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TASK **No.: JC-11 (A-556)**

TITLE: Non-destructive Assay System for Uranium and Plutonium in Reprocessing Input Solutions (Hybrid K-edge / XRF Densitometer)

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JASPAS JC-11 (A-556) ; Non-destructive Assay System for Uranium and Plutonium in Reprocessing Input Solutions - Hybrid K-edge/XRF Densitometer - (JASPAS JC-11 Final Report)

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要旨

Hybrid K-edge/XRF Densitometer は、JASPAS（日本の対 IAEA への保障措置技術開発支援計画）の一環で、使用済燃料溶解液中のウランおよびプルトニウム濃度を迅速かつ高精度に分析できる非破壊測定装置として、1991年以来、東海再処理工場で開発されてきたものである。

本装置は、K-エッジデンシトメータ（KED)とエネルギ分散型蛍光X線分析装置（XRF）を組み合わせたハイブリッド型のシステムである。測定原理は、KED 法によりウラン濃度を測定し、XRF 法により U／Pu 濃度比を測定して、これら両者の結果よりプルトニウム濃度を求めるものであり、タイムリーかつオンサイトでの分析が期待できることから保障措置上有効な検認技術であると考えられている。これまで、本装置を高放射性分析セルに設置した後、ホット試験を実施し、破壊分析（DA）である同位体希釈質量分析法（IDMS）との比較検討を続けてきたが、概ね良好な一致性が確認できたことから、本装置を実査察へ適用するために、査察側による受入試験（Acceptance Test）および性能試験（Performance Test）を行った。ここで測定精度として、ウラン：0.2％、プルトニウム：0.7％程度が得られており、査察機器としての性能を有することが評価された。

本ファイナルレポートは、試験結果および技術・操作を併せてまとめたものであり、これを以て、本 JASPAS 開発項目を終了することとする。

JASPAS JC-11 (A-556) ; Non-destructive Assay System for Uranium and Plutonium in Reprocessing Input Solutions - Hybrid K-edge/XRF Densitometer - (JASPAS JC-11 Final Report)

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ABSTRACT

As a part of JASPAS programme, a non-radioactive assay system for the accountability of uranium and plutonium in input dissolver solutions of a spent fuel reprocessing plant, called Hybrid K-edge/XRF Densitometer, has been developed at the Tokai Reprocessing plant (TRP) since 1991.

The instrument is the one of the hybrid type combined K-edge densitometry (KED) and X-ray fluorescence (XRF) analysis. The KED is used to determine the uranium concentration and the XRF is used to determine the U/Pu ratio. These results give the plutonium concentration in consequence. It is considered that the instrument has the capability of timely on-site verification for input accountancy. The instrument had been installed in the analytical hot cell at the TRP and the experiments comparing with Isotope Dilution Mass Spectrometry (IDMS) method have been carried out. As the results of measurements for the actual input solutions in the acceptance and performance tests, it was typically confirmed that the precision for determining uranium concentration by the KED was within 0.2%, whereas the XRF for plutonium performed within 0.7%.

This final report summarizes the design information and performance data so as to end the JASPAS programme.

Non-destructive Assay System for Uranium and Plutonium in Reprocessing Input Solutions -Hybrid K-edge/XRF Densitometer-
(Final Report of JASPAS JC-11)

CONTENTS

1. INTRODUCTION

Isotope Dilution Mass Spectrometric determination (IDMS) which is considered as one of the most accurate technique, has been so far employed for safeguards accountability analysis of the input dissolver solutions at the Tokai Reprocessing Plant (TRP). This method, as a destructive analysis, requires operator's skillful technique but also takes long time, a few days to complete a determination. In addition, the sample preparation is complicated for not only operators and also inspectors over the entire processing operation, involving spiking, sampling, dilution, aliquoting and chemical equilibration.

In order to achieve the timeliness and effectiveness of the inspection, the IAEA has insisted on the necessity of rapid determination, such as a Non-Destructive Assay (NDA), for the input verification analysis.

The development of NDA system, Hybrid K-edge/XRF Densitometer (H-KED), for the accountability of uranium and plutonium in input dissolver solutions initiated in 1991 at the TRP. The system was designed by PNC based upon the information of the report presented by Ottmar et al., KfK. [1)]

The examination for the hybrid instrument has been carried out since the system was installed in a shielded cell. The results of uranium and plutonium concentrations obtained by the H-KED have been compared with those by IDMS. This report mainly describes the design information and the experimental results.

2. INSTRUMENT

The original H-KED was pioneered at KfK Karlsruhe [1)]. The H-KED system consists of a K-Edge Densitometer (KED) and X-Ray Fluorescence (XRF) spectrometer. The KED is utilized to determine uranium concentration, while the XRF analysis is utilized to determine the ratio of U/Pu for obtaining a simultaneous determination. Plutonium concentration can be calculated from both the measurement values.

A similar system has been designed by PNC and installed at the TRP [2),3),4)]. The system shown in Photo. 1 consists of an X-ray generation tube

Photo.1 Mechanical set-up of the Hybrid KED/XRF instrument installed in the shielded cell.

(COMET, MXR-160 0.4-3.0, Max. 160 kV, 19 mA), two Ge detectors (EG & G, LOAX type), two sample holders (cells) and controller/electronics modules. The sample holder for KED has a 25 mm pass length and that for XRF is 10 mm diameter of cylindrical cell as shown in Photo. 2. They were connected each other with stainless steel tube to be simultaneously filled with only 8 ml of sample solution. This measurement cell system is a flow-type one which facilitates the remote operation of sample injection and is advantageous for a geometry reason. The instrument was designed to be installed compactly into a small hot cave of lead shielding(1m×1m×0.6m). The electronics, controller and CPU manufactured by SEIKO EG & G and NEC have been installed outside the shielded cell.

Primary X-ray from the generation tube was separated into the K-edge section and the XRF one by collimator. Each X-ray irradiates the sample holders filled with sample solution. The beam collimated less than 1 mm diameter to lower the intensity of primary X-ray, is absorbed and analyzed at the K-edge densitometry section. The XRF spectrum induced by the primary X-ray is measured by another detector. The X-ray beam geometry is shown in Fig. 1.

The schematic diagram around sample cells is also shown in Fig. 2.

