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Hungarian Academy of Sciences

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BUDAPEST

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INTRODUCTION

The structure and physical properties of tungsten metal can be appreciably affected by additives or trace impurities [1,2,3]. The most important activation analytical studies of such elements that have been reported in the past 15 years [4-24, 28] are summarized in Table 1. The weight of the samples used in these experiments, varied from mg to a few g.

The present report describes a nondestructive method for the determination of the axial concentration distributions of the residual additives Al, Si and K in relatively large sized sintered tungsten rods. The rods are irradiated with 14 MeV neutrons and the average concentration in the environment of a given point is determined from the fast, simultaneous, quantitative analysis of the activities arising from the additives. By measurements at different points of the rod the axial concentration distribution of the additives can be determined and this permits the useful length of the rod to be established before wires are drawn from it. The analysis of representative samples can be utilized in following the different phases of the production process. The method can be modified or supplemented for the analysis of other types of tungsten metal.

EXPERIMENTAL

A NA-2/125 kV/-type neutron generator [25,26] with an effective target diameter of 17 mm was used in the experiments. Measurements were performed at 5 points along the length of each rod using 14 MeV neutrons for activation. Appropriate standards composed of Al_2O_3, SiO_2 and $KHCO_3$ were prepared. Either 40 mg of 8 mm x 8 mm copper foil, or a polyamide disc was used as flux monitor. Comparative measurements with external and internal monitoring permitted a multiple check on the flux measurements and increased the reliability of the activity measurement.

A newly developed sample transfer system operating on pneumatic and gravitational principles secures transport of the samples to the target area, their automatic release and fast conveyance to the de-

tector, and their automatic positioning at the detector. The deformation undergone by rods during the sintering process raised the difficulty of fixing the samples at the target position in a reproducible manner, but the problem was overcome by encasing the rods in polyethylene holders of regular, rectangular shape. The rod in its holder and the monitor are propelled along separate channels of the transfer system, their position at the target area and detector being fixed by pneumatically controlled positioners. In the monitor channel the position is set by a single positioner, while in the sample channel there are 5 uniformly spaced positioners stopping the rod each time at a different point of exposure when propelled in 5 separate runs for activation and measurement. In this way the concentration distribution of the additives is measured at 5 uniformly spaced points along the rod. The equipment and measuring arrangement are shown in Figs 1,2 and 3.

The gamma spectra were obtained in 3" x 3" Nuclear Enterprises NaI/Tl/ crystal with a 9% resolution for ^{137}Cs. The spectra for Al, Si and K were accumulated in 256 channels of a NTA 512/A--type analyser covering the energy range from 0,04 to 2,5 MeV. The energy resolution was found to be satisfactory for Si and K. Investigations were made to see if an improved resolution would give a better evaluation of Al. For this purpose a 10 cm^3 Ge/Li/ detector was used. It was found that the Al lines could be distinguished from those of the tungsten matrix, however the detection efficiency was found to be inadequate. We used therefore a NaI/Tl/ crystal of 7,5% resolution and added a NE-273-type digital peak stabilizer to the measuring equipment.

EVALUATION

The $^{27}Al/n,p/^{27}Mg$, $^{28}Si/n,p/^{28}Al$ and $^{39}K/n,2n/^{38}K$ reactions were found to be the most suitable for the determination of Al, Si and K in the tungsten rods. The optimum irradiation, cooling and detection times were found to be 600, 60 and 500 sec, respectively. The complex spectrum obtained for this timing for a single point is shown in Fig.4. Before irradiating the rod at another point at least 2400 secs must be allowed for cooling, the irradiation, cooling and detection cycle is then restarted, timed as above.

The additive concentration in the tungsten rod was calculated from the average value of several parallel measurements by using the formula

$$\bar{w}_R = \frac{1}{j} \bar{A}_S \sum_{i}^{j} \bar{T}^*_{R,i} \qquad \text{where} \qquad \bar{A}_S = \frac{W_S}{\frac{1}{k} \sum_{i}^{k} T^*_{S,i}}$$

The subscripts R and S indicate the rod and the standard, respectively; W_R and W_S are the concentrations of the addive; T^x_R and T^x_S are the photopeak areas normalized to unit neutron yield; j and k are the numbers of repeated measurements on the sample and the standard, respectively. Thus, the average concentration $\bar{w}_R$ of any element in the rod is proportional to the area under the photopeak T_R of the isotope activated in this element. The proportionality factor A_s is obtained from the measurement on the standard under identical conditions. The error of the determination was assumed to have only two components: the correction for geometrical reproducibility and the deviation in the value of the normalized photopeak area T^x_R.

For a sample of regular geometry the results of about 1000 measurements yield a distribution curve of nearly Gaussian shape /Fig.5/. The error for geometrical reproducibility can be estimated in this case as 2,5 to 3,0%. This error is larger for deformed samples; 26 measurements on a deformed sample showed up to 10% deviation from the average.

The photopeak areas /T^x_R/ of scintillation spectra can be determined by using a gated single channel analyser /e.g. for Al [22] /or by a weighted least-square fit on computer. The latter is already extensively used for the analysis of complex gamma spectra [27]. This method is particularly convenient for the simultaneous determination of two close lying photopeak areas in a scintillation spectrum /e.g. Si and K/. In Figs 6,7,8,9 the spectra of the $^{28}Si/n,p/^{28}Al$ and $^{39}K/n,2n/^{38}K$ reactions in a tungsten matrix are shown along with the fitted analytical functions for different activation times. The error, as a computed parameter, is indicated for the evaluation of the measured data in Figs 7,9 and 10. The normalized residual spectrum obtained from the difference between the measured and computed values for the sample of Fig. 10 is shown in Table II. The variation of this spectrum with the channel number shows whether the measured spectrum contains non-random effects. The small systematic error between the channels 32 and 39 indicates the presence of an unidentified nucleus; this, however, does not interfere with the determination.

The interference from the reaction $^{56}Fe/n,p/^{56}Mn$ /1,81 MeV,

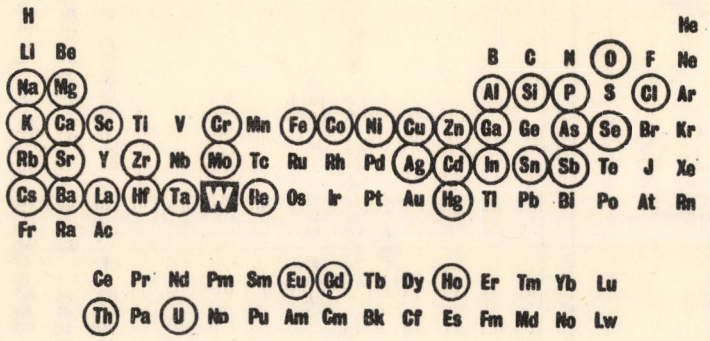
2.58 h/ can be eliminated by half-life measurement, as shown in Fig. 11. The interference from the $^{31}P/n,\gamma/$, $^{40}Ca/n,t/$ and $^{27}Al/n,\gamma/$ competing reactions was investigated and it was found that the P and Ca concentration in the samples was negligible as compared with that of the analyzed elements and that the interference from the $^{27}Al/n,\gamma/^{28}Al$ reaction can be corrected for by e.g. irradiating Al--foils simultaneously with the sample. The disturbing effects and the correction procedures will be discussed in a forthcoming paper.

The measured data were read from the analyser through a telephone line by a NTB-200/T-type digital data transmission unit which is connected through a magnetic tape store to ICT 1905 computer.

