Study of a non-destructive measurement method for highly enriched
U-Al alloy plate fuel

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CONTENT

1. Introduction

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## 1. Introduction

In the fabrication process of nuclear fuels, a simple, rapid and nondestructive method of uranium assay is desirable and useful. This report relates to nondestructive assay method for determining the amount of U-235 in the highly enriched U-Al alloy plate fuel. The method is to utilize the 185 KeV gamma ray from the decay of U-235. The standard uranium foil with uniform thickness, the U-235 content of which is evident and in which self-absorption can be disregarded, is fabricated by means of electrodeposition for th use in the assay. The intensities of gamma ray from U-235 in highly enriched uranium fuel plate is determined by applying self-attenuation correction in the use of the standard uranium foil, then the U-235 content in the plate is evaluated.

The uranium segregation in the fuel plate and the geometry of the measuring system are also examined. The estimation method is enough accurative for practical use.

## 2. Description of

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## 2. Description of U-Al alloy plate fuel

Highly enriched U-Al alloy plate fuel is used in the Material
Testing Reactor. The uranium in the fuel is disposed in the
parallel aluminium plates (fuel plate), about 30 mg of U-235 per
$cm^2$ and its enrichment is $90 \sim 93\%$. Each plate is composed of
0.5 mm U-Al alloy as the "meat" and aluminium plate "cover" in
the 0.4 mm thickness on either side. The plate has the following
dimensions: length about 700 mm, width about 70 mm and thickness
about 1.2 mm.

The U-235 content ratio in the U-Al alloy is about 20%. Some
plates are assembled into a "fuel element": the several mm space
between the plates in parallel is filled with the cooling and
moderating water.

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3. Principle of the method

3-1. The gamma ray for the detection

The method of determining the amount of U-235 based on gamma ray spectrometry is necessary for the gamma ray characteristic of U-235 with ample intensities for detection by NaI(Tl) detector.

The most useful gamma ray relating to U-235 has an energy of 185.7 KeV and an intensity of $4.3 \times 10^4$ $\gamma$/g-sec.

The gamma ray spectrum emitted by uranium is complex. There are many reports with respect to the gamma ray spectrum of uranium isotopes.

According to the reference[1], in the gamma ray scintillation spectrum with U-234, the maximum peak is found at 53 KeV and there are small peaks at approximately 95 KeV and 118 KeV. In the U-236 spectrum the primary photo-peak is at 49 KeV and with other peaks at 87 KeV. In the natural uranium spectrum, two peaks are prevalent at 96 KeV and 185 KeV. The peak at 185 KeV is due to U-235 in the sample.

Fig. 1 is a comparision of spectra of natural, 54% enriched, 72% enriched and 90% enriched uranium. It is easily clear from this figure that the intensities of the photo-peak are in proportion to an uranium-235 enrichment in the sample. Detecting the gamma ray from the uranium sample by means of a NaI(Tl) scintillation spectrometer, the photo-peaks are found in three regions at approximately 185 KeV, 94 KeV and 50 KeV.

The 185 KeV gamma rays result from the decay of U-235. The gamma rays in the 90 KeV region are due to the 94 KeV with

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the decay of U-235 and the 93 KeV with Th-234, the daughter products of U-238. The 50 KeV peak results from the decay of U-238.

Using a pulse height analyzer, it is possible to select the desired gamma rays emitted from the sample within the desired energy range and determine the counting rate of it.

In this study, the 185 KeV gamma ray from the decay of U-235 is detected to determine the U-235 content. It is clear from the spectra that there are no interfering gamma rays in this energy region from the decay of the other uranium isotopes or their daughter products.

3-2. Theory

The method in the measurement of the U-235 content by the gamma ray spectroscopy is based on the fact that the amount of U-235 is proportion to the number of gamma rays with the decay of it on the principle of the radioactive disintegration. The equation for radioactive disintegration is written as:

$$n = \frac{1}{\lambda} \cdot \left(-\frac{dn}{dt}\right)$$

$$= \frac{T}{0.693} \cdot \left(-\frac{dn}{dt}\right) \quad \cdots\cdots\cdots\cdots\cdots\cdots (1)$$

in which $n$ is the number of the U-235 nuclide, $\lambda$ is the disintegration constant of the U-235, $t$ is the time and $T$ is the half life of the U-235 which is $7.1 \times 10^8$ years. Therefore, $\left(-\frac{dn}{dt}\right)$ denotes the disintegration rate with which the gamma rays emmit.

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The amount of U-235 could be evaluated from this equation on the measurement of the disintegration rate. The relation between the disintegration rate and the absolute intensity of 185 KeV gamma ray from U-235 is the constant, which is given as 55 per hundred disintegrations. Namely, the specific activity of the 185 KeV gamma ray from the U-235 is $4.3 \times 10^4$ $\gamma$/g-sec.

In determining the disintegration rate by detecting the gamma rays with a scintillation spectrometer; it is necessary to know the factors which depend on the geometry of the measuring system, the detection efficiency for 185 KeV gamma rays, the 185 KeV self-absorption of the measured material and the specific activity of the 185 KeV gamma rays. It is difficult to determine all of the above factors, however, the evaluation of the amount of U-235 is possible with the appropriate detection system and by applying self-absorption correction.

