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METHODS FOR DE
OF THE RADION
OF STANDARD S
35_S, 32_P, $90_{Sr}+90_Y$, 147_{Pm}

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METHODS FOR THE DETERMINATION OF THE RADIONUCLIDIC PURITY OF STANDARD SOLUTIONS OF ^{35}S, ^{32}P, ^{90}Sr+^{90}Y, ^{147}Pm

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S u m m a r y

For the purpose of beta spectrometry a semiconductor spectrometer with one Si(Li) detector, cooled with liquid nitrogen, was constructed. The geometrical detection efficiency is about 10 % 4 sr. The achieved resolution for conversion electrons of ^{137m}Ba with an energy of 624 keV is 2,6 keV (FWHM). A program in the FORTRAN language for the correction of the deformation of the measured spectrum by backcatter was prepared for the analysis of contimuous beta spectra. The described method permits the determination of the maximum energy of the beta spectrum with a precision of $\pm$ 5 keV.

A method for the purity control of standard solutions of ^{35}S, ^{32}P, ^{90}Sr and ^{147}Pm is designed. The method is based on the use of the semiconductor beta spectrometer Si(Li). For the nuclides ^{35}S and ^{147}Pm, an overall standardization precision of 1,5 % for ^{35}S and 2,5 % for ^{147}Pm may be quarranteed. To ensure the required standardization precision of 1 % for the nuclides ^{90}Se and ^{32}P, the method of comparison with the spectrum of the pure nuclide has to be used.

PART I. – BETA SPECTROMETRY

1. Introduction

During the preceding period a 4π plastic scintillator[1] and a semiconductor spectrometer with a non-cooled Si(Li) detector [2] were experimentally tested in the ÚVVVR for the use in the spectrometry of beta particles. The disadvantage of these spectrometers was namely the inadequate resolution power. Therefore, a new spectrometer was developed, based on a liquid nitrogen cooled Si(Li) detector.

The primary intention was to construct a spectrometer with two cooled detectors, detecting the radiation from a solid angle near to 4πsr. It was presumed that such an arrangement will be a good approximation of the ideal case of 4πsr detection and that in this way the effect of backscatter will be suppressed. Experience has thought us that with the arrangement of the spectrometer with two Si(Li) detectors, the maximum possible detected solid angle is about 66 % 4πsr and that from the viewpoint of the reduction of the backscatter coefficient this arrangement is less advantageous than a spectrometer working with one Si(Li) detector in a narrow beam. The details on the performed tests with various detector arrangements are presented further. Therefore, the concept of the two detectors was abandoned and the final arrangement makes use of one detector only.

2. Cryostat and vacuum changer

A Dewar vessel built in the Nuclear Research Institute in Řež was used for the constructtion of the spectrometer. The cooled copper finger is led out through the bottom, perpendicular to the vessel axis. The vessel is filled with liquid nitrogen from the top side.

The copper finger is further led from the vessel bottom in a duralumin tube where it is centered by means of a laminated bracing. On the end of the duralumin tube, the measuring chamber with the detector is mounted. The chamber has a diameter of 65 mm and is 75 mm high. The location of the detector sideways of the other parts of the apparatus led to an enlargement

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of the pumped volume by about 1 liter, it enables, however, to add to the measurement chamber another Ge(Li) or Si(Li) detector in the Dewar vessel, e.g. for coincidence measurements.

The sample exchange is performed through a small chamber connected with the channel and measuring chamber. The connecting channel is closed by a slide valve during the sample exchange. After insertion of the sample and closing of the chamber, the chamber volume is evacuated by means of a rotary pump to 10 Pa (10^{-3} Torr), then the connecting channel is opened and the sample is pushed into the measuring position by means of a pull rod passing through a rubber bushing.

A substantial part of the spectrometric device is the specially constructed cryostat with a vacuum sample changer and a vacuum apparatus, the construction and putting into operation of which were the most difficult and time consuming tasks.

3. Vacuum apparatus

The original design of the vacuum apparatus presumed the use of a rotary oil pump to generate a pre-vacuum and of an oil diffusion pump to generate the working vacuum of the order of 10^{-3} to 10^{-4} Pa (10^{-5} to 10^{-6} Torr). Already a short, few days test operation has shown that such an arrangement of the vacuum apparatus is quite unfeasible. The recurrent diffusion of oil vapors even over the freezing trap between the pump and the pumped volume into the space around the detector and the ice accreation on the cooled parts was such that after a few days operation the input window of the detector was covered with visible oil drops. The oil deposit on the detector input window would lead to a permanent deterioration of the spectrometric properties and the use of the oil diffusion pump was, therefore, abandoned.

The diffusion pump was used only for the evacuation of the cryostat volume in a non-cooled state, when no condensation of vapors colud take place. Then the diffusion pump was shut and for the maintenance of the operation vacuum a Penning gauge is used which serves also as the vacuum indicator.

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The Penning gauge works here as a ion pump with a low pumping speed which, however, is quite sufficient to keep up the necessary vacuum. With the permanent operation of the Penning pump, a vacuum of 10^{-3} Pa (cca 10^{-5} Torr) is obtained over the time when the connecting channel between the measuring chamber and exchanger chamber is closed. During measurement, the attained vacuum is worse (0,01 to 0,05 Pa = 10^{-4} to 5.10^{-4} Torr), due to leakes caused by the rubber bushings of the pull rods for the handling of the valve and the sample holder.

After achieving this vacuum in the whole spectrometric system, the Dewar vessel shows a daily nitrogen evaporation of only about 2 kg which assures a permanent reliable performance.

4. Choice of the detector geometry

The greatest limiting factor in the study of the continuous beta spectra measured with the use of a Si-detector is the electron backscatter. This effect can be removed by the radiation detection in the 4πsr geometry, as was e.g. carried out by FLOTHMAN [3], who used two Si(Li) detectors in close touch with the sample prepared on a thin membrane. Such a spectrometer showed very good properties, but for routine work it is quite unsuited.

In the ÚVVVR there are two Si(Li) detectors available with an area of 200 mm^2 and thickness 2 and 5 mm, which were used for the choice of the best configuration that could be utilized for routine measurements.

Three possible variants were arranged:

1st variant:

The two detectors were used, put as close together as practically possible, i.e. on a distance of 4,5 mm. The sample on a thin membrane 30 to 40 $\mu g.cm^{-2}$ stretched on a supporting ring was inserted between the detectors. The obtained geometric efficiency was about 66 % 4πsr. The detectors were connected in parallel with the input of a charge sensitive preamplifier CI 970. In this connexion, a resolution of 6 keV (FWHM) was achieved for 624 keV electrons. The backscatter

coefficient p, defined as the ratio of the number of the partially absorbed electrons to the total number of the detected monoenergetic electrons, may be determined by the detection of the electron spectrum measured in coincidence with the photons of the characteristic radiation [2, 4-7]. The coefficient was determined for the 624 keV conversion electrons of the ^{137m}Ba with the result p = 0,33. This variant had a disadvantage in the necessity of the use of thin sample, prepared in the same way as for absolute measurement. The thin membranes often crashed when inserted and removed from the vacuum and even caused a contamination of one detector. Also the movement of the sample carrier in the close vicinity of the detector window was very disadvantageous. The detector window was poluted with dust particles and organic binder used for the fixing of the samples to the support.

2nd variant:

Only one detector was used, placed in a distance of about 2 mm from the sample. Other scattering objects (chamber walls, X-detector) were at least 30 mm remote from the sample. The geometric efficiency was about 33 % 4πsr. The backscatter coefficient for the 624 keV conversion electrons of ^{137m}Ba was p = 0,37. The resolution power was 2,6 keV (FWHM).

3rd variant:

Only one detector was used again, this time at a distance of 13 mm from the sample. The geometric detection efficiency was about 10 % 4πsr. The bacscatter coefficient for the 624 keV conversion electrons of ^{137m}Ba was p = 0,289. The resolution power was 2,6 keV (FWHM).