ACKNOWLEDGMENT

Thanks are due to Mrs. M. Baross-Sándory for technical assistance in the work.

Table I.



O - [18, 21]
Na - [5, 9, 13, 14, 17, 20]
Mg - [17]
Al - [11, 13, 22]
Si - [11, 17, 22]
P - [24]
Cl - [23]
K - [5, 11, 12, 14, 17, 20, 22]
Ca - [17]
Sc - [17 , 24]
Cr - [17, 24]
Fe - [5, 17, 24]
Co - [5, 17]
Ni - [17]
Cu - [5, 17, 24]
Zn - [5, 17, 24]
Ga - [17, 23]
As - [4, 5, 24]
Se - [17]
Rb - [12, 17]
Sr - [17]
Zr - [17]
Mo - [5, 6, 17, 24, 29]
Ag - [17]
Cd - [17]
In - [17, 23]
Sn - [5]
Sb - [17]
Cs - [17]
Ba - [12, 17]
La - [19]
Hf - [28]
Ta - [8, 17, 24, 28]
Re - [23]
Hg - [17]
Eu - [15]
Gd - [15]
Ho - [19]
Th - [10, 24]
U - [19]

Reported determination of trace elements in metallic tungsten and its alloys by activation analysis

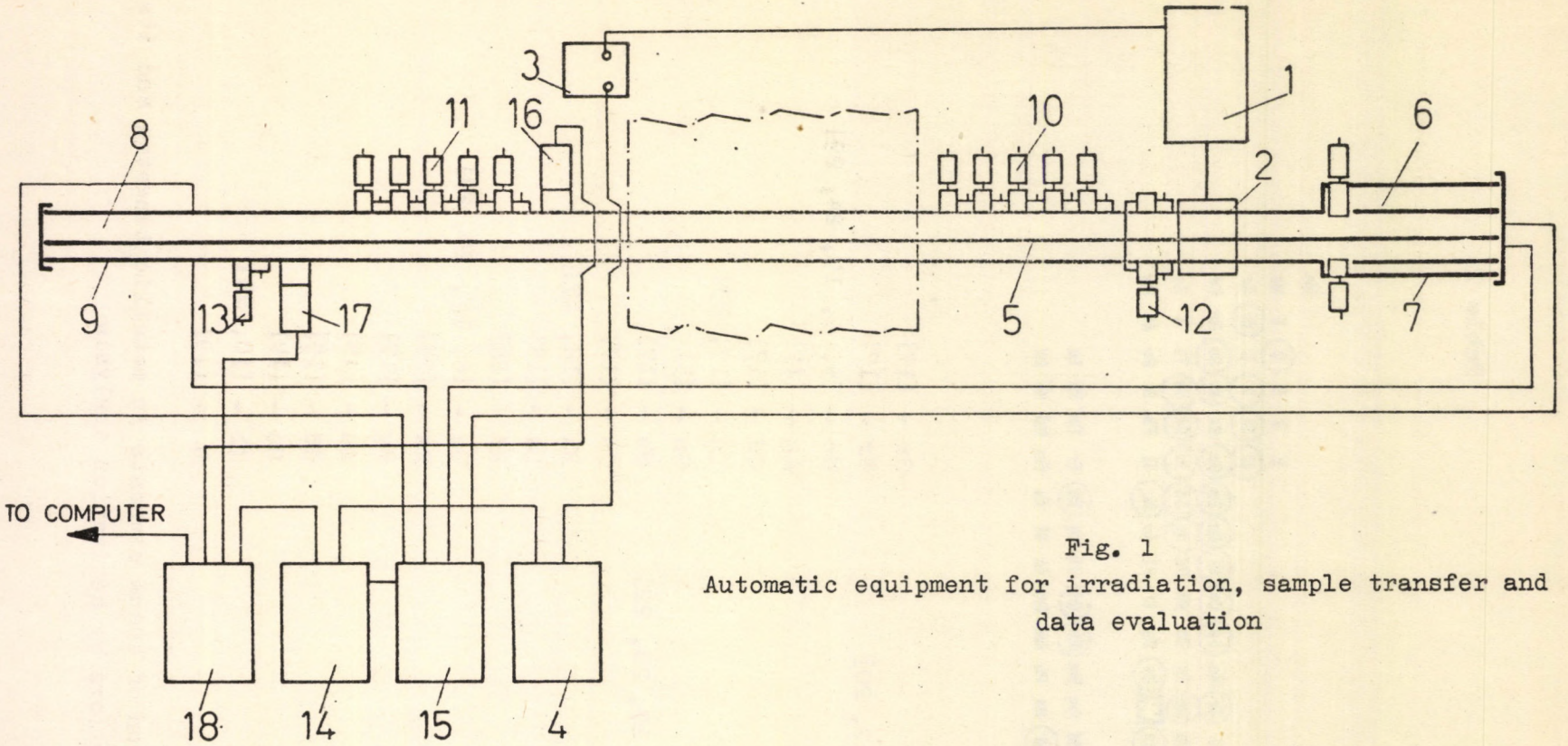


Fig. 1
Automatic equipment for irradiation, sample transfer and data evaluation

Legend: 1 - Neutron generator /NA-2, 125 kV/, 2 - target, 3 - 125 kV power supply, 4 - generator controller, 5 - pneumatic 2-channel sample conveyer, 6 - , 7 - stores for sample and monitor before irradiation 8 -, 9 stores after detection, 10 - , 11 pneumatic sample positioners, 12 -, 13 - pneumatic monitor positioners, 14 -, 15 - electronic and pneumatic program control unit, 16 - sample detector, 17 - monitor detector, 18 - stabilized multi-channel analyser /NTA-512/ and data transmitter /NTB-200/T/