The main characteristics of the system are as follows:

1. The flow-type cell is employed to facilitate the remote operation of sample injection.
2. The equipment is worked up compactly to install into the limited space.
3. Energy calibration for the KED is performed with X-ray fluorescence (Kα1) of lead (75.0 keV) used as shield from generator and K-edge point of uranium (115.6 keV), namely without additional isotopic sources. X-ray fluorescence of uranium, Kα1, Kα2 and Kβ1, are used for the energy calibration for XRF.
4. Most of the mechanical/electric units used are domestic products, which enable easy maintenance.

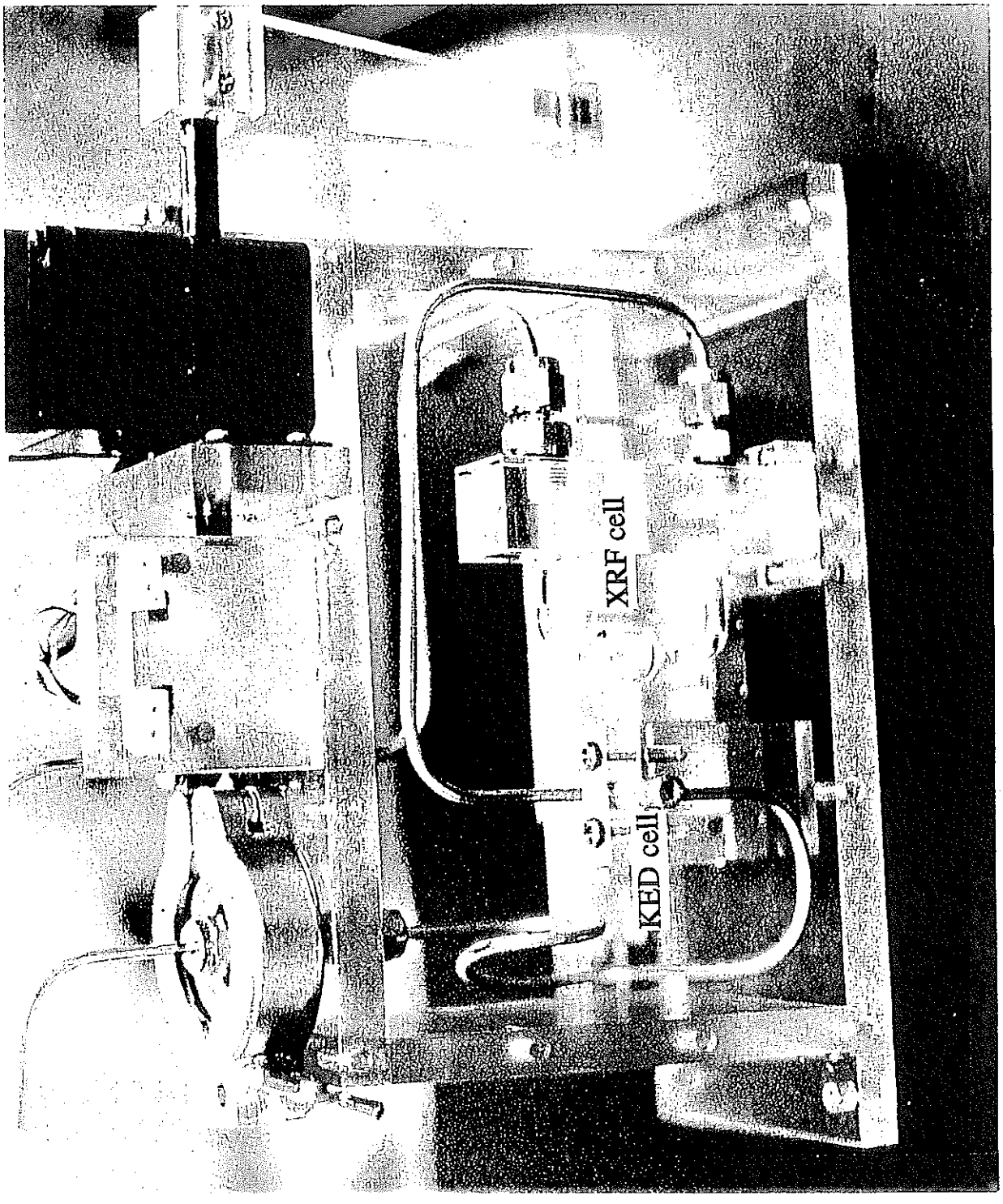

Photo.2 Measurement cell system.

