

NEUTRON ACTIVATION INVESTIGATION OF IMPURITIES OF HIGH PURITY GALLIUM

By

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The significance of the high purity gallium (99.9999% Ga) has extremely increased together with the rapid spreading of the semi-conductor technics (e. g. gallium activated by arsenium). The value of gallium as a by-product of the aluminium metallurgy is equal to that of noble metals in case of good purity. So the qualification, the analysis of high purity gallium is an important task from point of view of economy, too [1].

On the initiative of the Research Institute of Metal Industry (PAPP and co-workers) the neutron activation analysis of gallium has begun at our institute. Our programme was to explore the possibilities of employing the non-destructive test method.

The main point of the non-destructive test is that the activated sample is investigated without chemical separation, purifying, only on ground of the intensity, energy and change of radiation in time and from these dates the composition of the sample is determined either with the help of correspondingly chosen comparator samples (relative method) or calculating taking into consideration the activation circumstances (absolute method). The method is suitable, if e. g. the half-life of the active isotope of the element, building the principal mass of the studied material, is much shorter, than the half-life of the impurities and at the same time the nuclear chemical properties of the active products of the impurities are different enough to demonstrate them side by side with gamma-ray spectrometry, with measuring the absorption of gamma, beta radiation or determining the half-life [2].

In the literature of the activation analysis the destructive method is prevalent for the time being, but in the newest publication there is an obvious tendency to employ the non-destructive method, if it is possible theoretically, and the practical requirements (i. e. the length of time) permit it [3].

At the destructive activation tests [4—6] the activated impurity will be separated after the activation from the active principal mass of the material with chemical (i.e. precipitation), physicochemical (i.e. distillation, chromatography) or electrochemical (electrolysis) methods. For sake of the efficiency

of the separation, generally the method of the reverse isotope dilution is to be applied. Inactive isotope of known quantity is added to the active material, and this way abolishing its trace character the separation can be fulfilled effectively. The precondition in applying this method is that the half-life of the active isotope should not be so short, as to decompose practically during the separation and to have a suitable separation method. In most case the matter is the second precondition.

The destructive activation analysis can be regarded as a generally applicable method — the general applicability being its great advantage. But to carry it out can be often difficult, complicated, dangerous (radiation protection) consequently if possible, the non-destructive test must be regarded, as the more favourable. At the same time the destructive method has a disadvantage — rarely referred to in the literature — coming essentially from applying the isotope dilution method. The active material in question can be got from the mixture of active isotopes mainly with inverted dilution method. Because of the very sensible demonstrability of the active isotopes every active impurity (coming from adsorption, building of complexes, inclusions, splashing on, infiltration) can unintentionally be measured easily. As the principal mass of the material, from which the isotope in question is to be got, is radioactive, the danger of impurity rather effecting the results of the measuring is big and can be eliminated in many cases only by long, complicated purification processes [7, 8].

Our task was more closely to examine the possibilities of applying the non-destructive neutron activation method in connection with Zn, Fe, Hg and Cu impurities of six 9's purity gallium.

Table I shows the nuclear data of Ga, Zn, Fe, Hg and Cu important for the non-destructive investigation [2, 9].

Studying the literature about activation analysis and gallium no paper could be found about the activation investigation of the high purity gallium.

Comparing the nuclear data of the elements shown in table I it was found that in theory there is a possibility for the non-destructive activation determination of Zn, Fe and Hg, but Cu can be determined only with destructive method. (The suitable analytical method has been elaborated in the Analytical and Isotope Laboratory of the Research Institute for Metallurgy.) In determining Zn, Hg and Fe in Ga the interfering effects in activation were investigated and it could be stated, that in the present case an interfering possibility is to be taken in account. As the simultaneous presency of Cu and Zn has to be taken into consideration, we must have regard for a so-called second order reaction [2].

A secondary disturbing reaction is spoken about, when the decomposition product of an activated element of the sample (n, γ) gives the investigated isotope in the reaction:

Principal reaction: ${}^A Z (n, \gamma) {}^{A+1} Z$
 Interfering reaction: ${}^{A-1}(Z-1) (n, \gamma) {}^A(Z-1)$
 ${}^A(Z-1) \xrightarrow{\beta^-} {}^A Z (n, \gamma) {}^{A+1} Z$

In given case:

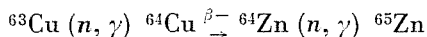


Table I

Target element	Occurrence %	(n, γ) isotope products	Activation cross section σ barn	Half-life t _{1/2}	Nature of radiation	Radiation Energy	
						β MeV	γ MeV
${}^{69}_{31}\text{Ga}$	60.2	${}^{70}\text{Ga}$	1.4	21.1m	β ⁻	1.65 (99%)	1.04
							0.17
${}^{71}_{31}\text{Ga}$	39.8	${}^{72}\text{Ga}$	4	14.3h	β ⁻	0.6 (40%)	0.63
						0.9 (32%)	0.83
						1.5 (11%)	2.2
						2.5 (8%)	3.1
						3.1 (9%)	2.5
${}^{64}_{30}\text{Zn}$	48.89	${}^{65}\text{Zn}$	0.5	250d	K β ⁻ (3%)	0.32	1.12
${}^{68}_{30}\text{Zn}$	18.56	${}^{69}\text{Zn}^*$	0.3	14.8h	I		
							${}^{69}\text{Zn}$
${}^{58}_{26}\text{Fe}$	0.31	${}^{59}\text{Fe}$	0.3	45.1d	β ⁻	0.46 (50%)	1.29
						0.26 (50%)	1.09
${}^{63}_{29}\text{Cu}$	69.1	${}^{64}\text{Cu}$	4	12.8h	K (42%)		1.35
${}^{65}_{29}\text{Cu}$	30.9	${}^{66}\text{Cu}$	2	5.1m	β ⁻ (39%)	0.57	
					β ⁺	0.66	
					β ⁻	2.6 (91%)	
						1.5	1.04
${}^{196}_{80}\text{Hg}$	0.14	${}^{197}\text{Hg}^*$	2900	23h	I		0.13
					K (3%)		0.16
							0.27
							0.19
${}^{198}_{80}\text{Hg}^*$	10.02	${}^{197}\text{Hg}$	20	65h	K		0.19 : 0.07
					I		0.15
${}^{202}_{80}\text{Hg}$	29.8	${}^{203}\text{Hg}$	2.45	47.9d	β ⁻	0.2	0.36
							0.28
${}^{204}_{80}\text{Hg}$	6.85	${}^{205}\text{Hg}$	0.3	5.5m	β ⁻	1.8	

The cross section of ${}^{63}\text{Cu} (n, \gamma) {}^{64}\text{Cu}$ reaction is 4 barn, the fractional isotopic abundance of ${}^{63}\text{Cu}$ is 69.1%, the half-life is 12.8 hours, 39% of the active isotope yield β-rays.

Taking the initial quantity of 10^{-5} g, the irradiation time 3—5 days, the quantity of the active Zn got in the second order reaction does not attain the detection level (10^{-2} — 10^{-3} μ C).

The number of active Cu atoms arising during the activation is

$$N_{\text{Cu}}^* = \frac{\Phi \cdot \sigma_{\text{Cu}} \cdot m \cdot N_A \cdot f_{\text{Cu}} \cdot t}{A_{\text{Cu}}} \approx 10^{12}$$

where Φ = neutron flux = 10^{13} n/cm² s

σ = reaction cross section = $4 \cdot 10^{-24}$ cm²/n

m = mass of the isotope = 10^{-5} g

N_A = Avogadro's number = $6 \cdot 10^{23}$ atoms/g-atom

A_{Cu} = atomic weight of copper = 63 g/g-atom

f_{Cu} = fractional isotopic abundance of Cu-63

t = activation time (80 hours) in seconds

Computing with all Cu arised during the activation, the number of Zn atoms:

$$N_{\text{Zn}} = N_{\text{Cu}}^* \cdot 0.39 \cdot \left[1 - \exp \left(- \frac{0.69 t}{t_{1/2 \text{ Cu}}} \right) \right] \approx 4 \cdot 10^{11}$$

The activity of Zn:

$$\omega_{\text{Zn}} = \Phi \cdot \sigma_{\text{Zn}} \cdot N_{\text{Zn}} \left[1 - \exp \left(- \frac{0.69 t}{t_{1/2 \text{ Zn}}} \right) \right] \approx 2 \cdot 10^{-2} \text{ tps}$$

After the data of Table I the trace elements have several stable isotopes, a great part of which can be activated parallel (competing nuclear reactions).

The scheme of the competing (n, γ) nuclear reactions [2]:

principal reaction: ${}^A Z (n, \gamma) {}^{A+1} Z$

competing reactions: ${}^{A \pm n} Z (n, \gamma) {}^{A \pm n + 1} Z$

i. e. ${}^{63}\text{Cu} (n, \gamma) {}^{64}\text{Cu}$ principal reaction

${}^{65}\text{Cu} (n, \gamma) {}^{66}\text{Cu}$ competing reaction

${}^{202}\text{Hg} (n, \gamma) {}^{203}\text{Hg}$ principal reaction

${}^{196}\text{Hg} (n, \gamma) {}^{197}\text{Hg}$ competing reaction

The mistake caused by the competing reactions is negligible in our case because only isotopes with a long half-life are investigated, but the products of the competing reactions are of short half-life.

Activation

Samples of gallium are placed in ground joint quartz vessels (15 mm dia, 25 mm height) in 0.1—1 g quantities for irradiation in reactor. The time of irradiation was between 2—5 days.

Neutron flux: 10^{13} n/cm² \times s

The efficiency of the activation, the number of the developed active nuclei

depends beside the neutron flux and the quantity of the material on the half-period of the developed active isotope, too. For short-lived activation products (if $t_{1/2}$ is of hour magnitude) about $10 t_{1/2}$ is the best duration of irradiation to get the saturation activation.

In this case — isotopes with long half-life (50—250 days) were investigated — we had to be satisfied with, 0.1—0.01 parts of the half-life (2—5 days), as an activation time.

Analytical methods [10, 11]

1. Determination of the half-lives.
2. Determination of the absorption coefficients.
3. Gamma-ray spectrometry.

The model diagrams of the determination of the half-life resp. the absorption coefficient are shown in Fig. 1—8.