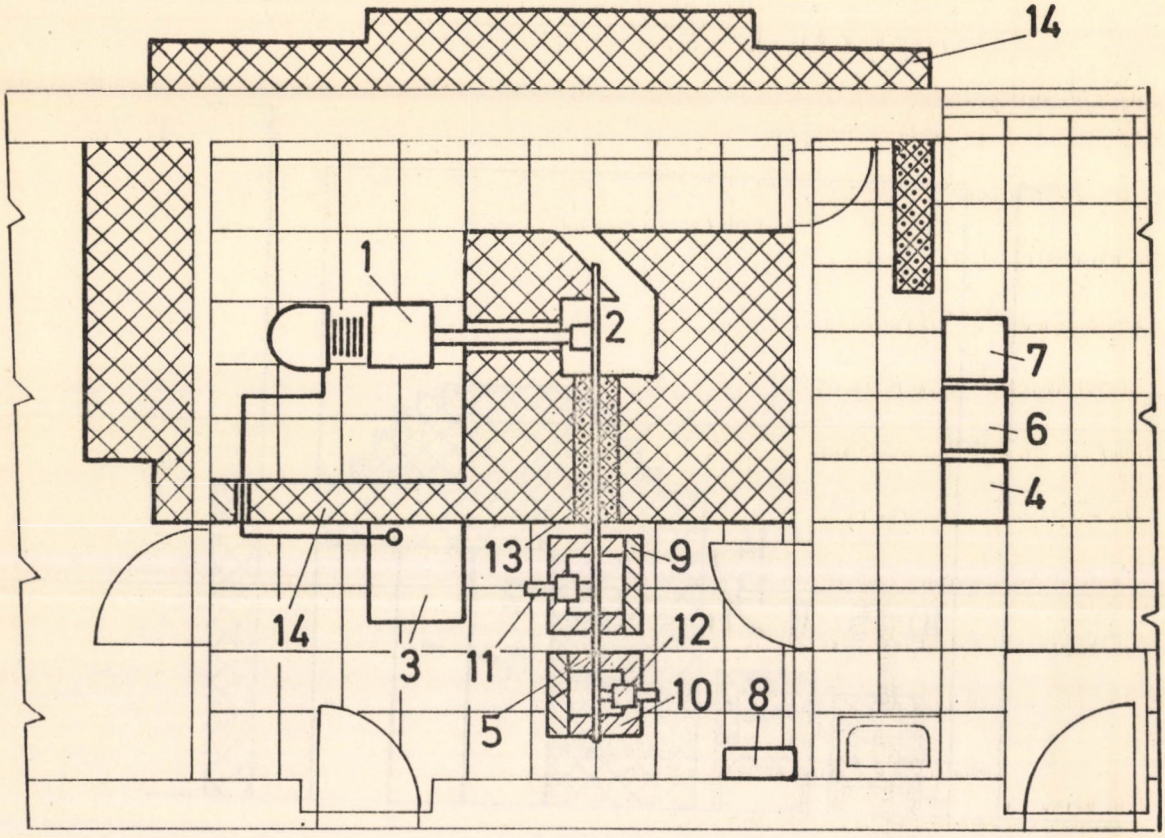


Fig. 2
Arrangement of an activation analytical laboratory equipped with a neutron generator

Legend: 1 - neutron generator, 2 - target, 3 - 125 kV power supply, 4 - generator controller, 5 - 2-channel sample conveyer, 6-electronic program control unit, 7 - multichannel analyser and data transmitter, 8 - pneumatic program control unit, 9 - low background measuring box for sample and 10 - for monitor, 11 - sample detector, 12 - monitor detector, 13 -, 14 - shielding

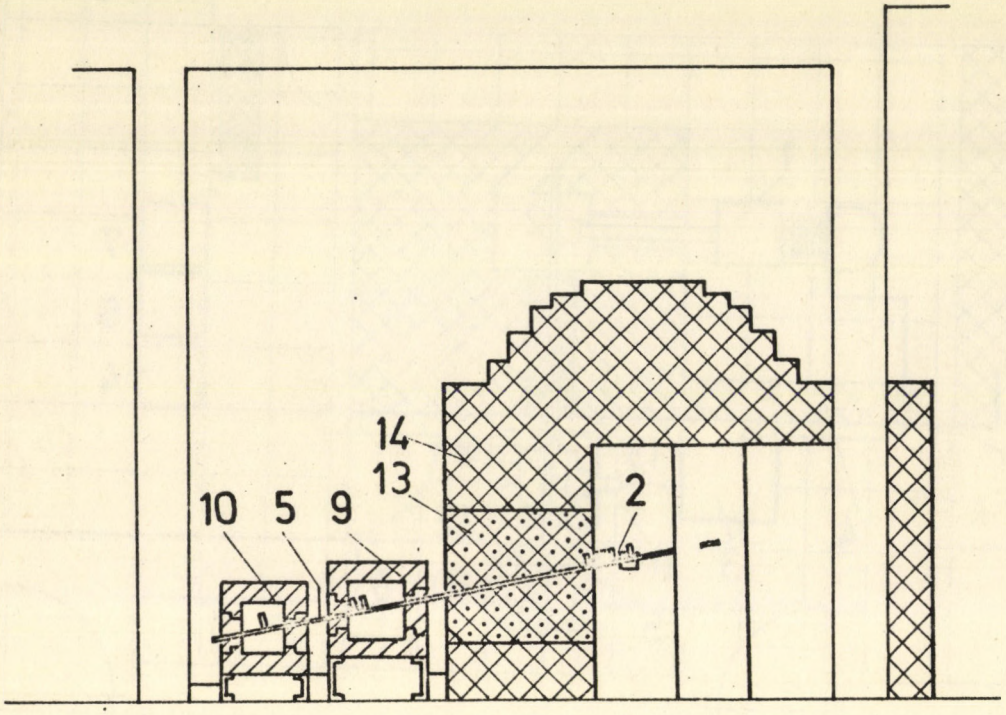


Fig. 3

Cross-section of activation analytical laboratory with a neutron generator

Legend: 2 - target, 5 - gravitational and pneumatic 2-channel sample conveyer, 9 - low background measuring box for sample, and 10 - for monitor, 13 -, 14 - shielding