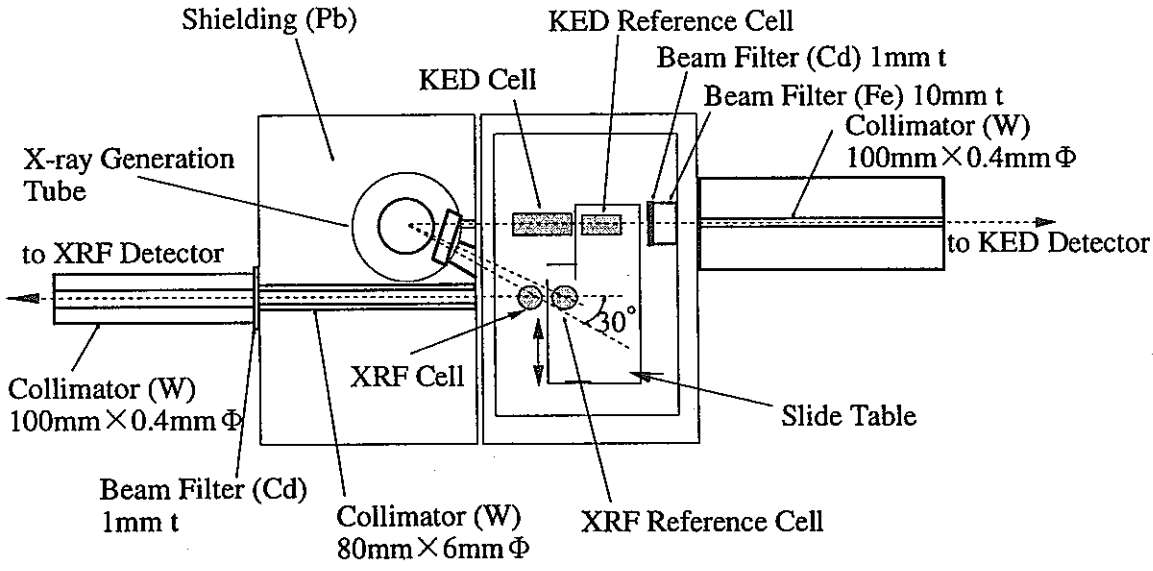

Fig. 1 X-ray beam geometry for KED and XRF in the Hybrid instrument

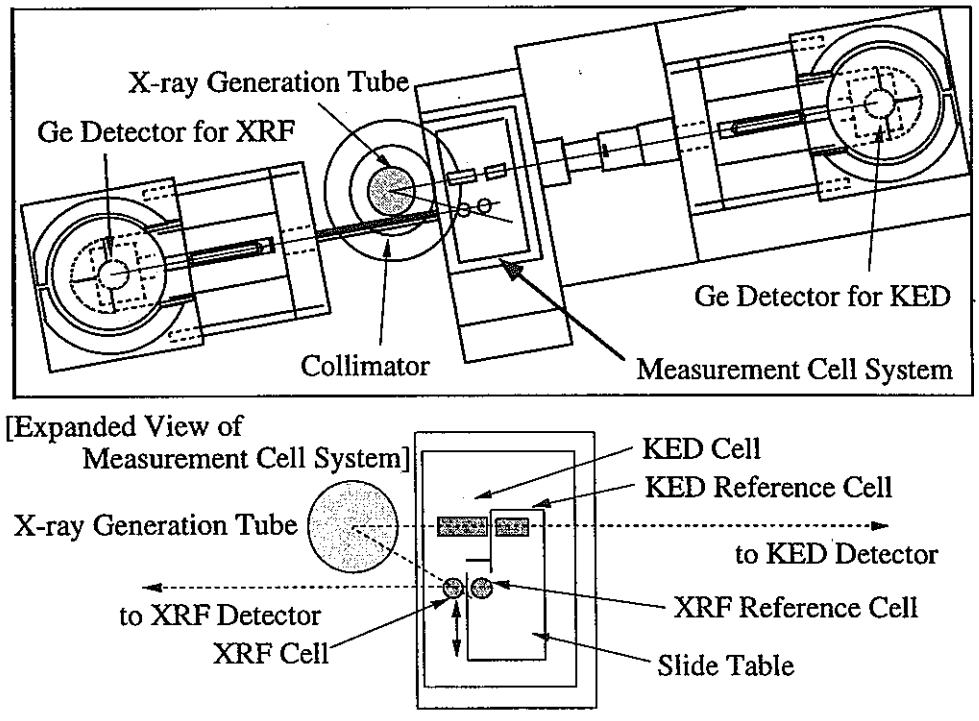

Fig. 2 Schematic diagram of Hybrid instrument installed in shielded cell

In addition, the following ideas have been employed for the quality control and authentication purpose.

1. Uranium foil (approx. 0.3 mm in thickness) remotely controlled is used for the measurement control of KED. Whereas, a solidified MOX standard composed of uranium, JAERI U-4, and plutonium, NBL CRM-126, for control measurement of the XRF is also set remotely at the appropriate position by means of a small magnet in the measurement cell system.
2. The flow-type cell can be observed from the outside by using a CCD camera to confirm whether input solution is properly filled with.

Since the measurement precision of the XRF depends on the magnitude of count rate of each peak, the distance between the focus of the X-ray generation tube and the measurement cell was modified from 16 cm to 6 cm in 1994.

Prior to the demonstration of the H-KED system for JNSB and IAEA, the system was designed to be applied to routine inspection of the TRP. The system shown in Fig. 3 has two control units including electronics module. One is for facility operation and the other completely isolated from the operator's is to offer the independent operation for the national / international safeguards. The unit is installed in a cabinet and under IAEA seal when the inspectors are not present. The application software is also under IAEA control for the authentication purpose.