The reason is that the other factors without a self-absorption factor are deemed as constant with the same detection system and cancel out of the evaluation. For the purpose to determine self-absorption correction factor, the measurement is made by using the standard uranium foil which has no self-absorption or can be ignore as permissible errors in the evaluation. The absorption factor for the unknown U-Al alloy plate is calculated from the following equation.

$$e^{-\mu t} = \frac{C-I}{I-B}$$

$$I_0 = I -$$

where

$\mu$ : the
$t$ : the
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$I$ : the
$B$ : the
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$$R_0 = \frac{1}{1}$$

where
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$$e^{-\mu t} = \frac{C-R}{I-B} \qquad \dots\dots\dots\dots\dots\dots (2)$$

$$I_0 = I - B \qquad \dots\dots\dots\dots\dots\dots (3)$$

where

$\mu$ : the absorption coefficient of U-Al alloy plate

t : the thickness of the plate

C : the counting rate of the plate on the standard foil

R : the counting rate of the plate

I : the counting rate of the standard uranium foil

B : the counting rate of the background

$I_0$ : the net counting rate of the standard foil without the self-absorption correction

$\mu t$ is obtained by using the table of the exponentials, because the values on the right side of the equation (2) is known in the measurement. The real counting rate of U-Al alloy plate, $R_0$, is calculated by applying self-absorption correction as:

$$R_0 = \frac{\mu t}{1 - e^{-\mu t}} \cdot R' \qquad \dots\dots\dots\dots\dots (4)$$

where

R' : the net counting rate of U-Al alloy plate

The above equation is get by considering the self-absorption. The ratio of the real counting rate of the standard foil and the U-Al alloy plate is equal to the ratio of the amount of U-235 in them on the constant area under the collimater. This ratio give us to determine the

amount of U-235 in the U-Al alloy plate.

$$W(u) = \frac{A(u)}{A(f)} \cdot \frac{R_0}{I'_0} \cdot W(f) \quad \dots\dots\dots\dots\dots\dots (5)$$

where

$W(u)$ : the U-235 weight of the U-Al alloy plate

$A(u)$ : the area of the plate

$A(f)$ : the area of the standard foil

$I'_0$ : the real counting rate of the foil

$W(f)$ : the U-235 weight of the foil

4. Standard uranium foi

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4. Standard uranium foil

4-1. Preparation of the standard uranium foil

For the preparation of the standard uranium foil which has
no or less self-absorption for the 185 KeV gamma ray, we
experimented to make the foil by means of the vacuum evaporation
method, powder rolling method and electrodepositing method.
It is found that the electrodepositing method is the most
suitable, therefore, we describe the way to make the foil by
this method as follows.

a) Equipment

The equipment of electrodeposition is shown in the Fig. 2.
The equipment was designed to electrodeposit the foil on
only one surface.

The cell of electrodeposition is placed in a water bath,
the temperature of the bath being kept constant by an
electric hot plate.

Electrical contact is made directly the foil to the
cathod and the platinum wire through the motor to the
anode.

b) Electrodepositing condition

The electrolyte was prepared in three kinds.
First consists of 500 ml of alkali salt carbonate
$(0.15M \ K_2CO_3 + 0.15M \ KOH)$ and 1 g uranyl nitrate,
second consists of 500 ml of fluoride $(0.1M \ LiF)$ and
1 g uranyl nitrate, and last one consists of 500 ml
of oxalate $(0.4M \ NH_4C_2O_3)$ and 1 g uranyl nitrate.
The electrodeposition was studied on the various cathode
(backing foil) at the temperature of 60°C and the

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current density 50 mA/cm$^2$.  It is found on the result
from this experiment that the combination of tin foil
and the electrolytic consisting of the oxalate is
disirable.

The electrodepositing conditions by the oxalate electrolyte
are described as follows:

i)  Influence of Electrodepositing Temperature

The result of the experiment is shown in Fig.3, from
which we found that the electrodepositing rate is low
at the temperature less than 60°C or more than 80°C.
As evaporation of the electrolyte at the temperature
more than 80°C is substantial, 70°C is considered
to be an optimum temperature in practical use.

ii)  Influence of the Ammonium Oxalate Concentration

The result of the experiment is shown in Fig. 4.  The
less is the ammonium oxalate concentration, the
higher is the rate of electrodeposition, however,
the electrodepositing surface is more easy to peel
off on the roughness.  At the concentrate more than
0.2N, the rate of electrodeposition is almost
constant, and the higher is the concentration, the
harder is peeled off the deposition (the 0.4N
ammonium oxalate is saturated solution at the room
temperature).

iii)  Influence of the Current Density

The result of experiment is shown in Fig. 5.  The

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electrodepositing rate is in proportion to the
current density in the experimental range. The
higher is the current density, the rougher becomes
the surface.