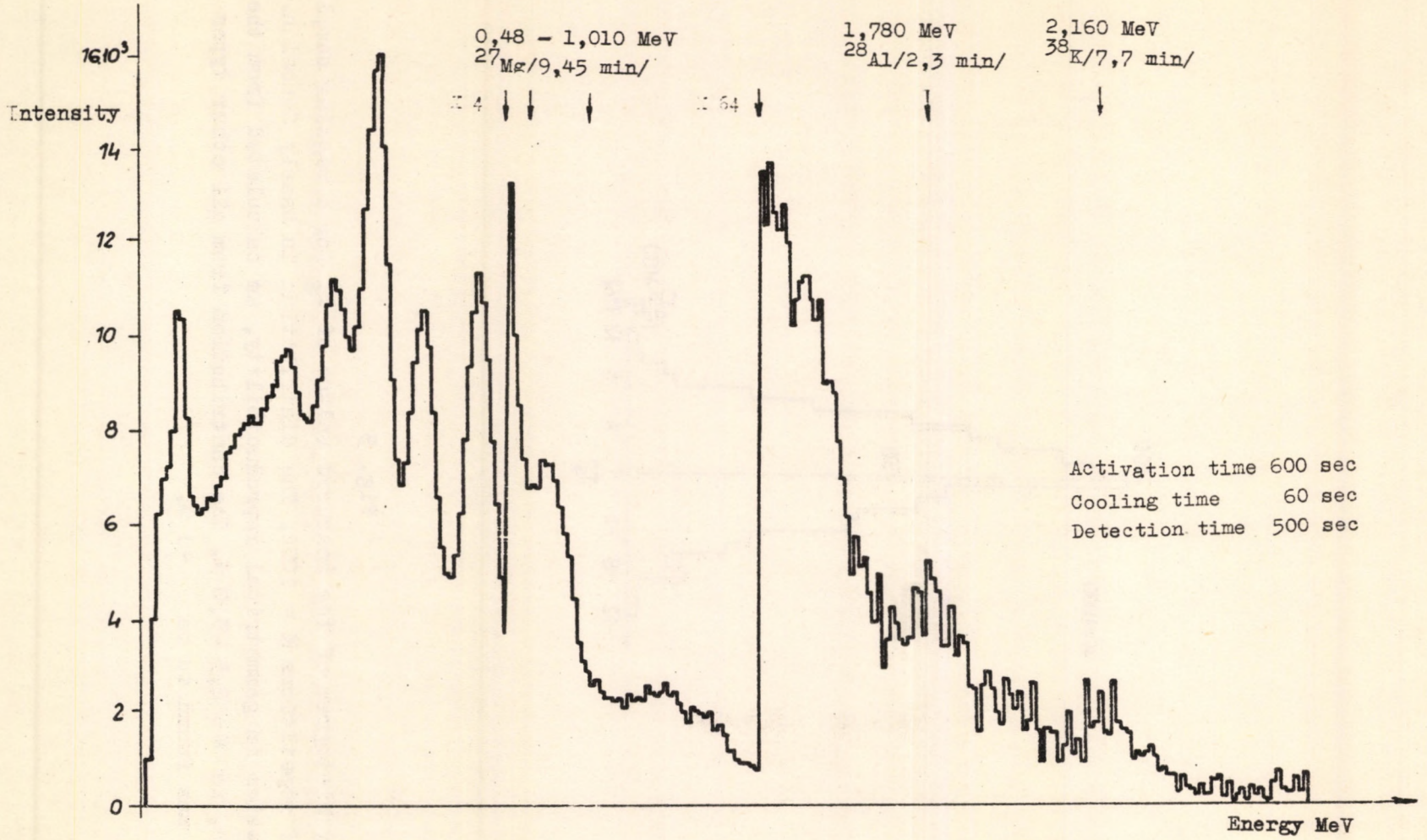


Fig. 4

Gamma spectrum of sintered tungsten metal rod containing Al, Si and K residual additives irradiated with 14 MeV neutrons. The measured reactions are ^{27}Al/n,p/^{27}Mg, ^{28}Si/n,p/^{28}Al, ^{39}K/n,2n/^{38}K

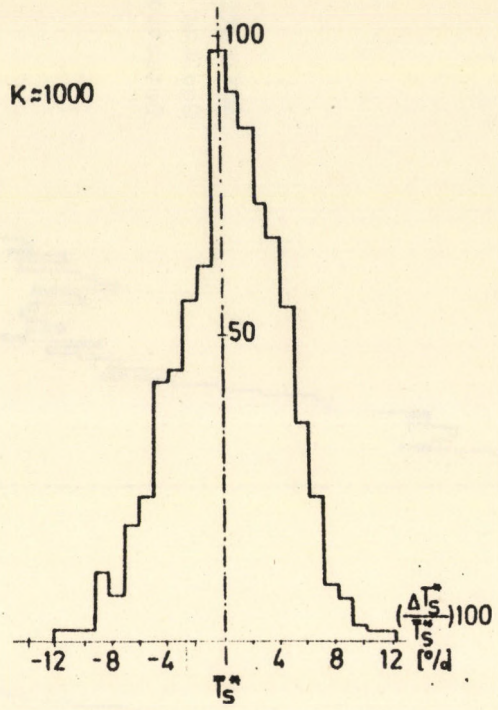


Fig. 5

Frequency histogram of the measured values of T_S^x for a regular sample. Number of repetitions K ≃ 1000. The distribution is nearly Gaussian. The deviation in geometrical reproducibility, as calculated from the histogram, is σ = 2,5 -3,0 %. The contribution from all other types of error was found to be ~1 %.

ORION magnetic tape 6, series 1, spectrum 22

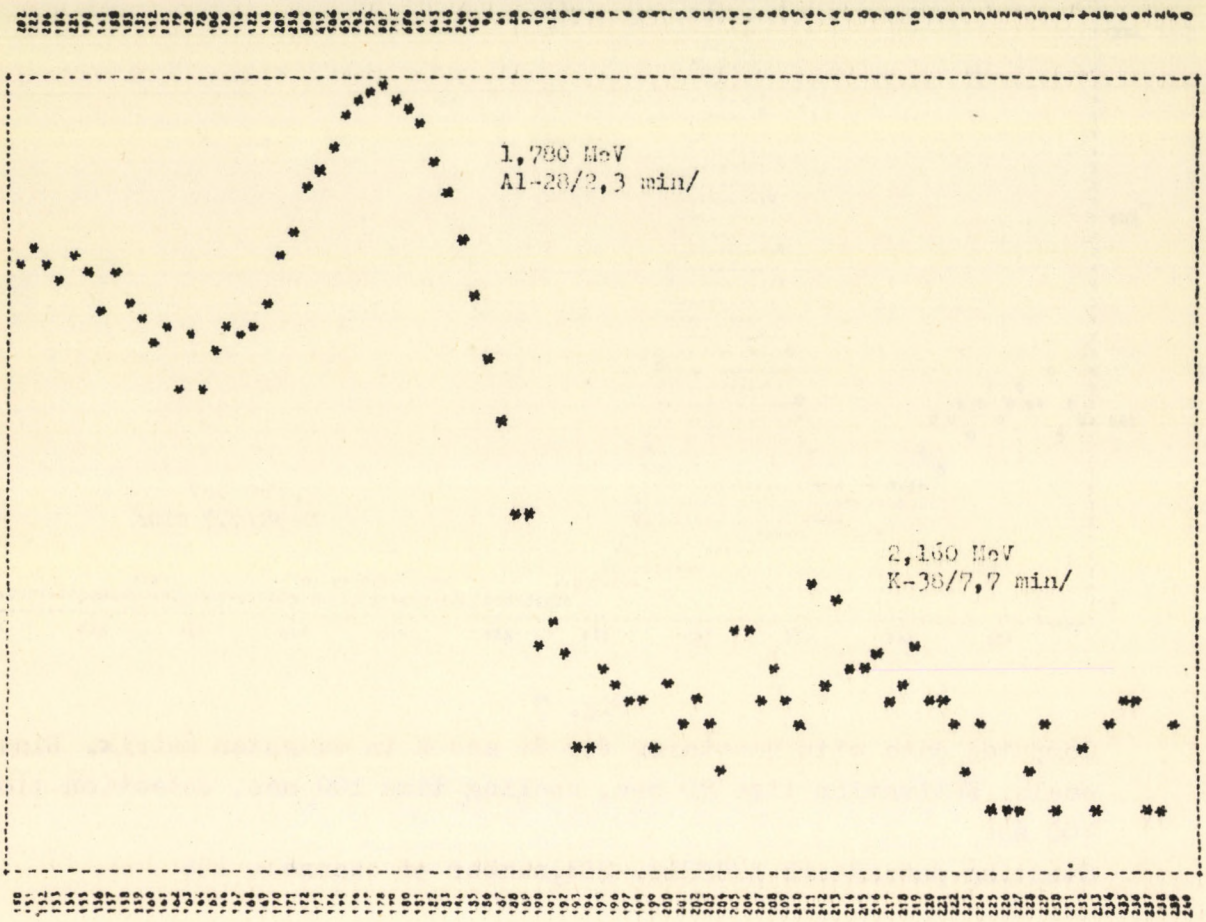


Fig. 6

Logarithmic plot of non-smoothed measured data for Si and K in a tungsten matrix with an activation time of 80 sec, a cooling time of 100 sec and a detection time of 400 sec.