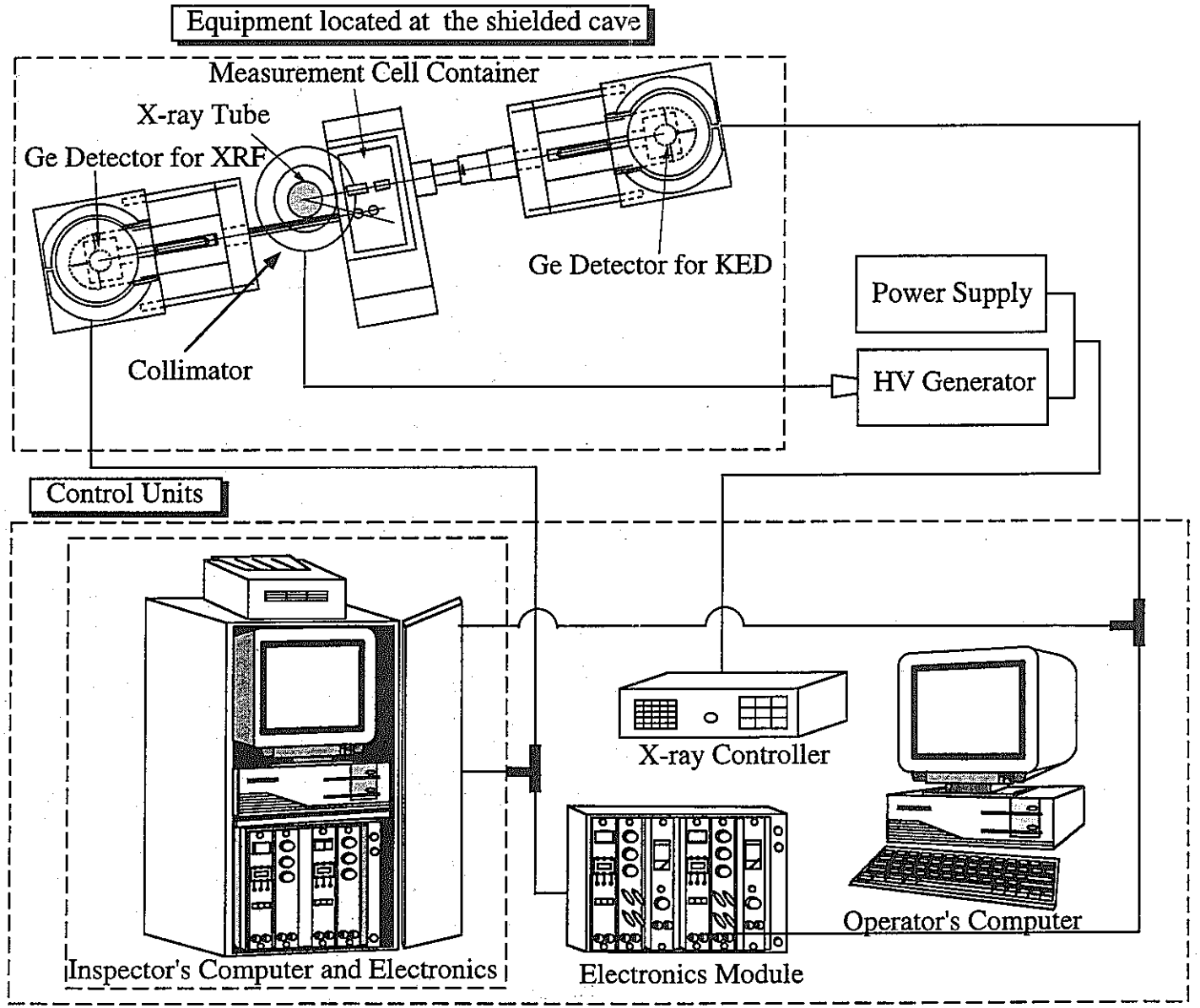

Fig 3 Block diagram of the HYBRID system

3. TECHNIQUE

3-1. K-edge Densitometer

Figure 4 shows the spectrum of dissolver solution (PWR spent fuel : 27 GWD/t, [U] = 188 g/L, [Pu] = 1.6 g/L) obtained with the KED, where the instrument was operated at a fixed power of 150 kV / 15 mA. The discontinuity observed is the K-edge absorption of the uranium, 115.6 keV.

The uranium concentration should be in proportion to the ratio of the detector counting at the upper energy (Eu) side to that at the lower energy (El) side in the K-edge measurement, as given in the following equation.

$$U = \frac{1}{\Delta\mu \cdot d} \cdot \ln\left\{\frac{C(Eu)}{C(El)}\right\} \qquad (1)$$

where d is the sample thickness, 25 mm, and $\Delta\mu$ denotes the difference of mass attenuation coefficients of uranium for the energies Eu and El.

As shown in Fig. 5, the uranium concentration can be determined with the counting ratio of C(Eu) and C(El) at the channel of the K-edge point, where C(Eu) and C(El) are obtained from the extrapolation of counting values observed in the windows which does not interfere with the K-edge positions of U/Pu.

Figure 6 shows an example to determine K-edge point. As is shown in this figure, the point of the K-edge is obtained by differential curve.

For the K-edge densitometer, the following procedure is adopted;

1. Energy calibration is performed with the channel of X-ray peak of Pb (74.97 keV) and the central channel of K-edge jump of transmission (115.6 keV).
2. Background counting arising from gamma ray, compton scatter, pile-up and so on due to the dissolver solution itself is subtracted from the spectrum of K-edge densitometry. The correction is made by the same method presented in the MGA code [5] as follows;