Table II

The employed scalars	EMG 1873	EKCO N 530 F
<i>Counting part</i>		
Electronic scaler	3 steps: 1, 10, 100	6 steps: 1—10 ⁵
Resolution of decades	5 μ sec	5 μ sec
Electromechanic counter	5 numbers	no
<i>Pulse amplifier</i>		
Amplifying	with min. 5 mV and max 700 mV input-signal in 6 steps: 100, 200, 500, 1000, 2000, 5000 \times	with min. 200 mV input-signal, 25 \times
<i>Discriminator</i>		
Resolution time	5 μ sec	5 μ sec
Voltage (continuously regulated) ..	5—70 V	5—50 V
Discriminating accuracy	± 0.3 V	± 0.2 V
<i>Applied scintillation detectors</i>		
Type of photomultiplier tube	Gamma F 029	EKCO N 664
Diameter of window mm	M 12 F S35	EMI 9514 S
	35	60
<i>Data of current supply</i>		
A-supply	6.3 V	6.3 V (1.3 A)
Anode voltage	250 V 10 mA	300 V 25 mA stabilized
Direct voltage	1500 V 0.5 mA	1500 V 20 mA
Amplification	—	25, 50, 100, 250, 500, 1000 \times

The basis of the model computing is the 46 and 250 days half-life resp. 0.16 and 1.2 cm half-thickness of the lead layer. The result of the calculation shows well that a difference of 2 orders of magnitude in the quantity of isotopes causes uncertainty in the determination.

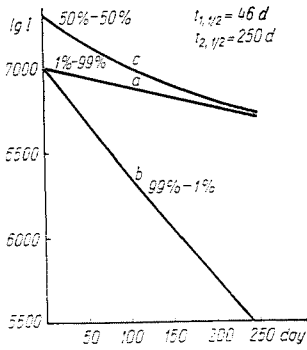


Fig. 1. Decay curves of mixtures of isotopes with half-lives of 46 and 250 days

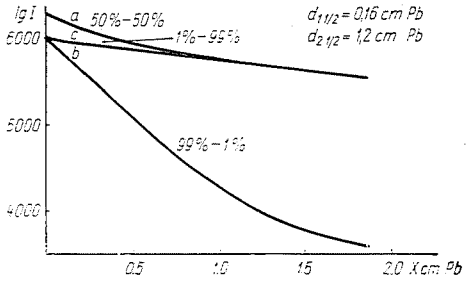


Fig. 2. Absorption curves of gamma rays of 0.2 and 1.1 MeV energy at different composition

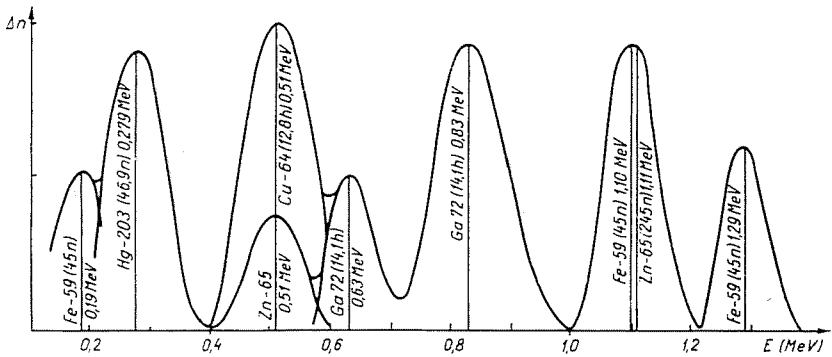


Fig. 3. Possibility of spectroscopic detection of ^{65}Zn , ^{59}Fe , and ^{203}Hg (10)

The possibilities of the spectral separation of the gamma radiation of different active isotopes are shown in Fig. 3 [10].

The measurements — at the present possibilities as pre-investigation — were made with “Labor” scaler (Type EMG 1873) fitted out with a scintillation detector, and EKCO scaler with its integrating discriminator (N 530 F type. Scaler, N 664 type scintillation detector).

The most important data of the equipments are given in Table II. The calibration of the measuring instruments was done with ^{137}Cs , ^{144}Ce , ^{60}Co , spectroscopic comparator and ^{65}Zn , ^{59}Fe , ^{131}I samples.

The resolution with EKCO apparatus on Cs-137 spectrum on 780 V is 8.6%.

Table III contains the characteristics of comparators and active isotopes of the concerning elements, important for the measuring of radiation [10].

Table III

Isotopé	Half-life	Energy of gamma-ray	Remarks
^{144}Ce	282	0.13; (0.08)	Spectr. comparator
^{137}Cs	33	0.66; 0.032	Spectr. comparator
^{131}J	8.1	0.36	
^{60}Co	5.27	1.17; 1.33	Spectr. comparator
^{59}Fe	45n	1.10; 1.29; 0.19; (0.14)	
^{65}Zn	245—250	1.11; (0.51)	
^{203}Hg	48	0.28; (0.07)	
^{64}Cu	12.8	0.51	
^{72}Ga	14.3	0.63; 0.83	

Investigation of gallium samples containing Zn and free from it

The task was primarily the control of the efficiency of dezincizing.

The irradiation time was 48 hours, the neutron flux: 10^{13} n/s.cm². Measurements began 12 days after finishing the activation, this time is $t = 0$.

According to the data of measurements it could be seen, that on the 7th day from the beginning of the measuring, Ga was so far decayed, that its radiation cannot be measured. (The original activity level diminished to 10^{-10} times.)

On the 17th day of the measuring the ratio of the specific activities (I_1/I_2) of the samples with Zn (1) and without it (2) was 16, on the 36th day 16.8, on the 53th day 20.6.

Data of the measurements are shown on Fig. 5.

The sample containing Zn and the dezincized one were activated during the same period and in the same circumstances. During the measurements ^{72}Ga was decayed at both. The difference greater than one order of magnitude at the specific activities can come exclusively from the impurities.