From the comparison of these three variants it follows that the least value of the parameter p is obtained for the third arrangement, where a sufficiently good resolution is ensured, too. The disadvantage of the lower detection efficiency is fully compensated for by a number of practical merits, namely a smaller risk for the detector. Therefore, this variant was chosen as the basis for the final arrangement, the properties of which were then investigated in detail and which is schematically shown on Fig. 1.
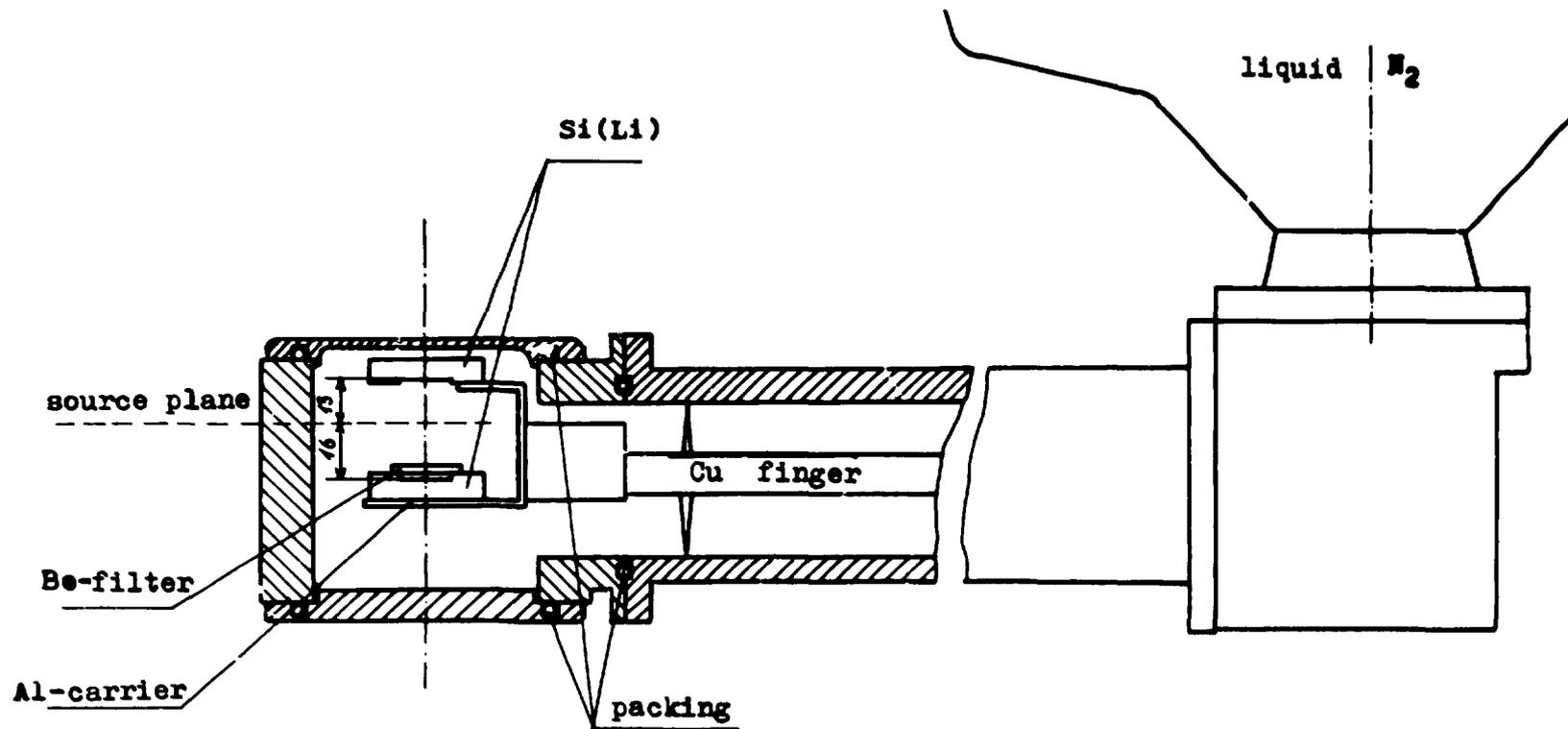
Si(Li)

source plane

Be-filter

Al-carrier

Cu   finger

packing

liquid   N$_2$

Fig. 1 Detector arrangement in the measuring chamber

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Both detectors, however, have been mounted in the cryostat.
The 5mm detector serves as electron detector, the second,
2mm detector, is covered with a Be-filter of a square weight
of 240 mg.cm^{-2} and may be used as an X-detector in coinciden-
ce measurements of nuclear constants.

5. Spectrometer properties

The Si(Li) detector produced by KEVEX has a designation
TR200X5, the area of the input window is 200 mm^2 and the
thickness of the sensitive layer is 5 mm. A charge sensitive
preamplifier CANBERRA CI 1408 C and a standard spectrometric
set ÚVVVR - linear amplifier CI 1417 B and a 4000-channel
analyzer BA 163 with a converter CT 102 (INTERTECHNIQUE), are
connected to the detector.

With the working voltage of +400 V on the detector the
best resolution (FWHM = 2,6 keV) was obtained with Gaussian
pulse shaping and time constant τ = 2 μs.

The detector linearity was verified by the measurement of
the conversion electron peak positions of the radionuclides
^{137m}Ba, ^{207m}Pb and ^{113m}In. An integral non-linearity less than
10^{-3} was found.

The spectra of mono-energetic electrons of different
energies were studied for the purpose of beta spectra analy-
sis. On the basis of the processing of these spectra, the
spectrometer response course was determined. The spectra were
measured by the coincidence method. The second Si(Li) detector
mounted in the cryostat was used as the X-detector. Table 1
presents the results of the determination of the parameter p
for different electron energies.

TABLE 1.

Parameter p measurement results

Source	Electron energy	p
^{99m}Te	119 keV	0,238 ± 0,005
^{113m}In	365 keV	0,263 ± 0,008
^{137m}Ba	624 keV	0,288 ± 0,005

Through the measured points the straight line

$$p = 0.2261 + E \times 0.0001005, \quad E = keV \qquad [1]$$

was interlaced and used for the extrapolation of p values for other energies.

6. Spectrum deformation through backscatter

The usual way of processing the continuous spectra is the construction of a Fermi-Kurie graph, from the curvature of which we can judge on impurities. In the consequence of electron scattering on the detector and its environment, the spectrum is deformed to such an extent that the Fermi-Kurie graphs show always a deviation from the linear dependence which makes itself apparent as excess pulse count rates in the low-energy part of the spectrum. The knowledge of the response of the spectrometer to mono-energetic electrons enables, however, to perform a correction of the continuous spectrum [5,10]. If

$M(E)$ = the measured spectrum

$N(E)$ = the real spectrum

$R(E, E')$ = the spectrometer response to electrons with an energy E, then

$$N(E) = \int_{0}^{E_{max}} N(E').R(E, E'). \, dE' \qquad [2]$$

The desired real spectrum $N(E)$ may be obtained by solution of the integral equation [2] by an iterative method. As a starting approximation of the real spectrum we may consider the measured spectrum, viz. $N_0(E) = M(E)$. As a further approximation we take the expression