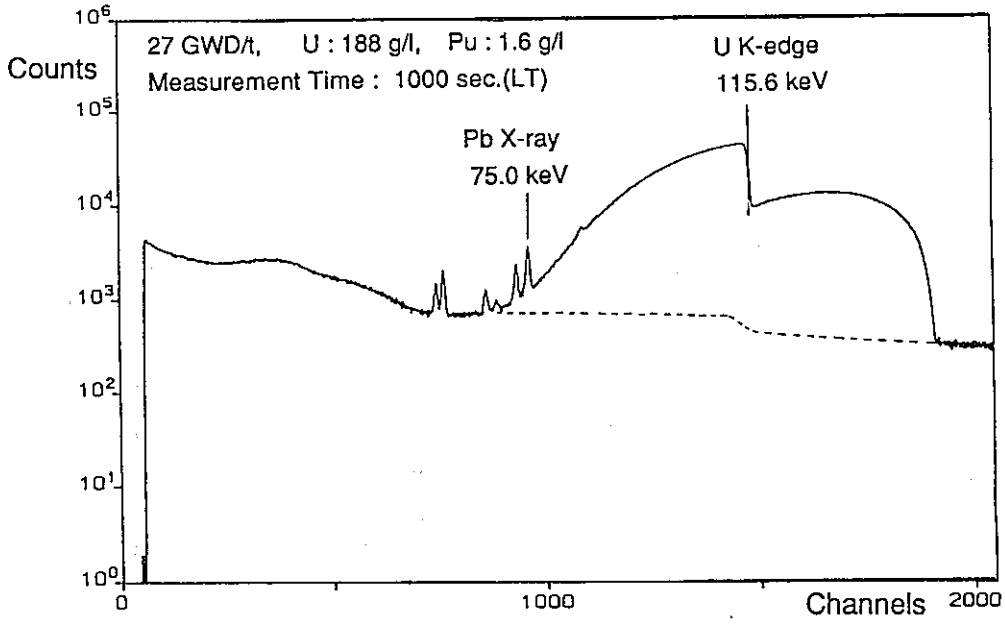

Fig. 4 Typical K-edge spectrum of spent-fuel dissolver solution

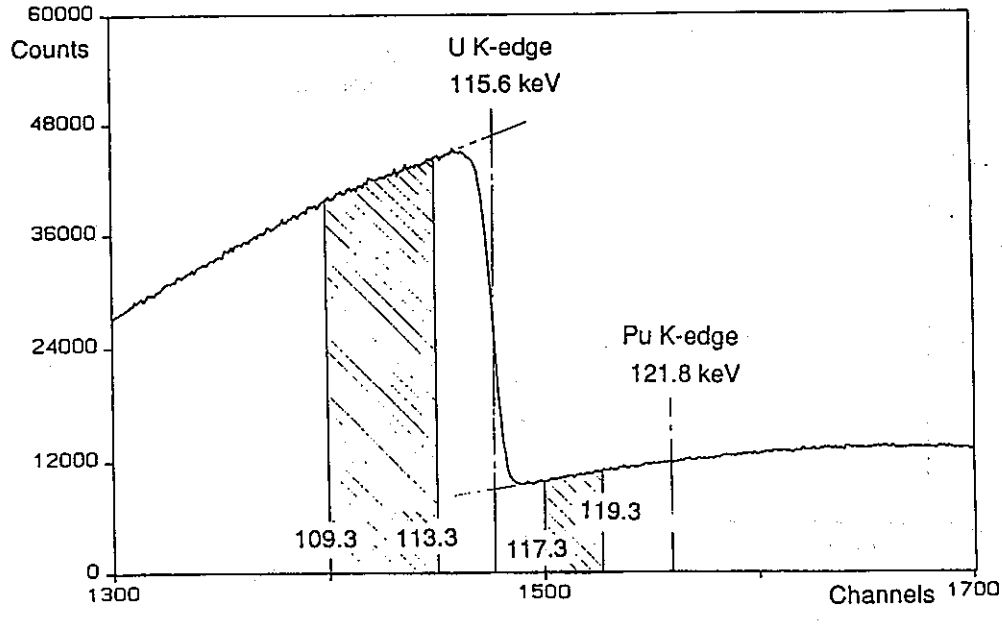

Fig. 5 Expanded K-edge spectrum of data fitting region

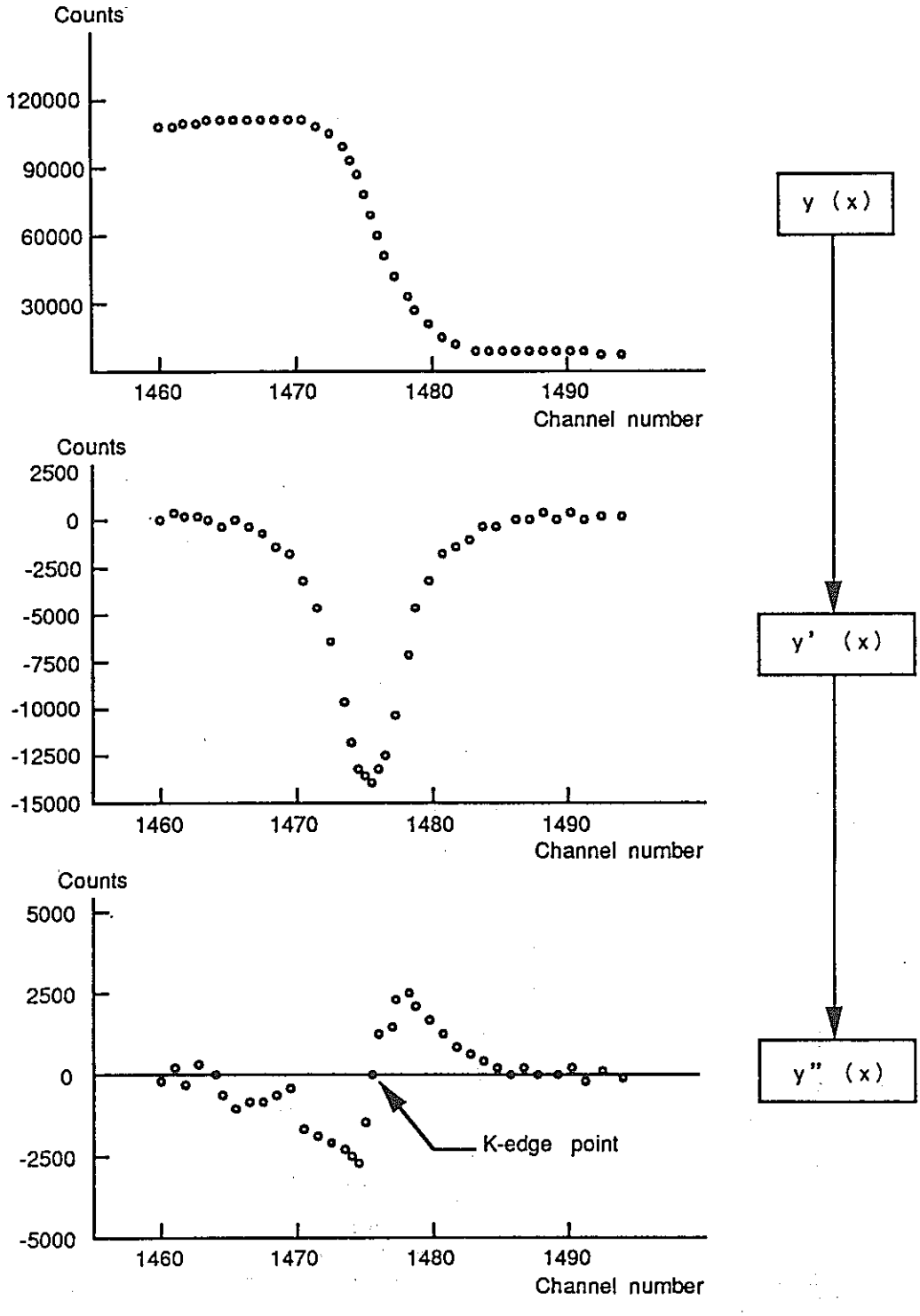

Fig. 6 Determination of K-edge point by differential curve

$$BG(I) = B_L + (B_R - B_L) \cdot \frac{\sum_{j=L}^{j=I} Y(j)}{\sum_{K=L}^{K=R} Y(K)} \qquad (2)$$

where,
BG(I) = computed background at channel I,
Y(I) = spectra counts of channel I,
B_L = average counts in left background window, and
B_R = average counts in right background window.

3. Linear curves are obtained for both region of below and above the K-edge jump of the spectrum b linear least-squares fit.
4. The transmission is obtained by the extrapolation of the fitted lines as shown above. The non-extrapolated way is also studied, where specific $\triangle \mu$ is obtained for the closest channels of the fitting regions to the K-edge.

The uranium concentration is calculated from the counting ratio C(Eu) / C(El) with a correction using factor of the mass attenuation coefficient of uranium.

3-2. X-Ray Fluorescence Spectrometer

The XRF spectrum of uranium and plutonium is shown in Fig. 7 where the peaks of U-K α 1 and Pu- K α 1 are 98.44 keV and 103.76 keV, respectively. The ratio of the concentration of uranium to that of plutonium is determined from the ratio of the net peak area, P(U-K α 1) / P(Pu- K α 1) . Therefore the U / Pu weight ratio, combined with the uranium concentration obtained by the KED, accurately yields the plutonium concentration.