The decay curve of sample 1 shows unambiguously the presence of the ^{65}Zn of long half-life ($t_{1/2} = 245$ days).

From the decay curve of the dezincized sample 2 it can be seen that the sample is in first approximation really free from Zn, the decay curve shows the presence of an or several isotopes with a half-life of 45—47 days (Hg, Fe).

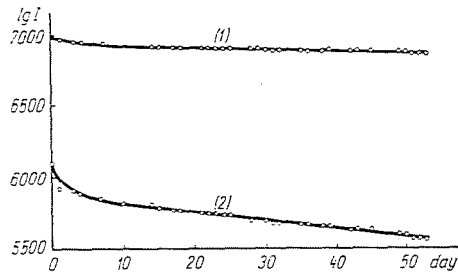
Fig. 4. Spectrum of ^{137}Cs on 780 V

Fig. 5. Decay of activated gallium samples contaminated by Zn(1) and dezincized (2)

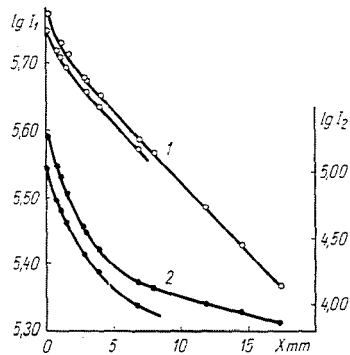


Fig. 6. Absorption of gamma-rays of active gallium contaminated by zinc (1) and dezincized (2) in lead

The shapes of the absorption curves of samples 1 and 2 announces that the energy of the radiation is rather different at the two samples (Fig. 6). The half thickness measured at sample 1 (12.2 mm) is in good agreement with the half thickness measured with ^{65}Zn (12.8). At sample 2 the half thickness is

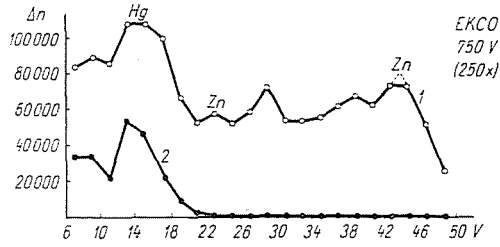


Fig. 7. Gamma spectrum of active gallium contaminated by zinc and dezinced, on 750 V



Fig. 8. Gamma spectrum of ⁶⁵Zn on 750 V

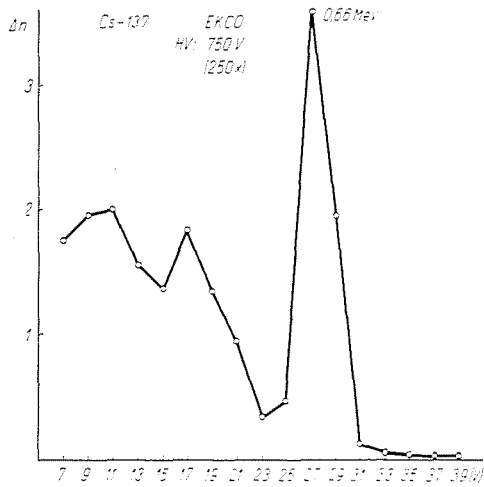


Fig. 9. Gamma spectrum of ¹³⁷Cs on 750 V

1 mm from the first part of the absorption curve, at the same time the literature half thickness of the 0.28 MeV gamma-rays (Hg) is 1.4 mm. So the absorption measurements verify the statements that we made as the results of the decay tests: there is mainly Zn impurity in sample 1 and in the first line Hg resp. Fe in sample 2.

From the spectrum (Fig. 7) got with EKCO apparatus, on ground of the calibration spectrums (Fig. 8, 9) it can be seen, that in sample 1 there is ^{65}Zn in great quantity, while the spectrum of the sample 2 shows the presence of ^{203}Hg .

Investigation of high purity gallium samples

Two samples of nearly the same weight (sample 1 1.1496 g, sample 2 1.0150 g) were examined after 72 hours irradiation. The aim of the test was to ascertain the possibilities of showing out faults caused by the possible distri-

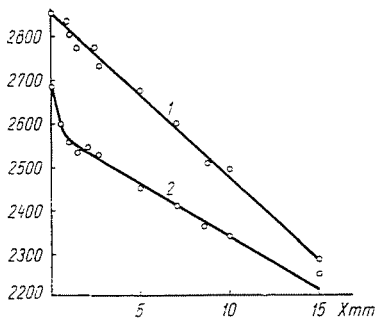


Fig. 10. Lead absorption of gamma rays of high purity gallium samples

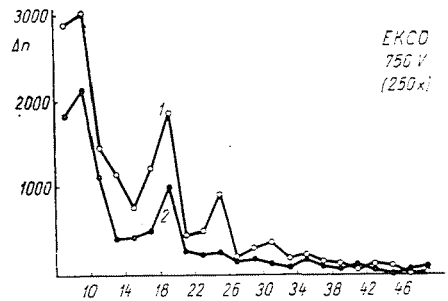


Fig. 11. Gamma spectra of high purity gallium samples on 750 V

bution inequality of the impurity or by the sampling. The half-lives were the same 46 days between the limit of error, but at the specific activities, the absorption curves (Fig. 10) and the spectra resp. (Fig. 11) there were differences easy to show out.

The ratio of the specific activities was 1.6.