$$N_1(E) = N_0(E) - (M_0(E) - M(E)) \qquad [3]$$

where $M_0(E)$ is the calculated spectrum with the use of the integral [2] and the zero approximation $N_0(E)$. The result is substituted in the integral, the next approximation is computed, etc. The convergence of the method is controlled by adjoining of the approximation of the experimental spectrum $M_1(E)$ to $M(E)$. The convergence is fast and 3 to 4 steps are enough to attain the approximation of the real spectrum, re-
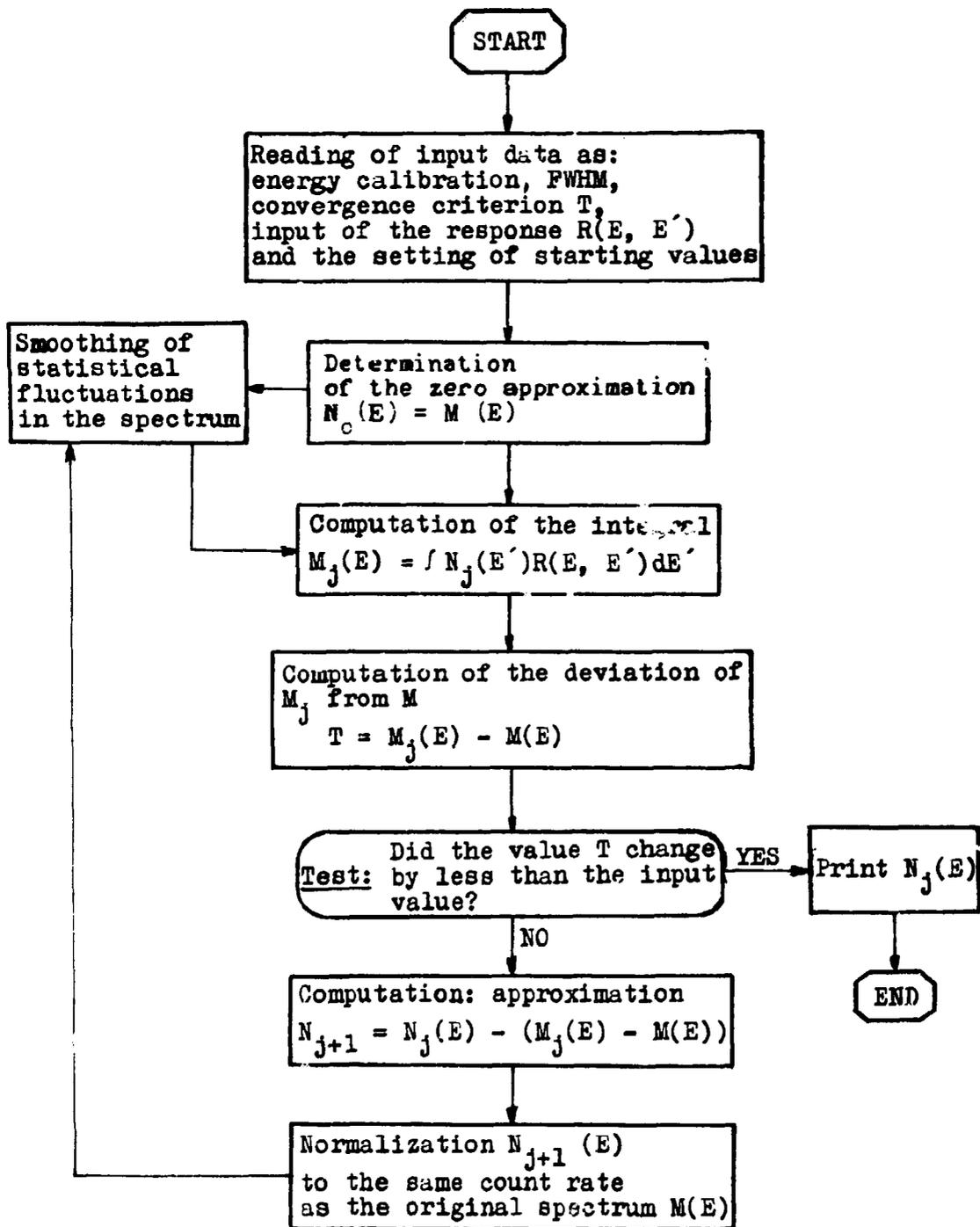
Fig. 2   Flow sheet of the program for the correction
         of the continuous spectrum.

producing the measured spectrum with deviations lower than
1 %.

The correction of spectra is carried out numerically on
the computer VARIAN 620-L with the use of the program, the
flow sheet of which is shown on Fig. 2. The time of the spec-
trum processing depends approximately on the square of the
channel number in the processed spectrum. A spectrum of 100
channels is processed for about 10 minutes, of 300 channels
about 2 hours and of 500 channels (maximum possible number)
7 hours.

The Figs. 3 - 10 show the Fermi-Kurie graphs of the cor-
rected and uncorrected spectra of the radionuclides ^{14}C, ^{147}Pm,
^{45}Ca, ^{32}P, ^{90}Sr, ^{90}Y and ^{60}Co.

It appears that for the nuclide with $E_{\beta max}$ lower than
1 MeV, the corrected Fermi-Kurie graphs are as far linear
that the deviations of the extrapolated values do not exceed
2 %. The drop of pulse count rates for energies lower than
50 to 90 keV is caused by the irreproducible absorption in
the samples. The count rate increase for lower energies in
^{32}P is due to the presence of ^{33}P. The linearity of the graphs
indicates also the correctly determined spectrometer response
$R(E, E')$ to the mono-energetic electrons up to 1 MeV. On the
other hand, the non-linearity of the Fermi-Kurie graphs for
the radionuclides ^{32}P and ^{90}Y is caused by the incorrect ex-
trapolation of the parameter p for higher energies using
equation [1] .

For higher energies, the parameter p rises apparently
faster than linearilly with increasing energy in consequence
of higher energy losses of the electron by bremsstrahlung and
increased electron escape from the marginal regions of the de-
tector.

The correction with a wrongly extrapolated p value leads
to the increase of count rates in the range about 250 to
1000 keV and this in turn to decreased count rates in the
energy range below 250 keV.

The Fermi-Kurie graph of ^{90}Sr, obtained by decomposition
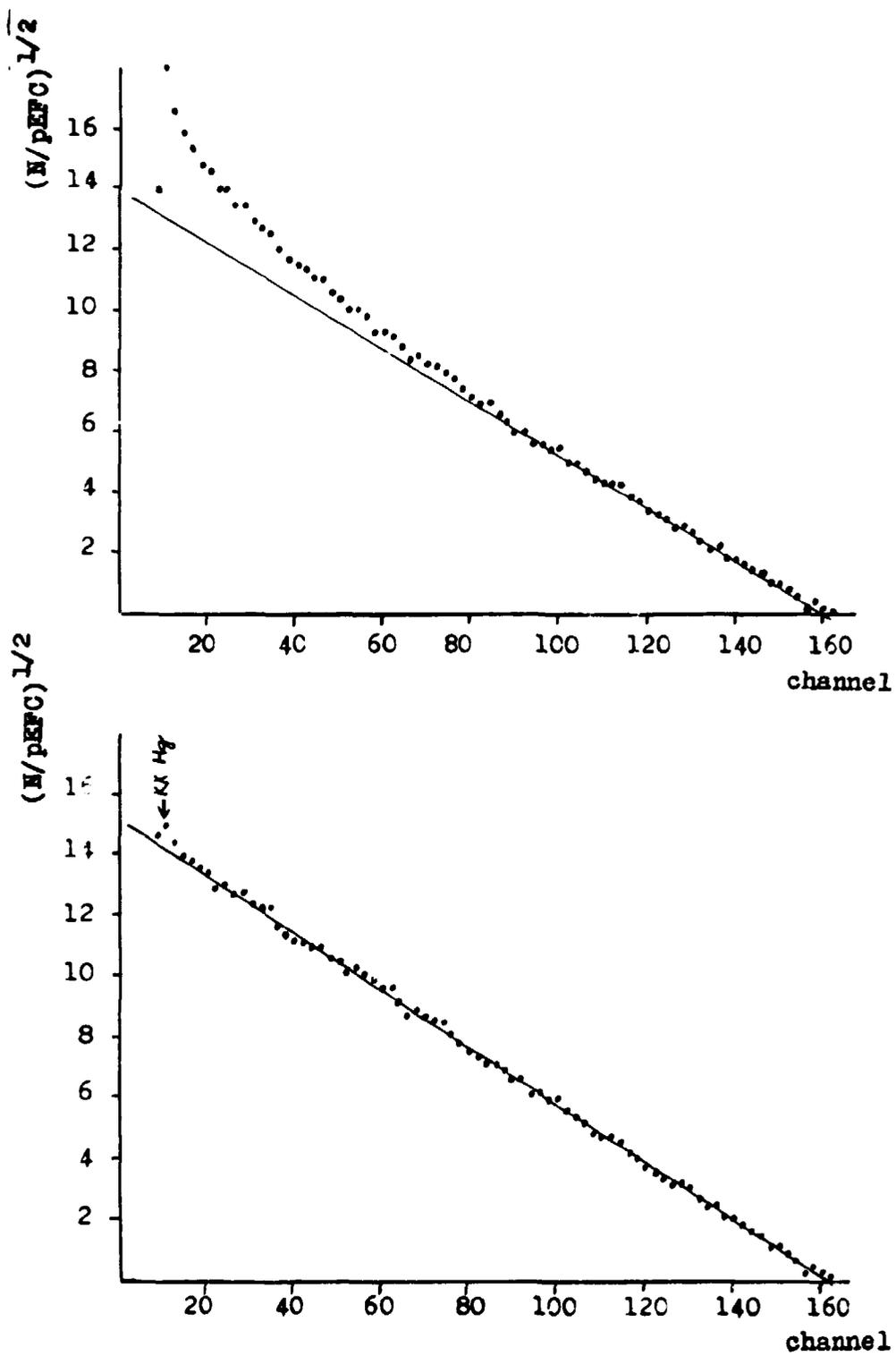of the complex spectrum $^{90}Se+^{90}Y$ is curved in consequence of the