Figure 7 shows the XRF spectrum of the same dissolver solution of spent fuel as that used in the above experiment for KED. The ratio of U / Pu is obtained from the ratio of net peak area of the U-K α 1 and Pu- K α 1 X-rays. Outline of the procedure for the calculation is as follows;

1. Energy calibration is performed with the channels of uranium K α 1, K α 2 and K β 1.
2. Background counting of the regions of interest is subtracted by the same manner as in the KED.
3. Energy resolution (FWHM) is determined by Gaussian fit to each peak of K α 1, K α 2 and K β 1, then the relation of FWHM-energy is calculated by a linear least squares fit.
4. The count rates of the peaks, U- K α 1 and Pu- K α 1 X-rays, are calculated from their area obtained from the background-subtracted spectrum.
5. A factor, F, based on the overall relative detection efficiency [1], is calculated, which should be determined by an initial calibration since F is dependent on the characteristic of detector and the shape of measurement cell.
6. The correction factor for self-absorption, $R_{U/Pu}$, is introduced, based upon the method proposed by Ottmar et al [1]. Mass ratio of uranium to plutonium is given as follows;

$$\frac{U}{Pu} = \frac{A(U)}{A(Pu)} \cdot \frac{P(U-K\alpha 1)}{P(Pu-K\alpha 1)} \cdot \frac{F}{R_{U/Pu}} \qquad (3)$$

Where,
A = atomic weight,
P = net peak area of the K α 1 X-rays,
F = overall factor, and
$R_{U/Pu}$ = ratio of excitation probabilities for emission of U- K α 1 and Pu- K α 1 X-ray in the primary X-ray beam.

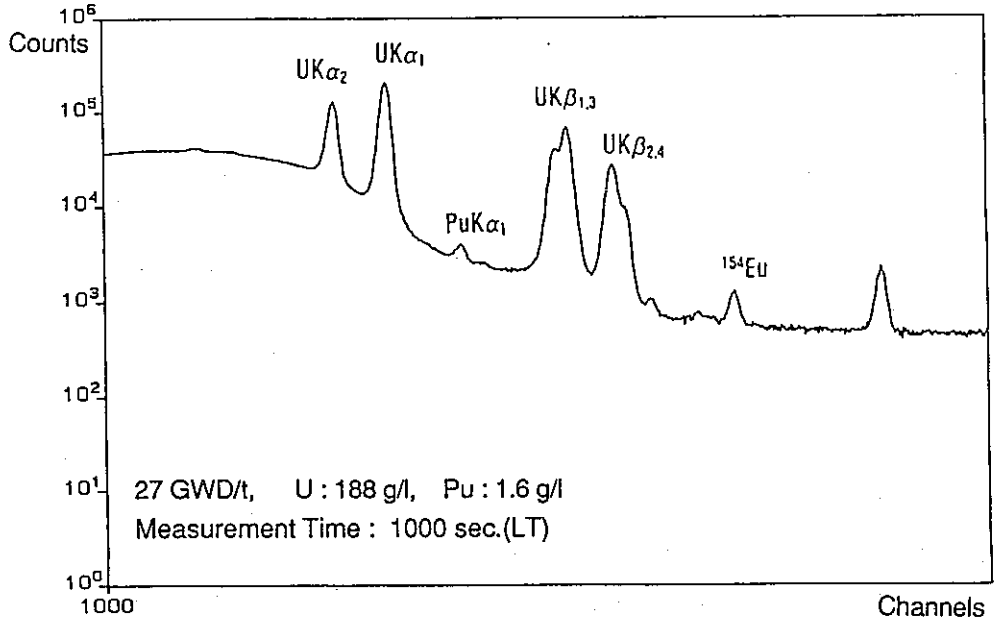

Fig. 7 Typical XRF spectrum of spent-fuel dissolver solution

4. EXPERIMENTAL

4-1. Control Sample

For the purpose of measurement control, namely, to check the instrument's stability, two reference samples, U-foil (approx. 0.3 mm in thickness) for KED and a sealed glass cuvette containing a U/Pu MOX powder for XRF, were prepared. These samples are mounted into a slide table beside the measurement flow-cell of sample solutions and remotely set at the appropriate position when a control measurement is conducted. The precision for determining U concentration (approx. 230 g/L) by KED (Non-extrapolated) is 0.2% for a 1000-s counting and the precision for determining U/Pu ratio (approx. 18 relatively) by XRF is 0.9% for a 1000-s counting. As shown in Fig. 8, the results of 45 control measurements of the U-foil reference sample during a period of 6 months indicate long-term stability. In the case of the MOX powder, good results were also obtained as shown in Fig. 9.

Table 1 lists the MC-Bias results obtained during the performance tests held in December 1995 and October 1996. We would preliminary fix the reference values and their reject limits according to these data and the calibration constants obtained above. The summary is shown below;

	U-Conc.	*U/Pu ratio*
Reference Value	232.03	18.83
Warning Limit (2 σ)	0.34 %	1.82 %
Action Limit (3 σ)	0.51 %	2.73 %