We assume that the optimum current density is $100 \sim$
$150 \ ^{mA}/cm^2$.

iv) Influence of the Uranium Nitrate Concentration

The result of experiment is shown in Fig. 6. In case
that the concentration is more than $4g/l$, black precipitation
of the uranium oxides is produced and the electrodepositing
surface becomes rougher with the decrease of electrode-
positing rate.

v) Electrodepositing Material on the foil

The chemical form of the electrodepositing material
on the foil is found to be $U_3O_8$ by the method of
oxidation.

4-2. The property of the standard uranium foil

a) Determination of electrodeposited uranium content

The quantity of the uranium on the standard foil was
determined to be calculated as $U_3O_8$ from the increase
in the weight of the foil on the electrodeposition.
In addition, the quantity of the uranium was confirmed
by means of the chemical analysis measurement of the
other foils under the same electrodepositing condition.
The amount of the uranium on the standard foil is

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19.73 mg/cm$^2$ as $U_3O_8$ or 15.007 mg/cm$^2$ as U-235.

b) Self-absorption

The self-absorption of the 185 KeV gamma ray in the
standard foil is calculated to be 0.1% by the self-
absorption equation: mass absorption coefficient
$\mu = 1.50$ cm$^2$/g. Therefore, it is unnecessary to apply
self-absorption correction in the foil.·
The relation between the quantity of uranium and the
counting rate from it is measured on several foils on
the use of the counting system shown in the next
paragraph.
The result of measurements is shown in Fig. 7.
In the range less than 20 mg/cm$^2$, self-absorption can
be disregarded as permissible errors in the measurement.
Example of actually measured value is shown as follows:

| Uranium (mg/cm$^2$) | Counting rate (ct/500 sec/mg) |
|---|---|
| 8.55 | 504.1 ± 22.5 |
| 12.60 | 506.9 ± 22.5 |
| 19.73 | 507.1 ± 22.5 |

5. Measuring syst
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### 5. Measuring system

The measurement diagram of gamma ray spectrometry is shown in
Fig. 8. The gamma ray is detected, through the defined collimeter,
by means of a $3"\emptyset \times 3"$ NaI(Tl) scintillation detector.
Gamma spectra are obtained by amplifying the detector output pulses
with a linear amplifier and then feeding the amplified pulses to
200-channel pulse height analyzer.
The spectrum is visible on the oscilloscope screen as it builds up,
and the data are printed out on a digital printer and simultaneously
plotted on a strip-chart recorder.
Then, the counting rate is obtained on the use of single channel
pulse height analyzer and scaler.

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## 6. Result

### 6-1. Quantitative analysis of U-235

i) Relation of counting rate and actual content of U-235

By measuring the foils with the same uranium content
in various enrichments, it is found that the counting
rate of the 185 KeV gamma ray are in proportion with
the U-235 content in the foil. (Fig. 9) The 185 KeV
gamma rays were counted through a single channel pulse
height analyzer with the window on the 184 KeV peak and
a window width of about 10 KeV.

The counting rate of the 185 KeV was corrected on
account of the contribution of U-238 and others.

ii) Quantitative Analysis of U-235 in U-Al Alloy Plate

The following four measurements were made:

Counting rate of the foil ................I

Counting rate of the U-Al alloy plate

put on the foil ..........................C

Counting rate of the U-Al alloy plate.....R

Counting rate of Background ..............B

In addition to the above, the following data are necessary:

Weight of U-235 contained in the

standard foil ...........................W(f)

Area of the foil ........................A(f)

Area of the U-Al Alloy plate ............A(u)

Correct factor to self-absorption

of the foil .............................$\alpha$

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Further, it is necessary to take care to decrease
the non-counting errors due to the dead time of the
instrument and the statistical errors of event.
The method is as follows.

(1)  $I-B = I_0$

(2)  $C-B = C (u + f)$

(3)  $R-B = C(u)$

(4)  From (1) formula, $I_0' = I_0 \cdot \alpha$

(5)  $\dfrac{C(u + f) - C(u)}{I_0} = e^{-\mu t}$  (the formula (2), (3) )

(6)  $\mu t$ is obtained from the mathematic table.

(7)  $\mu t / 1-e^{-\mu t}$ is obtained.
     This is absorption coefficient of U-Al alloy.

(8)  $C(u) \cdot \dfrac{\mu t}{1-e^{-\mu t}} = R_0$  (Please refer to the formula (4) )
     This is correction of self-absorption of U-Al alloy.

(9)  $\dfrac{R_0}{I_0}$ is obtained.  This is equal to the U-235
     content proportion to the same measured area of the
     foil and U-Al alloy.

(10)  $\dfrac{W(f)}{A(f)}$ is obtained.  This is the U-235 content in
     a unit area of the standard uranium foil.

(11)  From the formula (5), the content of U-235 in the U-Al
     plate can be obtained.

$$W(u) = A(u) \cdot \frac{W(f)}{A(f)} \cdot \frac{R_0}{I_0'}$$

In the above method, the standard uranium foil and uranium
U-Al alloy are measured in equal areas.  Therefore, the
area to be seen by the scintilation crystal through slit
must be less than the area of either the standard uranium
foil or U-Al alloy plate.

