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TITLE : High Resolution Gamma Spectrometer for Plutonium Isotopic Analysis

Field Test of New TASTEX System for Plutonium Product Verification at The Tokai Reprocessing Plant (Final Report of JASPAS JC-3)

JULY 1988

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Field Test of New TASTEX System for Plutonium Product Verification at The Tokai Reprocessing Plant

（ＪＡＳＰＡＳ　ＪＣ－３　ファイナルレポート）

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池田　久　和地　勇　林允之

要　旨

JASPAS（日本のIAEA保障措置支援プログラム）の１項目として1982年以来、開発されてきたγ－スペクトルによるPu同位体及び濃度分析システムについて、その成果を報告する。本装置は、高分解能γ－スペクトロメータによるPu同位体分析技術及びＫ－エッジデンシトメーターによるPu濃度分析技術を組み合わせたシステム（ニュータステックスシステムと呼ぶ）で、再処理工場のPu製品の同位体組成及び濃度を非破壊で、かつ迅速に分析するものである。これまで、数年にわたりフィールドテストを行い、DA（破壊分析）との比較を続けてきたが、比較的良好な精度で結果が得られることが確認できたため、フィールドテストを終了することとした。本レポートで取扱われているのは1985年～1988年各キャンペーン、インターキャンペーンにて行なわれた測定結果である。代表的な結果を記せばPu濃度測定については約10分の測定で0.7％（Fresh Pu）及び1.0％（Aged Pu）という精度が確認された。またPu同位体測定については、質量分析に対する相対偏差のバラツキがPu－238, 239, 240, 241, 242で各々1.6％, 0.4％, 0.5％, 1.1％, 8.0％（Fresh Pu, 30分測定）及び1.4 ％, 0.5％, 1.1％, 1.1％（Aged Pu , 60分測定、ただしPu-242は質量分析値を使用）であった。その他、本フィールドテストで得られた知見としては、高水準なソフトウェア、ハードウェアが用いられた施設設置タイプ保障措置システムにおいても長期的なシステマティックエラーというものは避けられず、装置に熟知した者によるシステムの管理が不可欠であるということである。なお、本装置の開発は米国ロスアラモス国立研究所及び同ローレンスリバモア国立研究所の協力により進められてきたものである。

FIELD TEST OF NEW TASTEX SYSTEM FOR PLUTONIUM PRODUCT VERIFICATION AT THE TOKAI REPROCESSING PLANT

(Final Report of JASPAS JC-3)

CONTENTS

JASPAS JC3;High Resolution Gamma Spectrometer For Plutonium Isotopic Analysis

FIELD TEST OF NEW TASTEX SYSTEM FOR PLUTONIUM PRODUCT,VERIFICATION AT THE TOKAI REPROCESSING PLANT

(Final Report of JASPAS JC-3)

Y. KUNO, K. SHIGEOKA, K. NISHIDA, H. IKEDA, I. WACHI, N. HAYASHI

ABSTRACT

This report describes the field test results of the New TASTEX system. This system consisting of the high resolution gamma spectrometer and the K-edge densitometer can measure both isotopic abundances and concentration of plutonium simultaneously. Entire system is controlled by the multi-channel analyser and a multi-user computer. The system was designed and built under the Japan Support Program for Agency Safeguards (JASPAS). The software of this system developed at Los Alamos National Laboratory (LANL) and Lawrence Livermore National Laboratory (LLNL) has been installed in the system assembled at the Tokai reprocessing plant (TRP) in July 1985. In the course of campaigns from 1985 till 1988, field tests have been carried out on plutonium product solutions of TRP. The results of plutonium concentration and isotopic abundances obtained by the K-edge densitometer and the high resolution gamma spectrometer (HRGS) have been compared with those by controlled potential coulometer and mass spectrometer respectively. Precision of plutonium determination with K-edge densitometer is estimated approximately 0.7% and 1.0% for the freshly processed plutonium and the aged plutonium respectively. The scatters in the relative differences between HRGS and the destructive

analysis (DA) detected on the results of freshly processed plutonium sample were 1.6%, 0.4%, 0.5%, 1.1%, 8.0% for Pu-238, Pu-239, Pu-240, Pu-241, Pu-242 respectively, whereas those on the results of aged sample were 1.4%, 0.5%, 1.1%, 1.1% for Pu-238, Pu-239, Pu-240, Pu-241 respectively.

1. INTRODUCTION

A few non-destructive assay techniques were introduced to the Tokai reprocessing plant in 1978 under the agreement between the United States and Japan called "TASTEX" (Tokai Advanced Safeguards Technology Exercise). The K-edge densitometry and the high resolution gamma spectrometry for concentration and isotopic composition analysis of plutonium were included in the TASTEX.

The K-edge densitometry technique developed at LANL has been implemented and used for routine inspection analysis since 1982 because of its fairly satisfactory results in the field test. Although good agreement between mass spectrometry and HRGS results were observed, it was difficult to properly maintain the LLNL spectrometer system in our environment. The development of a new system has therefore initiated in 1984 as a part of JASPAS (Japan Support Program for Agency Safeguards).

In order to improve and facilitate the measurements, the new system called "New TASTEX System" / "Combined system" consisting of not only high resolution gamma spectrometer but also K-edge densitometer has been developed. The purpose of the new system is to determine both concentration and isotopic composition of plutonium simultaneously for safeguards of the Tokai reprocessing plant.

It was found that thick cell used for the K-edge densitometer was not suitable for isotopic composition measurement from the viewpoint of self absorption of plutonium. Therefore the idea of two different cells was adopted in the system. The system consists of two HP-Ge detectors, two amplifiers, two different electronic modules including ADC and stabiliser and a multi-channel analyser (ND) and multi-user computer (DEC) commonly used.

Two programs of this system which were developed at both LANL and LLNL have been combined and installed in the system at TRP. Operators can use both programs simultaneously and independently by using two CRT terminals.

The absorption of the K-edge densitometry in the present application involves the measurement of gamma radiation. The transmission sources for the K-edge densitometer are the radioisotopes, Se-75 and Co-57. The 121.1 keV and 122.1 keV gamma rays emitted by the sources closely bracket the K-edge absorption edge of plutonium at 121.8 keV. The logarithm of the ratio of the measured transmissions at these two gamma-ray energies is directly proportional to the concentration of plutonium.[1)]

Plutonium isotopic abundances are obtained by analysing the peaks of passive gamma-ray emitted from plutonium solution. The energy region analysed is from 40 keV to 300 keV. The removal of U-237 and Am-241 from samples permits the prominent peaks of Pu-238, Pu-239 and Pu-240 at 43 keV, 51 keV, 45 keV, respectively, to be analysed and interpreted. The 148 keV peak is used to measure the abundance of Pu-241. For aged plutonium solution, several individual peaks of high energy plus the 94-104 keV complex of unresolved gamma and x-ray peaks are analysed because intense 59 keV peak of Am-241 obscures the Pu peaks of low energy.[2)] Pu-242 isotopic fraction is evaluated from an isotopic correlation based on Pu-239, Pu-240, Pu-241.[3)]

Calibration of the system was carried out in March 1986. The plutonium solution (about 250 g/l) used for the K-edge densitometer calibration was characterised by destructive analyses, controlled potential coulometry, AgO-Fe(II)-Cr(VI) titration and isotopic dilution method after being purified. On the other hand, calibration of high resolution gamma spectrometry was divided into two ways, namely initial calibration consisting of peak shape fitting and efficiency curve acquisition and long term calibration. The former calibration

had been done when the program was installed, while correction factors for long term calibration were obtained throughout the campaings.

The results of plutonium concentration and isotopic abundances obtained by the K-edge densitometer and high resolution gamma spectrometer have been compared with those by controlled-potential coulometer and mass spectrometer respectively for the last three years.

2. EQUIPMENT

1) Detector and Cell Characteristics

The location of sample cells and detectors is shown in Fig. 1.

[K-edge Densitometer]

The measurement station of K-edge densitometer consists of an intrinsic planar germanium detector (200 mm^2 by 7 mm) and a mechanical system. The Se-75 and Co-57 transmission sources, mounted in separate positions on a wheel, are rotated into the measurement position by a motor-driven Geneva mechanism. Collimators positioned between sample and the detector are rotated synchronously into the transmission path.

For the passive measurement, the sources are rotated out of the measurement position and shielded from the detector, and the collimation is enlarged. Fig. 2 is a conceptual view of the mechanical system and detector, while Fig. 3 is a detailed drawing of the measurement well and mechanical system. The entire mechanical system is automated and under computer control. A Cd-109 source mounted on the detector is used to correct losses of events resulting from changing count rates.

The signals from the germanium detector preamplifier are shaped, amplified, and digitised by NIM modules in the electronics rack. Pile-up pulses are electronically eliminated from the spectrum. A two-point digital stabiliser, under computer control, maintains a constant energy calibration.

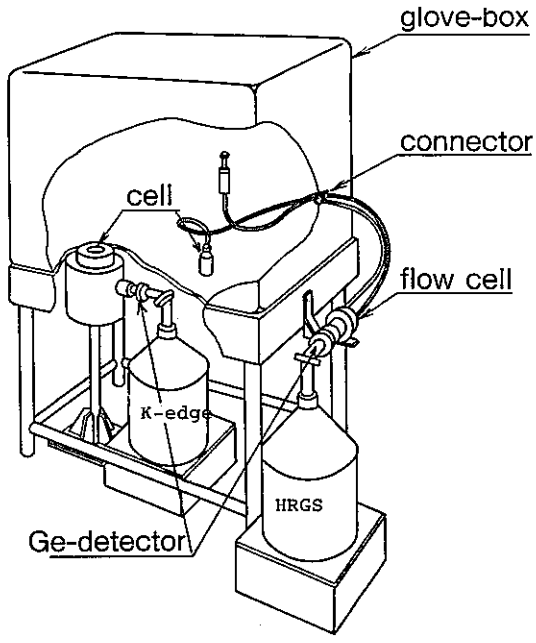


Fig. 1 Sample Cell & Detector

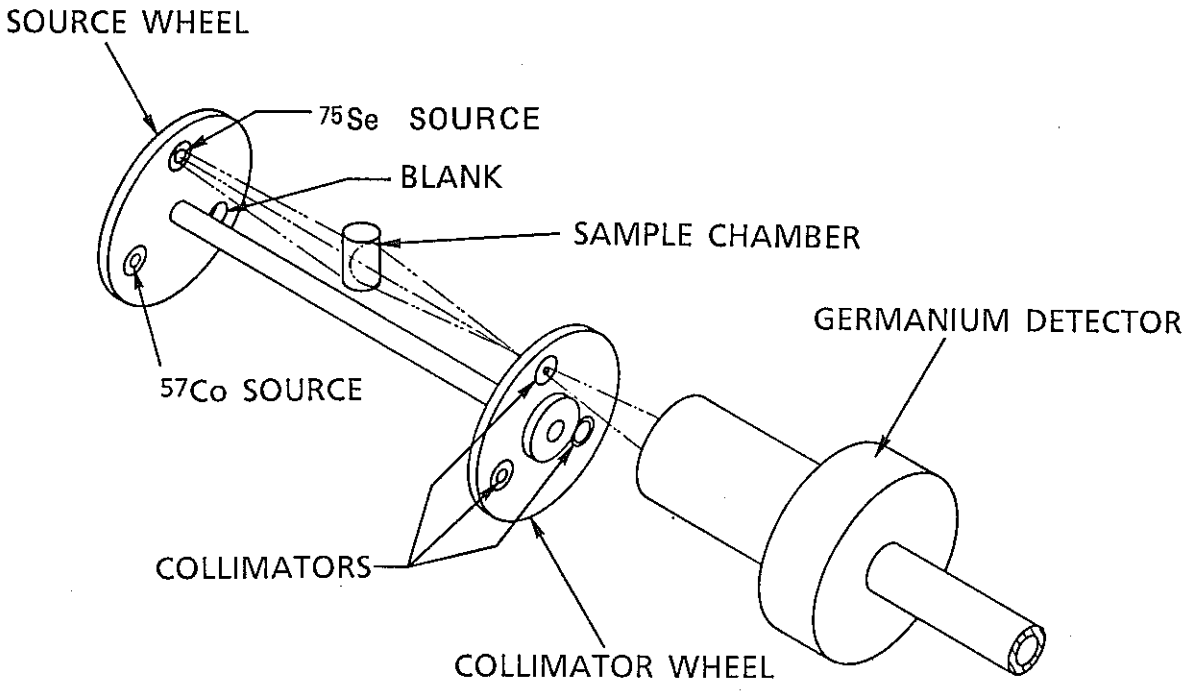


Fig. 2 Illustration of the concepts of K-edge (transmission) and passive measurements. The sample chamber is shown in position (in the counting well) for measurement of the transmissions of the ^{75}Se gamma rays.

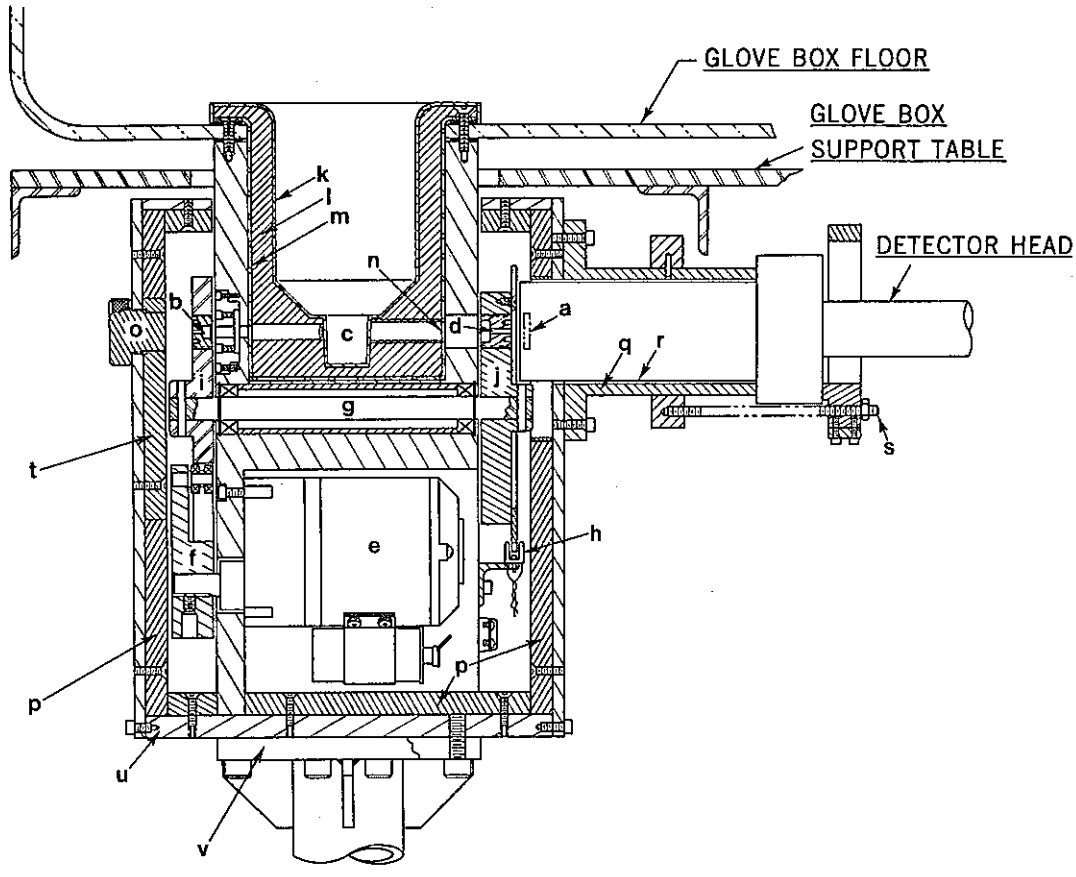


Fig. 3 Detailed features of the mechanical system:

(a) germanium crystal

(b) tungsten transmission source holder

(c) sample position

(d) tungsten collimator holder

(e) drive motor for Geneva mechanism

(f) Geneva mechanism

(g) common axle for source and collimator wheels

(h) position-sensing mechanism

(i) stainless steel source wheel

(j) tungsten collimator wheel

(k) polyurethane liner for measurement well

(l) tungsten shield

(m) stainless steel containment can

(n) 0.25-mm-thick stainless steel window

(o) removable tungsten plug for changing sources

(p) lead shielding

(q) tungsten detector shield

(r) polyethylene electrical shield

(s) detector clamping device

(t) tungsten shielding

(u) aluminum anchor plate

(v) aluminum support base

Solution samples contained in vials are introduced into the glovebox through pneumatic transport tubes following removal of the solution from the process. The solutions are transferred into disposable plastic measurement vials that are inserted into the well for assay. This disposable vial has the parallel side walls which can reduce the errors due to the positioning of sample vials.

[HRGS]

The detector is of a planar design with approximate dimensions of 200 mm^2 by 12 to 15 mm deep. The signals from germanium detector/preamplifier are processed without serious degradation of the resolution by controlling input count rates of up to 15000 cps. A pulses pile-up rejector is used to reduce the effects of chance coincidence.

The principal requirement of the analysis codes is that the sample be confined in a disk shaped geometry such that the projected surface density of the plutonium in the solution does not exceed approximately 0.02 g/cm^2. The flow-cell adopted here has 2.5 cm^2 effective area and 0.1 cm thickness. Fig. 4 is a vertical sectional view of the HRGS flow-cell. The sample container and detector are closely coupled (about 1 cm) to obtain the best counting efficiency.

The analysis code used for freshly separated plutonium solution measures peak areas in the 43-51 keV energy region; therefore the amount of absorbing material must be minimized. The presence of an intense 59 keV gamma ray in aged plutonium samples due to the in-growth of Am-241 requires the use of absorbers. Cadmium absorber around 0.1 cm is used not only to attenuate the gamma ray that directly incident upon the detector, but also to reduce the detection of Compton-scattered radiation.