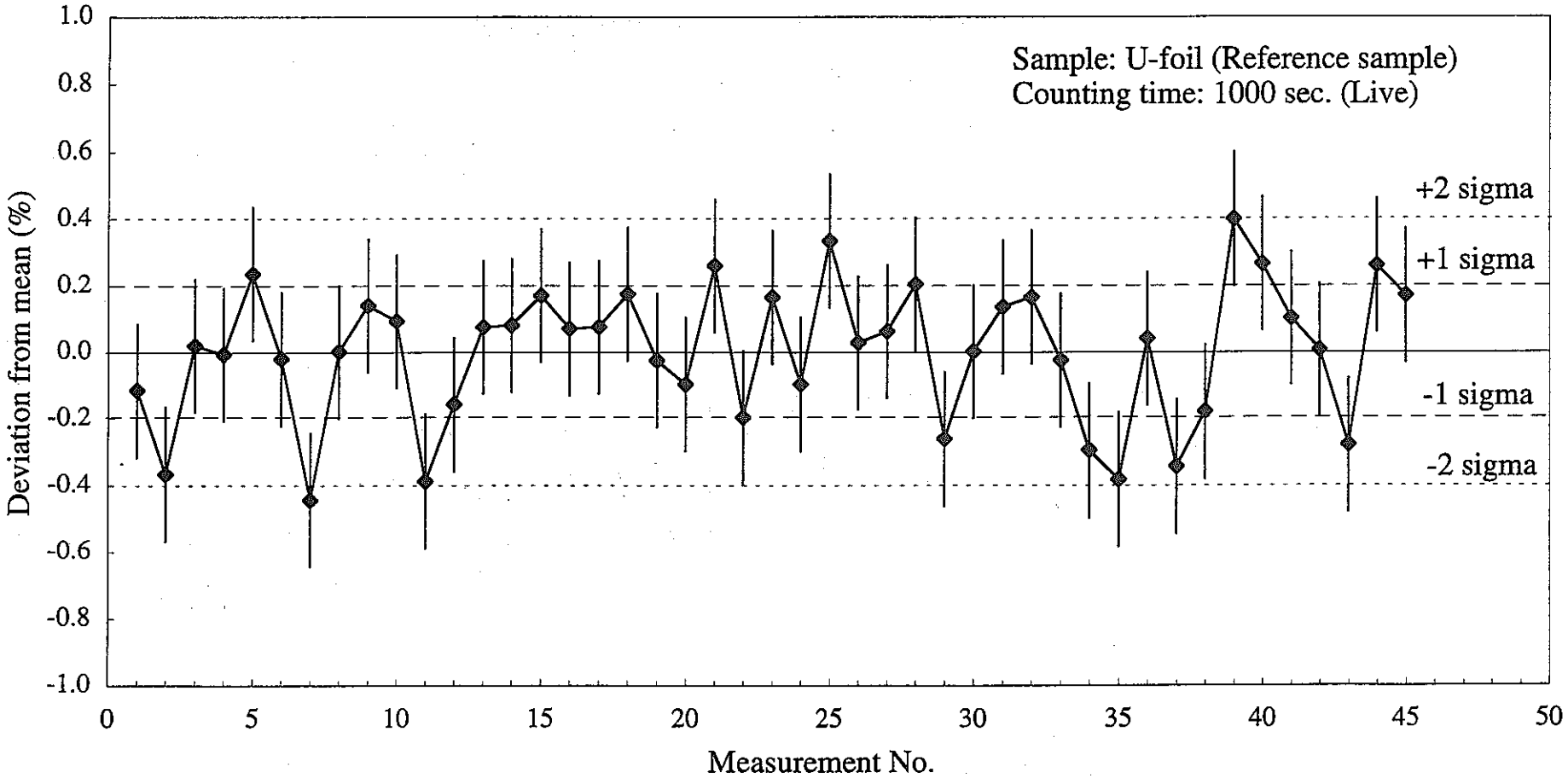

Fig. 8 Results of control measurement for KED

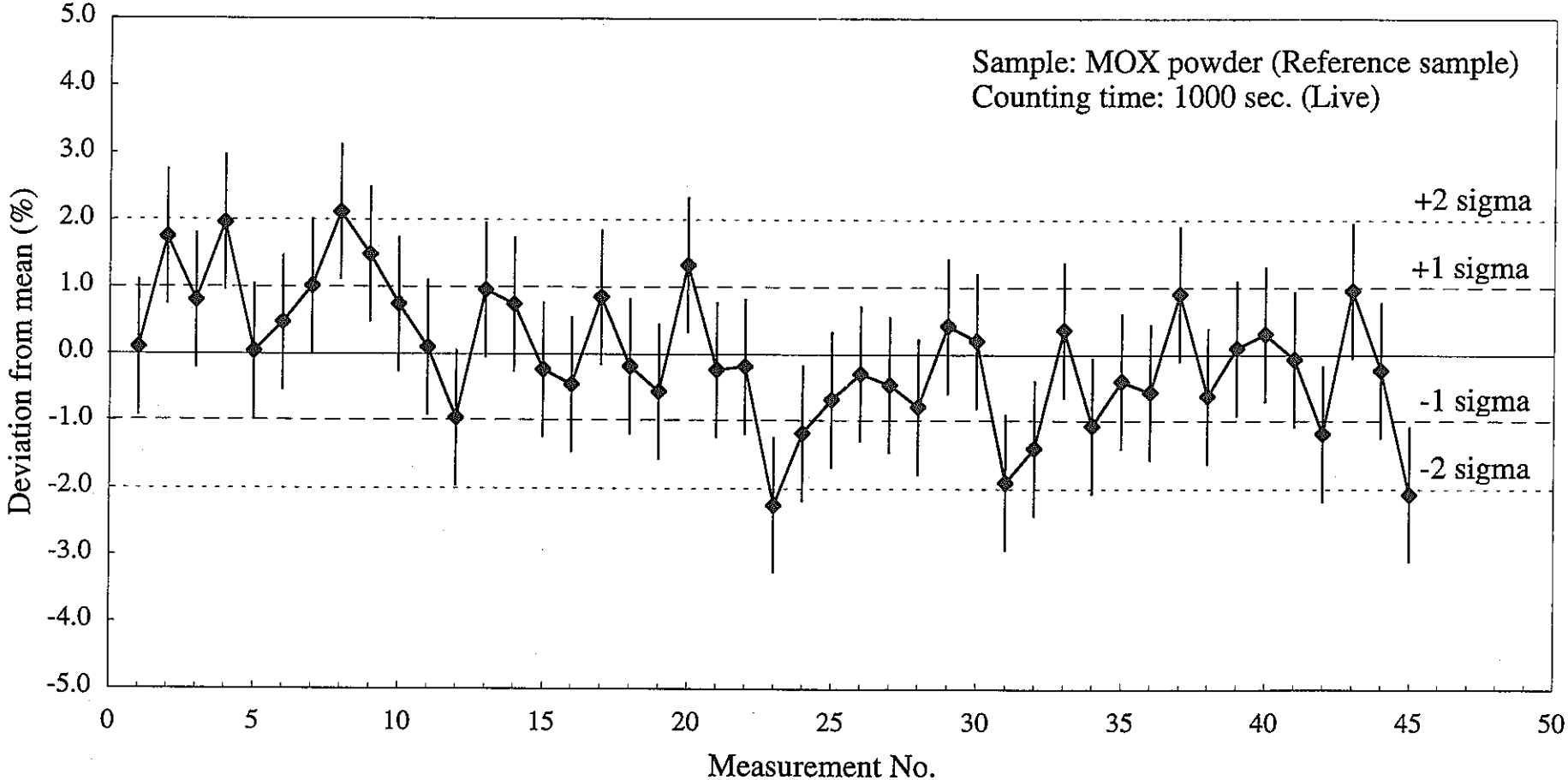

Fig. 9 Results of control measurement for XRF

Table 1. MC-Bias results

DATE	U Conc.	U/Pu Ratio
28-Apr-95	231.31	18.35
28-Apr-95	230.73	18.66
10-May-95	231.62	18.48
12-May-95	231.56	18.70
17-May-95	232.12	18.34
19-May-95	231.53	18.42
19-May-95	232.39	18.12
23-May-95	231.92	18.16
23-May-95	231.90	18.61
24-May-95	231.79	18.47
24-May-95	230.68	18.35
25-May-95	231.21	18.16
26-May-95	231.75	18.51
29-May-95	231.76	18.47
29-May-95	231.97	18.29
30-May-95	231.74	18.25
30-May-95	231.75	18.49
31-May-95	231.98	18.30
1-Jun-95	231.52	18.23
1-Jun-95	231.35	18.58
1-Nov-95	232.18	18.29
14-Dec-95	231.12	18.30
15-Dec-95	231.96	17.93
18-Dec-95	231.35	18.12
19-Dec-95	232.35	18.21
20-Dec-95	231.64	18.28
21-Dec-95	231.72	18.25
22-Dec-95	232.05	18.19
15-Oct-96	232.00	18.49
16-Oct-96	231.70	18.42
17-Oct-96	231.80	18.35
18-Oct-96	231.82	18.22
21-Oct-96	231.73	18.55
22-Oct-96	232.41	18.41
23-Oct-96	231.98	18.32
24-Oct-96	231.88	18.38
Ave.	231.73	18.35
S.D.	0.40	0.17
RSD%	0.17	0.91

4-2. Measurement Precision

To evaluate the reproducibility of the system and to verify that the hardware and software were constructed and installed as required, an acceptance test was held in 1995 in attendance with national and international inspectors. A typical input solution (177 gU/L and 1.7gPu/L) was employed for the test. The measurements were repeated 13 times continuously. As the results, the reproducibility (n=13) of replicate measurements for KED (Non-extrapolated) was estimated 0.1% RSD for 1000-s counting, whereas that of XRF was 0.6% RSD for 1000-s counting. These results satisfy the expected values ; less than 0.5% for U (with concentration around 200 gU/L) and less than 1.0% for Pu (with concentration around 2 gPu/L) based on no more than one hour real counting time.