ORION magnetic tape 6, series 1, spectrum 22

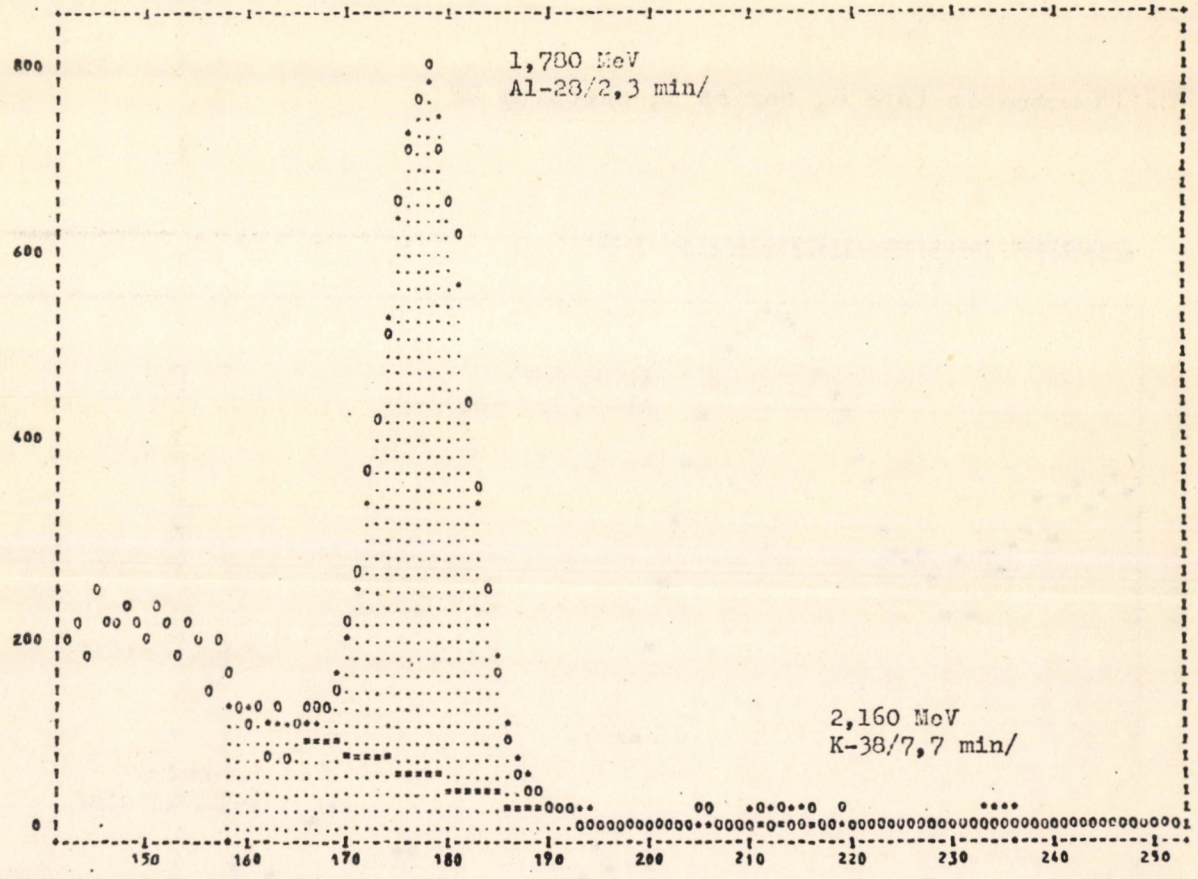


Fig. 7

Computed data with smoothing for Si and K in tungsten matrix. Linear scale, activation time 80 sec, cooling time 100 sec, detection time 400 sec

Starting parameters 178/216.-20:,number of channels 256

Computed parameters

Nr	Peak position	Peak area	Ampl.	Peak/Backg.	Width	H	χ^2	It.
1.	177.70±0.03	7352±66/0.90%/	730	14.61	9.46±0.08%/	G	-3.3/1.0/	8
2.	212.88±0.90	219±42/19.4%/	22	-2.92				

Time 44 sec

ORION magnetic tape 6, series 2, spectrum 23

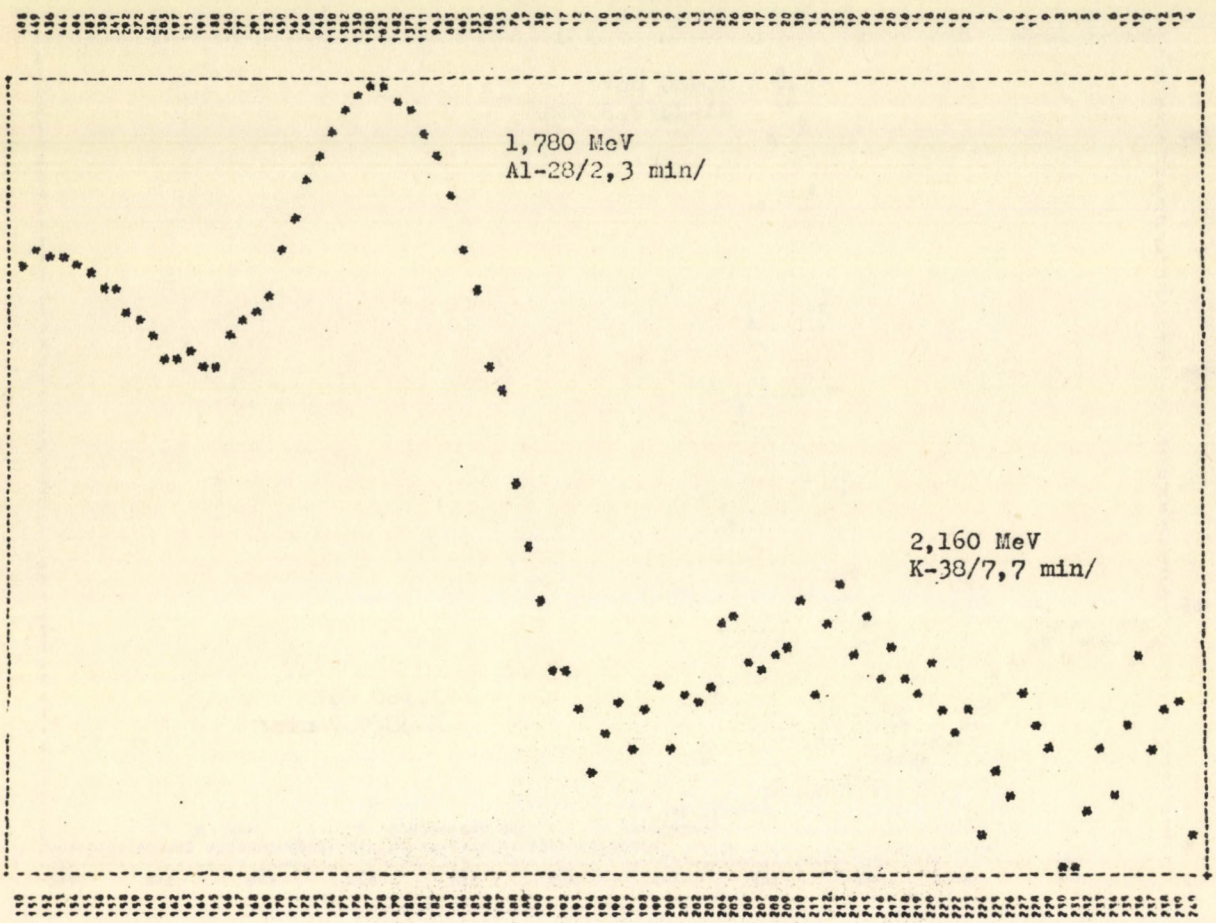


Fig. 8

Logarithmic plot of non-smoothed measured data for Si and K in tungsten matrix with an activation time of 300 sec, cooling time of 100 sec and a detection time of 500 sec.