Fig. 3 Fermi-Kurie graph of the ^{204}Tl spectrum
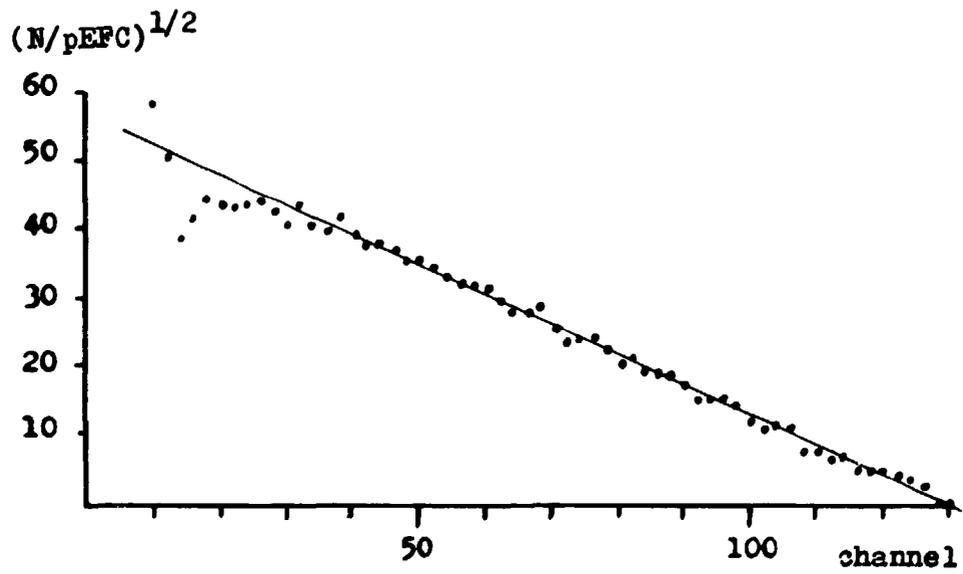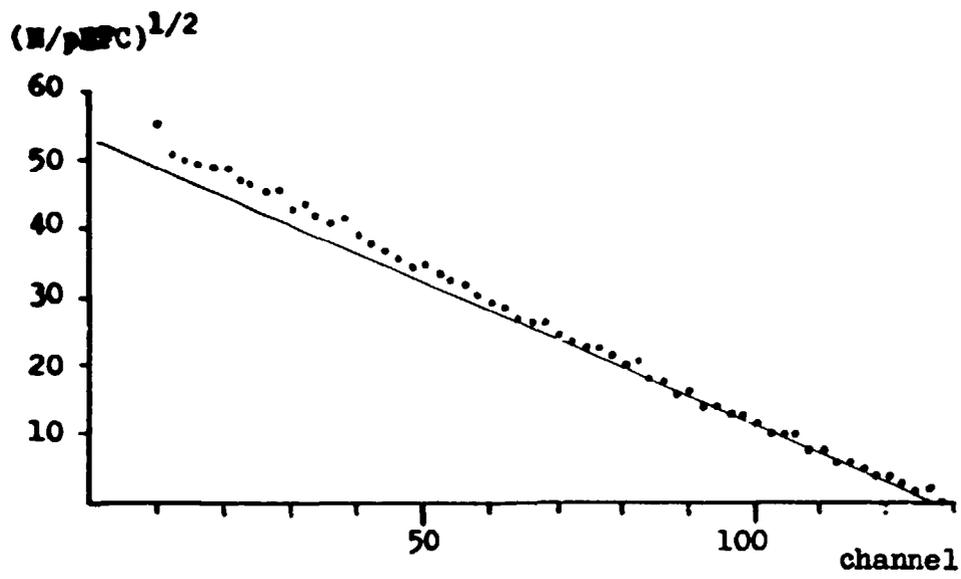uncorrected (top) and corrected.

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Fig. 4 Fermi-Kurie graph of the uncorrected (top) and
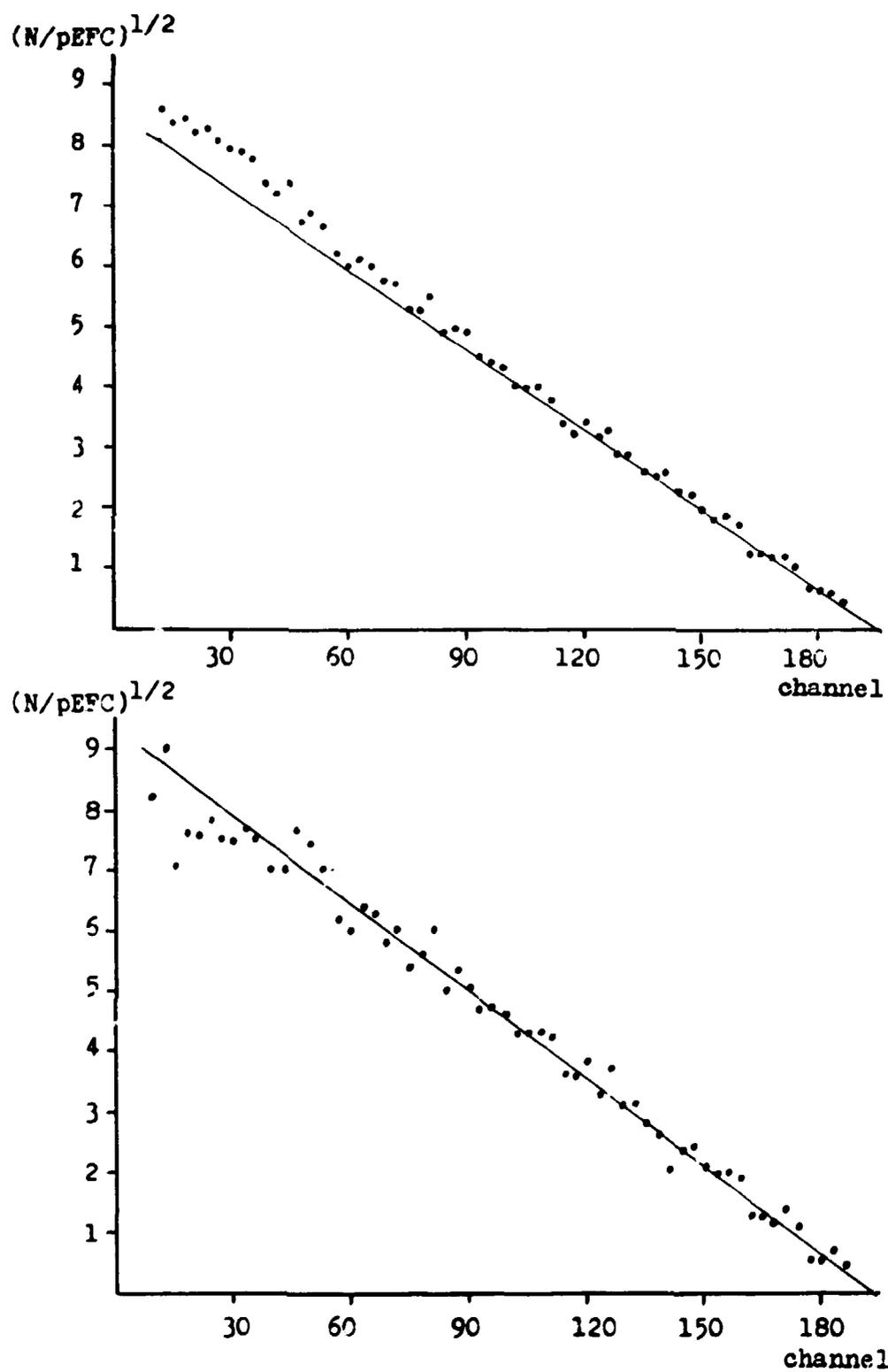corrected (low) ^{14}C spectrum.