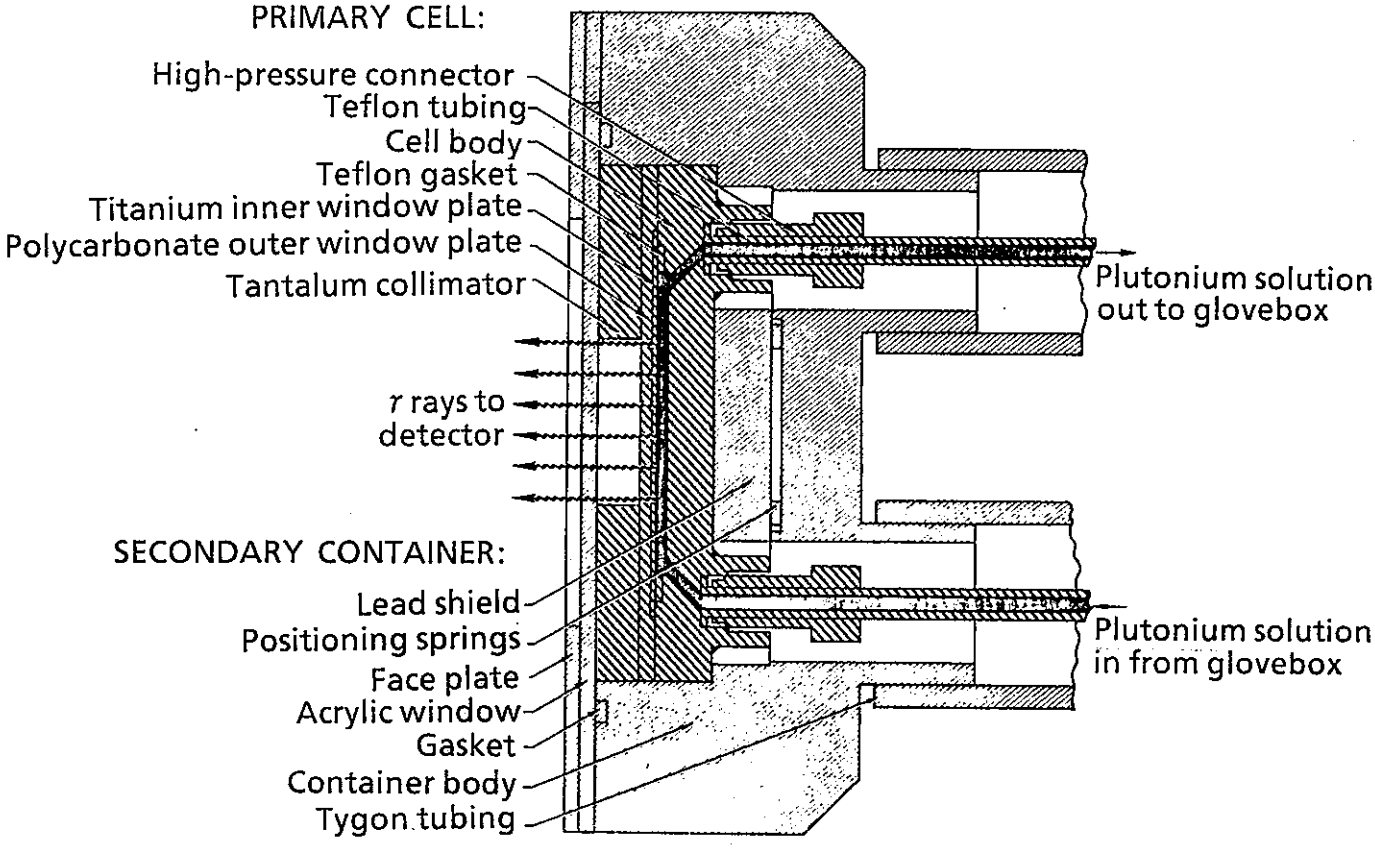


Fig. 4 Sectional View of HRGS Flow-cell

2) Spectrometer

The system consists of Nuclear Data 66 MCA with two analog to digital converters. The MCA is controlled by a Digital Equipment Corporation (DEC) PDP-11/23 plus computer with 256 k bytes of memory.

Fig. 5 Electronics & CPU

The software is RSX-11M from DEC. The system has two hard disk drivers (RL02) and two floppy disk drivers (RX02) for data and program storage. The time sharing nature of the software allows both the densitometer and the isotopic software to control the MCA simultaneously and to operate independently each other. Electronics and computer terminals of the system are shown in Fig. 5.

Fig. 6 describes the block diagram of the total system.

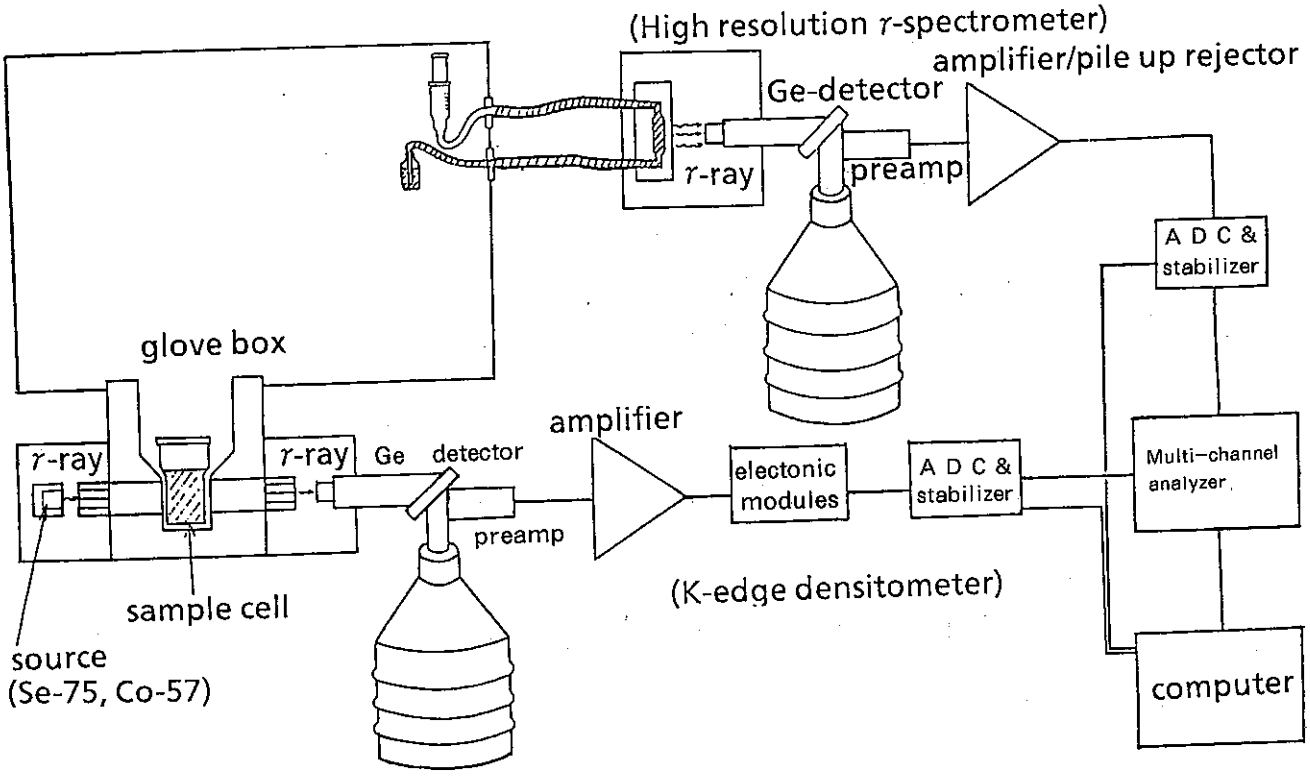
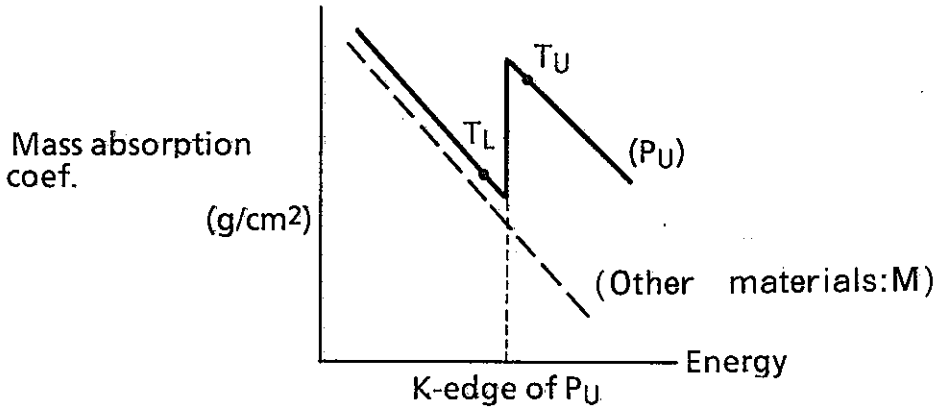


Fig. 6 Block Diagram of New TASTEX System

3. MEASUREMENT METHODS AND SOFTWARE

[K-edge Densitometer]

K-edge densitometry is based on the transmission properties of gamma radiation around the absorption edge. As the gamma ray energy is increased the transmission of the gamma ray through a material slowly increases until the binding energy of electron in the material is reached. The the transmission suddenly decreases, because the another electrons can absorb the incoming gamma radiation. At 121.8 keV, the plutonium K-edge energy (shown below), all other materials exhibit nearly constant transmission properties as the energy of the gamma radiation is varied.



Absorption of gamma ray from a source and all other materials is described as following equation.

$$I = I_0 \cdot \exp(-\mu_M \rho_M X) \cdot \exp(-\mu_{Pu} \rho_{Pu} X) \qquad (1)$$

where

I_0 : intensity of source

I : intensity of transmitted gamma ray of source

μ_{Pu} : mass absorption of coefficient for Pu

μ_M : mass absorption of coefficient for anything other than Pu

ρ_{Pu} : concentration (density) of Pu

ρ_M : concentration (density) of anything other than Pu

X : sample thickness.

The ratio of I to I_0 gives transmission of gamma ray.

$$T = I/I_0 \qquad (2)$$

Therefore transmissions of just above (U) and below (L) the K-edge of Pu are given as follows;

$$T_U = \exp(-\mu_{MU}\rho_M X)\cdot\exp(-\mu_{UPu}\rho_{Pu} X) \qquad (3)$$

$$T_L = \exp(-\mu_{ML}\rho_M X)\cdot\exp(-\mu_{LPu}\rho_{Pu} X) \qquad (4)$$

If (3) is divided by (4),

$$\frac{T_U}{T_L} = \frac{\exp(-\mu_{MU}\rho_M X)}{\exp(-\mu_{ML}\rho_M X)}\cdot\exp(-\Delta\mu_{Pu}\rho_{Pu} X) \qquad (5)$$

where

$\Delta\mu$ is difference of μ_U and μ_L. The first term of above equation can be regarded unity because μ_{MU} and μ_{ML} are nealy same.

$$\ell n\,(T_U/T_L) = -\Delta\mu_{Pu}\rho_{Pu} X \qquad (6)$$

and

$$\rho_{Pu} = -\frac{1}{\Delta\mu_{Pu} X}\ell n\frac{T_U}{T_L} \qquad (7)$$

In conclusion, ρ_{Pu} is independent of matrix (things other than Pu) absorption or self absorption.

To measure a transmission, it is first necessary to get a straight through value. Namely, the Se-75 and Co-57 measurement control spectra are acquired to obtain the unattenuated intensities (net peak areas in the absence of sample attenuation) of the gamma-ray lines required for transmission measurements.

Cd-109 source, 88 keV peak, is used as a "clock" to determine the actual collection time that the system has. Namely, transmmision value is corrected as follows;

$$\frac{T_U}{T_L} = \frac{I_U/I_{U_0}}{I_L/I_{L_0}} = \frac{\{A(122)/A(88)\} / \{A_0(122)/A_0(88)\}}{\{A(121)/A(88)\} / \{A_0(121)/A_0(88)\}} \quad ,$$

where

$A(121), A(122), A(88)$: count rate of Se-75, Co-57, and Cd-109 on sample measurement

$A_0(121), A_0(122), A_0(88)$: count rate of Se-75, Co-57, and Cd-109 on straight through measurment.

The sample thickness for the cell X used in the system is approximately 2 cm. The value of $\Delta\mu$ the K-edge of plutonium is 3.4 cm^2/g. Therefore, the expected value of $\Delta\mu X$ for these measurements should be approximately 6.8 cm^3/g. Detector relative efficiency check is performed using the net peak areas of 121.1 and 136.0 keV (Se-75), in addition to the resolution and gain checks applied to the Se-75 and Co-57 measurement control spectra when straight through measurement is carried out. The ratio of these two peak areas should remain constant for a given Se-75 source if the relative detection efficiency remains unchanged.

The new K-edge densitometer includes additional correction on plutonium determination which can minimize the effect of matrix based on the energy difference between 121.1 and 122.1 keV. In addition to the transmission at 121.1

and 122.1 keV, the measured transmission at 136.0 keV from the Se-75 source is also available with no additional measurement. The technique is, therefore, to extrapolate from 136 and 122 keV to 121.8 keV to find the transmission above the edge. (Refer to Fig. 7)

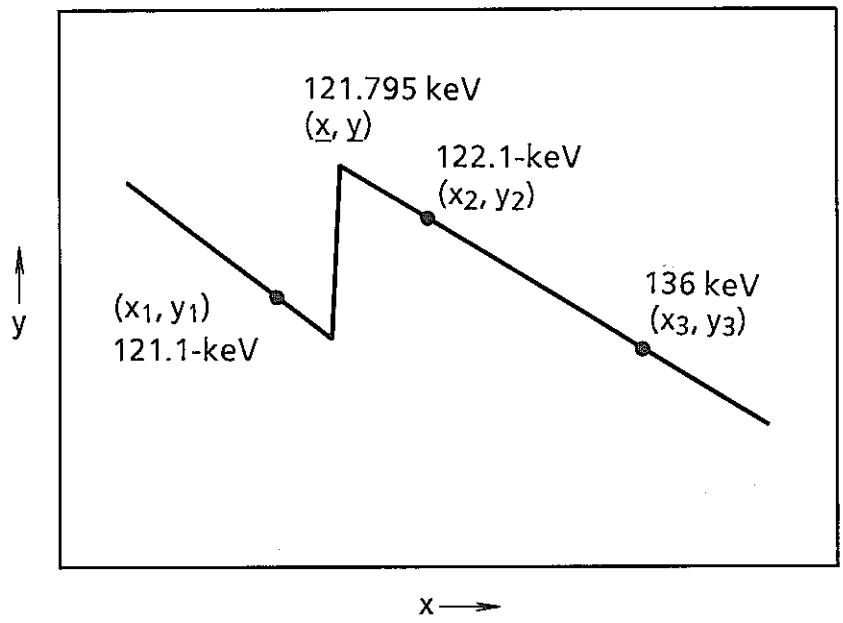


Fig. 7 This figure illustrates the extrapolation. The x-axis is the ℓn E variable, and the y-axis is the ℓn μ variable.

For a short energy range near the K-absorption edge, μ vs E is approximately a straight line in ℓn-ℓn space. By assuming the same slope below the K-edge, the extrapolation below the edge can also be performed. The extrapolation is reasonable because of the short distance to be extrapolated (from 121.1 to 121.8 keV). Details are described elsewhere.[4)]

The K-edge densitometer software consists of two programs.[4)] The main program, DENSIT, controls the MCA, the Geneva source wheel, and the digital stabiliser. The program also performs data analysis and writes (or reads) data to (or from) the disk. In addition to these operations, the program performs the

measurement control function by promoting operators to perform bias or precision checks on a scheduled basis. In this program, net peak areas are determined by subtraction of straight-line backgrounds obtained from two regions of interest that bracket the peak region. The peak region is, typically, three times the peak FWHM. The background regions are, typically, one-third the peak region width. A formal description of the methods used for peak area determination is given elsewhere.[5)]

Following the acquisition of each spectrum, the energy resolution and gain are evaluated from the data. A reference peak FWHM and centroid are determined and compared with limits specified by the program for these peaks.

The DENSIT program uses a set of parameters, and this set is prepared and modified by the ancillary program, CDENSIT. Program options of the K-edge densitometer measurement included in DENSIT are divided into operators options and supervisor options. The former options are the routine operations used in the course of normal data acquisition and analysis. The latter options are used only by personnel who are fully knowledgeable in the operation of the programs.

Several improvements have been made in the upgrade in addition to the extrapolation shown above. Operator interaction in the revised software is much more intuitive and user friendly.

[HRGS]

The typical spectra of freshly processed plutonium and aged one are shown in Fig. 8 and Fig. 9 respectively.

The spectral data acquired by using intrinsic germanium detector are analysed and interpreted by the program[6] which is divided into two parts. One analyses spectra taken of freshly separated solutions, and the other spectra of aged solutions. The program called LEPA is used to analyse spectra of recently processed solutions.[7] Because such soultions are relatively free of Am-241 and U-237, the spectral features associated with these isotopes are largely absent, permitting other features to be more easily detected. This is particularly true of the 59-keV gamma ray that is principally due to Am-241. Its absence allows gamma rays of lower energy to be clearly visible in a spectrum.

The program LEPA takes advantage of this condition and measures the intensities of three low-energy gamma rays, at 43, 45, and 51 keV, from which it calculates abundances for the plutonium isotopes 238, 240 and 239 respectively. The Pu-241 abundance is calculated from the 148 keV peak intensity. A confirmatory value for Pu-241 can be calculated from the intensity of the 94 keV X-ray peak. the 129 keV peak is also used as an additional measure of the abundance of Pu-239. The Pu-242 isotope in the plutonium solutions can not be detected by gamma ray spectrometry. However, its abundance is usually less than 5% in first-cycle fuel, and therefore a highly accurate measure of its abundance is not required. The correlation between the abundance of Pu-242 and the abundances of Pu-239, Pu-240 and Pu-241 is given by the relationship[8]

$$[\text{Pu-242}] = k\,[240][241]\,/\,[239]^2,$$

where [241] is decay-corrected to the reactor discharge time.

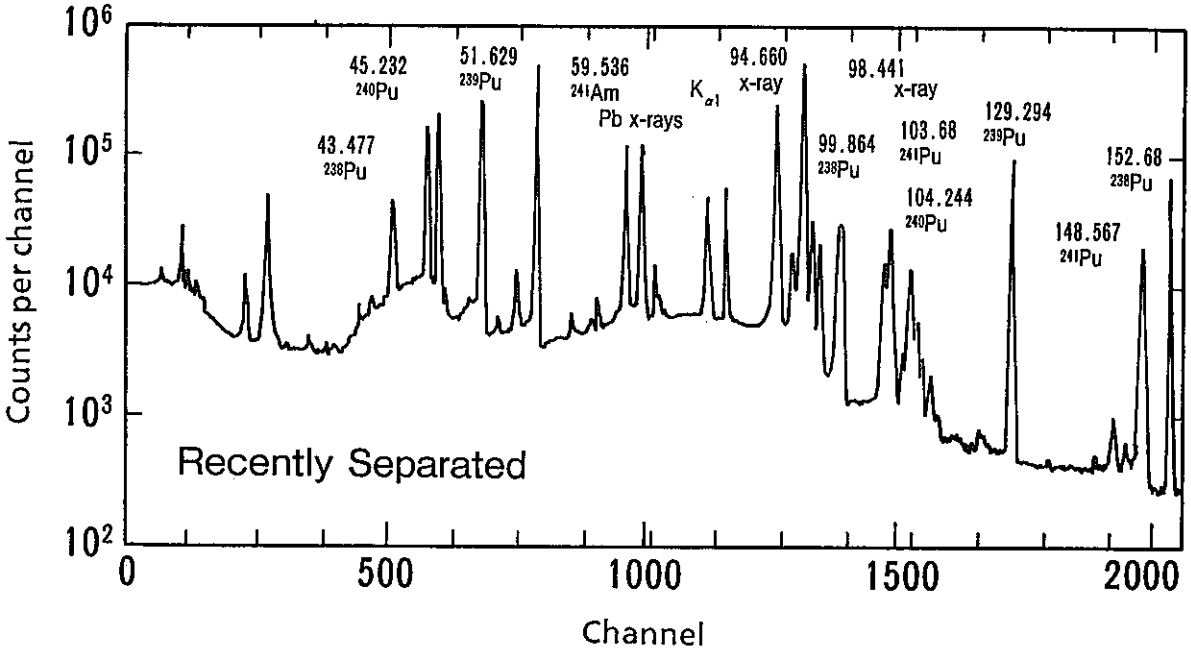


Fig. 8 Gamma spectrum of fresh plutonium

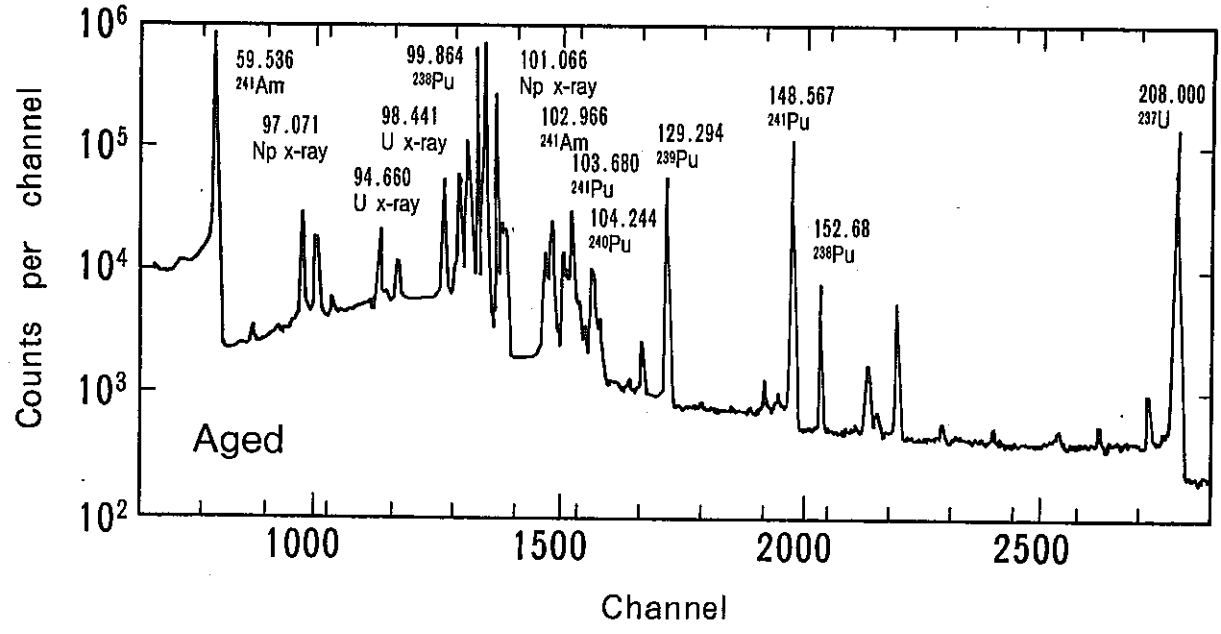


Fig. 9 Gamma spectrum of aged plutonium

In addition to reading in appropriate portions of the spectrum to be analysed, the program also reads two additional files used for the analysis. The first is a file containing peak shape constants generated by the PKSNAL program. These constants are used to describe the peak shapes as a function of energy. The second file contains entries defining detector efficiencies for the six peaks that will be analysed, sample cell parameters, bias correction factors and other constants. Each entry is automatically generated when the calibration program LECAL is executed.