6-2.  Control of U.
    U-235 conten
    alloy plate.  A
        $B = 4$ ct,
        $C = 3,96$

Therefore,

        $e^{-\mu t} = ($

        $\mu t / 1-e^{-\mu}$

        $R_0 = 6,0$

        $\dfrac{R_0}{I_0'} = 19$

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## 6-2. Control of U-235 in the Process

U-235 content is estimated by measuring a part of the U-Al alloy plate. An example is shown as follows:

$$B = 4\ {}^{ct}/2\ \text{minutes}, \qquad I = 309\ {}^{ct}/2\ \text{minutes},$$
$$C = 3,963\ {}^{ct}/2\ \text{minutes} \qquad R = 3,851\ {}^{ct}/2\ \text{minutes}$$

Therefore,

$$e^{-\mu t} = 0.3672 \qquad \therefore\ \mu t = 0.995$$

$$\mu t / 1 - e^{-\mu t} = 1.5724$$

$$R_0 = 6,049$$

$$\frac{R_0}{I_0'} = 19.833 \qquad \therefore\ W(u) = 15.35\ (g) \pm 0.24\ (g)$$

The above value is close to the value 15.41 g $\pm$ 0.05 g obtained by the conventional density measuring method, and also to the value 15.38 g obtained from the chemical analysis and enrichment of uranium.

In another case, the measured value by this method was 15.59 g which was differed from the value by the conventional density measuring method, 13.79 g $\pm$ 0.25 g.

The reason of this difference was studied and found that the test piece had the segregation of uranium in the material.

## 6-3. Measurement of the Uranium Content, Enrichment and Segregation

As mentioned above, absorption coefficient is obtained from the following equation:

$$\frac{C(u + f) - C(u)}{I_0} = e^{-\mu t}$$

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By measuring the intensity of 184 KeV $\gamma$-ray emitted from the foil and absorbed in the U-Al alloy plate, $e^{-\mu t}$ of the U-Al alloy plate can be obtained. $e^{-\mu t}$ is in proportion to the uranium content in the test pieces and, according to the result of our various experiments, the absorption coefficient relates only to the uranium in U-Al alloy plate. Therefore, total uranium content can be obtained by measuring the absorption coefficient.

Fig. 10 shows the relation of the uranium content and absorption coefficient measured. This coincides with the theoretically calculated value of the uranium metal.

Measurement was made by using the 184 KeV $\gamma$-ray emitted from the enriched uranium foil.

Mass absorption coefficient obtained from the experiment:

$$\mu = 1.42 \ cm^2/g$$

If the U-235 content contained in the foil is known, the U-235 content in the U-Al alloy plate can be obtained by the following equation, simultaneously with the measurement of the mass absorption coefficient.

$$W(\text{U-235 in U-Al}) = A(u) \cdot \frac{W(f)}{A(f)} \cdot \frac{R_0}{I_0} \quad \text{(mentioned above)}$$

Then, the enrichment of uranium-235 can be calculated, using the uranium content measured previously and U-235 content obtained by the above equation, with following equation.

$$\text{Enrichment} \ (\%) = \frac{\text{U-235 content}}{\text{U content}} \times 100$$

The self absorption of the test piece is an important factor. When the content of uranium in the U-Al alloy plate was

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increased, the self absorption would increase, and then, the
error of the measurement would increase.

The counting rate was measured with various uranium content,
and the result are shown in Fig. 11.

The counting rate would exponentially reach a constant value
with the U-235 content increased.

The U-235 content in the uranium aluminum alloy plate
produced in the actual processes are in the range between 10
and 500 $mg/cm^2$, however, in this range, the counting rate has
a proportional relationship with U-235 content, then, this
method can be utilized.

The uranium aluminum alloy plate which has a segregation of
uranium was measured by this method. The segregation was
defined by the X-ray radiograph.

The uranium aluminum alloy plate specimen which has U-235
segregation of about 35 mm diameter was scanned and the
result is shown in Fig. 12.

The uranium content of the U-Al alloy plate is 250 $mg\text{-}U^{235}/cm^2$
in average and the content in the segregation part is
260 $mg\text{-}U^{235}/cm^2$.

By the result of the scanning measurement, the uranium content
in the U-Al alloy plate was obtained as 248 $mg\text{-}U^{235}/cm^2$ from
the measured value 1420 cpm and Fig. 11.

By the same method, we obtained the segregated uranium content
260 $mg\text{-}U^{235}/cm^2$ from the measured value 1480 cpm and Fig. 11.

The above experimental result shows that, by this method, we
can measure precisely the segregated U-235 content in the
U-Al alloy plate.

8. Conclusion

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8. Conclusion

The advantages using this method in the process control are as follows;

1) It requires very short time to measure uranium content.

2) The precision of the measurement is usual. (It depends on the condition of the measurement, and it is as high as ±0.5%.)

3) The U-235 weight in fuel plate can be mensured by this method.

4) The total uranium content can be measured by this method, and the enrichment of U-235 can be calculated from the measured value of total uranium content and U-235 content.

