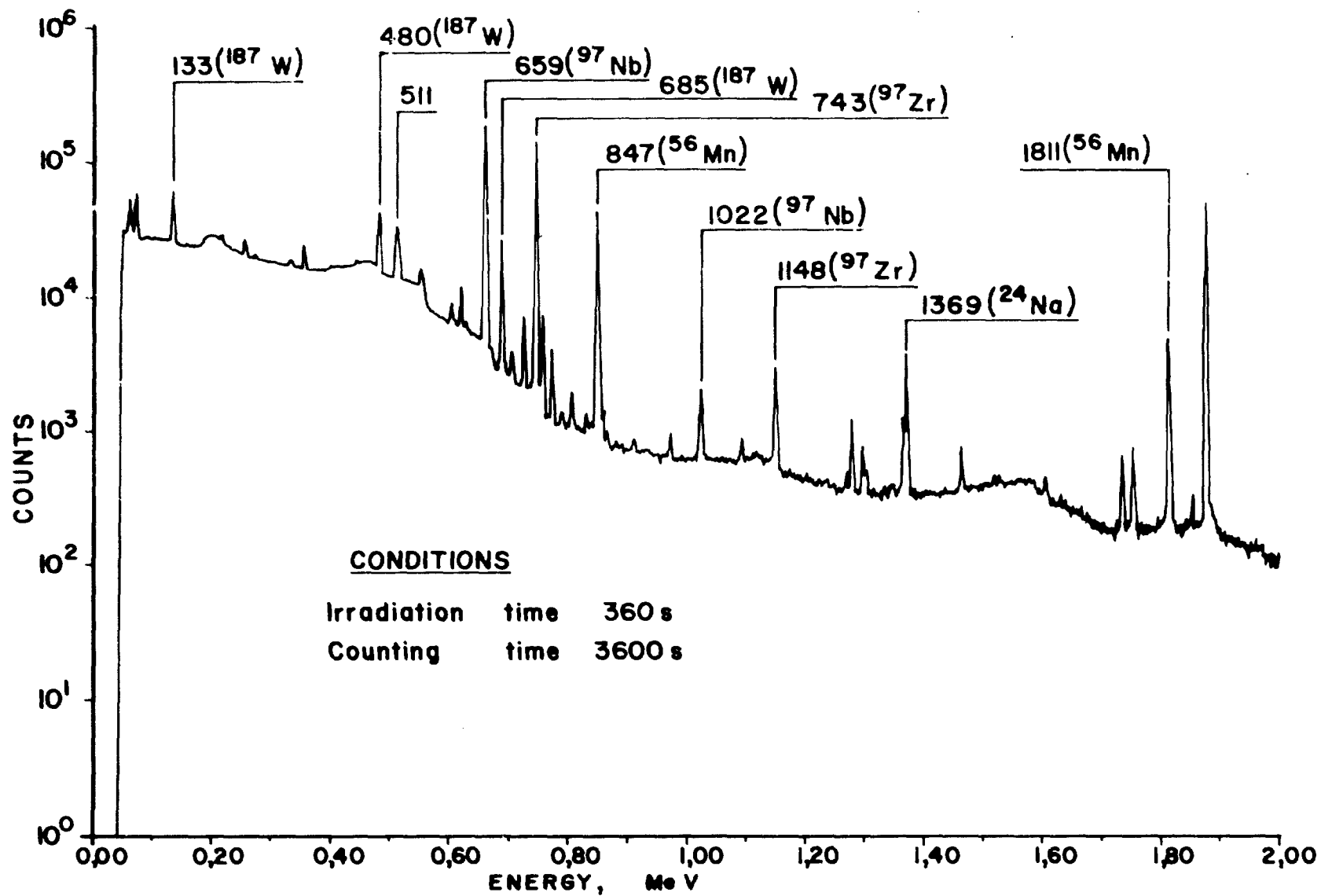


FIGURE 2 Spectrum of sample Ezs 17 at a decay time of 0,46 days