The program called PUAN is used to analyse the spectra of aged solutions. Such solutions generally exhibit an intense peak at 59 keV due to Am-241. The Compton distribution from this peak usually obscures all peaks of low energy. Also since the intensity of this peak tends to dominate the total counting rate, a cadmium absorber is used to reduce its intensity.

The presence of U-237 in aged solutions is also easily observable. This activity comes into equilibrium with its Pu-241 parent after about eight weeks. In its normal mode of operation, PUAN assumes that this equilibrium is in effect. However, an internal check is made, and if the solution is suspected or determined to be out of equilibrium, a message is given and appropriate adjustments are automatically made in the program.

The PUAN program uses more constants than does LEPA. A file is generated automatically when the calibration program PUCAL is run that contains entries corresponding to the peak energies, the efficiency coefficients for these peaks, and a bias correction factor for each isotope.

The program mentioned above are menu driven, and the menu options shown in Fig. 10 are similar to that of theK-edge system as much as it is practical.

Fig. 10 The List of Program Options

OPTIONS of K-edge

OPERATOR OPTIONS

A	-	Assay	AU	-	AUtocycle
SB	-	St thru - Backgr			
MB	-	Measurement control - Bias			
MP	-	Measurement control - Precision			

SUPERVISOR OPTIONS

AD	-	Assay from Disk	AUD	-	AUtocycle from Disk
SBU	-	St thru - Backgr from Disk			
C	-	Calibration	CD	-	Calib from Disk
D	-	Default			
LA	-	List Assay log	LM	-	List MC log
LR	-	List combined Results file			
OU	-	change OUtput Listing device			
R	-	Read data from disk	W	-	Write data to disk
X	-	Exit from program			

OPTIONS OF HRGS

OPERATOR OPTIONS

A	-	Assay
MB	-	Measurement control - Ba133 Source
MP	-	Measurement control - precision

SUPERVISOR OPTIONS

AD	-	From disk
AU	-	Autocycle
AUD	-	Autocycle from disk
C	-	Calibration mode
D	-	Default settings
LA	-	List assay log
LM	-	List measurement control log
OU	-	Change output listing device
ST	-	Report System Status
R	-	Read data into analyzer from disk
W	-	Write data from analyzer to disk
X	-	Exit from program

4. EXPERIMENTAL RESULTS AND DISCUSSION

[K-edge Densitometer]

(1) Measurement Control

Tantalum reference foils are used to check the total performance of the K-edge densitometer. The combination of two non-active tantalum foils which have different thickness can simulate absorption edge.

Fig. 11 is the results of the tantalum measurement control obtained in 1988-1 campaign of the Tokai Reprocessing Plant. The effective concentration in g/ℓ is plotted in order of measurements performed throughout the campaign. Each solid point is the result of the single measurement carried out prior to every plutonium assay. Clock count times for the Se-75 and Co-57 measurements were 300 seconds (each). The average of the results is 209.3 effective plutonium g/ℓ, which is slightly higher than the data expected from the standard value determined on the existing system. However, the standard deviation of all the results is 0.74% which shows reasonable performance as compared with that of routine system.

This figure may show the possibility of short term systematic bias. For instance, the result has been slightly decreasing vs time from the point No. 30, in which the isotopic sources were renewed. This might be due to the difference of both count rates.

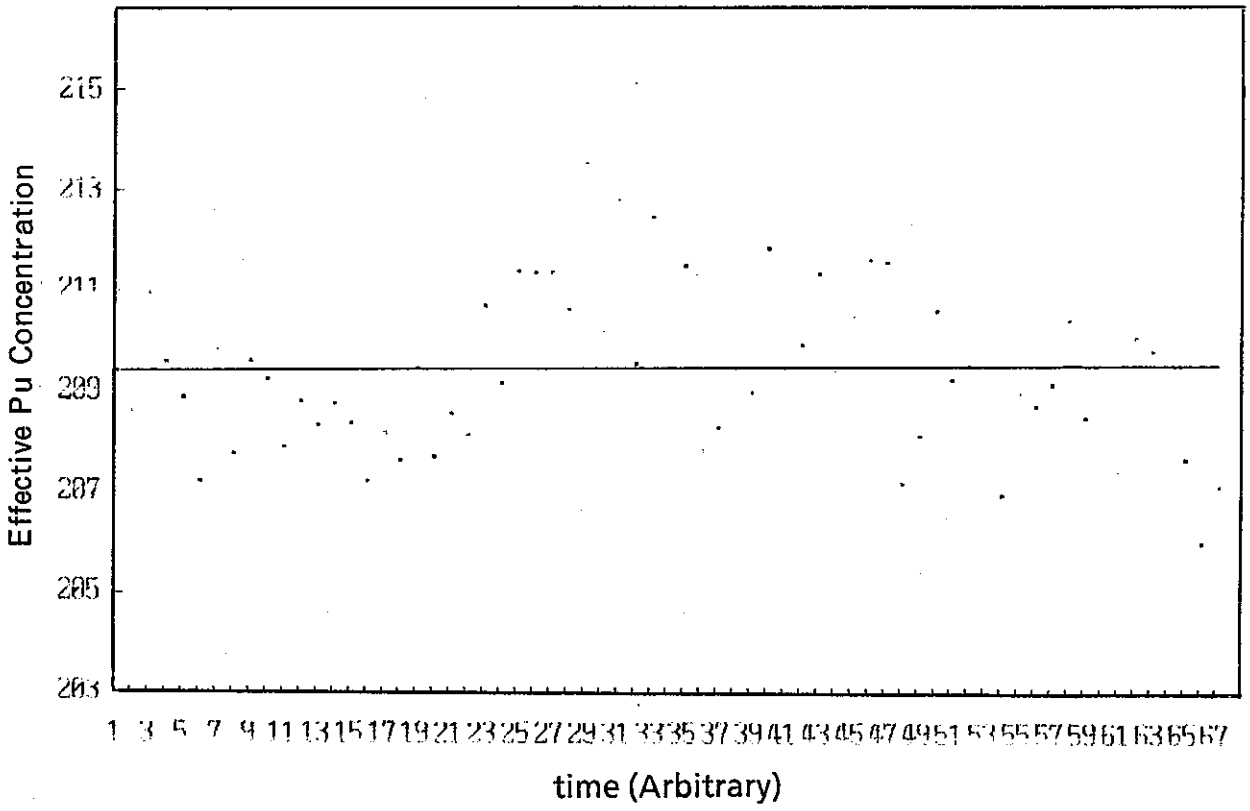


Fig. 11 Ta-Foil Control Chart (1988-1 Campaign)

(2) Plutonium Concentration Measurement

The K-edge assay results for both freshly separated plutonium and aged plutonium are discussed relative to the results of destructive analysis (DA) and those of the existing system (called OLD system) routinely used. Clock count times for the plutonium assay were the same as those for the measurement control described above, while 1800 second count period (each for Se, Co, BG) was used for the straight through measurment. As DA method for the plutonium concentration determination, the controlled potential coulometry has been adopted, that can measure plutonium concentration very accurately within 0.1% without any interference.[9]

Table 1 and 2 show the average ratios of K-edge densitometer result/DA result and the standard deviations of them obtained in the campaign of 1985-2, 1986-1, 1987-1 and 1988-1. Detailed raw data obtained with K-edge denstometer, OLD system and DA are given in Attachment 1.

Differences between the results of the K-edge densitometer (here called "New" K-edge meter) and those of DA obtained in 1985-2 plus 1986-1, and 1988-1 campaigns for freshly separated plutonium are plotted in Fig. 12 and 13 respectively. The plotted values are obtained from the average of 3-cycle measurements. Difference between the results of the K-edge densitometer ("New" meter) and those of existing K-edge densitometer (here called "Old" K-edge meter) are shown in the same figures.

The same comparisons between "New" K-edge meter and DA, and between "New" and "Old" K-edge meters have been carried out on aged plutonium measurement. The results are shown in Fig. 14. It is noted on freshly separated plutonium measurement that "New" K-edge meter has shown better performance than that of "Old" K-edge meter over last three years. Specially mean biases of "New" system calculated from each campaign result show less biases than those of "Old" system. Looking at the performance of both systems, each K-edge meter shows its own systematic errors, which should agree with the tendency of tantalum measurements. This sort of systemtic bias is supposed to be difficult to correct insitu measurement.

Table 1. K-edge Densitometer Measurement Results: Fresh Samples

Campaign Name	1985 - 2 Campaign	1986 - 1 Campaign	1988 - 1 Campaign
Number of Samples	22 Samples	6 Samples	28 Samples
Av. of Ratio (K/C)* (Rel. Difference)	1.0028 (+0.28 %)	0.9919 (-0.81 %)	1.0027 (+0.27 %)
CV%	0.71 %	0.38 %	0.61 %

* Ratio of K-edge Densitometry / Coulometry.

Table 2. K-edge Densitometer Measurement Results: Aged Samples

Sampling Point	Pu-Storage (1987)	Pu-Storage (1988)
Number of Samples	13 Samples	6 Samples
Av. of Ratio (K/C)* (Rel. Difference)	0.9978 (-0.22 %)	0.9999 (-0.01 %)
CV%	1.08 %	0.70 %

* Ratio of K-edge Densitometry / Coulometry.

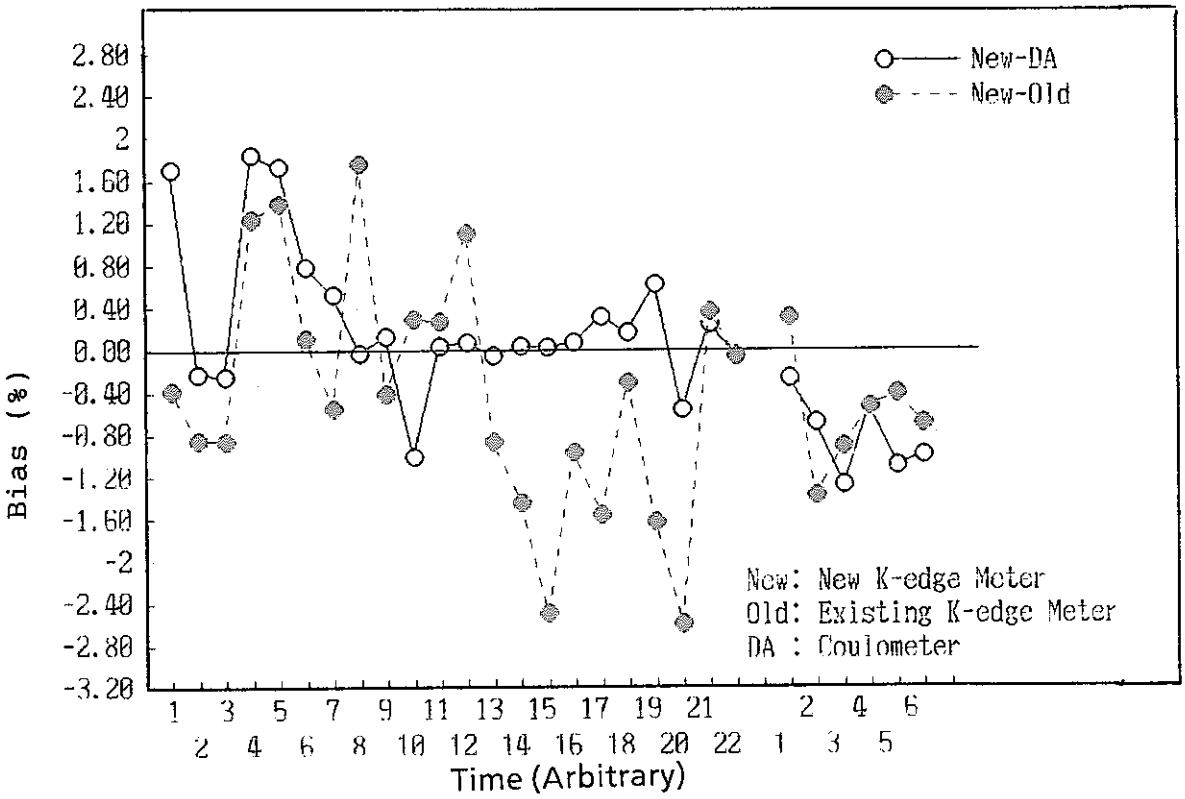


Fig. 12 Difference between New System & DA, Difference between New & Old System Fresh Pu (85-2 & 86-1 Campaign)

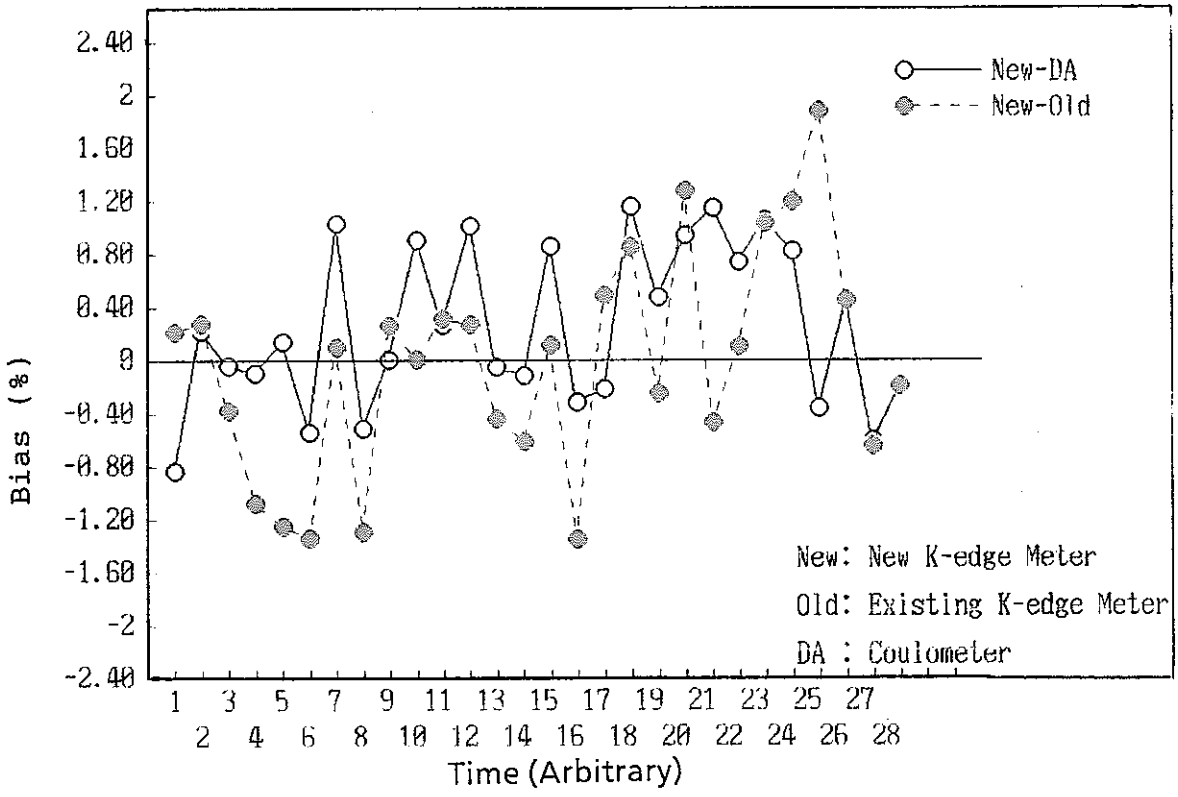


Fig. 13 Difference between New System & DA Difference between New & Old System Fresh Pu (88-1 Campaign)

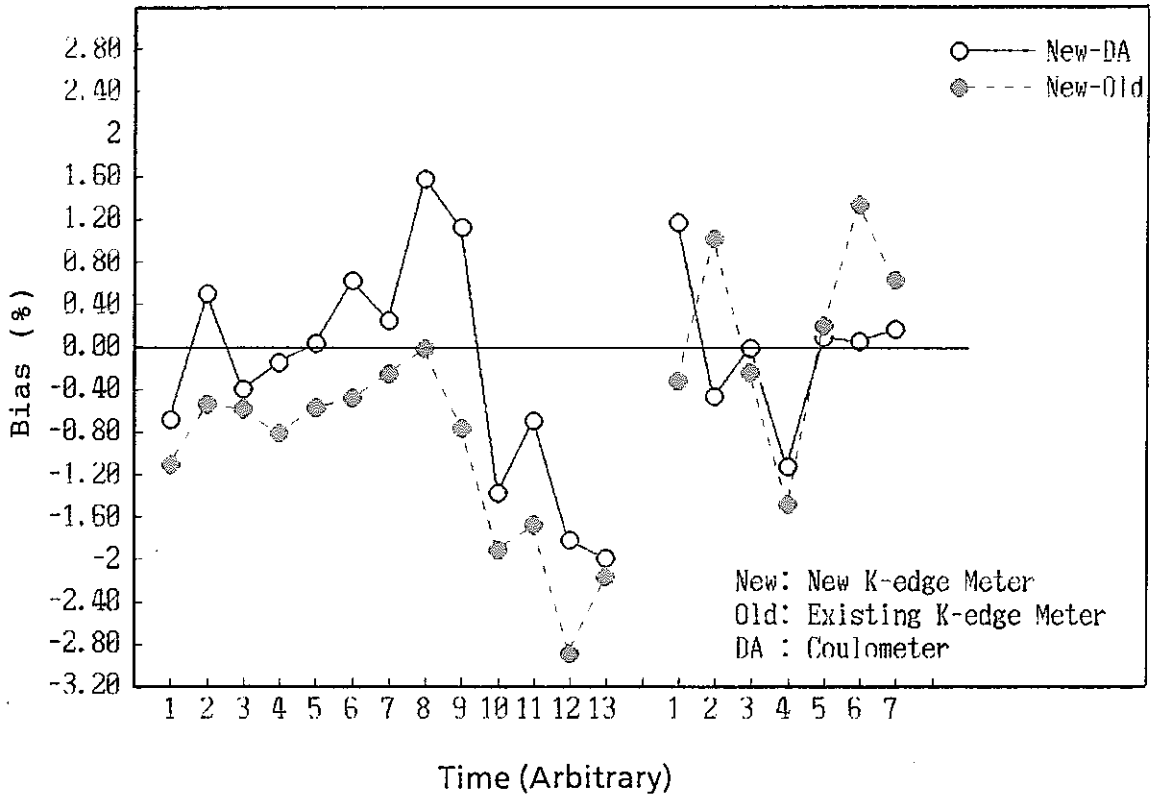


Fig. 14 Difference between New System & DA, Difference between New & Old System Aged Pu (87-2 & 88--1 Campaign)