ORION magnetic tape 6, series 2, spectrum 23

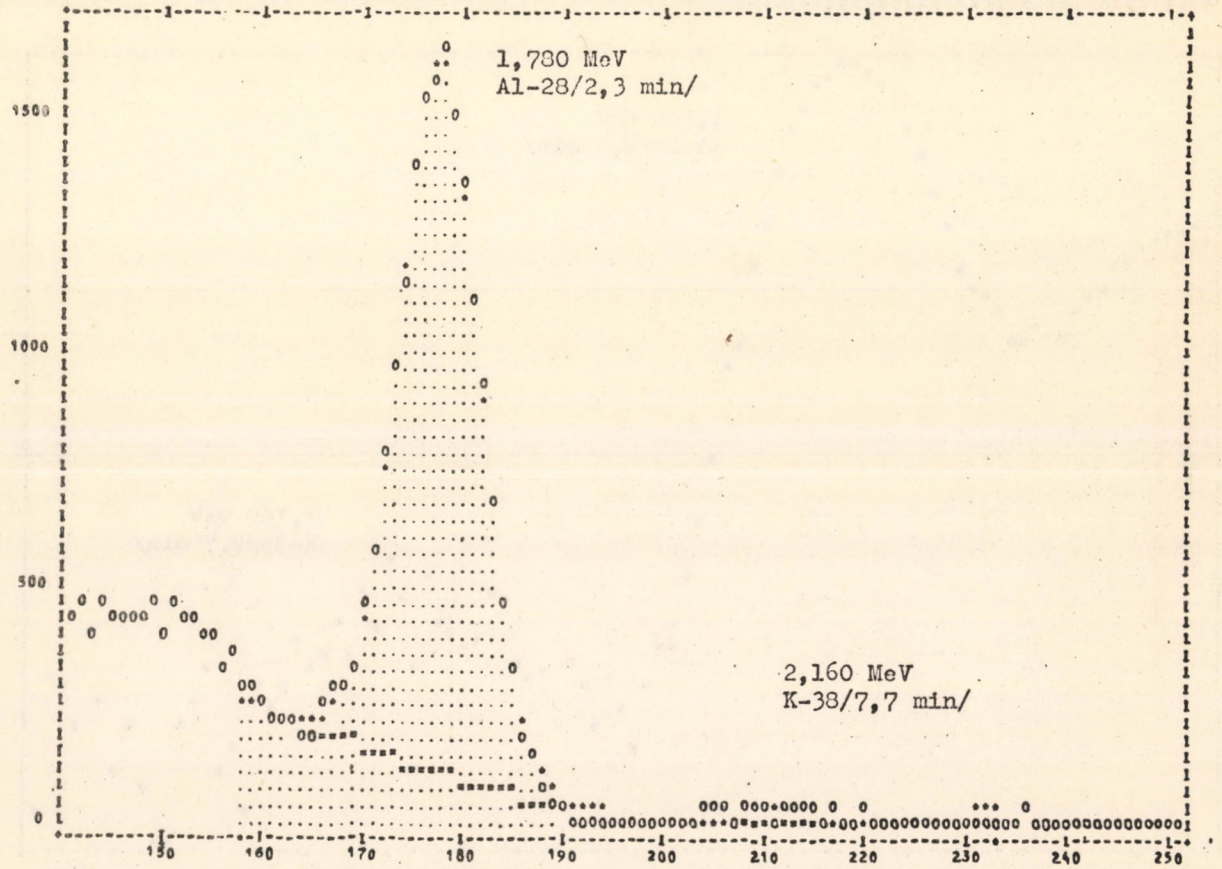


Fig. 9

Computed data with smoothing for Si and K in tungsten matrix with an activation time of 300 sec, cooling time of 100 sec and a detection time of 500 sec.

Starting parameters 178/213.-20:, number of channels 256

Computed parameters

Nr	Peak position	Peak area	Ampl.	Peak/Backg.	Width	H	χ^2	It.
1.	177.47±0.03	15454±101/0.66%/	1515	15.05	9.58±0.06/0.60%/	G	-0.9/1.0/	5
2.	211.90±0.54	450± 52/11.5%/	44	-3.94				

Time: 31 sec

Magnetic tape 37, series 3, spectrum 780

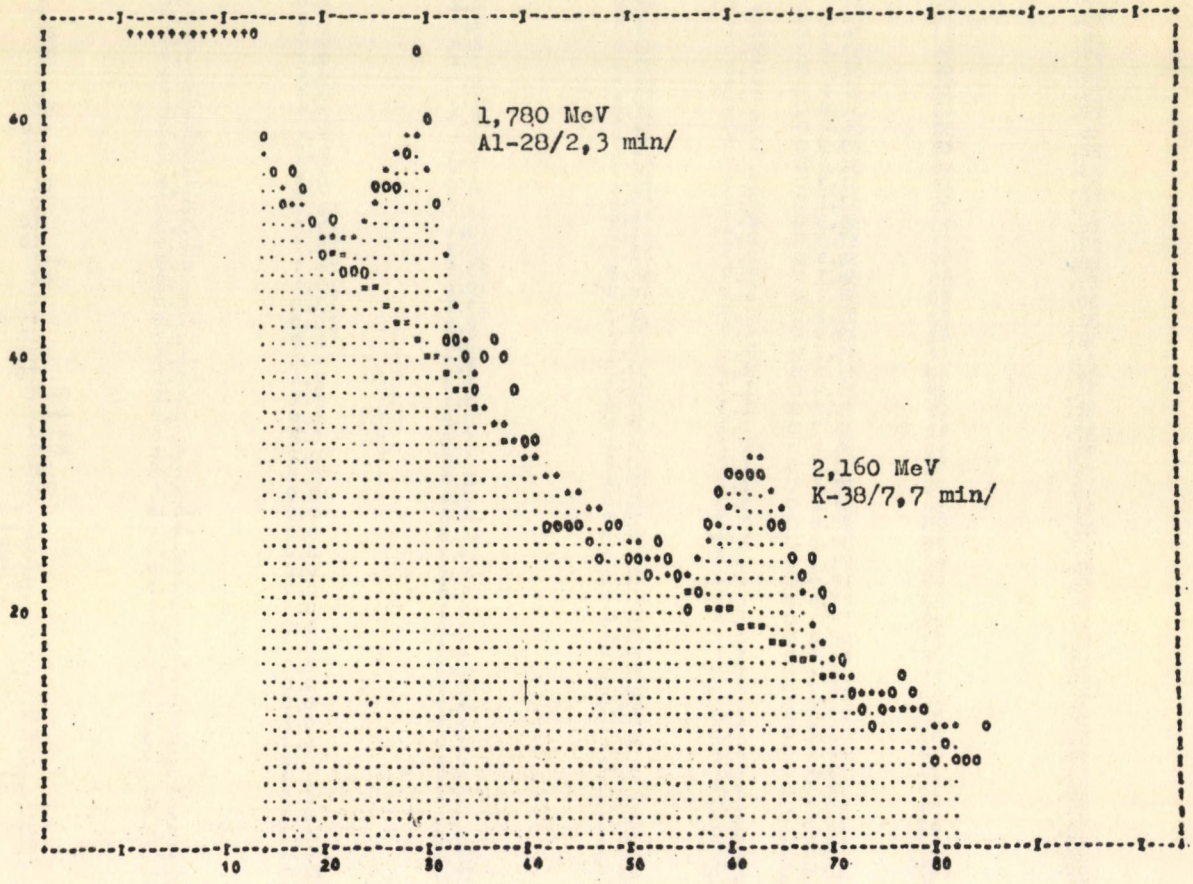


Fig. 10

Computed data with smoothing for Si and K residual additives in sintered tungsten metal rod. Linear scale, activation time of 600 sec, a cooling time of 60 sec and a detection time of 500 sec.