5) Developing the scanning method, the segregation of U-235 can be measured.

6) There are good reproducebility in this method compared with density measuring mothod.

The disadvantages of this method are follows:

1) The equipment is expensive.

2) It need precise attention to manufacture and maintain the standard foil.

3) The error depends on the geometry.

In conclusion, this method seems to be very useful, and we are going to study using this method in the manufacturing process of our plant, comparing with the method density measurement.
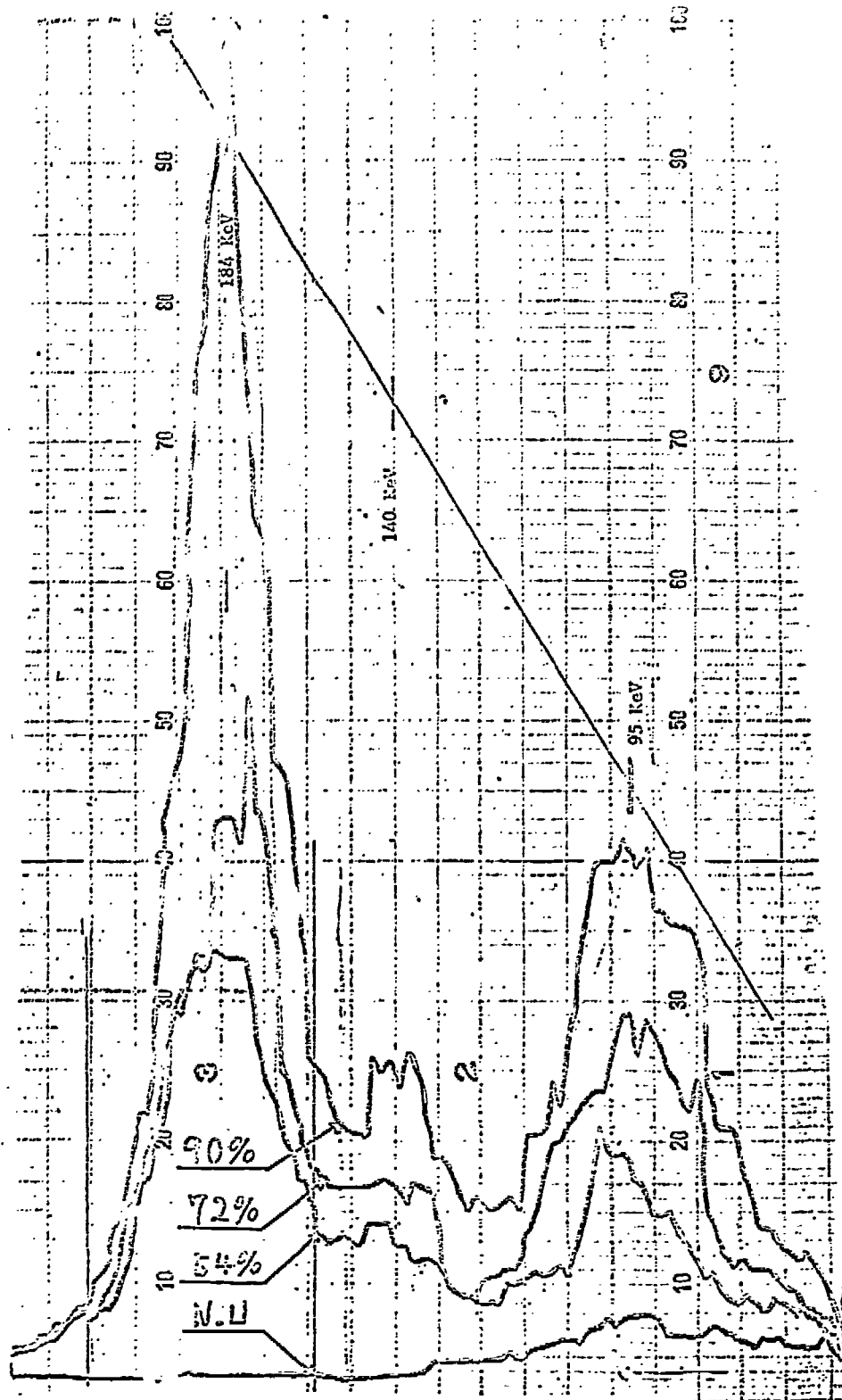
Figure   Captions

Fig. 1.   The gamma ray

Fig. 1. The gamma ray spectra of uranium

Fig. 2.