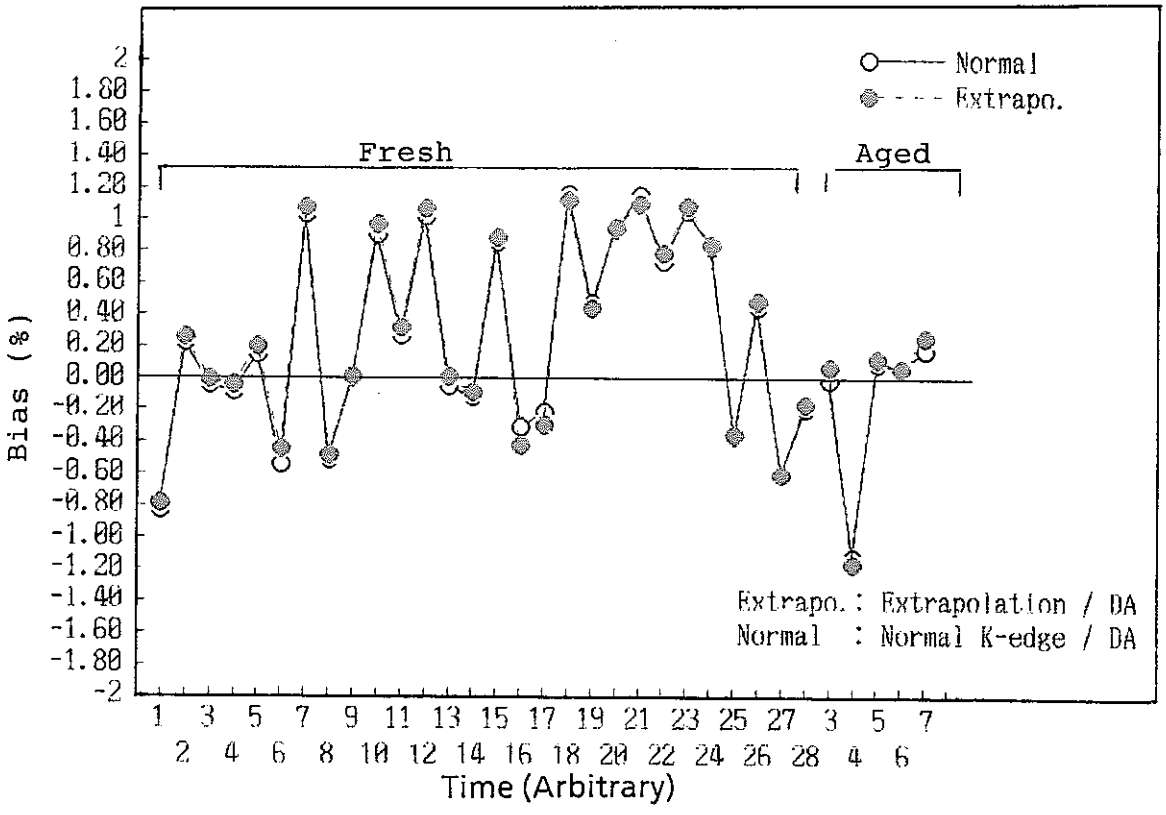


Fig. 15 Normal & Extrapolation Calculation Fresh & Aged Pu (88-1 Campaign)

Comparing the results of "New" system with those of DA, the precision of the "New" system on the mean value of three repetitions can be estimated approximately 0.7 %. On the other hand, repeatability of the system was approximately 0.4 % obtained from 14 repeated measurements as shown in Table 3. Differences of the above two estimations can result from the systematic error. With respect to the aged plutonium measurements, "Old" system used to show large systematic bias which seemed to be due to increase of count rate arising from generated Am-241. However, Fig. 14 gives no outstanding bias on the results of "New" system in spite of the fact that measurement precision of "New" system is estimated approximately 1.0 % which is worse than that obtained in the case of freshly separated plutonium. Americium effect on plutonium concentration measurement with the system was therefore examined.

Table 4 shows the results of the americium test. Here four different concentrations of americium were examined, while every plutonium concentration of the sample was same. The results show that the precision of the plutonium measurement got worse with increasing americium concentration, although no significant error was found. This phenomenon may be

Table 3. Repeatability of K-edge Densitometer

Meas. No.	Measured Data (g/ℓ)
1	244.14 ± 1.00
2	243.84 ± 1.00
3	243.42 ± 1.00
4	242.39 ± 1.00
5	241.97 ± 1.00
6	241.99 ± 1.00
7	242.21 ± 1.01
8	244.97 ± 1.01
9	244.22 ± 1.01
10	243.29 ± 1.01
11	243.74 ± 1.01
12	243.30 ± 1.01
13	245.23 ± 1.01
14	243.33 ± 1.01
Mean ± S.D.	243.43 ± 1.03 (0.42 %)

due to the increase of count rates.

Fig. 15 gives the comparison between normal calculation and extrapolation calculation on "New" system. Each plotted point is the mean of three cycle measurements. No significant difference can be observed because the plutonium treated here did not contain large amount of impurities. It could be effective on the samples which contain large amount of other elements such as uranium.

Table 4 Americium Effect on Plutonium Measurement

Measurement Number	Am-241 Concentration (g/l)			
	0 (no Am)	0.3	0.9	1.5
1	100.60	101.53	104.33	105.96
2	101.29	102.18	100.78	101.20
3	101.29	98.57	100.79	101.84
4	101.41	100.87	98.89	99.34
5	100.22	100.65	99.64	97.67
6	-	98.63	101.58	101.80
7	-	101.37	98.89	98.50
8	-	102.41	100.46	100.16
Av.	100.96	100.78	100.67	100.81
S.D. (%)	0.52	1.46	1.75	2.56

[HRGS]

(1) Measurement Control

Ba-133 source is used to monitor the ratio of counting efficiency of low-energy gamma rays relative to higher-energy ones as a function of time. Namely this measurement can verify the stability of the shape of relative efficiency curve. The peak regions used are at 31 keV (K-alpha X rays), 35 keV (K-beta X rays), 53 keV, 79-81 keV doublet, 276 keV, and 303 keV. The code determines the ratio of the various peak intensities relative to the 79-81 keV intensity and then compares these values with the corresponding intensity ratios determined at the time the system was calibrated and put into operation. Any significant differences are an indication that the shape of the efficiency curve is changing. Clock times for Ba-133 were 1800 seconds.

Fig. 16 shows the control status of the HRGS obtained over the 1988-1 campaign, where a point (No. 20) gives the possibility of change of detection efficiency on high energy relative to low energy, because both peaks of 276 keV and 303 keV show significant differences compared with the calibration data. Fig. 16, however, suggests that the system status immediately recovered. A recalibration may be necessary when this phenomenon is detected repeatedly. Some points between No. 9 and No. 21 on 53 keV give significant differences despite the fact that other close energy peaks such as 31 keV and 35 keV do not show any differences. These are therefore not supposed to indicate the shape change of the efficiency curve.

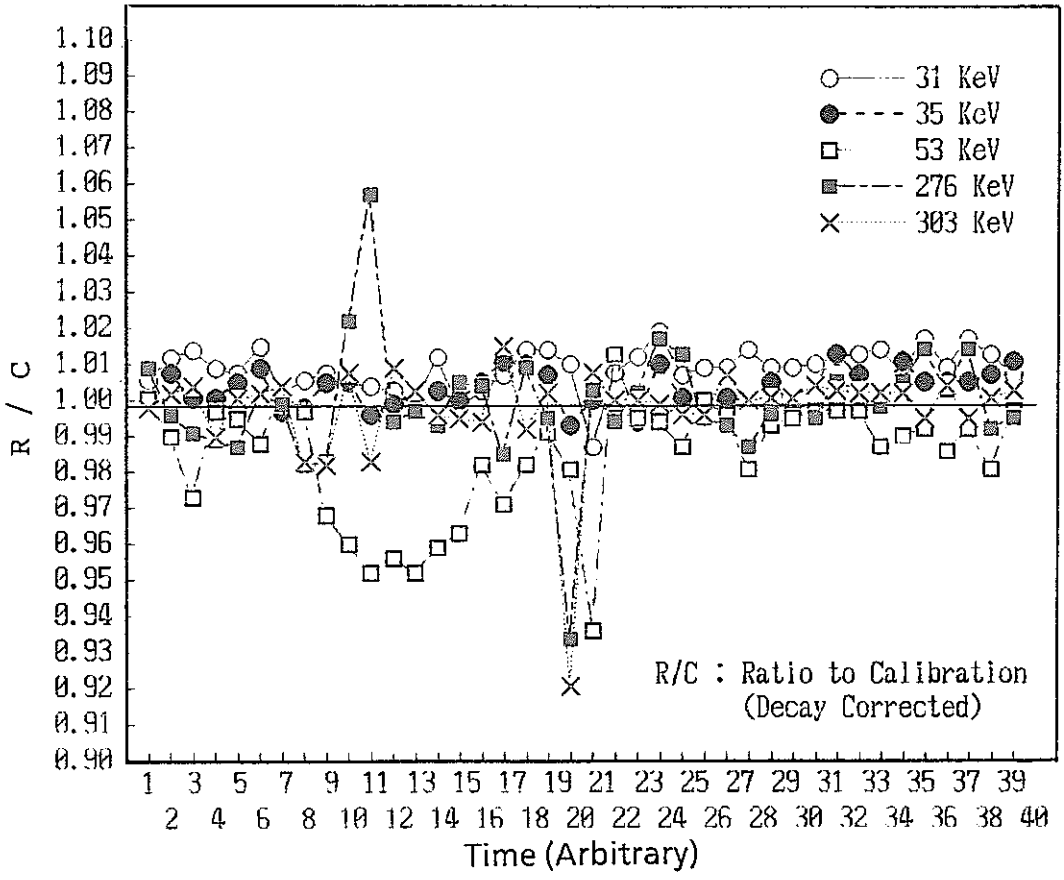


Fig. 16 Ba-133 Measurement Control

(2) Plutonium Isotopic Abundances Measurement

[Freshly Processed Plutonium]

Table 5 gives the summary results of the 1986-1 campaign, 1986-2 campaign, and 1988-1 campaign results; the means and standard deviation of the ratios of HRGS / mass spectrometer. Fig. 17 and Fig. 18 show the same ratios on each isotopic abundance obtained in the campaign of 1986-1, 1986-2 and 1988-1 respectively. Detailed raw data obtained with HRGS and mass spectrometer are

Table 5. HRGS Isotopic Measurement Results : Fresh Samples

Campaign Name	Ratio (HRGS / Mass Spectrometry)					
	Pu-Isotope	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242
1986 - 1 Campaign (5 Samples)	Average (Rel. Diff.)	1.0861 (+8.61 %)	1.0040 (+0.40 %)	1.0108 (+1.08 %)	0.9470 (-5.30 %)	- -
	CV %	17.45 %	0.30 %	0.15 %	0.23 %	-
1986 - 2 Campaign (27 Samples)	Average (Rel. Diff.)	1.0412 (+4.12 %)	0.9979 (-0.21 %)	0.9901 (-0.99 %)	0.9991 (-0.09 %)	0.9419* (-5.81 %)
	CV %	9.20 %	1.23 %	1.09 %	1.70 %	1.71 %
1988 - 1 Campaign (32 Samples)	Average (Rel. Diff.)	0.9905 (-0.95 %)	1.0083 (+0.83 %)	0.9937 (-0.63 %)	0.9814 (-1.86 %)	0.9548 (-4.52 %)
	CV %	1.60 %	0.41 %	0.50 %	1.10 %	7.96 %

* Calculated except ATR Results

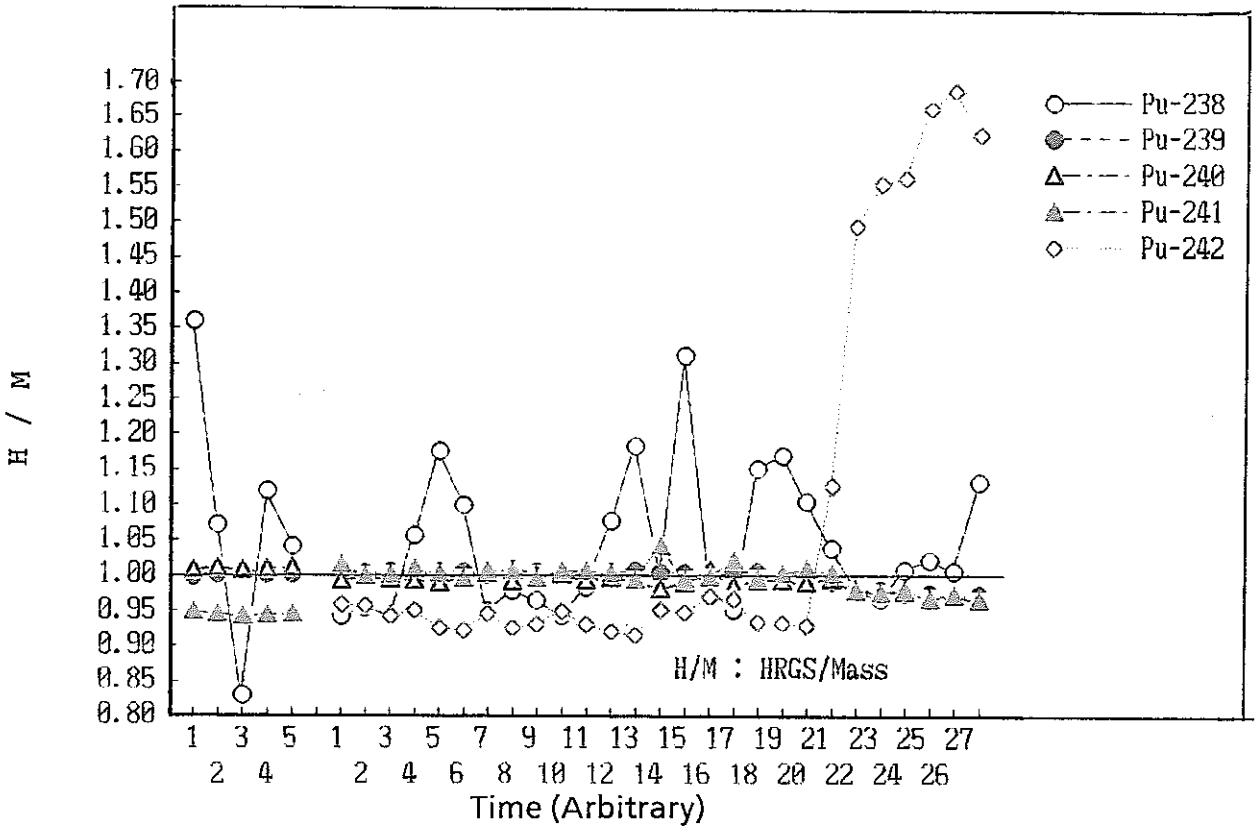


Fig. 17 Comparison between HRGS & Mass-spectrometry
Fresh Pu (86-1, 86-2 Campaign)

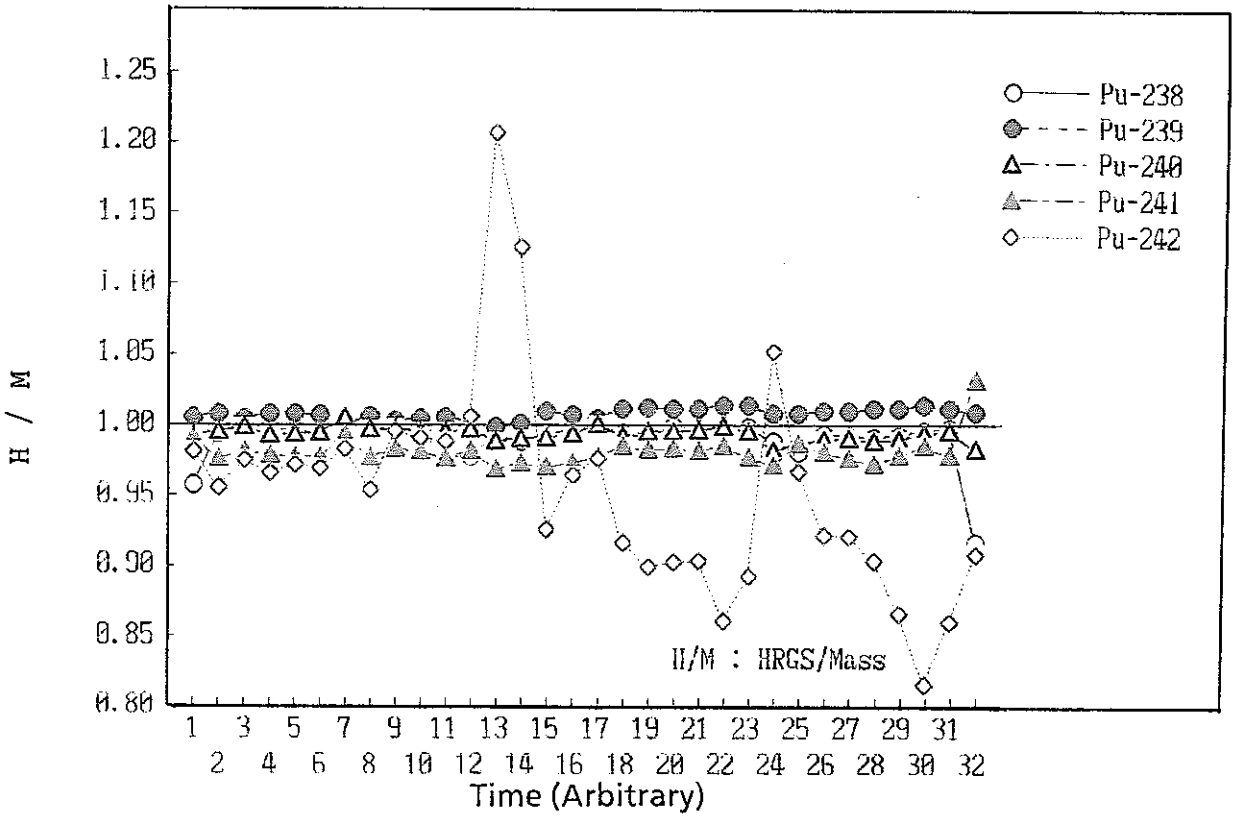


Fig. 18 Comparison between HRGS & Mass-spectrometry
Fresh Pu (88-1 Campaign)

given in Attachment 2. Clock count times for one determination by ASSAY program were 1800 seconds.

In 1986-2 campaign, the bias factors estimated from the results of previous campaign (1986-1 campaign) were used. The results of HRGS on Pu-239, Pu-240 and Pu-241 are in good agreement with those of mass spectrometry, while large scatter was observed on the results of Pu-238. The results on Pu-238 shown in Fig. 17 are larger than expected. That is probably due to the accuracy of the alpha-spectrometry technique used to determine Pu-238 at the time. With respect to Pu-242, the data obtained between No. 1 to No. 21 give negative bias, whereas the data after No. 22 show large positive bias. The former may be due to the fact that the spent fuels processed here were BWR type, which can cause negative bias, because the coefficient "53" used for the calculation of Pu-242 was approximately 2.5 % higher than the coefficient obtained by our measurements. The coefficients for BWR and PWR are estimated in Fig. 19 and 20 respectively, where the correlations were obtained from the measured results on mass spectrometry and cooling time information. The mean and standard deviation (%) of the correlation coefficient on BWR type fuel are 54.3 and 3.3 respectively, while those on PWR type fuel are 50.9 and 1.8 respectively. Pu-242 calculation is therefore expected to be improved by using separate coefficients. In latter case (after No. 22 in Fig. 17), the fuels processed at the plant were ATR (Advanced Thermal Reactor) type, where initial fuel includes plutonium. We therefore suggest that the Pu-242 isotopic correlation be modified for the thermal reactor fuels.