The cause of the difference between the two high purity Ga samples can be the impurity got during sampling resp. the unequal distribution of the impurities. The differences between the result of the measurements call attention to the importance of sampling carefully, free from impurities before activation, at the same time they show the sensibility of the measuring method as well.

Non-destructive investigation of Zn, Fe, and Hg impurities

The activation analysis of 8 samples of different purity Ga and of a mixture of Fe—HgO—Zn was done with the three methods mentioned above.

The half-lives were calculated from the values measured between 17—49th days. On the 17th day there was no ^{72}Ga in measurable quantity.

In Table V the measured gross half-lives and the content of Zn calculated from the measured half-lives are shown.

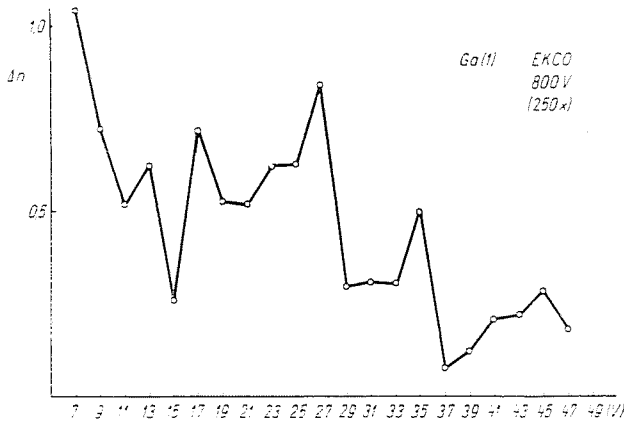


Fig. 12. Gamma spectrum of activated "pure" gallium [Ga(1)] on 800 V

Knowing the gross half-lives the content of Zn was calculated as follows:

$$I = I_1^0 \exp\left(-\frac{0.693}{t_{1,1/2}} t\right) + I_2^0 \exp\left(-\frac{0.693}{t_{2,1/2}} t\right)$$

$$I^0 = I_1^0 + I_2^0$$

$$t_{1,1/2} = 46 \text{ days (Fe, Hg)}$$

$$t_{2,1/2} = 250 \text{ days (Zn)}$$

$$t = 32 \text{ days}$$

$$I_2^0 = \frac{I - 0.61 I^0}{0.3}$$

From the intensity characteristic of the Zn content the real quantity of Zn was calculated. Data necessary for the calculation are:

1. Counting efficiency.
2. The activity of ^{65}Zn got at the activation circumstances.

The counting efficiency measured with ^{137}Cs and ^{60}Co standards of known activity was 7% with EKCO apparatus on 800 V at 10 V discriminator voltage.

The activity of the active isotopes (ω) got by t time radiation can be calculated:

$$\omega = \frac{\Phi \cdot \sigma \cdot m \cdot N_A \cdot f \left[1 - \exp\left(-\frac{0.693}{t_{1/2}} t\right) \right]}{A}$$

$$I = \omega \cdot \varphi$$



Fig. 13. Gamma spectrum of activated gallium contaminated by zinc [Ga(2)] on 800 V



Fig. 14. Gamma spectrum of activated gallium contaminated by zinc [Ga(3)] on 800 V



Fig. 15. Gamma spectrum of activated "pure" gallium [Ga(4)] on 800 V

where $m = \text{mass(g)}$; $N_A = \text{Avogadro's number}$; $A = \text{atomic weight (g)}$; $t_{1/2} = \text{half-life (day)}$; $f = \text{fractional isotopic abundance of the target nuclid (g/g)}$; $\Phi = \text{neutron flux (n/cm}^2 \cdot \text{s)}$; $\sigma = \text{the reaction cross section (cm}^2/\text{n)}$; $t = \text{time of irradiation (day)}$; $\varphi = \text{counting efficiency}$; $I = \text{measured intensity (see Table IV)}$.

Table IV

	Zn	Fe	Hg
Φ	10^{13}	10^{13}	10^{13}
σ	$5 \cdot 10^{-25}$	$3 \cdot 10^{-25}$	$2.5 \cdot 10^{-24}$
m	10^{-3}	10^{-3}	10^{-3}
N_A	$6 \cdot 10^{23}$	$6 \cdot 10^{23}$	$6 \cdot 10^{23}$
A	64	58	202
f	0.49	0.003	0.3
t	3.3	3.3	3.3
$t_{1/2}$	245	45	48
$I \left(\frac{\text{counts}}{100 \text{ s. mg}} \right)$	$1.6 \cdot 10^5$	$3.2 \cdot 10^4$	$7.8 \cdot 10^5$

Hg and Fe cannot be separated according to the half-life, therefore it, must be supposed in the first step, that there is no Hg and we calculate on Fe,

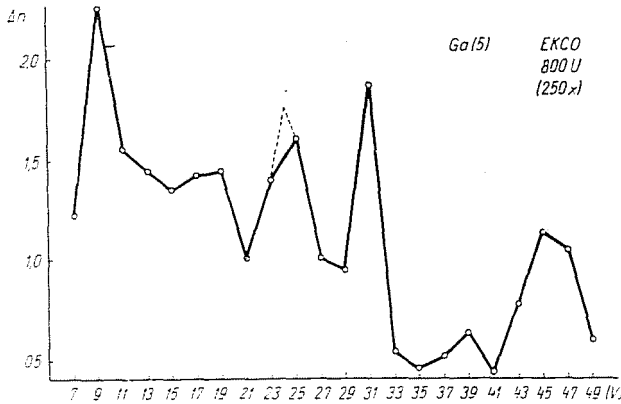


Fig. 16. Gamma spectrum of activated gallium contaminated by Fe [Ga(5)] 800 V

or we try to determine the relative quantities of Hg and Fe from spectrometric data.