Fig. 5 Fermi-Kurie graph of the uncorrected (top)
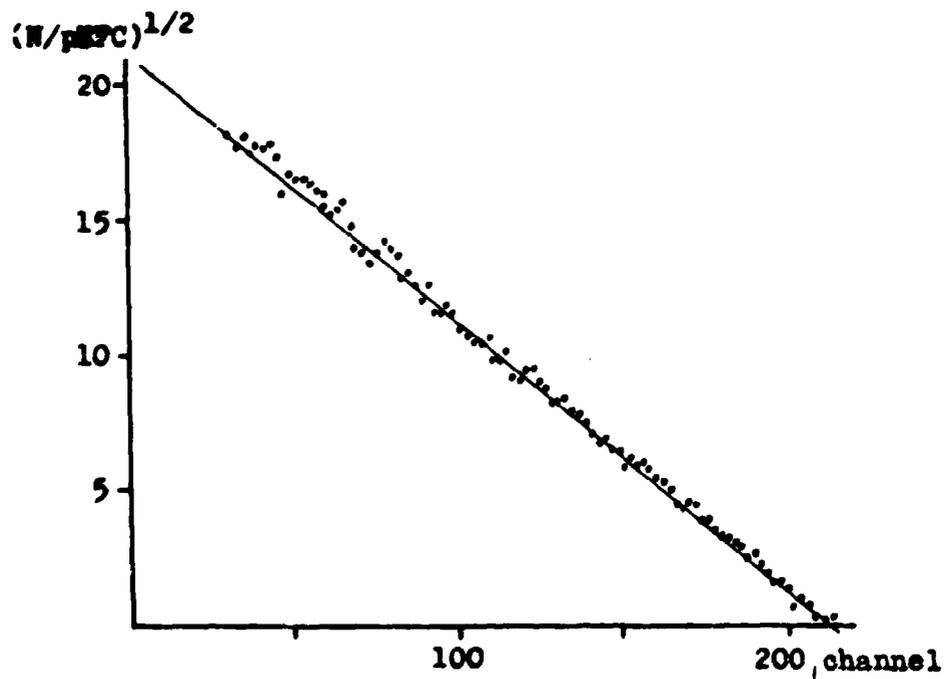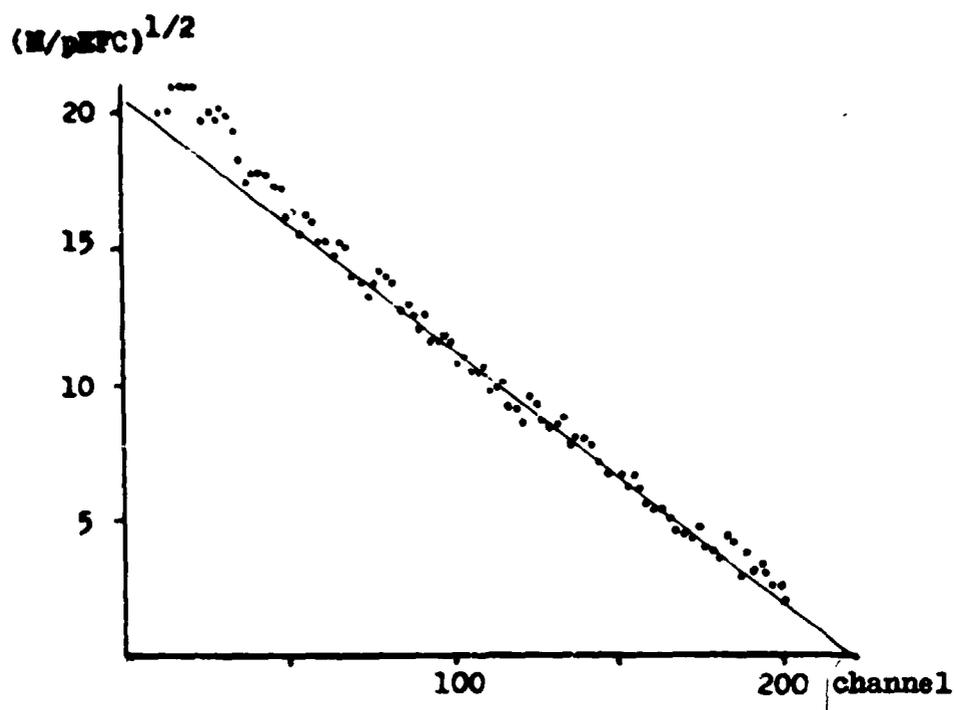and corrected (low) ^{147}Pm spectrum.

Fig. 6 Fermi-Kurie graph of the uncorrected (top)
and corrected (low) ^{45}Ca spectrum.

Fig. 7 Fermi-Kurie graph of the uncorrected (top)
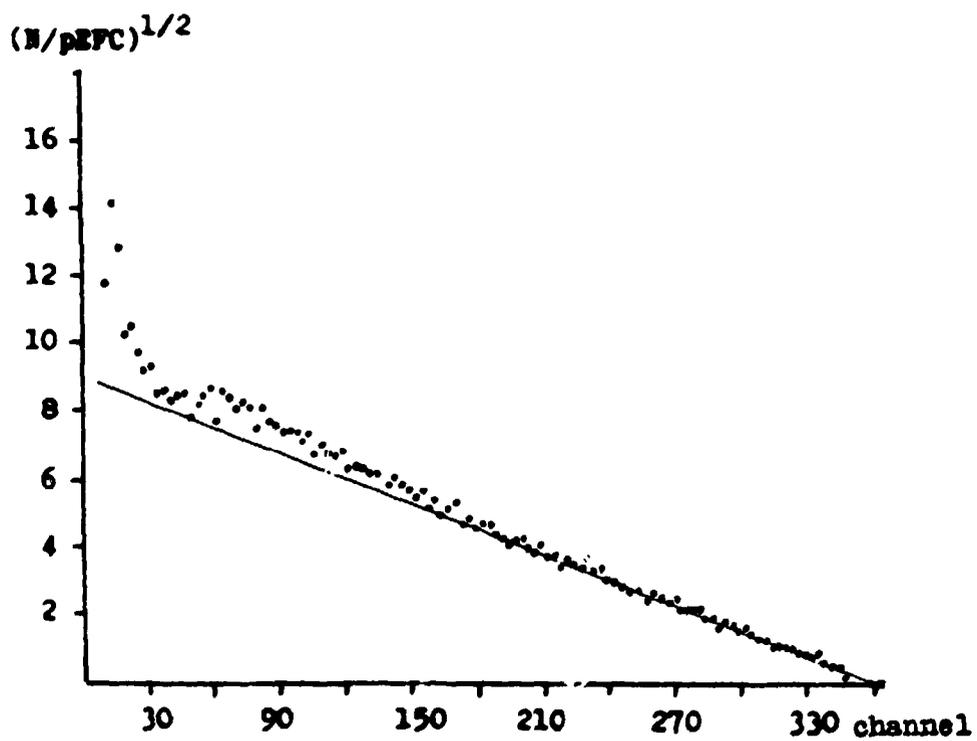and corrected (low) ^{32}P spectrum. (Traces of ^{33}P).