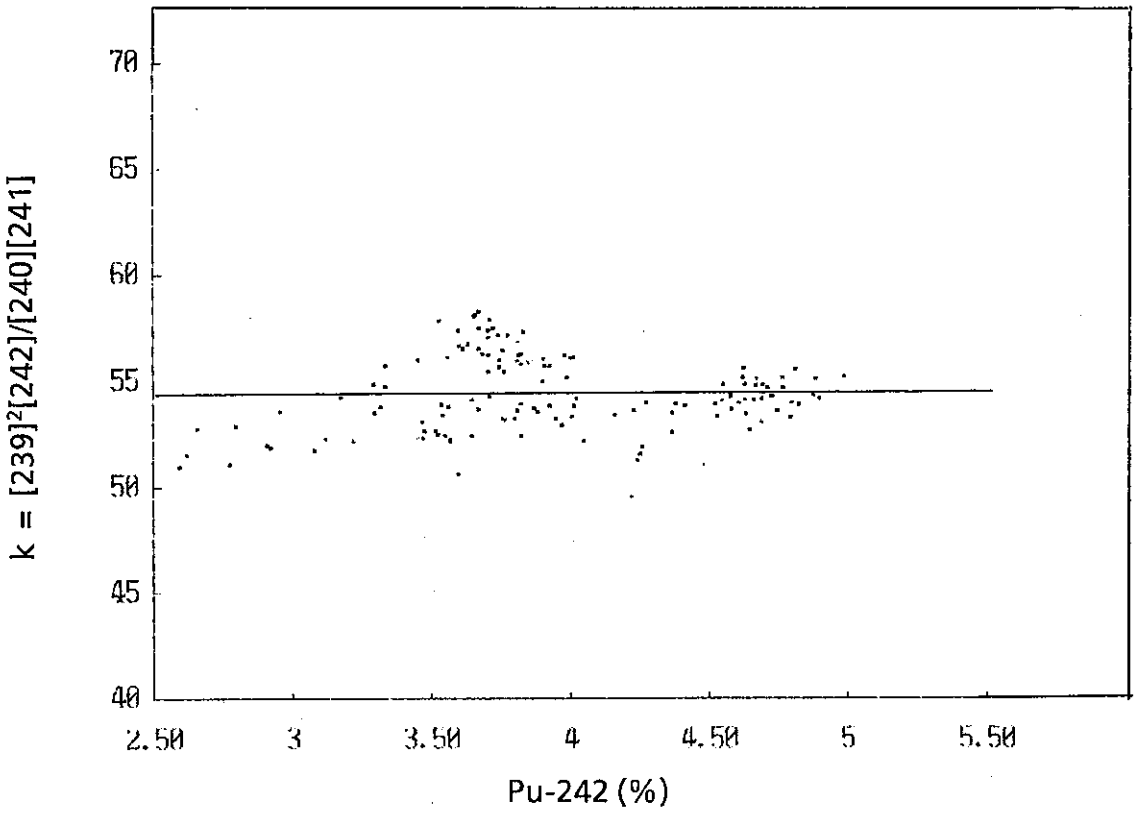


Fig. 19 Correlation Coefficient of 242 (BWR)

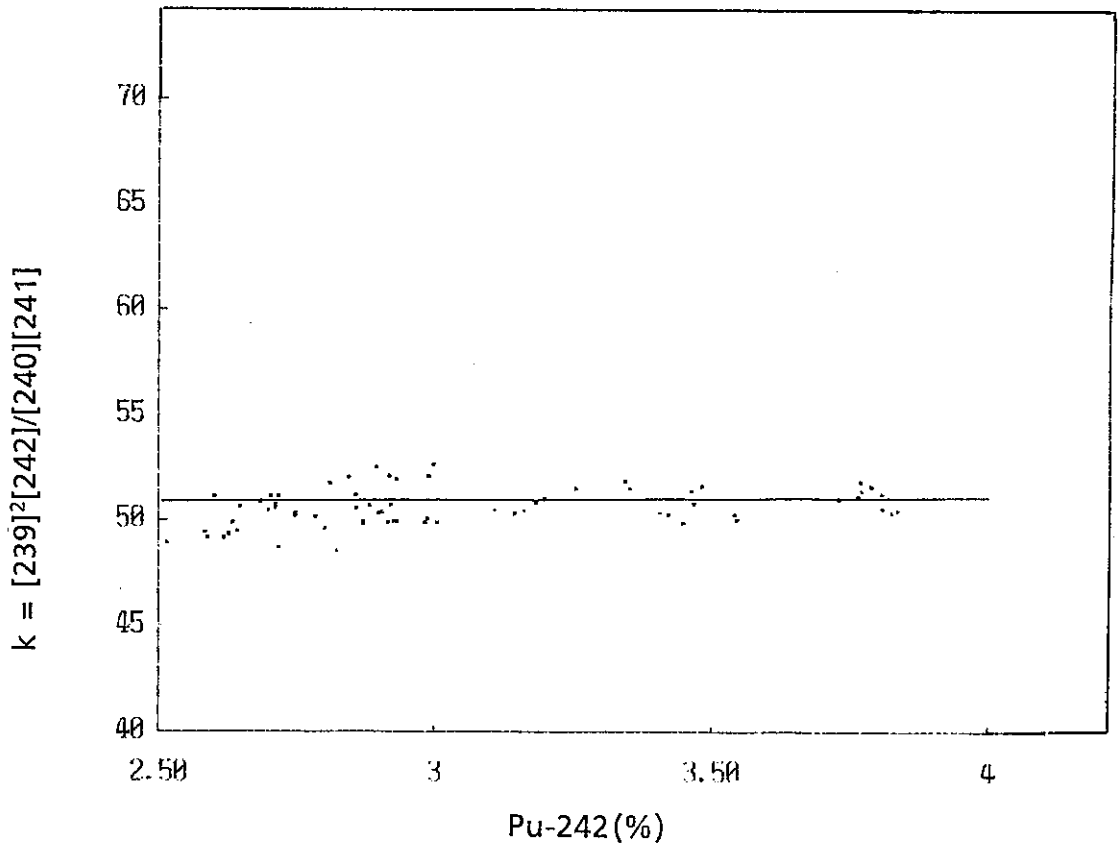


Fig. 20 Correlation Coefficient of 242 (PWR)

Prior to the 1988-1 capaign, a calibration was performed using a well characterised sample because of long interval in 1987. Every bias factor is therefore unity. Good precisions have been obtained throughout the capaign except the results of Pu-242. The fuels processed here are PWR type (No. 1 to No. 13 in Fig. 12) and BWR type (No. 14 to No. 32). Pu-242 plotted data are about 5% lower than previous data (given in Fig. 17) all over the campaign. On the other hand, Pu-240 and Pu-241 results have a few percent negative bias (approx. 1% and 3% respectively), while Pu-239 results show slightly positive bias (approx. 1%). Reviewing the equation of Pu-242 correlation;

$$[Pu242] = 53 \times [Pu240][Pu241] / [Pu239]^2 \quad \text{(original equation)},$$

Pu-242 data obtained from the first half of the 1988-1 campaign can be recalculated and estimated by following equation.

$$[Pu242] = 51 \times [Pu240 \times 1.01][Pu241 \times 1.02] / [Pu239 \times 0.99]^2 \quad \text{(PWR fuel)},$$

$$= 1.01 \times \text{(original equation)}$$

Those of second half of the campaign can be also done by next equation.

$$[Pu242] = 53 \times [Pu240 \times 1.01][Pu241 \times 1.02] / [Pu239 \times 0.99]^2 \quad \text{(BWR fuel)},$$

$$= 1.07 \times \text{(original equation)}$$

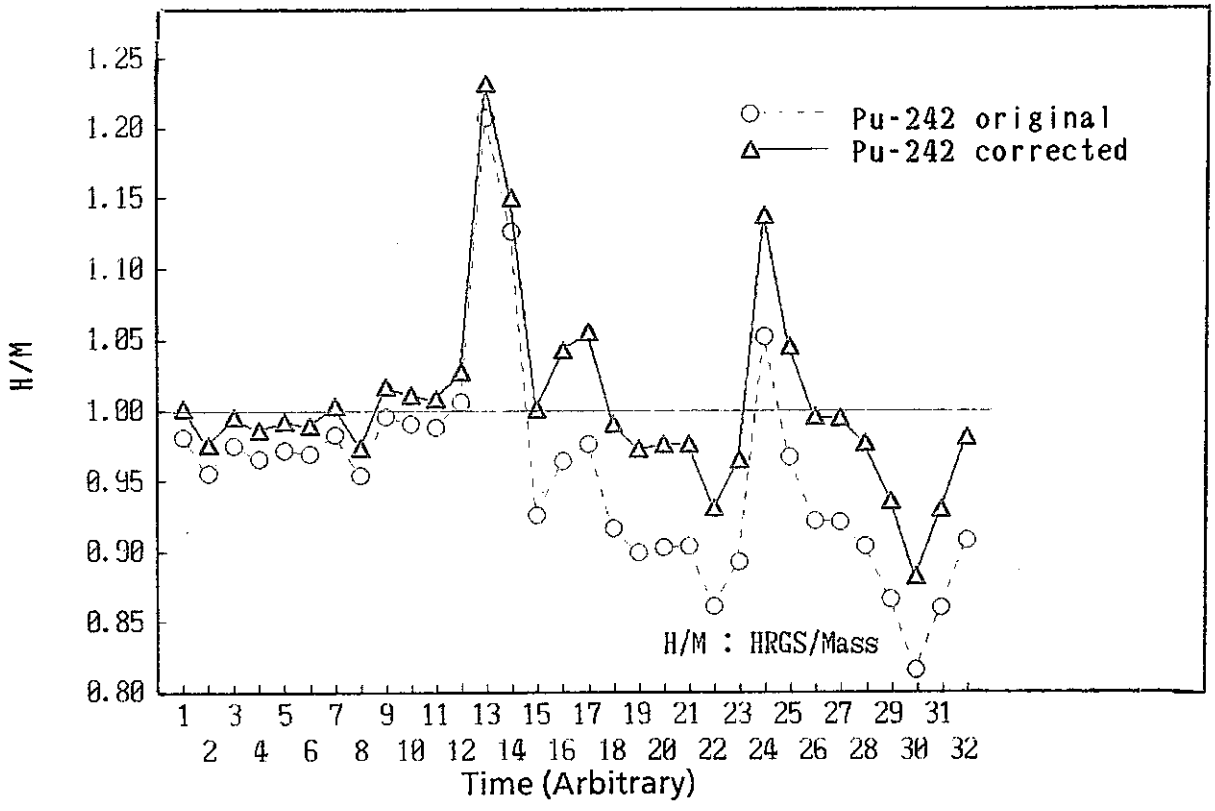


Fig. 21 Pu-242 Correction: Fresh Pu (88-1 Campaign)

Corrected Pu-242 of the campaign can be, therefore, shown in Fig. 21. Biases on Pu-240, Pu-241 and Pu-239 are suspected to be due to the errors arising from short term calibration. This suggests that longer study of bias corrections is important. A few outlying points have been detected in Fig. 18/20. These correspond to the changing of fuel types.

Table 6. Average of S.D. (%) Values (Each value is calculated from 10 repeated measurements.)

No.	Pu-238	Pu-239	Pu-240	Pu-241
1	0.193	0.222	0.334	0.561
2	0.251	0.424	0.477	1.045
3	0.914	0.550	1.114	2.756
4	0.097	0.224	0.278	0.339
5	0.462	0.441	0.515	1.333
6	0.163	0.318	0.369	1.049
7	0.166	0.173	0.211	0.257
8	0.158	0.299	0.189	0.637
9	0.412	0.335	0.444	1.384
10	0.154	0.396	0.366	0.659
11	0.207	0.249	0.214	0.646
12	0.180	0.332	0.244	0.852
13	0.114	0.413	0.309	0.695
14	0.109	0.292	0.309	0.695
15	0.764	0.547	0.843	2.136
16	0.274	0.402	0.417	0.847
17	0.287	0.312	0.322	1.313
18	0.332	0.347	0.418	1.590
Ave	0.388	0.361	0.477	1.140

Repeatability has been evaluated by repeating sample measurement 10 times. Table 6 gives the results obtained from 18 different samples, where each line (each sample result) shows standard deviation (%) of 10 repeated measurements. The values shown at the bottom of the table are the arithmetic mean of 18 data. In regard to Pu-239, 240 and 241, the estimated repeatability is nearly in accord with the relative standard deviation of 1988-1 campaign shown in Table 4.

[Aged Plutonium]

The average and the standard deviation of the ratios of HRGS / mass-spectrometry evaluated in the 1985-2, 1986-1, 1986-2 and 1988-1 campaign are shown in Table 7. Same ratios (HRGS vs mass spectrometry) obtained in the campaigns of 1985-2, 1986-1, 1986-2 and 1988-1 are plotted in Fig. 22, Fig. 23 and

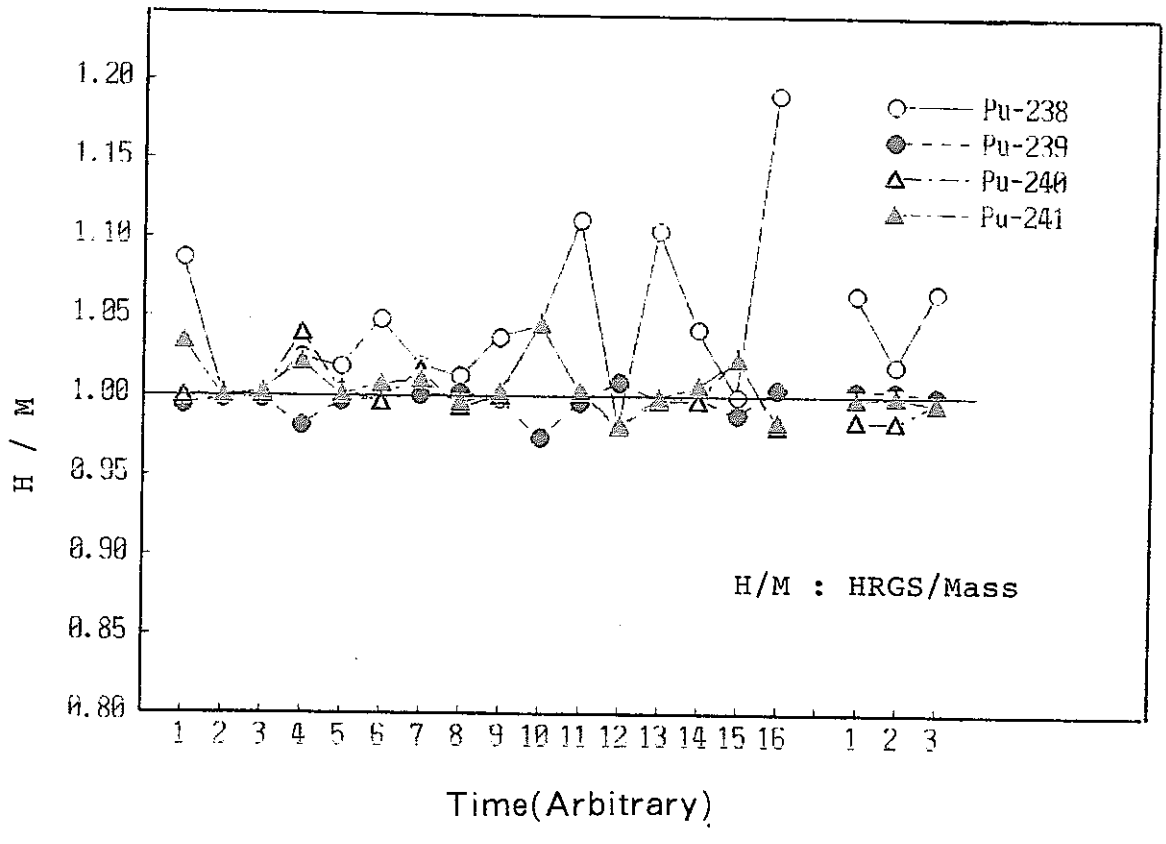


Fig. 22 Comparison between HRGS & Mass-spectrometry Aged Pu (85-2 & 86-1 Campaign)

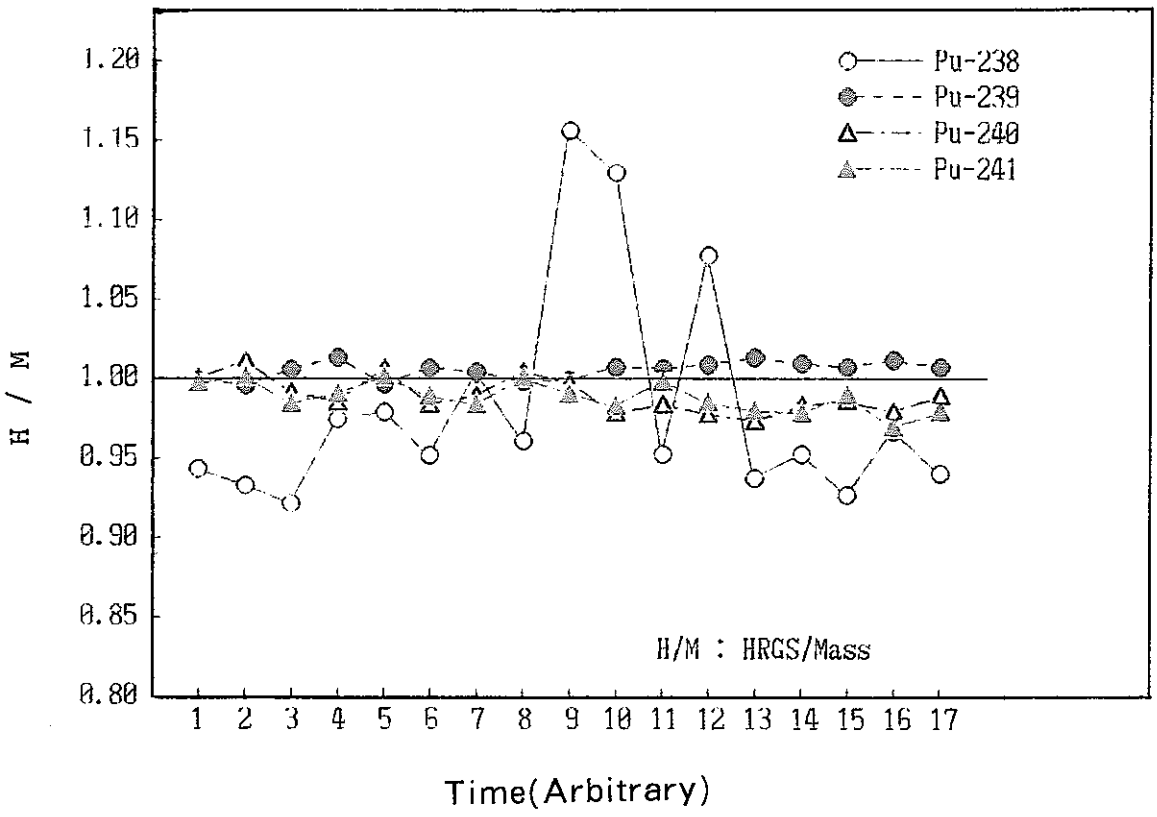


Fig. 23 Comparison between HRGS & Mass-spectrometry Aged Pu (86-2 Campaign)