After the data of Table V the followings can be stated:

1. The half-life shows well the presence of Zn. (The half-life of "pure" Ga is 55 days, that of the samples contaminated by Zn 176 resp. 64 days.)
2. The value of the specific activities is proportional to the eventual change in quantity not only of Zn, but of Fe and Hg, too (column 4).
3. The simultaneous determination of Fe and Hg is inambiguously possible only with gamma spectrometry.

Table V

Number of Sample	Given composition	I_{10}/I_{30} *	I_c/I_p **	$t_{1/2}^{***}$ (day)	Calculated Zn $1.6 \cdot 10^5$ c/100 s = 10^{-3} g/gZn
1. Ga-(1)	pure Zn < 0.001%	1.77	1	55	$6 \cdot 10^3$ c/100 s = $4 \cdot 10^{-5}$ g Zn/g
2. Ga-(2)	+0.025% Zn	1.53	14.3	176	$4.6 \cdot 10^5$ c/100 s = $3 \cdot 10^{-3}$ g Zn/g
3. Ga-(3)	+0.003% Zn	1.65	1.53	64	$2 \cdot 10^4$ c/100 s = 10^{-4} g Zn/g
4. Ga-(4)	pure	1.92	1	55	$3 \cdot 10^3$ c/100 s = $2 \cdot 10^{-5}$ g Zn/g
5. Ga-(5)	+0.005% Fe	1.73	5.03	47	$3 \cdot 10^3$ c/100 s = $2 \cdot 10^{-5}$ g Zn/g
6. Ga-(6)	+0.001% Fe	1.81	1.25	45	$< 10^{-5}$ g Zn/g
7. Ga-(7)	pure	2.02	1	45	$< 10^{-5}$ g Zn/g
8. Ga-(8)	+Zn + Fe	1.76	4.1	57	$2 \cdot 10^4$ c/100 s = 10^{-4} g Zn/g
9. Fe-Zn-Hg****	92.4% Fe (50%) 4.6% Zn (12%) 3.0% Hg (38%)	3.74	—	62	28%*****
^{65}Zn	—	1.63	—	—	—
^{59}Fe	—	1.77	—	—	—
^{60}Co	—	1.42	—	—	—
^{137}Cs	—	1.95	—	—	—

* Ratio of intensities measured at 10 and 30 V discriminator voltages.

** Ratio of specific intensities of contaminated Ga and so-called pure Ga.

*** Half-life determined between 17–49th days.

**** Isotope and activity composition at the end of the activation.

***** On the 90th day from the end of the activation 28% of the whole activity is the Zn activity from measured and calculated half-lives as well.

4. From the data of the columns 2 and 6 we see, that the content of Zn calculated from the half-lives with absolute method (column 6) and that given by the Research Institute of Metal Industry (column 2) do not agree. But it is to see, too that the contents of Zn in the samples change in parallel. So the employing of the relative method gives results in any case.

The usage of the evaluation of the absorption curves for analysis has great difficulties in case of more than one impurity present the evaluation of the results of the measurements from point of view of the absorption coefficients is shown in Table VI.

Table VI

Sign of Sample	Absorption coefficient (cm^{-1}) (lead absorbent)	
	I	II
Ga(1)	1.88	0.69
Ga(2)	1.72	0.69
Ga(3)	1.72	0.64
Ga(4)	2.11	0.74
Ga(5)	2.6	0.78
Ga(6)	2.4	0.70
Ga(7)	2.6	0.69
Ga(8)	1.97	0.60
9(Zn—Fe—Hg) ..	3.9 1.22	0.55
^{60}Zn	1.6	0.53
^{59}Fe	1.65	0.48
^{137}Cs	—	0.92
^{60}Co	—	0.45

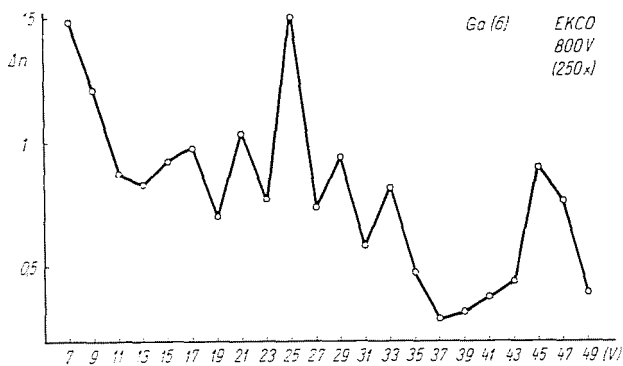


Fig. 17. Gamma spectrum of activated gallium contaminated by Fe [Ga(6)] 800 V

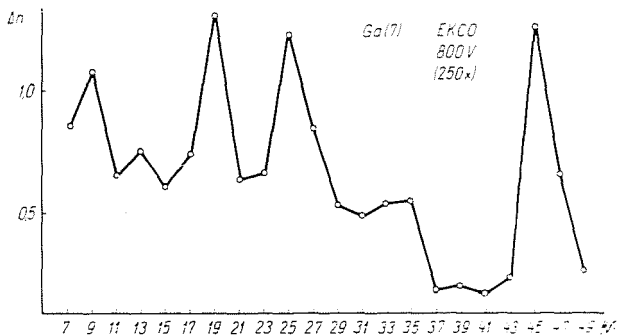


Fig. 18. Gamma spectrum of activated "pure" gallium [Ga(7)] on 800 V

On the absorption diagrams generally two sections with rather different slope can be distinguished. From the absorption coefficients given in Table VI the values determined from section one are marked with I, those determined from section two with II.