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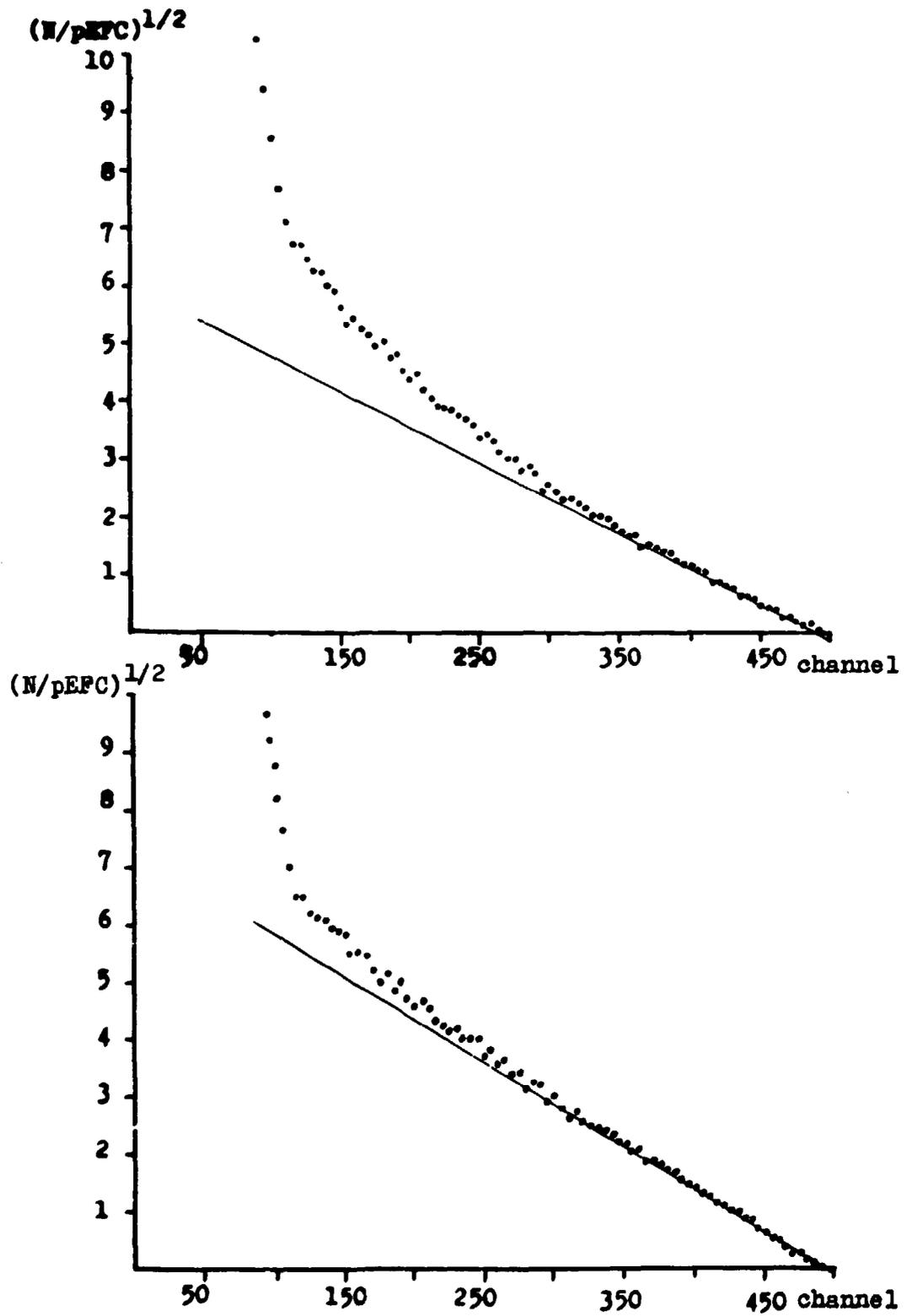
Fig. 8 Fermi-Kurie graph of the uncorrected (top)
and corrected ^{90}Y spectrum (traces of ^{90}Sr).
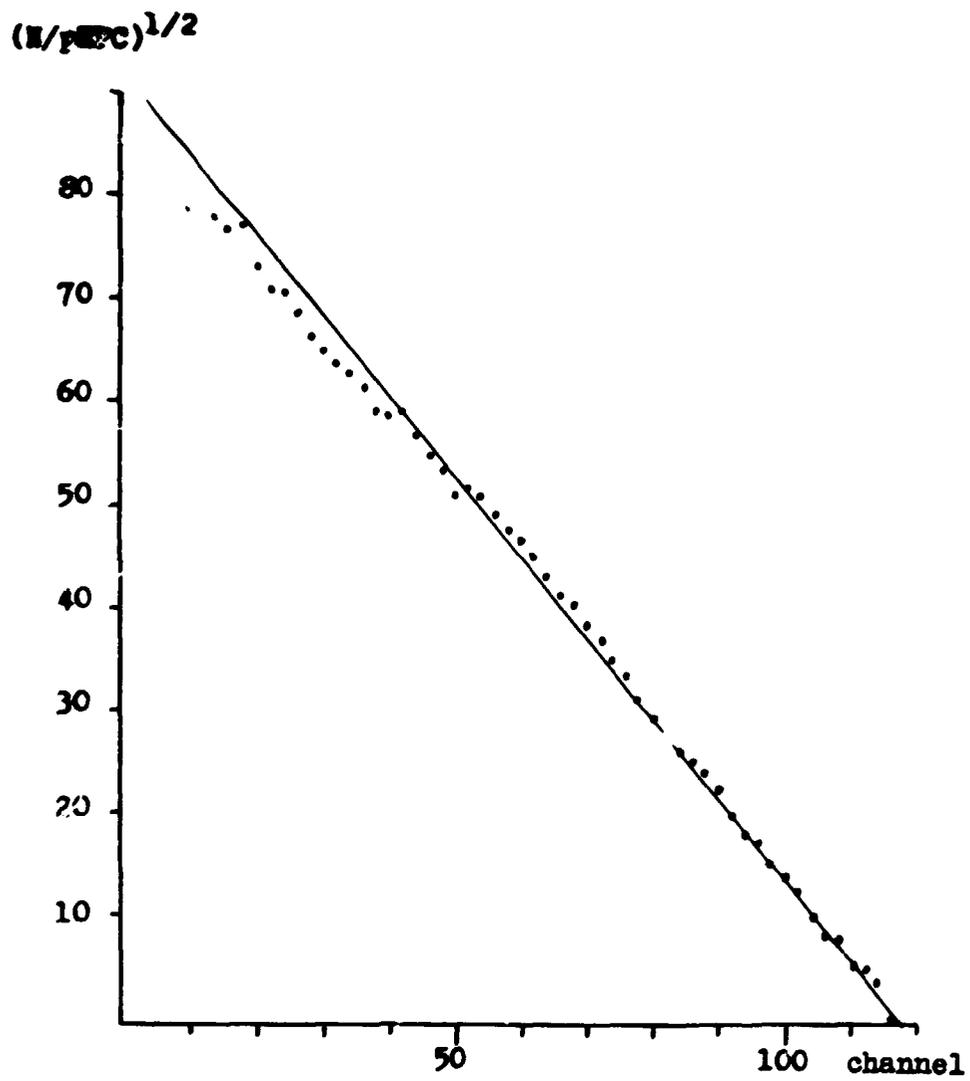
Fig. 9 Fermi-Kurie graph of ^{90}Sr corrected for electron backscatter. The non-linearity of the graph is caused by the non-linearity of the ^{90}Y Fermi-Kurie graph, subtracted from the complex spectrum of ^{90}Sr+^{90}Y.