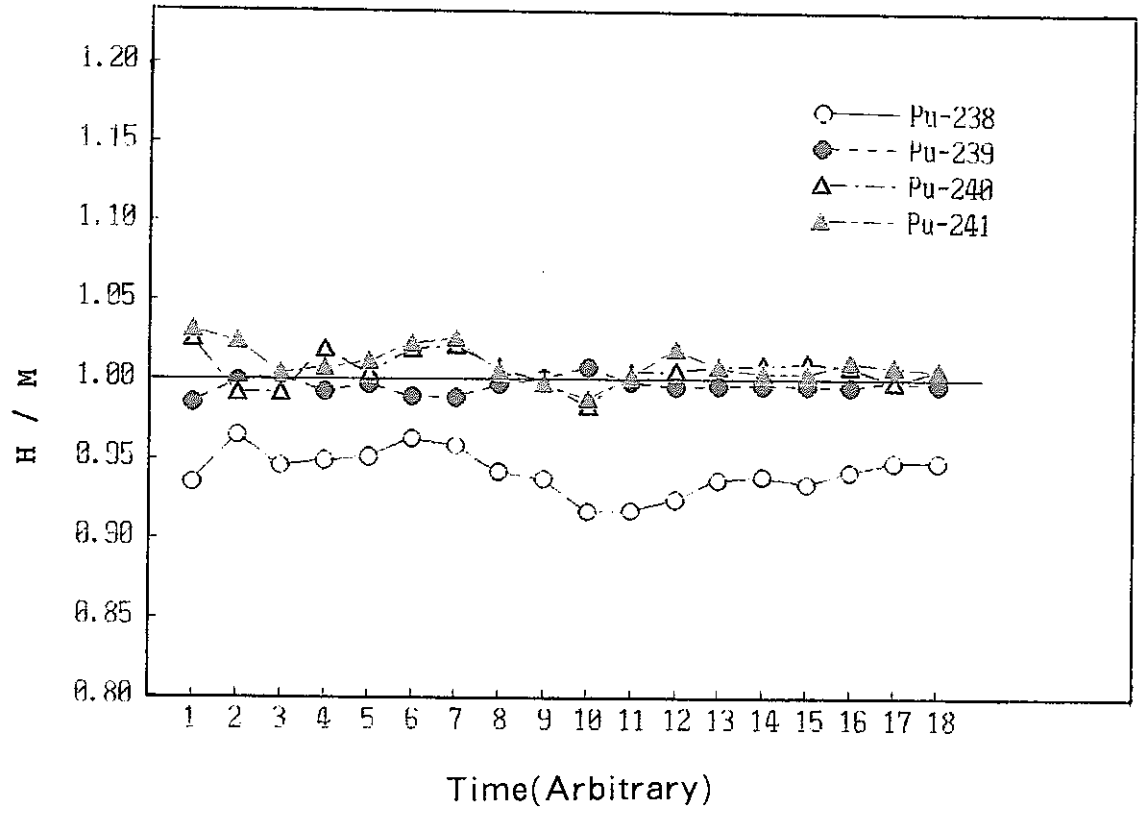


Fig. 24 Comparison between HRGS & Mass-spectrometry Aged Pu (88-1 Campaign)

Table 7. HRGS Isotopic Measurement Results : Aged Samples

Campaign Name	Ratio (HRGS / Mass Spectrometry)				
	Pu-Isotope	Pu-238	Pu-239	Pu-240	Pu-241
1985 - 2 Campaign (16 Samples)	Average (Rel. Diff.)	1.0459 (+4.59 %)	0.9967 (-0.33 %)	1.0055 (+0.55 %)	1.0082 (+0.82 %)
	CV %	5.16 %	0.85 %	1.83 %	1.66 %
1986 - 1 Campaign (4 Samples)	Average (Rel. Diff.)	1.0349 (+3.49 %)	1.0026 (+0.26 %)	0.9908 (-0.92 %)	0.9986 (-0.14 %)
	CV %	3.66 %	0.18 %	0.70 %	0.21 %
1986 - 2 Campaign (17 Samples)	Average (Rel. Diff.)	0.9837 (-1.63 %)	1.0057 (+0.57 %)	0.9901 (-0.99 %)	0.9891 (+1.09 %)
	CV %	7.17 %	0.52 %	1.11 %	0.94 %
1988 - 1 Campaign (18 Samples)	Average (Rel. Diff.)	0.9427 (-5.73 %)	0.9968 (-0.32 %)	1.0067 (+0.67 %)	1.0105 (+1.05 %)
	CV %	1.43 %	0.52 %	1.09 %	1.08 %

Fig. 24 respectively. Detailed data obtained with HRGS and mass spectrometer are given in attachment 2. Clock count times for one determination on aged plutonium measurement were 3600 seconds.

Table 8. Average of S.D. (%) Values (Each value is calculated from 10 repeated measurements.)

No.	Pu-238	Pu-239	Pu-240	Pu-241
1	0.538	0.184	0.423	0.476
2	0.662	0.351	1.026	0.660
3	0.664	0.241	0.647	0.671
4	0.880	0.381	0.889	0.970
5	0.804	0.379	0.971	0.641
6	0.859	0.477	1.214	0.711
7	0.812	0.427	1.116	0.637
8	0.997	0.345	0.789	0.815
AVE.	0.777	0.349	0.878	0.716

Fairly good agreements are observed except on the result of Pu-238. Pu-238 value on Fig. 22 and 23 shows large scatter/bias. That seems due to uncertainty of the alpha-spectrometry data like the Pu-238 scatter previously shown in Fig. 17. As for Pu-242, it is nearly impossible to calculate the cooling time for Pu-241 correction because of the mixture of plutonium products. Here, Pu-242 values determined by mass-spectrometer were therefore used for the isotopic composition calculation.

Repeatability on the aged plutonium measurement has been also evaluated by the same way as that performed for freshly processed plutonium. Table 8 shows the results obtained from 8 samples. The value given at the bottom of the table is the arithmetic mean of 8 data. Comparing the precisions obtained in the table with those estimated in the last two campaigns of the Table 7, slight differences are observed. Those can be errors based upon the mass spectrometry uncertainty.

5. CONCLUSIONS

The field test of New TASTEX System (Combined System) consisting of K-edge densitometer and high resolution gamma spectrometer has been carried out for three years. Measurement performance has been improved at the last campaign as compared with that of initial campaign.

Precisions of plutonium concentration determination with the K-edge densitometer were estimated approximately 0.7% and 1.0% for freshly processed plutonium and aged plutonium respectively. It should be noted that short term systematic bias is possibly detected. That sometimes causes larger errors than expected. The precision of plutonium concentration measurment tends to be affected by the presence of americium.

The scatters in the relative differences between HRGS and DA detected on the results of freshly processed plutonium measurements were 1.6%, 0.4%, 0.5%, 1.1% and 8.0% for Pu-238, Pu-239, Pu-240, Pu-241 and Pu-242 respectively. Those on aged plutonium were 1.4%, 0.5%, 1.1%, and 1.1% for Pu-238, Pu-239, Pu-240 and Pu-241 respectively. With regard to Pu-242 determination, following points should be noted.

1) It is expected that the Pu-242 determination can be improved by discriminating between the correlation coefficients of BWR and PWR.

2) Isotopic correlation for Pu-242 estimation can be, effective especially in the case fuel type is not changed throughout the campaign. In contrast, plutonium product samples corresponding to the transition of fuel types can give outstanding error.

3) The correlation can neither be applied to the fuel containing plutonium initially such as ATR fuel, nor plutonium storage of reprocessing plant.

It does not seem to be necessary that calibration for K-edge densitometer be repeated periodically as far as Ta-foil measurement control provides good performance. Instead, just when series of Ta-foil measurements give biased results, recalibration of the system should be performed. With respect to HRGS, Ba check source can give similar information. However, precise measurement requires long term bias correction, namely it is desired that bias correction factor for each isotope be evaluated from previous campaign results. Calibration for HRGS should be performed in the case either Ba check source shows significant bias or long term bias correction factors turn to large value.

NDA system such as New TASTEX System can easily give results when system is in good control status. However this also means that even NDA system must be always checked and controlled by the operator who is fully knowledgeable about the system according to our years' experience of insitu NDA measurement.

ACKNOWLEDGEMENT

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Attachment 1 Data of K-edge Densitometer Measurement

(1) 85-2 Campaign : Fresh Sample

No.	Sample ID	K-edge (New)	Extrapol.	Coulometry	Old K-edge	K-edge/ Coulo.	Extr./ Coulo	K-edge New/Old
1	PUA224	219.066	-	215.37	219.894	1.01716	-	0.99623
2	PUA226	209.255	-	209.72	211.031	0.99778	-	0.99158
3	PUA227	203.846	-	204.34	205.609	0.99758	-	0.99143
4	PUA242	222.602	-	218.64	219.971	1.01853	-	1.01237
5	PUA243	204.652	-	201.16	201.862	1.01736	-	1.01382
6	PUA244	227.902	-	226.11	227.638	1.00793	-	1.00116
7	PUA245	222.785	-	221.609	224.004	1.00531	-	0.99456
8	PUA246	204.234	-	204.275	200.701	0.99980	-	1.01760
9	PUA247	213.062	-	212.772	213.943	1.00136	-	0.99588
10	PUA248	214.677	-	214.778	214.911	0.99953	-	0.99891
11	PUA249	211.918	-	214.069	211.306	0.98995	-	1.00290
12	PUA250	206.480	-	206.385	205.923	1.00046	-	1.00270
13	PUA251	203.932	-	203.774	201.702	1.00078	-	1.01106
14	PUA252	215.679	-	215.150	213.792	1.00246	-	1.00883
15	PUA253	187.189	-	187.270	188.830	0.99957	-	0.99131
16	PUA254	204.265	-	204.168	207.256	1.00048	-	0.98557
17	PUA255	221.171	-	221.108	226.825	1.00028	-	0.97507
18	PUA256	206.368	-	206.212	208.380	1.00076	-	0.99034
19	PUA257	222.950	-	222.240	226.472	1.00319	-	0.98445
20	PUA258	206.756	-	206.408	207.408	1.00169	-	0.99686
21	PUA259	213.329	-	212.019	216.863	1.00618	-	0.98370
22	PUA260	186.825	-	187.863	191.791	0.99447	-	0.97411
					Average	1.00284	-	0.99638
					CV%	0.707 %	-	1.194%

(2) 86-1 Campaign : Fresh Sample

No.	Sample ID	K-edge (New)	Extrapol.	Coulometry	Old K-edge	K-edge/ Coulo.	Extr./ Coulo	K-edge New/Old
1	PUA263	219.123	-	219.7	218.327	0.99737	-	1.00365
2	PUA264	217.503	-	219.0	220.551	0.99316	-	0.98618
3	PUA265	201.786	-	204.4	203.639	0.98721	-	0.99090
4	PUA266	207.886	-	209.0	208.988	0.99467	-	0.99473
5	PUA267	212.958	-	215.3	213.835	0.98912	-	0.99590
6	PUA268	109.706	-	110.8	110.471	0.99013	-	0.99308
					Average	0.99194	-	0.99407
					CV%	0.383%	-	0.584%

(3) 88-1 Campaign : Fresh Sample

No.	Sample ID	K-edge (New)	Extrapol.	Coulometry	Old K-edge	K-edge/ Coulo.	Extr./ Coulo	K-edge New/Old
1	PUA373	168.445	168.523	169.845	168.074	0.99176	0.99222	1.00221
2	PUA374	206.088	206.086	205.530	205.421	1.00271	1.00271	1.00325
3	PUA375	219.918	220.044	219.993	220.734	0.99966	1.00023	0.99630
4	PUA376	210.305	210.407	210.478	212.576	0.99918	0.99966	0.98932
5	PUA377	221.546	221.672	221.206	224.341	1.00154	1.00211	0.98754
6	PUA378	196.325	196.520	197.374	198.984	0.99469	0.99567	0.98664
7	PUA379	208.341	208.443	206.195	208.120	1.01041	1.01090	1.00106
8	PUA380	224.499	224.559	225.628	227.426	0.99500	0.99526	0.98713
9	PUA381	226.158	226.178	226.133	225.551	1.00011	1.00020	1.00269
10	PUA382	225.344	225.489	223.293	225.300	1.00919	1.00983	1.00020
11	PUA383	230.428	230.550	229.792	229.686	1.00277	1.00330	1.00323
12	PUA384	229.532	229.645	227.200	228.900	1.01026	1.01076	1.00276
13	PUA385	215.679	215.816	215.770	216.610	0.99958	1.00021	0.99570
14	PUA386	211.137	211.196	211.362	212.423	0.99894	0.99921	0.99395
15	PUA387	217.228	217.301	215.361	216.970	1.00867	1.00901	1.00119
16	PUA388	231.369	231.116	232.071	234.510	0.99698	0.99588	0.98661
17	PUA389	218.759	218.585	219.215	217.681	0.99792	0.99713	1.00495
18	PUA391	215.281	215.210	212.794	213.451	1.01169	1.01135	1.00857
19	PUA394	201.284	201.219	200.310	201.764	1.00486	1.00454	0.99762
20	PUA395	203.099	203.025	201.096	200.434	1.00996	1.00959	1.01330
21	PUA396	230.788	230.679	228.140	231.860	1.01161	1.01113	0.99538
22	PUA397	207.142	207.249	205.603	206.907	1.00749	1.00801	1.00114
23	PUA398	200.776	200.840	198.662	198.711	1.01064	1.01096	1.01039
24	PUA399	215.684	215.718	213.897	213.118	1.00835	1.00851	1.01204
25	PUA400	217.511	217.527	218.273	218.546	0.99651	0.99658	0.99526
26	PUA401	213.579	213.659	212.606	213.488	1.00458	1.00495	1.00043
27	PUA402	213.307	213.313	214.687	216.599	0.99357	0.99360	0.98480
28	PUA403	206.545	206.609	206.933	209.701	0.99812	0.99843	0.98495
					Average	1.00274	1.00293	0.99816
					CV%	0.614%	0.614%	0.829%

(4) Pu-product Storage (1987) : Aged sample

No.	Sample ID	K-edge (New)	Extrapol.	Coulometry	Old K-edge	K-edge/ Coulo.	Extr./ Coulo	K-edge New/Old
1	(Jul.) 267V10	194.192		195.5	196.346	0.99331		0.98903
2	267V12	238.605		237.4	239.874	1.00508		0.99471
3	267V13	229.101		230.0	230.418	0.99609		0.99428
4	267V14	205.905		206.2	207.549	0.99857		0.99208
5	267V15	222.904		222.8	224.158	1.00047		0.99441
6	267V16	232.269		230.8	233.370	1.00636		0.99528
7	(Sep.) 267V10	172.471		172.03	172.877	1.00256		0.99765
8	267V11	235.791		232.10	235.785	1.01590		1.00003
9	267V12	239.286		236.59	241.085	1.01140		0.99254
10	267V13	227.632		230.78	232.051	0.98636		0.98096
11	267V14	214.186		215.67	217.792	0.99312		0.98344
12	267V15	229.263		233.48	236.065	0.98194		0.97119
13	267V16	225.253		229.79	230.197	0.98026		0.97852
					Average	0.99780		0.98955
					CV%	1.084%		0.859%

(5) Pu-product Storage (1988) : Aged sample

No.	Sample ID	K-edge (New)	Extrapol.	Coulometry	Old K-edge	K-edge/ Coulo.	Extr./ Coulo	K-edge New/Old
1	(Jan.) 267V13	223.298		220.688	223.991	1.01183		0.99691
2	267V15	173.839		174.636	172.060	0.99544		1.01034
3	(Feb.) 267V10	199.484		199.487	199.949	0.99998		0.99767
4	267V11	225.769		228.320	229.131	0.98882		0.98533
5	267V13	225.973		225.734	225.487	1.00106		1.00216
6	267V14	237.781		237.618	234.619	1.00069		1.01348
7	267V15	177.458		177.135	176.319	1.00182		1.00646
					Average	0.99995		1.00176
					CV%	0.696%		0.950%

Attachment 2 Raw Data of HRGS Measurement

(1) 86-1 Campaign: Fresh Sample

No. 1/1

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	1.071		64.665		21.325		9.744		~~(3.195)~~	
	PUF262	Assay (2)	1.080		64.265		21.622		9.838			
		HRGS Av.	1.0755	1.36139	64.465	1.00034	21.4735	1.00947	9.791	0.95058	~~(3.195)~~	1.
		Mass Spec.	0.790		64.443		21.272		10.300			
2		Assay (1)	1.009		65.350		21.012		9.646		~~(2.982)~~	
	PUF264	Assay (2)	1.017		65.348		20.994		9.658			
		HRGS Av.	1.013	1.07423	65.349	1.00349	21.003	1.01185	9.652	0.94665	~~(2.982)~~	1.
		Mass Spec.	0.943		65.122		20.757		10.196			
3		Assay (1)	0.979		65.689		20.690		9.725		(2.917)	
	PUF265	Assay (2)	0.978		65.764		20.669		9.672			
		Assay (3)	0.978		65.718		20.622		9.766			
		HRGS Av.	0.9783	0.83189	65.7237	1.00887	20.6603	1.00930	9.721	0.94461	~~(2.917)~~	1.
		Mass Spec.	1.176		65.146		20.470		10.291			
4		Assay (1)	0.996		65.397		20.854		9.824		(2.929)	
	PUF266	Assay (2)	0.989		65.659		20.677		9.745			
		Assay (3)	0.994		65.471		20.717		9.888			
		HRGS Av.	0.993	1.12077	65.509	1.00362	20.7493	1.01049	9.819	0.94614	~~(2.929)~~	1.
		Mass Spec.	0.886		65.273		20.534		10.378			
5		Assay (1)	1.050		64.802		21.015		10.045		(3.087)	
	PUF267	Assay (2)	1.053		64.743		21.112		10.006			
		Assay (3)	1.049		64.813		21.111		9.941			
		HRGS Av.	1.0507	1.04236	64.786	1.00387	21.0793	1.01294	9.9973	0.94680	~~(3.087)~~	1.
		Mass Spec.	1.008		64.536		20.810		10.559			
		Average	1.08613		1.00404		1.01081		0.94696			1.
		CV%	17.449%		0.304%		0.154%		0.233%			

(2) 86-2 Campaign : Fresh Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	1.032		62.844		24.372		8.444		3.308	
	PUF286	Assay (2)	1.028		63.070		24.266		8.387		3.249	
		HRGS Av.	1.030	0.94409	62.957	1.00311	24.319	0.99391	8.4155	1.01796	3.2785	0.96087
		Mass Spec.	1.091		62.762		24.468		8.267		3.412	
2		Assay (1)	1.065		62.733		24.683		8.207		3.312	
	PUF287	Assay (2)	1.074		62.559		24.799		8.217		3.351	
		HRGS Av.	1.0695	0.95662	62.646	1.00352	24.741	0.99839	8.212	1.00098	3.3315	0.95981
		Mass Spec.	1.118		62.426		24.781		8.204		3.471	
3		Assay (1)	1.076		62.484		24.918		8.200		3.322	
	PUF288	Assay (2)	1.077		62.757		24.751		8.159		3.256	
		HRGS Av.	1.0765	0.94513	62.6205	1.00556	24.8345	0.99597	8.1795	1.00165	3.289	0.94349
		Mass Spec.	1.139		62.274		24.935		8.166		3.486	
4		Assay (1)	1.067		62.211		25.072		8.255		3.395	
	PUF289	Assay (2)	1.066		62.369		25.044		8.177		3.344	
		HRGS Av.	1.0665	1.05909	62.290	1.00314	25.058	0.99314	8.216	1.01083	3.3695	0.95211
		Mass Spec.	1.007		62.095		25.231		8.128		3.539	
5		Assay (1)	1.177		60.835		25.025		9.115		3.848	
	PUF290	Assay (2)	1.176		60.561		25.228		9.117		3.918	
		HRGS Av.	1.1765	1.17768	60.698	1.00540	25.1265	0.99068	9.116	1.00419	3.883	0.92717
		Mass Spec.	0.999		60.372		25.363		9.078		4.188	
6		Assay (1)	1.243		59.353		25.677		9.432		4.295	
	PUF291	Assay (2)	1.244		59.616		25.492		9.424		4.224	
		HRGS Av.	1.2435	1.10044	59.4845	1.00581	25.5845	0.99717	9.428	0.99672	4.2595	0.92337
		Mass Spec.	1.130		59.141		25.657		9.459		4.613	
7		Assay (1)	1.276		58.738		25.800		9.668		4.518	
	PUF292	Assay (2)	1.270		58.930		25.785		9.574		4.441	
		HRGS Av.	1.273	0.95071	58.834	1.00274	25.7925	1.00372	9.621	1.00649	4.4795	0.94664
		Mass Spec.	1.339		58.673		25.697		9.559		4.732	
8		Assay (1)	1.266		59.236		25.511		9.618		4.369	
	PUF293	Assay (2)	1.275		59.136		25.401		9.759		4.429	
		HRGS Av.	1.2705	0.97957	59.186	1.00840	25.456	0.99244	9.6885	1.00764	4.399	0.92708
		Mass Spec.	1.297		58.693		25.650		9.615		4.745	