Starting parameters 30/66.-16:, number of channels 85

Computed parameters

Nr	Peak position	Peak area	Ampl.	Peak/Backg.	Width	H	χ^2	It
1.	28.89±0.67	128±29/22.5%/	16	0.39	7.36±1.32/17.9%/	G	-5-4/1.0/	7
1.		124±44						
2.	62.80±0.78	111±27/24.3%/	14	0.81				
2.		116±40						

Time: 44 sec

Table II.

The normalized residual spectrum obtained from the difference between the measured and computed data of the spectrum shown in Fig. 10. Significant deviations of the measured from calculated values are indicated by ⟶.

Magnetic tape 37, series 3, spectrum 780

CHANNEL N°	MEASURED INTENSITY	COMPUTED INTENSITY		BACKGROUND	MEASURED BACKGROUND	COMPUTED BACKGROUND	MEASURED	— COMPUTED
14.	58	56.8 □	2.3	56.8	1.2	0.0	1.2	OI
15.	56	55.8 □	2.2	55.8	0.2	0.0	0.2	O
16.	53	54.7 □	2.1	54.7	-1.7	0.0	-1.7	IO
17.	56	53.6 □	2.0	53.6	2.4	0.0	2.4	OI
18.	54	52.6 □	1.8	52.6	1.4	0.0	1.4	OI
19.	52	51.6 □	1.7	51.5	0.5	0.1	0.4	O
20.	49	50.8 □	1.6	50.5	-1.5	0.3	-1.8	IO
21.	52	50.2 □	1.5	49.5	2.5	0.7	1.8	OI
22.	48	49.9 □	1.6	48.5	-0.5	1.4	-1.9	IO
23.	48	50.3 □	1.9	47.5	0.5	2.8	-2.3	IO
24.	48	51.3 □	2.3	46.5	1.5	4.8	-3.3	IO
25.	55	53.1 □	2.6	45.5	9.5	7.6	1.9	OI
26.	55	55.3 □	2.7	44.6	10.4	10.7	-0.3	O
27.	54	57.3 □	2.7	43.6	10.4	13.7	-3.3	IO
28.	57	58.4 □	2.7	42.7	14.3	15.7	-1.4	IO
29.	65	58.1 □	2.7	41.8	23.2	16.4	6.9	←I
30.	60	56.2 □	2.7	40.9	19.1	15.4	3.8	OI
31.	53	53.0 □	2.7	39.9	13.1	13.0	0.0	O
32.	42	49.0 □	2.7	39.1	2.9	10.0	-7.0	I⟶
33.	42	45.1 □	2.5	38.2	3.8	6.9	-3.1	IO
34.	40	41.6 □	2.2	37.3	2.7	4.3	-1.6	IO
35.	38	38.9 □	1.7	36.5	1.5	2.4	-0.9	IO
36.	41	36.8 □	1.3	35.6	5.4	1.2	4.2	O—I
37.	42	35.3 □	1.1	34.8	7.2	0.6	6.7	←I
38.	40	34.2 □	1.2	34.0	6.0	0.2	5.8	←I
39.	38	33.2 □	1.2	33.2	4.8	0.1	4.8	←I
40.	34	32.4 □	1.3	32.4	1.6	0.0	1.6	OI
41.	34	31.6 □	1.3	31.6	2.4	0.0	2.4	O—I
42.	26	30.8 □	1.3	30.8	-4.8	0.0	-4.8	I⟶
43.	26	30.0 □	1.3	30.0	-4.0	0.0	-4.0	I—O
44.	26	29.3 □	1.4	29.3	-3.3	0.0	-3.3	I-O
45.	26	28.6 □	1.4	28.6	-2.6	0.0	-2.6	I-O
46.	25	27.8 □	1.4	27.8	-2.8	0.0	-2.8	I-O
47.	24	27.1 □	1.4	27.1	-3.1	0.0	-3.1	I-O
48.	27	26.4 □	1.4	26.4	0.6	0.0	0.6	O
49.	26	25.7 □	1.4	25.7	0.3	0.0	0.3	O
50.	24	25.0 □	1.3	25.0	-1.0	0.0	-1.0	IO
51.	23	24.4 □	1.3	24.4	-1.4	0.0	-1.4	IO
52.	22	23.7 □	1.3	23.7	-1.7	0.0	-1.7	IO
53.	25	23.2 □	1.3	23.1	1.9	0.1	1.8	OI
54.	24	22.7 □	1.2	22.4	1.6	0.3	1.3	OI
55.	22	22.4 □	1.2	21.8	0.2	0.6	-0.4	O
56.	19	22.5 □	1.4	21.2	-2.2	1.3	-3.5	I—O
57.	21	23.1 □	1.8	20.6	0.4	2.5	-2.1	IO
58.	27	24.3 □	2.2	20.0	7.0	4.3	2.7	OI
59.	29.	26.2 □	2.6	19.4	9.6	6.7	2.8	OI
60.	30	28.3 □	2.7	18.9	11.1	9.4	1.7	OI
61.	30	30.3 □	2.7	18.3	11.7	12.0	-0.3	O
62.	31	31.4 □	2.7	17.8	13.2	13.7	-0.4	O
63.	31	31.3 □	2.7	17.2	13.8	14.1	-0.3	O
64.	27	29.9 □	2.7	16.7	10.3	13.1	-2.9	IO
65.	26	27.3 □	2.7	16.2	9.8	11.0	-1.3	O
66.	23	24.1 □	2.6	15.7	7.3	8.4	-1.1	O
67.	22	21.0 □	2.4	15.2	6.8	5.7	1.0	O
68.	23	18.3 □	2.1	14.8	8.2	3.5	4.7	O-I
69.	21	16.3 □	1.6	14.3	6.7	2.0	4.7	O—I
70.	19	14.9 □	1.3	13.9	5.1	1.0	4.1	O—I
71.	15	13.9 □	1.2	13.4	1.6	0.5	1.1	OI
72.	13	13.2 □	1.2	13.0	-0.0	0.2	-0.2	O
73.	11	12.7 □	1.3	12.6	-1.6	0.1	-1.7	IO
74.	10	12.2 □	1.4	12.2	-2.2	0.0	-2.2	I-O
75.	11	11.8 □	1.4	11.8	-0.8	0.0	-0.8	IO
76.	13	11.4 □	1.5	11.4	1.6	0.0	1.6	OI
77.	14	11.1 □	1.6	11.1	2.9	0.0	2.9	O-I
78.	12	10.7 □	1.7	10.7	1.3	0.0	1.3	OI
79.	11	10.4 □	1.8	10.4	0.6	0.0	0.6	O
80.	7	10.0 □	1.9	10.0	-3.0	0.0	-3.0	I-O
81.	8	9.7 □	2.0	9.7	-1.7	0.0	-1.7	IO
82.	6	9.4 □	2.1	9.4	-3.4	0.0	-3.4	I-O
AT THE PEAKS								
28.9	64.4	58.2 □	2.8	41.9	22.5	16.4	6.2	O-I
62.8	31.2	31.5 □	2.7	17.3	13.9	14.1	-0.2	O