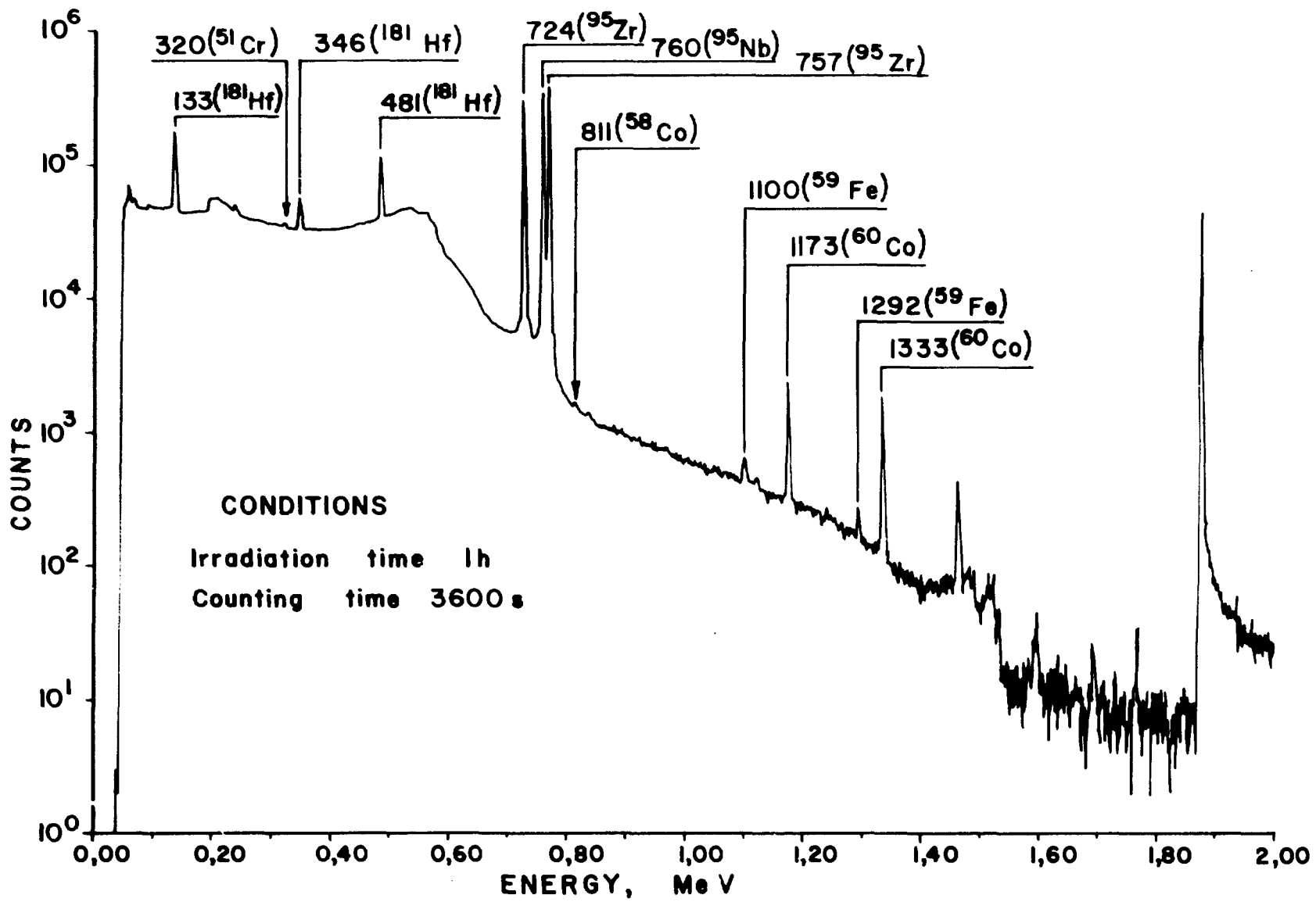


FIGURE 3 Spectrum of sample Ezs 17 at a decay time of 37 days