Fig. 3.

electrodepositing
rate ( mg/120min
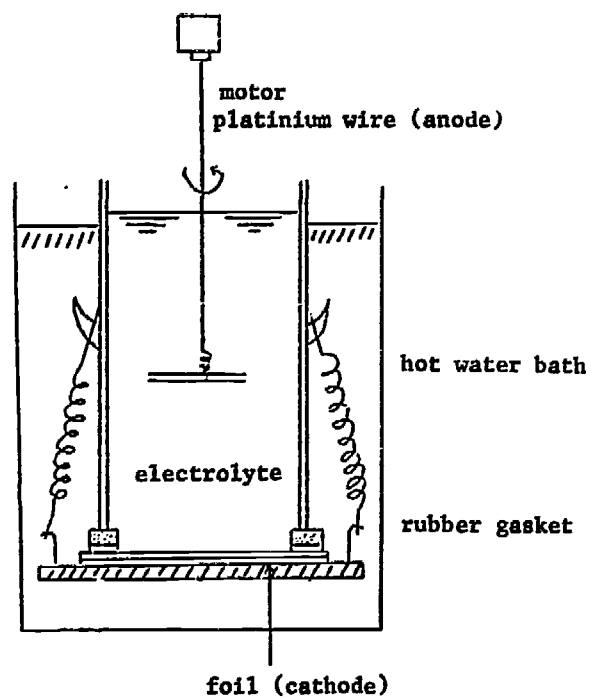
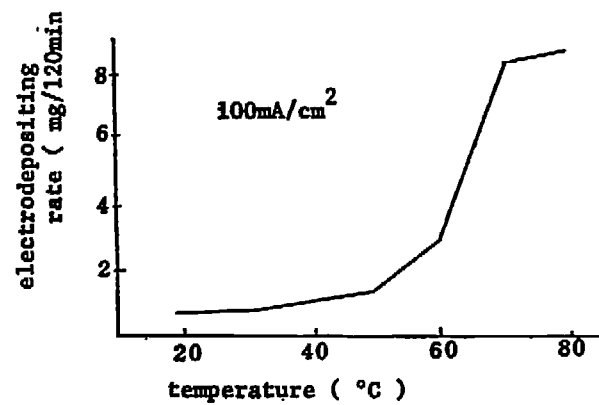Fig. 2. The equipment of electrodeposition



100mA/cm$^2$

Fig. 3. Influence of electrodepositing temperature

Fig. 4. In

Fig. 5.

Fig. 4. Influence of the ammonium oxalate concentration
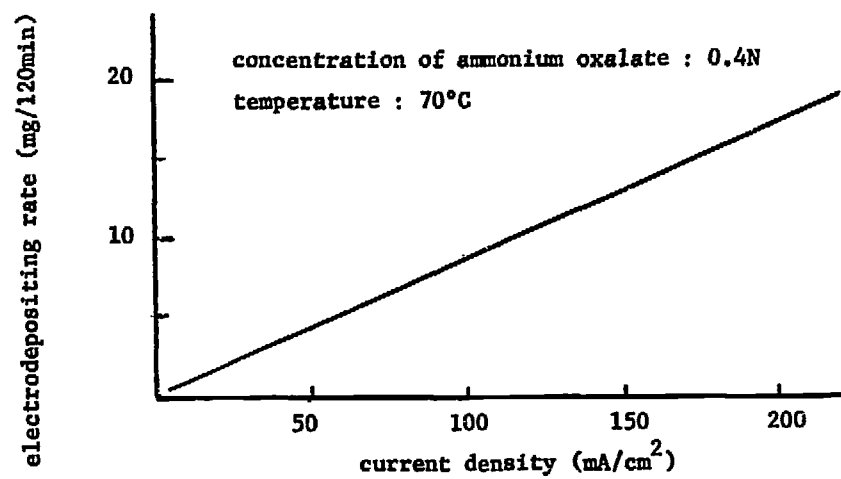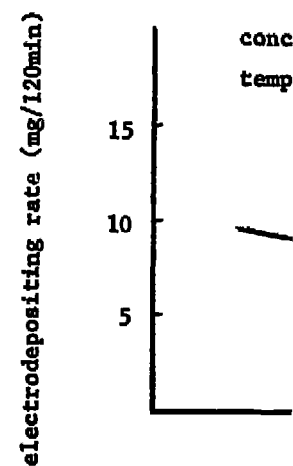
electrodepositing rate (mg/120min)

current density : 100mA/cm$^2$

temperature : 70°C

0.1    0.2    0.3    0.4

concentration (N)



Fig. 5. Influence of the current density

electrodepositing rate (mg/120min)

concentration of ammonium oxalate : 0.4N

temperature : 70°C

20

10

50    100    150    200

current density (mA/cm$^2$)



Fig. 6. Influen

electrodepositing rate (mg/120min)

conc

temp

15

10

5



Fig. 7. The rela

U-235 content (g/cm$^2$)