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Fig. 10 Fermi-Kurie graph of the uncorrected (top) and
corrected spectrum of ^{60}Co. The spectrum is deformed
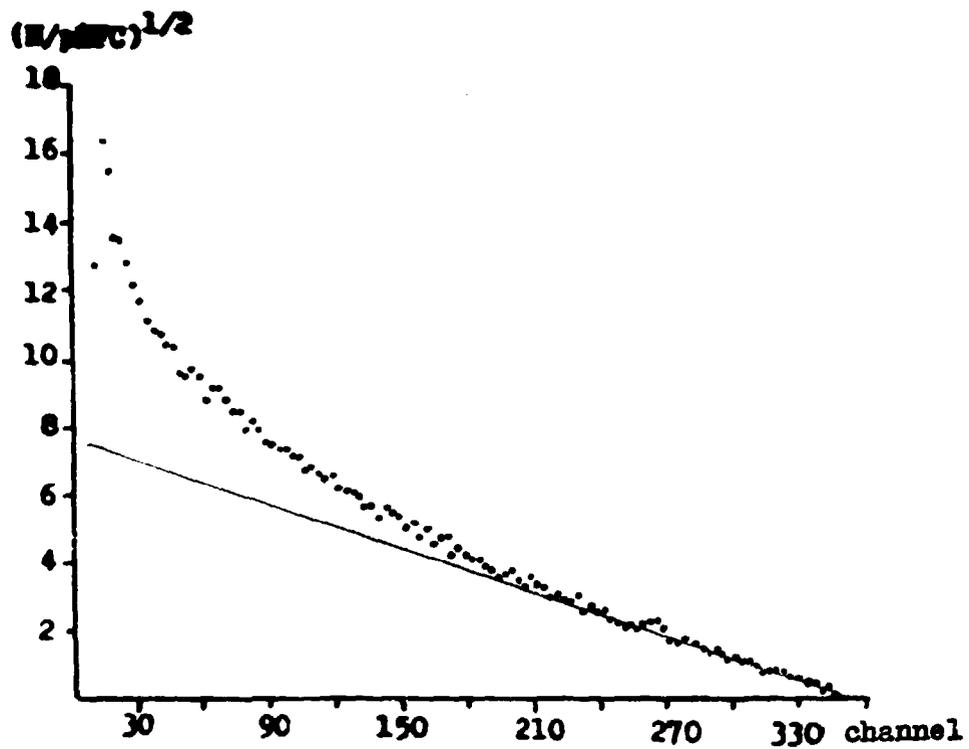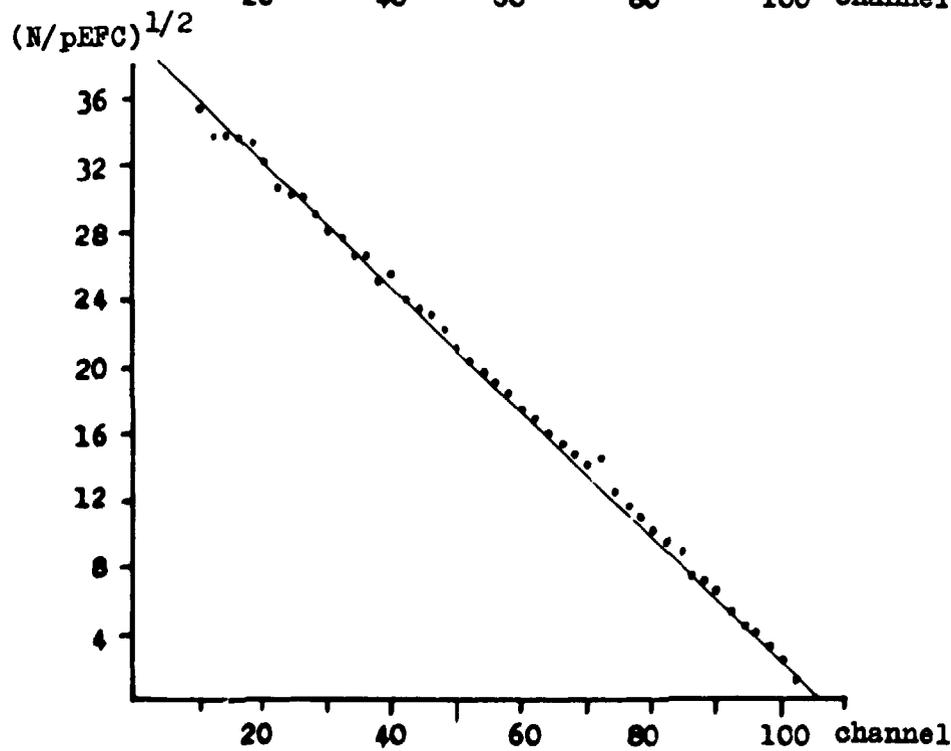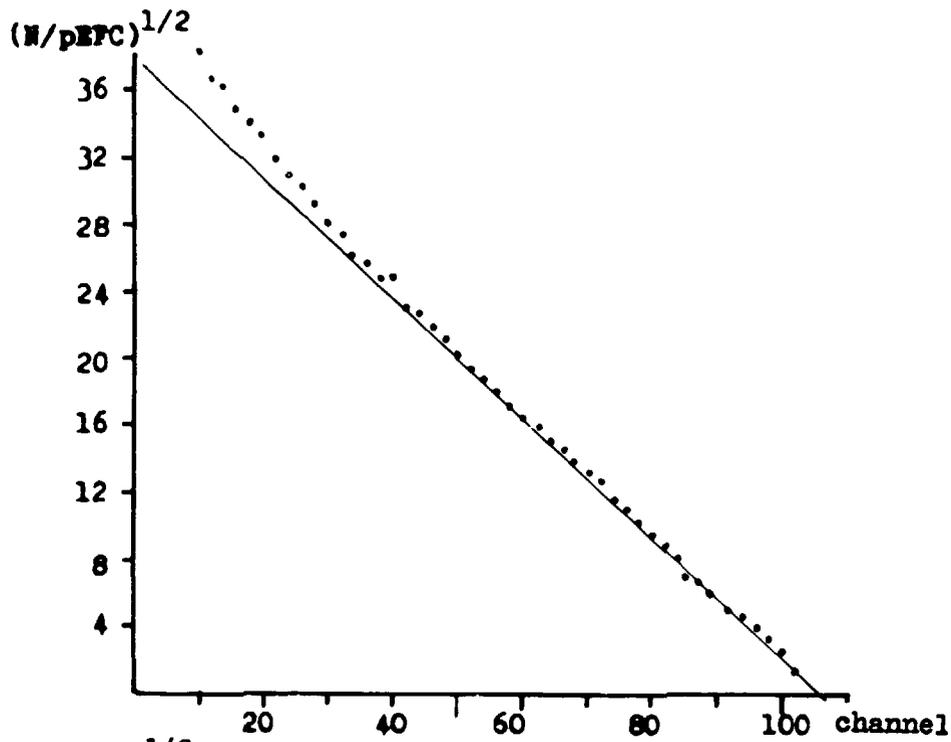both by backscattering and imperfect compensation of
the gamma background by the subtraction of the Al
covered sample spectrum.

curvature of the ^{90}Y Fermi-Kurie graph subtracted from the complex spectrum.

To obtain information from the course of the parameter p for energies greater than 1 MeV experimentally is very difficult, and it will be therefore necessary to carry out in future at least a theoretical estimation.

The obtained Fermi-Kurie graphs may be used for the determination of the maximum energy of the spectrum and for the computation of the total pulse count rate for the given spectrum. The method was checked with the plastic scintillator and was published (1).

On the Table 2 the results of the determination of $E_{\beta max.}$ are presented. For better understanding, the detection efficiency values are given for the individual radionuclides. In all cases, the energy calibration was performed by means of ^{207m}Pb conversion electrons.

The constructed spectrometer is suitable for the purpose of identification of the individual nuclides, as long as $E_{\beta max.}$ of the nuclide is greater than 100 keV. This limitation

TABLE 2.

Determination of $E_{\beta max.}$ and efficiencies for different nuclides.

Nuclide	Tabelated [11-13] $E_{\beta max.}$	Determined $E_{\beta max.}$	Detection efficiency
^{14}C	$157,0 \pm 2$	155	0,070
^{147}Pm	$244,4 \pm 0,5$	230	0,078
^{45}Ca	$252,6 \pm 1$	256	0,100
^{60}Co	$314,3 \pm 4,5$	314	0,102
^{204}Tl	$765,7 \pm 4,5$	760	0,105
^{90}Sr	$544,0 \pm 3$	548	0,099
^{90}Y	$2271,0 \pm 8$	2267	0,102

is given solely by the absorption of electrons in the sample
and will be removed by the introduction of a new method of
sample preparation by electrostatic deposition.

7. Conclusion

The construction of a beta spectrometer with a cooled
Si(Li) detector means a great extension of the experimental
possibilities of the spectrometric laboratory of the ÚVVVR.

The first experience with this spectrometer entitle the
authors to the statement, that in future it will be possible
to control beta impurities in the produced radiation stan-
dards, at least for $E_{\beta max.}$ > 100 keV. The control of nuclides
with lower $E_{\beta max.}$ is for this time limited only by the inade-
quate preparation of samples by dropping. This problem will
be solved by the introduction of a new method of sample pre-
paration with low selfabsorption by the method of electrosta-
tic deposition.

The second problem which still must be followed, is the
equipment of the cryostat with a suitable vacuum unit, based
on the use of the cryosorption and ionic pump.

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PART II. – DETERMINATION OF RADIONUCLIDIC PURITY

1. Introduction

After the solution of the problem of the determination of gamma impurities in basic materials used for the preparation of standards, the study of the determination of impurities of pure beta emitters was started. From the metrological standpoint, the determination of the quantity of impurities is very important, because the errors of the absolute method of activity measurement are usually within the order of one per cent and it is therefore necessary to distinguish and determine even small amounts of impurities to maintain the overall error of standardization.

A good example for the illustration of the importance of impurity determination is the case of ^{32}P which is always accompanied by an admixture of ^{33}P (1 to 12 %, most often about 3 %) and nonetheless this impurity was not controlled as yet, on the certificate of standard solutions the resulting error of activity is given with the value 1 %. This precision is achieved, of course, in the determination of the total activity of $^{32}+^{33}P$, but if we want to warrant the activity of the basic nuclide on the same level of precision, we have to determine very precisely the activity ratio of the two nuclides, too.

2. Effect of impurities on activity measurement

The activity of solutions of ^{35}S, ^{32}P, ^{90}Sr and ^{147}Pm is measured by means of $4\pi\beta$-counter. The pulse count rate N_β is given by the equation

$$N_\beta = N_0\varepsilon_\beta + N_{oi}\varepsilon_{\beta i} = N_0\varepsilon_\beta \left[1 + \frac{N_{oi}}{N_0} \cdot \frac{\varepsilon_{\beta i}}{\varepsilon_\beta} \right] \quad [1]$$

where the quantities denoted with an index i relate to the impurity.