(2) 86-2 Campaign : Fresh Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
9		Assay (1)	1.292		58.609		25.971		9.596		4.532	
	PUF294	Assay (2)	1.298		58.646		25.912		9.617		4.527	
		HRGS Av.	1.295	0.96570	58.6275	1.00588	25.9415	1.00226	9.6065	0.99694	4.5295	0.93296
		Mass Spec.	1.341		58.285		25.883		9.636		4.855	
10		Assay (1)	1.296		58.540		26.087		9.540		4.537	
	PUF295	Assay (2)	1.299		58.080		26.434		9.523		4.664	
		HRGS Av.	1.2975	0.94432	58.310	1.00332	26.2605	1.00231	9.5315	1.00649	4.6005	0.95071
		Mass Spec.	1.374		58.117		26.200		9.470		4.839	
11		Assay (1)	1.289		58.949		25.693		9.623		4.446	
	PUF296	Assay (2)	1.295		58.922		25.714		9.618		4.451	
		HRGS Av.	1.292	0.98476	58.9355	1.00770	25.7035	0.99364	9.6205	1.00633	4.4485	0.93162
		Mass Spec.	1.312		58.485		25.868		9.560		4.775	
12		Assay (1)	1.250		59.782		24.893		9.888		4.187	
	PUF297	Assay (2)	1.253		59.701		25.013		9.837		4.196	
		HRGS Av.	1.2515	1.07888	59.7415	1.00528	24.953	0.99621	9.8625	1.00433	4.1915	0.92243
		Mass Spec.	1.160		59.428		25.048		9.820		4.544	
13		Assay (1)	1.186		60.050		24.923		9.828		4.013	
	PUF298	Assay (2)	1.186		59.958		25.018		9.807		4.031	
		HRGS Av.	1.186	1.18363	60.004	1.00671	24.9705	0.99294	9.8175	0.99559	4.022	0.91722
		Mass Spec.	1.002		59.604		25.148		9.861		4.385	
14		Assay (1)	1.092		60.189		24.483		10.206		4.030	
	PUF299	Assay (2)	1.092		60.337		24.334		10.239		3.998	
		HRGS Av.	1.092	1.00460	60.263	1.00428	24.4085	0.98069	10.2225	1.04237	4.014	0.95322
		Mass Spec.	1.087		60.006		24.889		9.807		4.211	
15		Assay (1)	1.211		59.205		25.205		10.044		4.335	
	PUF300	Assay (2)	1.205		59.468		25.109		9.971		4.247	
		HRGS Av.	1.208	1.31162	59.3365	1.00414	25.157	0.99016	10.0075	0.99448	4.291	0.94997
		Mass Spec.	0.921		59.092		25.407		10.063		4.517	
16		Assay (1)	1.227		59.206		25.085		10.213		4.269	
	PUF302	Assay (2)	1.228		59.349		24.961		10.228		4.427	
		HRGS Av.	1.2275	0.98436	59.2775	0.99866	25.023	1.00928	10.2205	0.99917	4.2515	0.97199
		Mass Spec.	1.247		59.357		24.793		10.229		4.374	

(2) 86-2 Campaign : Fresh Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
17		Assay (1)	1.084		60.644		24.150		10.175		3.947	
	PUF303	Assay (2)	1.087		60.617		24.253		10.104		3.939	
		HRGS Av.	1.0855	0.95303	60.6305	1.00506	24.2015	0.98564	10.1395	1.02306	3.943	0.96856
		Mass Spec.	1.139		60.325		24.554		9.911		4.701	
18		Assay (1)	1.119		60.300		24.740		9.904		3.937	
	PUF304	Assay (2)	1.118		60.483		24.668		9.851		3.880	
		HRGS Av.	1.1185	1.15191	60.3915	1.00539	24.704	0.99325	9.8775	0.99692	3.9085	0.93482
		Mass Spec.	0.971		60.068		24.872		9.908		4.181	
19		Assay (1)	0.933		62.502		24.057		9.150		3.358	
	PUF305	Assay (2)	0.932		62.462		24.115		9.128		3.363	
		HRGS Av.	0.9325	1.17001	62.482	1.00366	24.086	0.99356	9.139	1.00351	3.3605	0.93347
		Mass Spec.	0.797		62.254		24.242		9.107		3.600	
20		Assay (1)	0.884		63.045		23.796		9.047		3.228	
	PUF306	Assay (2)	0.887		63.113		23.758		9.032		3.210	
		HRGS Av.	0.8855	1.10549	63.079	1.00460	23.777	0.99058	9.0395	1.01068	3.219	0.92981
		Mass Spec.	0.801		62.790		24.003		8.944		3.462	
21		Assay (1)	0.742		59.280		27.506		8.478		3.994	
	PUF307	Assay (2)	0.741		59.087		27.665		8.469		4.038	
		HRGS Av.	0.7415	1.03997	59.1835	0.99320	27.5855	0.99572	8.4735	1.00456	4.016	ATR (1.12841)*
		Mass Spec.	0.713		59.589		27.704		8.435		3.559	
22		Assay (1)	0.575		54.402		31.727		7.691		5.605	
	PUF308	Assay (2)	0.575		54.533		31.584		7.728		5.580	
		HRGS Av.	0.575	0.98123	54.4675	0.98050	31.6555	0.98163	7.7095	0.97923	5.5925	ATR (1.49452)*
		Mass Spec.	0.586		55.551		32.248		7.873		3.742	
23		Assay (1)	0.532		53.467		32.599		7.521		5.881	
	PUF309	Assay (2)	0.532		53.530		32.515		7.549		5.874	
		HRGS Av.	0.532	0.96727	53.4985	0.98017	32.557	0.97561	7.535	0.97667	5.8775	ATR (1.55366)*
		Mass Spec.	0.550		54.581		33.371		7.715		3.783	
24		Assay (1)	0.527		53.054		32.931		7.548		5.940	
	PUF310	Assay (2)	0.526		53.018		32.942		7.557		5.957	
		HRGS Av.	0.5265	1.00862	53.036	0.97697	32.9365	0.97769	7.5525	0.98135	5.9485	ATR (1.56211)*
		Mass Spec.	0.522		54.286		33.688		7.696		3.808	

(2) 86-2 Campaign: Fresh Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
25		Assay (1)	0.573		51.967		33.007		7.800		6.653	
	PUF311	Assay (2)	0.572		52.064		32.934		7.808		6.622	
		HRGS Av.	0.5725	1.02232	52.0155	0.97464	32.9705	0.96946	7.804	0.96728	6.6375	ATR (1.66187)*
		Mass Spec.	0.560		53.369		34.009		8.068		3.994	
26		Assay (1)	0.588		51.884		32.856		7.923		6.749	
	PUF312	Assay (2)	0.589		51.441		33.187		7.882		6.901	
		HRGS Av.	0.5885	1.00771	51.6625	0.97048	33.0215	0.97074	7.9025	0.97345	6.825	ATR (1.68643)*
		Mass Spec.	0.584		53.234		34.017		8.118		4.047	
27		Assay (1)	0.628		51.230		33.172		7.953		7.017	
	PUF313	Assay (2)	0.628		51.023		33.185		8.024		7.140	
		HRGS Av.	0.628	1.13357	51.1265	0.97376	33.1785	0.96702	7.9885	0.96561	7.0785	ATR (1.62388)*
		Mass Spec.	0.554		52.504		34.310		8.273		4.359	
		Average	1.04120		0.99785		0.99014			0.99906		0.9419*
		CV%	9.204%		1.233%		1.089%			1.697%		1.708%

* Calculated except ATR Results

(3) 88-1 Campaign : Fresh Sample

No. 1/5

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	1.152		62.462		21.871		10.885		3.630	
	PUF373	Assay (2)	1.157		62.609		21.854		10.799		3.581	
		HRGS Av.	1.1545	0.95809	62.5355	1.00604	21.8625	0.99271	10.842	0.99095	3.6055	0.98216
		Mass Spec.	1.205		62.160		22.023		10.941		3.671	
2		Assay (1)	1.165		62.518		22.037		10.693		3.587	
	PUF374	Assay (2)	1.156		62.788		21.903		10.637		3.516	
		HRGS Av.	1.1605	0.99358	62.653	1.00839	21.970	0.99542	10.665	0.97692	3.5515	0.95676
		Mass Spec.	1.168		62.132		22.071		10.917		3.712	
3		Assay (1)	1.192		62.176		21.988		10.942		3.702	
	PUF375	Assay (2)	1.193		62.127		22.059		10.911		3.710	
		HRGS Av.	1.1925	1.00126	62.1515	1.00484	22.0235	0.99943	10.9265	0.98225	3.706	0.97603
		Mass Spec.	1.191		61.852		22.036		11.124		3.797	
4		Assay (1)	1.202		62.182		21.931		10.980		3.705	
	PUF376	Assay (2)	1.204		62.230		21.915		10.961		3.690	
		HRGS Av.	1.203	0.99834	62.206	1.00833	21.923	0.99320	10.9705	0.97907	3.6975	0.96667
		Mass Spec.	1.205		61.692		22.073		11.205		3.825	
5		Assay (1)	1.215		61.981		21.977		11.062		3.765	
	PUF377	Assay (2)	1.214		62.088		22.015		10.960		3.723	
		HRGS Av.	1.2145	0.99631	62.0345	1.00800	21.996	0.99403	11.011	0.97763	3.744	0.97297
		Mass Spec.	1.219		61.542		22.128		11.263		3.848	
6		Assay (1)	1.173		62.274		21.948		10.927		3.678	
	PUF378	Assay (2)	1.170		62.750		21.722		10.810		3.548	
		HRGS Av.	1.1715	0.99448	62.512	1.00777	21.835	0.99472	10.8685	0.97765	3.613	0.97019
		Mass Spec.	1.178		62.030		21.951		11.117		3.724	
7		Assay (1)	1.118		62.779		21.888		10.685		3.530	
	PUF379	Assay (2)	1.119		62.943		21.915		10.551		3.472	
		HRGS Av.	1.1185	1.00134	62.861	0.99984	21.9015	1.00604	10.618	0.99401	3.501	0.98343
		Mass Spec.	1.117		62.871		21.770		10.682		3.560	
8		Assay (1)	0.946		64.315		21.626		10.010		3.103	
	PUF380	Assay (2)	0.949		64.152		21.787		9.979		3.133	
		HRGS Av.	0.9475	0.99737	64.2335	1.00691	21.7065	0.99717	9.9945	0.97765	3.118	0.95468
		Mass Spec.	0.950		63.793		21.768		10.223		3.266	

(3) 88-1 Fresh

No. 2/5

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
9		Assay (1)	0.801		64.035		21.983		10.034		3.147	
	PUF381	Assay (2)	0.803		64.140		21.973		9.969		3.115	
		HRGS Av.	0.802	0.99627	64.0875	1.00342	21.978	0.99823	10.0015	0.98363	3.131	0.99682
		Mass Spec.	0.805		63.869		22.017		10.168		3.141	
10		Assay (1)	0.716		64.972		21.608		9.776		2.928	
	PUF382	Assay (2)	0.717		64.977		21.704		9.688		2.914	
		HRGS Av.	0.7165	0.99652	64.9745	1.00463	21.656	0.99595	9.732	0.98154	2.921	0.99118
		Mass Spec.	0.719		64.675		21.744		9.915		2.947	
11		Assay (1)	0.696		65.465		21.529		9.514		2.796	
	PUF383	Assay (2)	0.698		65.480		21.538		9.494		2.790	
		HRGS Av.	0.697	0.97893	65.4725	1.00563	21.5335	0.99558	9.504	0.97687	2.793	0.98902
		Mass Spec.	0.712		65.106		21.629		9.729		2.824	
12		Assay (1)	0.695		65.320		21.525		9.621		2.839	
	PUF384	Assay (2)	0.699		65.008		21.571		9.666		2.906	
		HRGS Av.	0.697	0.97756	65.164	1.00350	21.623	0.99751	9.6435	0.98203	2.8725	1.00683
		Mass Spec.	0.713		64.937		21.677		9.820		2.853	
13		Assay (1)	0.681		65.830		21.316		8.760		3.413	
	PUF385	Assay (2)	0.681		66.094		21.265		8.631		3.329	
		HRGS Av.	0.681	0.99271	65.962	0.99909	21.2905	0.98883	8.6955	0.96940	3.371	1.20781
		Mass Spec.	0.686		66.022		21.531		8.970		2.791	
14		Assay (1)	0.672		66.640		21.392		8.176		3.120	
	PUF386	Assay (2)	0.673		66.772		21.240		8.215		3.100	
		HRGS Av.	0.6725	0.98897	66.706	1.00125	21.316	0.99057	8.1955	0.97345	3.110	1.12722
		Mass Spec.	0.680		66.623		21.519		8.419		2.759	
15		Assay (1)	0.676		66.525		21.257		9.011		2.531	
	PUF387	Assay (2)	0.680		66.238		21.392		9.095		2.595	
		HRGS Av.	0.678	0.99268	66.3815	1.01028	21.3245	0.99096	9.053	0.97062	2.563	0.92694
		Mass Spec.	0.683		65.706		21.519		9.327		2.765	
16		Assay (1)	0.873		65.628		20.645		10.161		2.693	
	PUF389	Assay (2)	0.878		65.683		20.659		10.105		2.675	
		HRGS Av.	0.8755	0.99715	65.6555	1.00782	20.652	0.99365	10.133	0.97330	2.684	0.96512
		Mass Spec.	0.878		65.146		20.784		10.411		2.781	

(3) 88-1 Fresh

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
17		Assay (1)	1.140		62.113		23.149		9.925		3.673	
	PUF390	Assay (2)	1.140		61.974		23.226		9.949		3.711	
		HRGS Av.	1.140	1.00088	62.0435	1.00485	23.1975	1.00050	9.937	0.97776	3.692	0.97724
		Mass Spec.	1.139		61.744		23.176		10.163		3.778	
18		Assay (1)	1.261		60.286		24.934		9.454		4.065	
	PUF391	Assay (2)	1.260		60.199		25.065		9.402		4.074	
		HRGS Av.	1.2605	0.99487	60.2425	1.01200	24.9995	0.99196	9.428	0.98547	4.0695	0.91738
		Mass Spec.	1.267		59.528		25.202		9.567		4.436	
19		Assay (1)	1.320		59.512		25.598		9.340		4.230	
	PUF392	Assay (2)	1.325		59.596		25.601		9.284		4.194	
		HRGS Av.	1.3225	0.99138	59.554	1.01296	25.5995	0.99535	9.312	0.98269	4.212	0.90019
		Mass Spec.	1.334		58.792		25.719		9.476		4.679	
20		Assay (1)	1.339		59.267		25.973		9.172		4.249	
	PUF393	Assay (2)	1.338		59.169		26.033		9.182		4.278	
		HRGS Av.	1.3385	0.99963	59.218	1.01229	26.003	0.99564	9.177	0.98402	4.2635	0.90348
		Mass Spec.	1.339		58.499		26.117		9.326		4.719	
21		Assay (1)	1.352		58.856		26.267		9.169		4.356	
	PUF394	Assay (2)	1.338		59.259		26.134		9.049		4.220	
		HRGS Av.	1.345	0.99703	59.0575	1.01221	26.2005	0.99660	9.109	0.98231	4.288	0.90407
		Mass Spec.	1.349		58.345		26.290		9.273		4.743	
22		Assay (1)	1.356		59.256		26.145		9.167		4.076	
	PUF395	Assay (2)	1.350		59.423		26.000		9.187		4.040	
		HRGS Av.	1.353	1.00000	59.3395	1.01402	26.0725	0.99883	9.177	0.98508	4.058	0.86175
		Mass Spec.	1.353		58.519		26.103		9.316		4.709	
23		Assay (1)	1.340		59.485		25.946		9.064		4.165	
	PUF396	Assay (2)	1.339		59.362		25.999		9.094		4.206	
		HRGS Av.	1.3395	0.99888	59.4235	1.01426	25.9725	0.99523	9.079	0.97729	4.1855	0.89357
		Mass Spec.	1.341		58.588		26.097		9.290		4.684	
24		Assay (1)	1.235		59.936		25.484		8.629		4.716	
	PUF397	Assay (2)	1.242		59.942		25.547		8.573		4.696	
		HRGS Av.	1.2385	0.98843	59.939	1.00845	25.5155	0.98186	8.601	0.97143	4.706	1.05303
		Mass Spec.	1.253		59.437		25.987		8.854		4.469	

(3) 88-1 Fresh

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
25		Assay (1)	0.883		64.161		24.911		6.851		3.194	
	PUF398	Assay (2)	0.881		64.408		24.826		6.766		3.119	
		HRGS Av.	0.882	0.98109	64.2845	1.00835	24.8685	0.98728	6.8085	0.98703	3.1565	0.96766
		Mass Spec.	0.899		63.752		25.189		6.898		3.262	
26		Assay (1)	0.846		64.799		25.005		6.410		2.940	
	PUF399	Assay (2)	0.849		64.627		25.137		6.414		2.973	
		HRGS Av.	0.8475	0.99355	64.713	1.01002	25.071	0.98958	6.412	0.98118	2.9565	0.92218
		Mass Spec.	0.853		64.071		25.335		6.535		3.206	
27		Assay (1)	0.819		64.531		25.488		6.226		2.936	
	PUF400	Assay (2)	0.815		64.785		25.339		6.185		2.876	
		HRGS Av.	0.817	0.99271	64.658	1.01015	25.4135	0.99039	6.2055	0.97648	2.906	0.92137
		Mass Spec.	0.823		64.008		25.660		6.355		3.154	
28		Assay (1)	0.790		65.207		25.185		6.055		2.763	
	PUF401	Assay (2)	0.800		64.911		25.371		6.092		2.826	
		HRGS Av.	0.795	0.99127	65.059	1.01196	25.278	0.98843	6.0735	0.97269	2.7945	0.90437
		Mass Spec.	0.802		64.290		25.574		6.244		3.090	
29		Assay (1)	0.762		65.690		25.046		5.983		2.519	
	PUF402	Assay (2)	0.765		65.692		25.069		5.961		2.513	
		HRGS Av.	0.7635	0.99285	65.691	1.01214	25.0575	0.98952	5.972	0.97854	2.516	0.86699
		Mass Spec.	0.769		64.903		25.323		6.103		2.902	
30		Assay (1)	0.950		64.054		24.945		7.184		2.867	
	PUF403	Assay (2)	0.951		64.183		24.906		7.130		2.830	
		HRGS Av.	0.9505	0.99633	64.1185	1.01418	24.9255	0.99420	7.157	0.98500	2.8485	0.81689
		Mass Spec.	0.954		63.222		25.071		7.266		3.487	
31		Assay (1)	0.951		63.922		24.237		7.909		2.981	
	PUF404	Assay (2)	0.948		64.157		24.014		7.938		2.943	
		HRGS Av.	0.9495	0.99842	64.0395	1.01208	24.1255	0.99536	7.9235	0.97857	2.962	0.86130
		Mass Spec.	0.951		63.275		24.238		8.097		3.439	
32		Assay (1)	1.009		62.895		23.551		9.125		3.420	
	PUF405	Assay (2)	1.017		62.571		23.710		9.196		3.506	
		HRGS Av.	1.013	0.91757	62.733	1.00928	23.6305	0.98243	9.1605	1.03205	3.463	0.90869
		Mass Spec.	1.104		62.156		24.053		8.876		3.811	