0.06

0.04

0.02

200

coun

concentration of ammonium oxalate : 0.4N
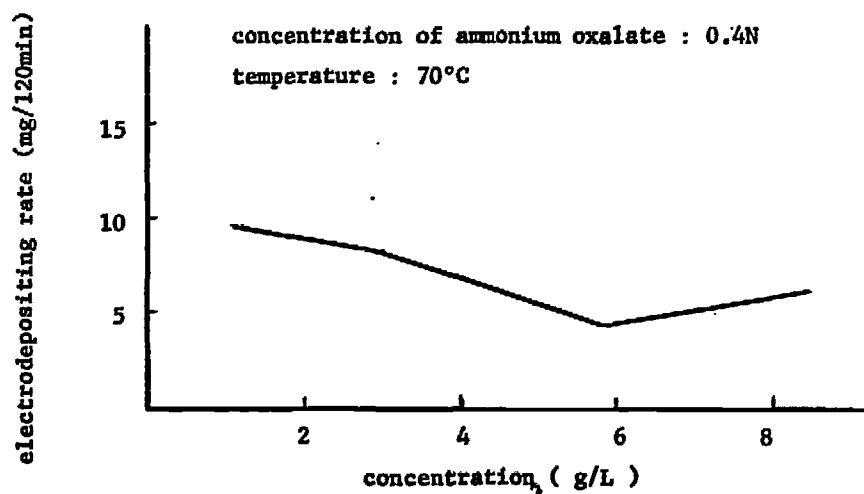
temperature : 70°C
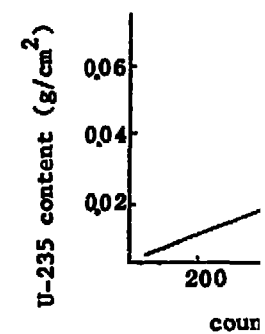
Fig. 6. Influence of the uranium nitrate concentration
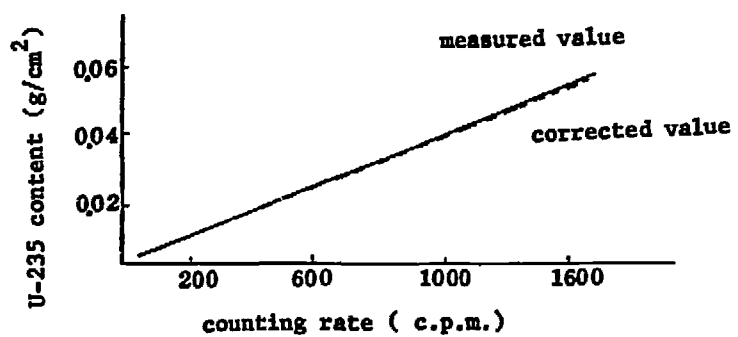
Fig. 7. The relation between counting rate and U-235 content

```
┌─────────────────────┐   ┌──────────┐
│ single channel      │───│ scaler   │
│ pulse hight analyzer│   └──────────┘
└─────────────────────┘
┌─────────────────────┐   ┌──────────┐
│ 200 channel         │───│ recorder │
│ pulse hight analyzer│   └──────────┘
└─────────────────────┘
┌─────────────────────┐
│ linear amplifier    │
└─────────────────────┘
┌─────────────────────┐   ┌──────────────────┐
│ pre-amplifier       │───│ high-voltage     │
└─────────────────────┘   │ power supply     │
                          └──────────────────┘
```

← photomultiplier tube
← NaI(Tl)scintillator
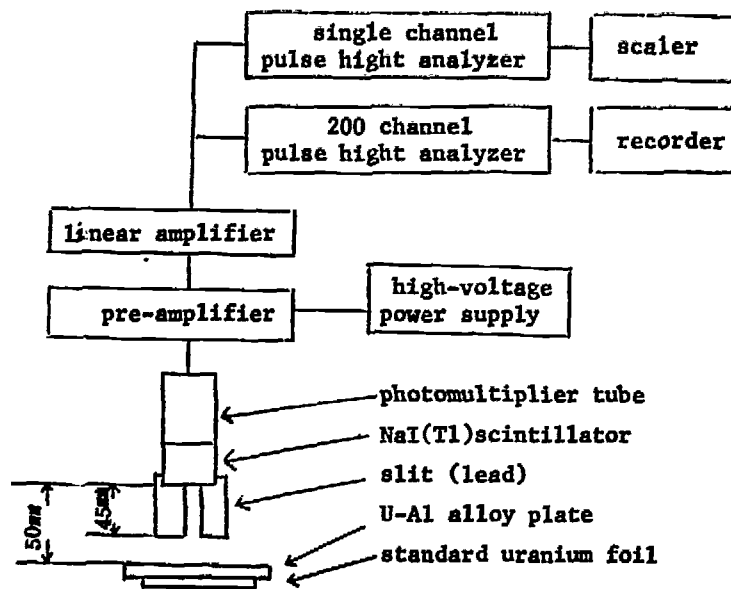← slit (lead)
← U-Al alloy plate
← standard uranium foil

50mm   45mm

Fig. 8.   measurement diagram of gamma ray spectrometry
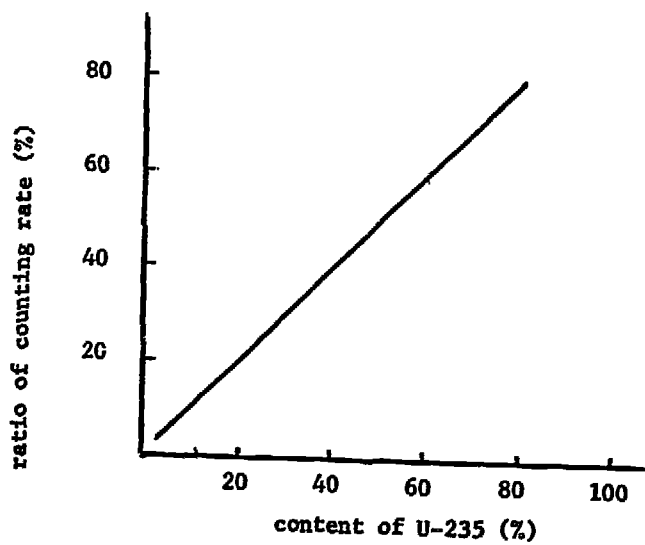