The main source of error in the standardization of pure beta emitters is usually the error in the determination ε_β. The magnitude of this error is indirectly proportional to

$E_{\beta max.}$ and, in addition, depends very strongly on the determination method of ε_β. This error lies within the range of 0,2 to about 2 % and represents the major part of the total standardization error. The total standardization error is hitherto defined as the quadratic sum of the partial errors lying approximately on the same level of significance and includes both random errors and the so called systematic errors, experimentally estimated or determined. The magnitude of the total error, quoted for the individual radionuclides e.g. in the catalog of standards, does in the majority of pure beta emitters not consider the effect of the admixture of another pure beta emitter. Only in some unique cases, where the presence of such an impurity was logical and inevitable, a certain reserve in precision was created, based usually on a rough estimate leaning e.g. on the approximate knowledge of the production technology of radioactive preparations.

The many years experience has shown that the improvement of the metrological parameters of radioactivity standards in this respect may be achieved only through a consistent control of the parameters of the processed materials and achievement of a complete independence on the producer's data.

If we now want to quote (in addition to activity data) also quantitative data on the nuclidic compositions of the standard and maintain the contemporaneous precision level, it is necessary to ensure a high precision of the determination of impurities.

As follows from equation [1] , the correction for impurities is expressed by the term $[1+(\varepsilon_{\beta 1}/\varepsilon_\beta).(N_{o1}/N_o)]$. The determination error of this correction must have such a value which practically does not affect the quadratic sum, i.e. it must be at least three times smaller than the total standardization error. As the precision of the ratio $\varepsilon_{\beta 1}/\varepsilon_\beta$ may be high, the total error of this correction is given by the error in the determination of the ratio of the individual activities N_{o1}/N_o. This requirement is rather severe for a method using

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the beta spectrometry and as far as it can not be fulfiled insome concrete cases, it will be necessary to achieve the reduction of the partial errors of standardization e.g. by the use of some more precise methods for the determination of ε_β .

In Table 1 the total standardization errors are summarized for ^{35}S, ^{32}P, ^{90}Sr and ^{147}Pm, as well as the impurities typical for the individual nuclides and the necessary precision of the ratio N_{oi}/N_1 for attaining the required standardization precision.

TABLE 1.

Potential impurities in standard solutions.

Nuclide	Required precision of activity determination	Potential impurities	The necessary reached determination precision of the ratio N_{oi}/N_o for ensuring the required precision
	[%]		[%]
^{35}S	1,5	^{32}P	0,5
^{32}P	1	^{33}P	0,3
$^{90}Sr+^{90}Y$	1	^{147}Pm	0,3
^{147}Pm	2,5	$^{90}Sr+^{90}Y$	0,7
^{90}Y	1	^{90}Sr	0,3
^{89}Sr	1,8	$^{90}Sr+^{90}Y$	0,6

3. Experimental determination of impurities

By means of the new constructed beta spectrometer it is possible to carry out the analysis of beta spectra corrected for the electron backscatter (see the description in Part I. of this report), using the technique of the Fermi-Kurie graphs [3]. At this time this method can be applied only to nuclides with $E_{\beta max.}$ greater than cca 100 keV. For lower energies, the beta spectrum is considerably distorted by self-absorption in the sample.

Relatively easy is the case of determination of a nuclide with $E_{\beta max.}$ greater than is the $E_{\beta max.}$ of the basic material. Such a case is the determination of the quantity of $^{90}Sr+^{90}Y$ in ^{147}Pm or $^{90}Sr+^{90}Y$ in ^{89}Y. The determination error is substantially given by background fluctuations in the energy window where the detected impurity is found. In the case of $^{90}Sr+^{90}Y$ in ^{147}Pm the ^{90}Y is detected in the window of about 500 keV to 2300 keV, where the fluctuations of the background during the measurement time of 500 s do not exceed 0,01 % of the ^{147}Pm pulse count rate. The construction of the Fermi-Curie graph permits to determine more precisely the maximum energy and with sufficient precision the contaminating nuclide.

More difficult is the reversed case, where in the basic material a nuclide with a lower $E_{\beta max.}$ is present. A typical example is ^{33}P which is always present in ^{32}P. In such cases it is necessary to carry out the resolution of the spectrum in two components by means of the Fermi-Kurie graph and to determine the end point and the activites corresponding to the two components. The result will be burdened with both statistical fluctuations in the spectrum and the systematic deviation of the Fermi-Kurie graph from the straight, due to the imperfect spectrum correction for backscatter. The procedure was tested in models e.g. on the example of Pm determination in Tl. Table 2 shows the results for samples containing varying amounts of ^{147}Pm.

TABLE 2.

Determination of the ^{147}Pm content in ^{204}Tl.

Sample	Real content of ^{147}Pm	Determined content of ^{147}Pm
	[%]	[%]
1	0	1,8
2	1	2,5
3	5	6,4

The results of ^{147}Pm determination are evidently burdened
with a systematic error of about 2 % due to the non-linearity
of graphs. Similar systematic errors were found in the pro-
cessing of pure spectra. For ^{90}Sr, the content of ^{147}Pm was
determined with the result -2,1 % and for ^{60}Co the content
of ^{14}C was determined with the result +1,7 %. In the impurity
determination of similar combinations by the direct method,
no significantly better determination accuracy may be ex-
pected.

From Table 1, where the required precisions of impurity
determination are listed, it is evident that the achieved
precision is not sufficient in the cases of ^{32}P and ^{90}Sr+^{90}Y.
In these cases another method has to be used which is based
on the comparison of the spectrum of the investigated material
with the spectrum of the pure radionuclide. The spectra are
used to construct a differential spectrum which is then fur-
ther processed by means of the Fermi-Kurie graph. This pro-
cedure enables to reveal a great number of impurities present
in concentrations of 0,2 % and more with a precision of the
same order, depending mainly on statistical fluctuations.

The method was tested again on the example of ^{204}Tl
contaminated with the nuclide ^{147}Pm. In a sample containing
really 1 % of ^{147}Pm the determined value was (1,2 $\pm$ 0,6) %
and in a sample containing really 5 % of ^{147}Pm the determined
value was (4,6 $\pm$ 0,6) %. In a sample of ^{90}Sr+^{90}Y containing
1 % of ^{147}Pm the value of (0,9 $\pm$ 0,5) % was found by this
method for measuring times of 500 s.

A necessary condition of the method is the use of pure
material which is not always available. This difficulty can
be avoided by continuous elaboration of a spectra catalog of
the processed radionuclides. The comparative method only is
successful for nuclides emitting complex beta spectra and in-
volving also gamma photons, where the complexity and deforma-
tion by gamma background makes the analysis by means of Fer-
mi-Kurie graphs impossible. In these cases the preparation
of a spectra catalog is indispensable.

The comparative method was successfull/ checked also in

determinations of the ^{3}H in ^{14}C (and vice versa). In this case, however, instead of the semiconductor spectrometer a spectrometer with a liquid scintillator (Packard-TRI-CARB) commonly used for routine activity measurements of labelled compounds. By comparison of the sample spectrum with the spectrum of the pure standard a tritium concentration of more than 0,3 % may be readily detected. The sensitivity of the ^{14}C content determination in tritiated compounds is naturally higher - the detection limit is about 0,01 % .

4. Conclusion

The introduction of beta spectrometry in the dosimetry department of the ÚVVVR enabled to control the contamination of radionuclides with pure beta emitters. The purity control is easy in the cases of ^{35}S and ^{147}Pm, where impurities of radionuclides are expected which have a higher maximum energy of the beta spectrum than the basic material. In these radionuclides the maximum achieved precision of the impurity determination is ± 0,1 % and is sufficient to ensure the required standardization precision [4]. The determination method in these cases is simple and approximately equally laborious as in determinations of gamma impurities with the use of a Ge(Li) spectrometer. On the other hand in the case of ^{32}P and ^{90}Sr+^{90}Y it is necessary to use a more elaborate method of the comparison of the spectrum investigated with the spectrum of the pure standard. The precision attainable by this method is in the order of 0,2 to 0,3 % and is satisfactory, too.

The substatial advantage of both methods is that they do not require the use of chemical separation methods which has been formerly the only possible method how to determine impurities of pure beta emitters [2].

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