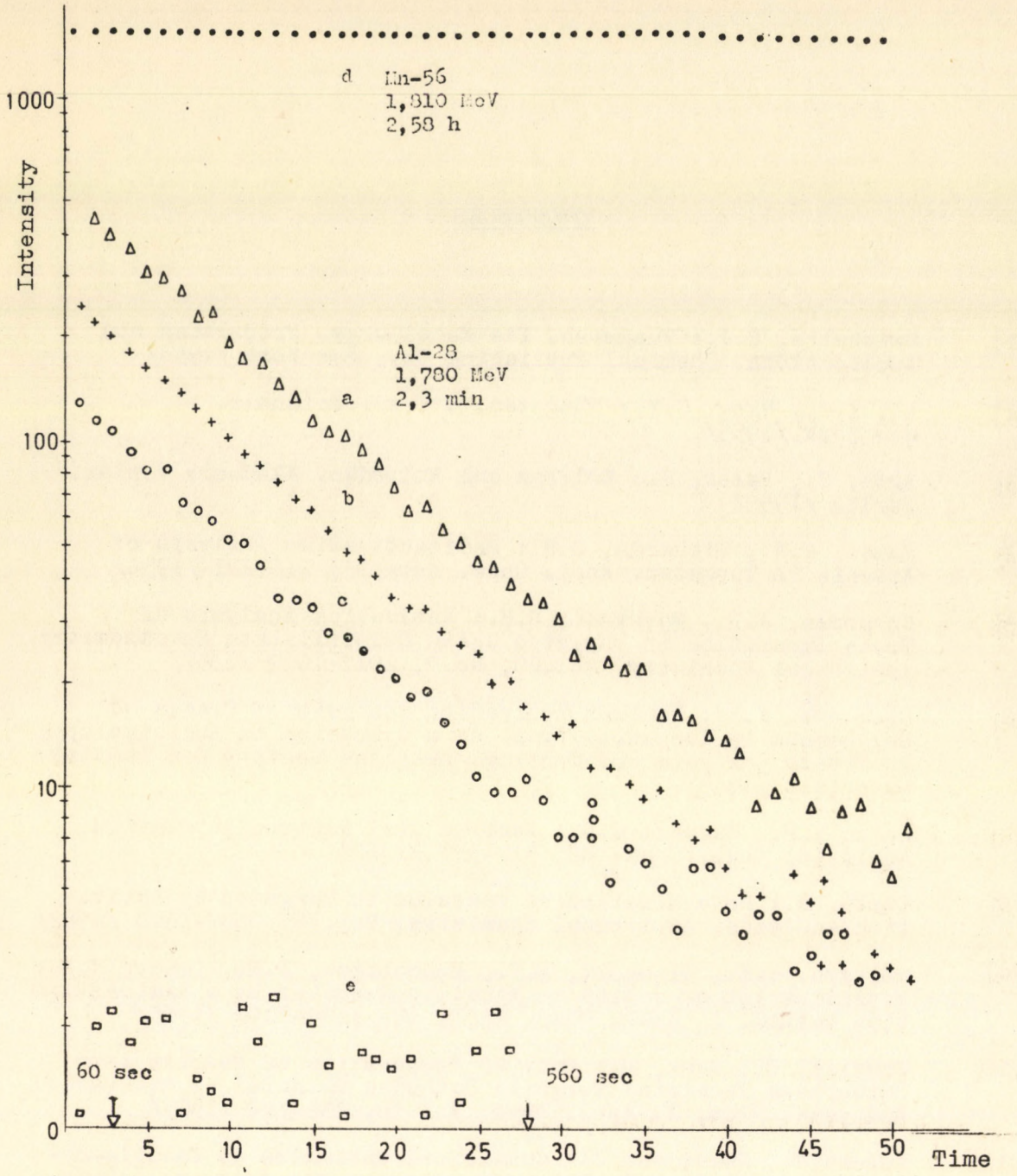


Fig. 11

Half-life measurement of the 1,78 MeV photopeak of $^{28}Si/n,p/^{28}Al$ reaction in tungsten matrix with multiscaler analyser /a,b,c/. /10 parts on the time axis represent 200 secs/ For comparison the decay of the most probable interference from $^{56}Fe/n,p/\ ^{56}Mn$ reaction /1,81 MeV line//d/ and the background intensity /e/ are also shown.

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ABSTRACT

A nondestructive activation analytical method developed for the determination of the axial concentration distribution of Al, Si and K residual additives in sintered tungsten rods is briefly described. A 14 MeV neutron generator is used to produce the reactions $^{27}Al/n,p/^{27}Mg$, $^{28}Si/n,p/^{28}Al$ and $^{39}K/n,2n/^{38}K$ and the activities are compared with standards. A weighted least-squares fitting program is used for peak area determination of the scintillation spectra. A scheme of the irradiating and measuring arrangement with a pneumatic sample-transfer system is presented. The system is applicable to large-scale routine analysis and lends itself well to automation. The results of preliminary measurements with this arrangement are given.

РЕЗЮМЕ

В работе коротко описывается метод активационного определения Al, Si и K, оставшихся в спеченных стержнях из металлического вольфрама, без разложения образца, с помощью нейтронного генератора по реакциям Al-27/n,p/Mg-27, Si-28/n,p/Al-28, K-39/n,2n/K-38. С помощью этого метода можно проверить аксиальное распределение концентрации присадочных материалов вдоль стержня. Исследование аксиального распределения в случае рутинных измерений может быть в значительной мере автоматизировано. Описывается блок-схема системы автоматического облучателя, транспортера образца и анализатора результатов измерений. Предварительные исследования были проведены на 15 стержнях. Был использован нейтронный генератор мощностью 14 Мэв. Полученные активности были сравнены с активностью образцов известного состава. Для определения площадей под фотопиком сцинтилляционных спектров была использована согласующая программа, обосновывающаяся на методе взвешанных наименьших квадратов.

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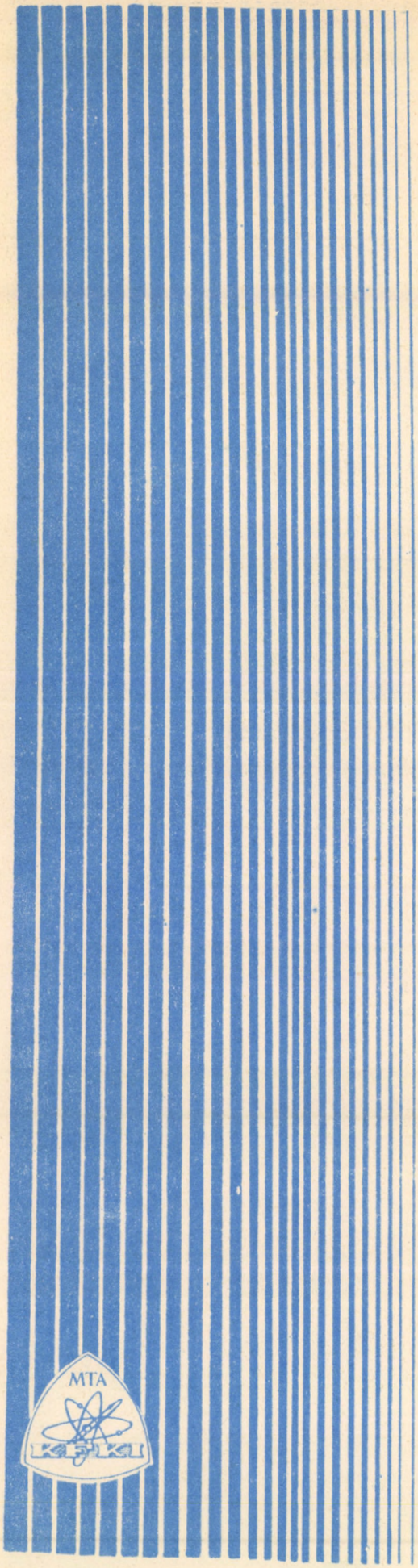

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