(3) 88-1 Fresh

No. 5/5

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
			Average	0.99051		1.00828		0.99366		0.98139		0.95481
			CV%	1.604 %		0.409 %		0.500 %		1.103 %		7.958 %

(4) 85-2 Campaign : Aged Sample

No. 1/3

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	1.296		63.258		22.704		9.154			
	PUA251	Assay (2)	1.363		63.119		22.450		9.084			
		Assay (3)	1.327		63.142		22.321		9.224			
		HRGS Av.	1.329	1.08756	63.173	0.99540	22.492	1.00018	9.154	1.03564	~~(3.882)~~	1.
		Mass Spec.	1.222		63.465		22.488		8.839			
2		Assay (1)	1.278		63.473		22.514		8.830			
	PUA253	Assay (2)	1.266		63.820		22.269		8.740			
		Assay (3)	1.193		63.864		22.298		8.738			
		HRGS Av.	1.246	1.00080	63.719	0.99892	22.360	1.00260	8.769	1.00126	~~(3.906)~~	1.
		Mass Spec.	1.245		63.788		22.302		8.758			
3		Assay (1)	1.308		62.861		22.843		8.940			
	PUA254	Assay (2)	1.296		63.233		22.583		8.841			
		Assay (3)	1.213		63.458		22.491		8.789			
		HRGS Av.	1.272	1.00236	63.184	0.99902	22.639	1.00124	8.857	1.00363	~~(4.048)~~	1.
		Mass Spec.	1.269		63.246		22.611		8.825			
4		Assay (1)	1.289		61.834		23.744		9.135			
	PUA255	Assay (2)	1.280		62.186		23.506		9.027			
		Assay (3)	1.196		62.434		23.404		8.968			
		HRGS Av.	1.255	1.02533	62.151	0.98194	23.551	1.04028	9.043	1.02239	~~(3.999)~~	1.
		Mass Spec.	1.224		63.294		22.639		8.845			
5		Assay (1)	1.310		62.361		23.002		9.278			
	PUA256	Assay (2)	1.299		62.712		22.760		9.182			
		Assay (3)	1.210		63.138		22.564		9.039			
		HRGS Av.	1.273	1.01922	62.737	0.99771	22.775	1.00463	9.167	1.00175	~~(4.048)~~	1.
		Mass Spec.	1.249		62.881		22.670		9.151			
6		Assay (1)	1.336		62.448		22.691		9.547			
	PUA257	Assay (2)	1.324		62.821		22.437		9.441			
		Assay (3)	1.231		63.442		22.068		9.282			
		HRGS Av.	1.297	1.04850	62.903	0.99903	22.399	0.99653	9.423	1.00845	~~(3.978)~~	1.
		Mass Spec.	1.237		62.964		22.477		9.344			

(4) 85-2 Aged

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
7		Assay (1)	1.347		61.996		23.047		9.555			
	PUA258	Assay (2)	1.335		62.341		22.811		9.459			
		Assay (3)	1.256		62.158		23.110		9.421			
		HRGS Av.	1.313	1.02020	62.165	1.00116	22.989	1.01721	9.478	1.01077	~~(4.055)~~	1.
		Mass Spec.	1.287		62.680		22.600		9.377			
8		Assay (1)	1.346		61.365		23.451		9.422			
	PUA259	Assay (2)	1.331		61.812		23.142		9.299			
		Assay (3)	1.247		62.162		22.933		9.242			
		HRGS Av.	1.308	1.01317	61.780	1.00279	23.175	0.99336	9.321	0.99637	~~(4.416)~~	1.
		Mass Spec.	1.291		61.608		23.330		9.355			
9		Assay (1)	1.357		61.425		23.416		9.469			
	PUA260	Assay (2)	1.344		61.808		23.153		9.363			
		Assay (3)	1.266		61.107		22.976		9.318			
		HRGS Av.	1.322	1.03768	61.780	0.99867	23.182	1.00000	9.383	1.00364	~~(4.333)~~	1.
		Mass Spec.	1.274		61.862		23.182		9.349			
10		Assay (1)	1.404		60.076		24.449		9.777			
	PUA261	Assay (2)	1.395		60.353		24.256		9.703			
		Assay (3)	1.313		60.695		24.054		9.644			
		HRGS Av.	1.371	1.04577	60.375	0.97470	24.253	1.04665	9.708	1.04601	~~(4.293)~~	1.
		Mass Spec.	1.311		61.942		23.172		9.281			
11		Assay (1)	1.303		62.644		22.846		9.156			
	267V10	Assay (2)	1.300		62.654		22.867		9.127			
		Assay (3)	1.214		62.910		22.790		9.034			
		HRGS Av.	1.272	1.11189	62.736	0.99624	22.834	1.00325	9.106	1.00386	~~(4.052)~~	1.
		Mass Spec.	1.144		62.973		22.760		9.071			
12		Assay (1)	1.207		64.799		22.539		8.098			
	267V11	Assay (2)	1.195		65.174		22.273		8.001			
		Assay (3)	1.120		65.386		22.180		7.957			
		HRGS Av.	1.174	0.98243	65.120	1.00911	22.330	0.98115	8.019	0.98308	~~(3.357)~~	1.
		Mass Spec.	1.195		64.532		22.759		8.157			

(4) 85-2 Aged

No. 3/3

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
13		Assay (1)	1.299		61.070		24.548		8.814			
	267V12	Assay (2)	1.291		61.268		24.449		8.724			
		Assay (3)	1.211		61.542		24.303		8.676			
		HRGS Av.	1.267	1.10462	61.293	0.99920	24.433	0.99735	8.738	0.99931	~~(4.269)~~	1.
		Mass Spec.	1.147		61.342		24.498		8.744			
14		Assay (1)	0.995		65.858		21.963		8.078			
	267V13	Assay (2)	0.987		66.105		21.807		7.996			
		Assay (3)	0.926		66.141		21.883		7.945			
		HRGS Av.	0.969	1.04306	66.035	0.99944	21.884	0.99727	8.006	1.00717	~~(3.106)~~	1.
		Mass Spec.	0.929		66.072		21.944		7.949			
15		Assay (1)	0.831		66.642		21.991		8.011			
	267V14	Assay (2)	0.826		66.830		21.780		7.958			
		Assay (3)	0.770		67.175		21.587		7.862			
		HRGS Av.	0.809	1.00000	66.882	0.98929	21.760	1.02530	7.944	1.02424	~~(2.606)~~	1.
		Mass Spec.	0.809		67.606		21.223		7.756			
16		Assay (1)	1.365		66.124		21.467		7.905			
	267V15	Assay (2)	1.358		66.206		21.368		7.931			
		Assay (3)	1.277		66.465		21.239		7.880			
		HRGS Av.	1.333	1.19124	66.265	1.00487	21.358	0.98112	7.905	0.98443	~~(3.139)~~	1.
		Mass Spec.	1.119		65.944		21.769		8.030			
			Average	1.04586		0.99672		1.00551		1.00824		1.
			CV%	5.159%		0.848%		1.829%		1.662%		-

(5) 86-1 Campaign : Aged Sample

No. 1/1

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	0.975		65.389		20.411		10.150			
	PUA263	Assay (2)	0.979		64.971		20.772		10.192			
		HRGS Av.	0.977	1.06469	65.180	1.00391	20.592	0.98498	10.171	0.99892	~~(3.074)~~	1
		Mass Spec.	0.912		64.926		20.906		10.181			
2		Assay (1)	0.960		65.296		20.556		10.206			
	PUA264	Assay (2)	0.964		65.539		20.319		10.195			
		HRGS Av.	0.962	1.02015	65.418	1.00455	20.438	0.98463	10.201	1.00049	~~(2.982)~~	1
		Mass Spec.	0.943		65.122		20.757		10.196			
3		Assay (1)	0.946		65.119		20.629		10.377			
	PUA266	Assay (2)	0.944		65.567		20.273		10.287			
		HRGS Av.	0.945	1.06659	65.343	1.00107	20.451	0.99596	10.332	0.99557	~~(2.929)~~	1
		Mass Spec.	0.886		65.273		20.534		10.378			
4		Assay (1)	0.997		64.579		20.708		10.628			
	PUA267	Assay (2)	0.995		64.625		20.814		10.478			
		HRGS Av.	0.996	0.98810	64.602	1.00102	20.761	0.99765	10.553	0.99943	~~(3.087)~~	1
		Mass Spec.	1.008		64.536		20.810		10.559			
		Average		1.03488		1.00264		0.99080		0.99860		1
		CV%		3.658%		0.184%		0.703%		0.213%		-

(6) 86-2 Campaign : Aged Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)	0.661		64.700		21.417		10.392			
	PUA269	Assay (2)	0.657		64.716		21.426		10.371			
		HRGS Av.	0.659	0.94413	64.708	1.00049	21.422	1.00115	10.382	0.99832	~~(2.830)~~	1
		Mass Spec.	0.698		64.676		21.397		10.399			
2		Assay (1)	0.653		64.294		21.676		10.488			
	PUA270	Assay (2)	0.651		64.038		21.938		10.484			
		HRGS Av.	0.652	0.93410	64.166	0.99649	21.807	1.01178	10.486	1.00172	~~(2.889)~~	1
		Mass Spec.	0.698		64.392		21.553		10.468			
3		Assay (1)	0.824		64.331		21.144		10.672			
	PUA271	Assay (2)	0.840		64.280		21.105		10.747			
		HRGS Av.	0.832	0.92239	64.306	1.00631	21.125	0.99171	10.710	0.98560	~~(3.029)~~	1
		Mass Spec.	0.902		63.902		21.301		10.866			
4		Assay (1)	0.977		64.198		20.726		11.059			
	PUA272	Assay (2)	0.954		64.835		20.392		10.780			
		HRGS Av.	0.966	0.97525	64.516	1.00637	20.559	0.98623	10.920	0.99115	(3.039)	1
		Mass Spec.	0.990		64.108		20.846		11.017			
5		Assay (1)	1.052		63.355		21.131		11.287			
	PUA273	Assay (2)	1.038		63.534		21.068		11.184			
		HRGS Av.	1.045	0.98030	63.445	0.99757	21.100	1.00723	11.236	1.00210	~~(3.175)~~	1
		Mass Spec.	1.066		63.599		20.948		11.212			
6		Assay (1)	1.084		65.452		20.196		10.424			
	PUA274	Assay (2)	1.059		66.068		19.739		10.290			
		HRGS Av.	1.072	0.95244	65.760	1.00724	19.968	0.98449	10.357	0.98996	~~(2.844)~~	1
		Mass Spec.	1.125		65.287		20.282		10.462			
7		Assay (1)	1.123		66.536		19.932		9.770			
	PUA275	Assay (2)	1.109		66.878		19.700		9.674			
		HRGS Av.	1.116	1.00450	66.707	1.00527	19.816	0.98981	9.722	0.98471	~~(2.639)~~	1
		Mass Spec.	1.111		66.357		20.020		9.873			
8		Assay (1)	1.066		65.088		21.293		9.737			
	PUA277	Assay (2)	1.055		65.156		21.297		9.677			
		HRGS Av.	1.061	0.96192	65.122	0.99877	21.295	1.00524	9.707	1.00113	~~(2.815)~~	1
		Mass Spec.	1.103		65.202		21.184		9.696			

(6) 86-2 Aged

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
9		Assay (1)	0.967		63.459		23.670		8.801			
	PUA278	Assay (2)	0.973		63.515		23.578		8.831			
		HRGS Av.	0.970	1.15752	63.487	0.99992	23.624	0.99785	8.816	0.99145	~~(3.103)~~	1
		Mass Spec.	0.838		63.492		23.675		8.892			
10		Assay (1)	0.966		63.639		23.541		8.644			
	PUA279	Assay (2)	0.961		63.307		23.853		8.669			
		HRGS Av.	0.964	1.13087	63.473	1.00839	23.697	0.97950	8.657	0.98369	~~(3.210)~~	1
		Mass Spec.	0.852		62.945		24.193		8.800			
11		Assay (1)	0.989		62.781		24.140		8.763			
	PUA280	Assay (2)	0.975		62.908		24.073		8.715			
		HRGS Av.	0.982	0.95340	62.845	1.00699	24.107	0.98442	8.739	0.99920	~~(3.328)~~	1
		Mass Spec.	1.030		62.408		24.488		8.746			
12		Assay (1)	1.014		62.577		24.233		8.704			
	PUA281	Assay (2)	1.013		62.462		24.361		8.693			
		HRGS Av.	1.014	1.07819	62.520	1.00950	24.297	0.97834	8.699	0.98595	~~(3.471)~~	1
		Mass Spec.	0.940		61.931		24.835		8.823			
13		Assay (1)	1.024		62.853		23.778		8.759			
	PUA282	Assay (2)	1.040		62.216		24.295		8.863			
		HRGS Av.	1.032	0.93818	62.535	1.01422	24.037	0.97424	8.811	0.98074	~~(3.586)~~	1
		Mass Spec.	1.100		61.658		24.672		8.984			
14		Assay (1)	1.005		62.656		24.503		8.333			
	PUA283	Assay (2)	1.016		62.328		24.709		8.444			
		HRGS Av.	1.011	0.95330	62.492	1.01045	24.606	0.98337	8.389	0.97894	~~(3.503)~~	1
		Mass Spec.	1.060		61.846		25.022		8.569			
15		Assay (1)	1.066		62.719		24.567		8.122			
	PUA284	Assay (2)	1.068		62.571		24.686		8.148			
		HRGS Av.	1.067	0.92783	62.645	1.00783	24.626	0.98693	8.135	0.99050	~~(3.527)~~	1
		Mass Spec.	1.150		62.158		24.952		8.213			
16		Assay (1)	1.085		62.826		24.447		8.077			
	PUA285	Assay (2)	1.086		62.781		24.551		8.017			
		HRGS Av.	1.086	0.96747	62.804	1.01283	24.499	0.97961	8.047	0.96999	~~(3.565)~~	1
		Mass Spec.	1.122		62.008		25.009		8.296			

(6) 86-2 Aged

No. 3/3

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
17		Assay (1)	1.051		63.061		24.441		7.976			
	PUA287	Assay (2)	1.053		62.753		24.624		8.099			
		HRGS Av.	1.052	0.94097	62.907	1.00771	24.533	0.98997	8.038	0.97971	~~(3.471)~~	1.
		Mass Spec.	1.118		62.426		24.781		8.204			
			Average	0.98369		1.00567		0.99011		0.98911		1.
			CV%	7.174%		0.522%		1.107%		0.935%		-

(7) 88-1 Campaign : Aged Sample

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
1		Assay (1)										
	PUA373	Assay (2)										
		HRGS Av.	1.128	0.93610	61.305	0.98625	22.602	1.02629	11.295	1.03236	~~(3.671)~~	1.
		Mass Spec.	1.205		62.160		22.023		10.941			
2		Assay (1)										
	PUA374	Assay (2)										
		HRGS Av.	1.128	0.96575	62.087	0.99928	21.888	0.99171	11.186	1.02464	~~(3.712)~~	1.
		Mass Spec.	1.168		62.132		22.071		10.917			
3		Assay (1)										
	PUA375	Assay (2)										
		HRGS Av.	1.127	0.94626	62.033	1.00293	21.868	0.99238	11.176	1.00467	~~(3.797)~~	1.
		Mass Spec.	1.191		61.852		22.036		11.124			
4		Assay (1)										
	PUA376	Assay (2)										
		HRGS Av.	1.144	0.94938	61.245	0.99275	22.495	1.01912	11.291	1.00768	~~(3.825)~~	1.
		Mass Spec.	1.205		61.692		22.073		11.205			
5		Assay (1)										
	PUA377	Assay (2)										
		HRGS Av.	1.160	0.95242	61.365	0.99712	22.229	1.00456	11.398	1.01199	~~(3.848)~~	1.
		Mass Spec.	1.219		61.542		22.128		11.263			
6		Assay (1)										
	PUA378	Assay (2)										
		HRGS Av.	1.132	0.96350	61.395	0.98976	22.372	1.01918	11.377	1.02339	~~(3.724)~~	1.
		Mass Spec.	1.178		62.030		21.951		11.117			
7		Assay (1)										
	PUA379	Assay (2)										
		HRGS Av.	1.071	0.95882	62.168	0.98882	22.242	1.02168	10.959	1.02593	~~(3.560)~~	1.
		Mass Spec.	1.117		62.871		21.770		10.682			
8		Assay (1)										
	PUA380	Assay (2)										
		HRGS Av.	0.896	0.94316	63.617	0.99724	21.940	1.00790	10.281	1.00567	~~(3.266)~~	1.
		Mass Spec.	0.950		63.793		21.768		10.223			

(7) 88-1 Aged

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
9		Assay (1)										
	PUA381	Assay (2)										
		HRGS Av.	0.755	0.93789	63.971	1.00160	21.993	0.99891	10.145	0.99774	~~(3.141)~~	1.
		Mass Spec.	0.805		63.869		22.017		10.168			
10		Assay (1)										
	PUA382	Assay (2)										
		HRGS Av.	0.660	0.91794	65.202	1.00815	21.390	0.98372	9.801	0.98850	~~(2.947)~~	1.
		Mass Spec.	0.719		64.675		21.744		9.915			
11		Assay (1)										
	PUA383	Assay (2)										
		HRGS Av.	0.654	0.91854	65.024	0.99879	21.739	1.00509	9.758	1.00298	~~(2.824)~~	1.
		Mass Spec.	0.712		65.106		21.629		9.729			
12		Assay (1)										
	PUA384	Assay (2)										
		HRGS Av.	0.660	0.92567	64.671	0.99590	21.808	1.00604	10.008	1.01914	~~(2.853)~~	1.
		Mass Spec.	0.713		64.937		21.677		9.820			
13		Assay (1)										
	PUA385	Assay (2)										
		HRGS Av.	0.643	0.93732	65.797	0.99659	21.723	1.00892	9.046	1.00847	~~(2.791)~~	1.
		Mass Spec.	0.686		66.022		21.531		8.970			
14		Assay (1)										
	PUA386	Assay (2)										
		HRGS Av.	0.639	0.93971	66.417	0.99691	21.729	1.00976	8.455	1.00428	~~(2.759)~~	1.
		Mass Spec.	0.680		66.623		21.519		8.419			
15		Assay (1)										
	PUA387	Assay (2)										
		HRGS Av.	0.639	0.93558	65.471	0.99642	21.761	1.01125	9.364	1.00397	~~(2.765)~~	1.
		Mass Spec.	0.683		65.706		21.519		9.327			
16		Assay (1)										
	PUA388	Assay (2)										
		HRGS Av.	0.663	0.94310	65.365	0.99605	21.450	1.00851	9.830	1.01205	~~(2.691)~~	1.
		Mass Spec.	0.703		65.624		21.269		9.713			

(7) 88-1 Aged

No.	Sample I.D.	Isotope	Pu-238		Pu-239		Pu-240		Pu-241		Pu-242	
				HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass		HRGS/Mass
17		Assay (1)										
	PUA389	Assay (2)										
		HRGS Av.	0.833	0.94875	65.130	0.99975	20.756	0.99865	10.499	1.00845	(2.781)	1.
		Mass Spec.	0.878		65.146		20.784		10.411			
18		Assay (1)										
	PUA390	Assay (2)										
		HRGS Av.	1.081	0.94908	61.580	0.99734	23.332	1.00673	10.229	1.00649	(3.778)	1.
		Mass Spec.	1.139		61.744		23.176		10.163			
			Average	0.94272		0.99676		1.00669		1.01047		1.
			CV%	1.431%		0.517%		1.091%		1.083%		-