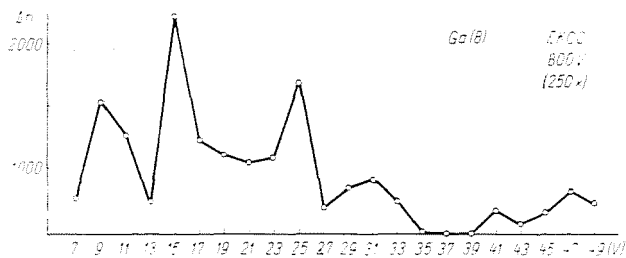


Fig. 19. Gamma spectrum of activated gallium contaminated by Zn and Fe [Ga(8)] on 800 V

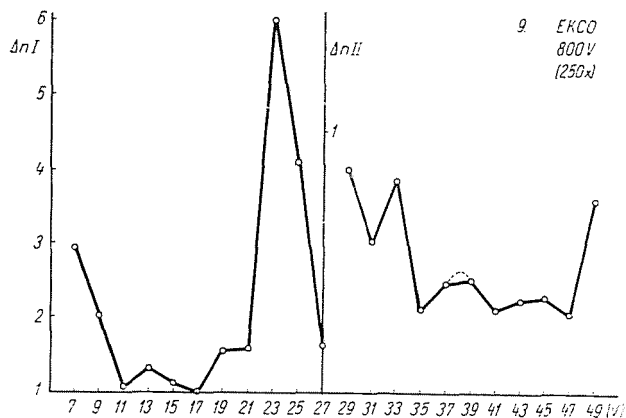


Fig. 20. Gamma spectrum of activated Fe—Zn—Hg mixture on 800 V



Fig. 21. Gamma spectrum of ^{59}Fe on 750 V



Fig. 22. Gamma spectrum of active "pure" gallium [Ga(1)] on 750 V



Fig. 23. Gamma spectrum of activated gallium contaminated by Zn [Ga(2)] on 750 V

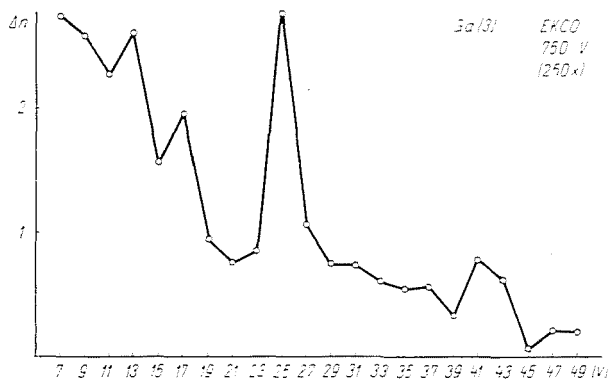


Fig. 24. Gamma spectrum of activated gallium contaminated by Zn [Ga(3)] on 750 V

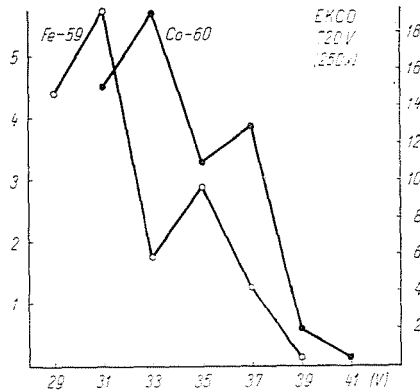


Fig. 25. Gamma spectrum ^{60}Co and ^{59}Fe on 720 V

Evaluating the results the values determined by us with ^{137}Cs , ^{60}Co , ^{59}Fe and ^{65}Zn must be taken into consideration and not the literatural ones.

The spectra were taken with EKCO apparatus at 800, 750 and 720 V voltage values, with amplification 250, between 6—50 V discriminator voltages (further dv), with 2 V discriminator channel.

On ground of the calibration data it could be stated, that the cutoff voltage is 800 V for 0.6 MeV gamma-rays, 750 V for 1.2 MeV and 720 V for 1.4 MeV (i. e. at 50 V discriminator position the intensity diminishes to the order of magnitude of the background). The spectra taken on 800 V are shown on Fig. 12—20.

The gamma-rays of ^{203}Hg (0.28 MeV) can be shown with good certainty at 800 V (25 dv), at the same time the 0.51 MeV rays of ^{65}Zn can be found (weakly; 39—40 dv). The 0.19 MeV rays of ^{59}Fe , too, can be found in the spectrum (17—19 dv); on the end of the spectrum the 0.63 MeV rays of the ^{72}Ga can be found distinctly (45 dv), as well.