Fig. 9.   Relation of counting rate and actual content of U-235



exponential mass absorption coefficient ($e^{-t}$)

1.0
0.5
0.3
0.2
0.1

0.1

Fig. 10.   The relatio

Fig. 10.　The relation of the uranium content and absorption coefficient
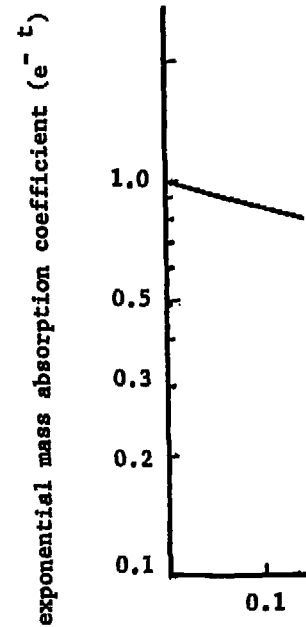


Fig. 11. The relat:
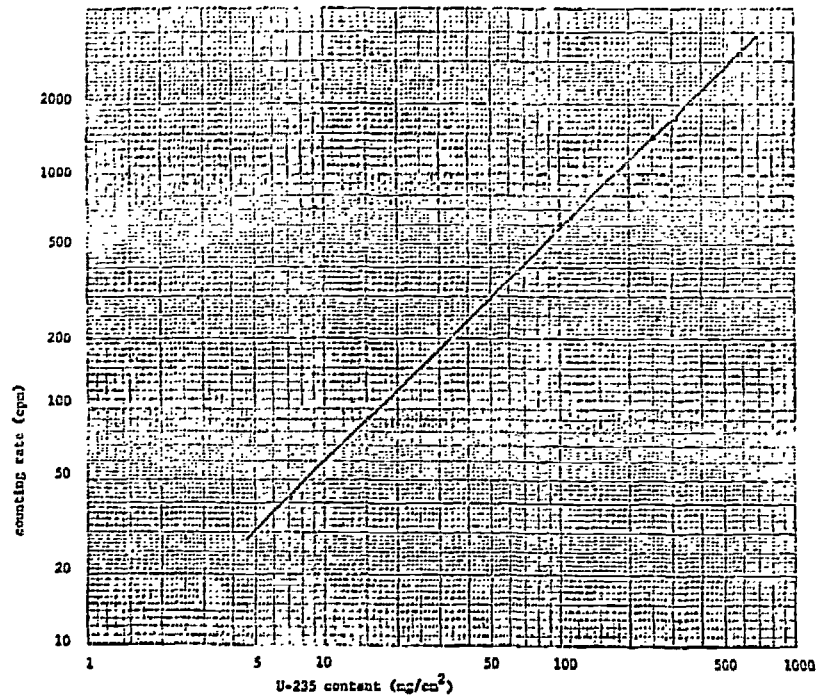


Fig. 12. Example o:

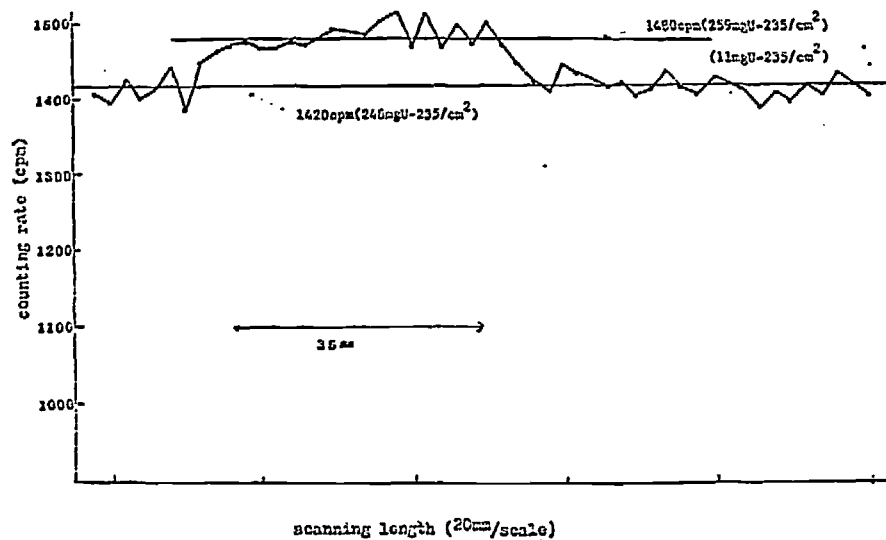Fig. 11. The relation of the counting rate and uranium-235 content



Fig. 12. Example of measurement of the segregation of U-235 in U-Al alloy plate.