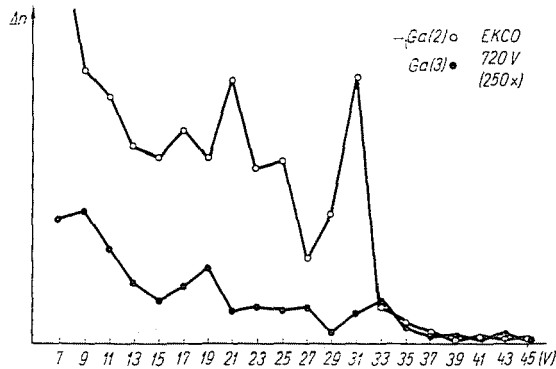


Fig. 26. Gamma spectra of active gallium contaminated by zinc [Ga(2,3)] on 720 V

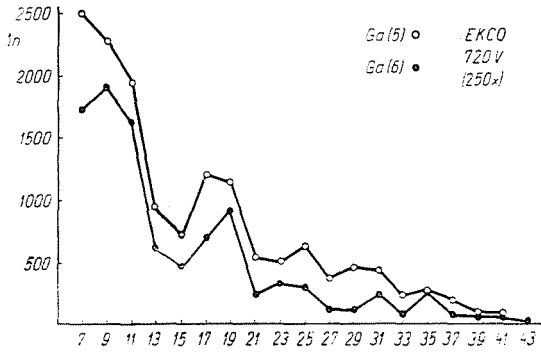


Fig. 27. Gamma spectra of active gallium contaminated by iron [Ga(5,6)] on 720 V

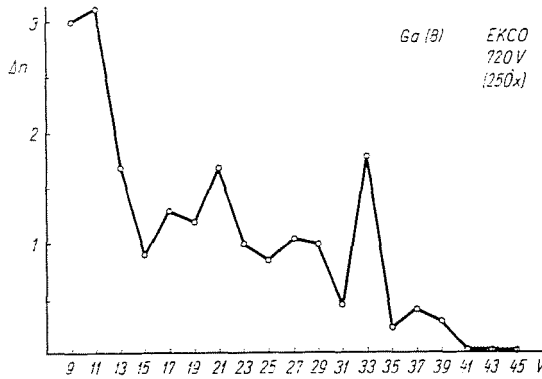


Fig. 28. Gamma spectrum of activated gallium contaminated by iron and zinc [Ga(8)] on 720 V

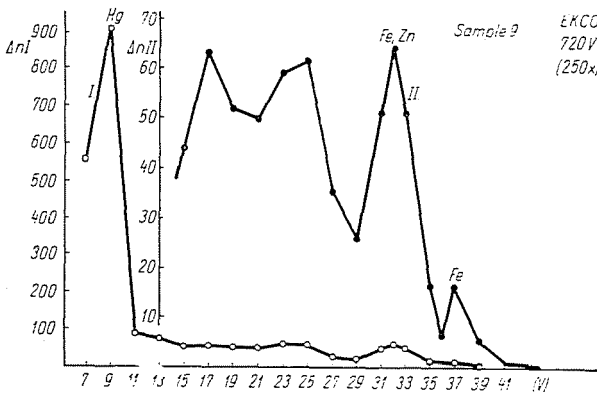


Fig. 29. Gamma spectrum of active Fe-Zn-Hg mixture on 720 V

The spectra show quite well, that on the 28th day from the end of the irradiation the 0.63 MeV peak of ^{72}Ga (45 dv) still strongly appears. 36 days after the activation the ^{72}Ga can be still shown, although the falling of the intensity is remarkable. It must be said, that no active gallium can be found already with other methods.

The 0.28 MeV (25 dv) top of ^{203}Hg appears clearly at the 8 Ga samples and at the Fe—Hg—Zn sample as well. So in each gallium sample there is Ga of well remarkable quantity.

The 0.19 MeV rays of ^{59}Fe can be found in each spectrum, but it is more difficult, uncertain to evaluate, than the ^{203}Hg .

The spectra on 750 V (Fig. 21—24) are in the first line for detecting ^{65}Zn (1.11 MeV), but there, too, the 1.10 MeV peak of ^{59}Fe can be found. For ^{65}Zn the apparatus blocks on 750 V, in presence of ^{59}Fe it does not. (The apparatus did not block at either sample, so there is ^{59}Fe in each one.) The peak of ^{65}Zn can be found at 45 dv, that of ^{59}Fe at 41—42 dv. The 0.51 MeV peak of ^{65}Zn is at 21—22 V dv. (weak).

The spectrum on 720 V is in the first line for pointing out the 1.10 and 1.29 MeV energies of ^{59}Fe . The calibration standard generally accepted is ^{60}Co of spectroscopic quality (Fig. 25—29).

It is interesting, that the radiations of the 3 so-called „pure” Ga samples are different. It can be seen 1. in the specific activities 2. in the half-lives, 3. in the absorption quantities and 4. in the spectra.

The differences shown reproducibly with several methods call the attention emphasized to the carefulness with measuring and handling the high purity samples before irradiation and raise the possibility of the unequal distribution of the impurities.

The evaluation of the spectra in simultaneous presence of Fe, Zn and Hg, the distorting, disturbing effect of the simultaneous peaks, the simultaneous quantitative determination of Hg, Fe and Zn, want still further research. On ground of the gamma spectrometric results (only pre-experiments because of the strongly limited efficiency of our measuring devices) it can be said that the task can be solved with amplitude analysator of suitable sensibility.

Summary

The qualitative and — with suitable standards — quantitative determination of Zn, Fe and Hg impurities in high purity gallium are possible with the so-called non-destructive neutron activation method. After the decay of the bulk of the activated gallium, the Zn, Fe and Hg can be simultaneously detected determining the half-lives, the absorption coefficients and the gamma-ray spectrum.

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