For obtaining the reproducibility of the system on the samples reprocessed during campaign as usual, several sets of duplicate measurements were conducted as a part of performance rest in 1995 and 1996. Table 2 shows the individual results of the duplicate measurements, their averages and the corresponding standard deviations. The average standard deviations of 0.20 % rel. and 0.73 % rel. are estimates of the measurement precision of the KED (Non-extrapolated) and the XRF, respectively.

Table 2. HYBRID KED/XRF Performance

No.	Batch No.	U Conc.(Non-extrapolated)				U/Pu Ratio				Pu Conc.			
		1st Meas.	2nd Meas.	Ave.	Std. Dev. (% rel.)	1st Meas.	2nd Meas.	Ave.	Std. Dev. (% rel.)	1st Meas.	2nd Meas.	Ave.	Std. Dev. (% rel.)
1	SH1-223	150.68	150.66	150.67	0.01	137.01	138.05	137.53	0.53	1.0998	1.0913	1.0956	0.54
2	SH1-224	167.46	167.11	167.29	0.15	126.09	127.77	126.93	0.94	1.3281	1.3079	1.3180	1.08
3	SH1-225	185.70	185.38	185.54	0.12	130.56	131.23	130.90	0.36	1.4223	1.4126	1.4175	0.48
4	SH1-226	166.49	166.14	166.32	0.15	133.00	136.44	134.72	1.81	1.2518	1.2177	1.2347	1.95
5	SH1-227	177.45	176.48	176.97	0.39	133.50	132.62	133.06	0.47	1.3292	1.3307	1.3300	0.08
6	SH1-228	163.39	163.74	163.57	0.15	126.97	126.55	126.76	0.23	1.2868	1.2939	1.2904	0.39
7	SH1-229	174.32	173.91	174.12	0.17	129.17	127.89	128.53	0.70	1.3495	1.3598	1.3547	0.54
8	SH1-230	170.54	169.48	170.01	0.44	132.33	129.73	131.03	1.40	1.2887	1.3064	1.2976	0.96
9	SH1-231	177.89	176.46	177.18	0.57	136.42	135.38	135.90	0.54	1.3040	1.3034	1.3037	0.03
10	SH1-232	166.59	166.99	166.79	0.17	130.74	129.57	130.16	0.64	1.2742	1.2888	1.2815	0.81
11	SH1-233	171.79	171.89	171.84	0.04	124.46	128.06	126.26	2.02	1.3803	1.3423	1.3613	1.98
12	SH1-234	163.59	163.76	163.68	0.07	131.39	131.16	131.28	0.12	1.2451	1.2486	1.2468	0.20
13	TK2-391	149.84	149.72	149.78	0.06	125.89	125.44	125.67	0.25	1.1902	1.1936	1.1919	0.20
14	TK2-392	162.52	161.32	161.92	0.52	115.08	117.28	116.18	1.34	1.4122	1.3755	1.3939	1.86
15	TK2-393	153.42	153.45	153.44	0.01	123.28	124.88	124.08	0.91	1.2445	1.2288	1.2366	0.90
16	TK2-394	154.17	153.35	153.76	0.38	126.82	125.39	126.11	0.80	1.2157	1.2230	1.2193	0.42
17	TK2-395	145.61	145.39	145.50	0.11	120.69	121.83	121.26	0.66	1.2065	1.1934	1.1999	0.77
18	TK2-396	165.27	164.96	165.12	0.13	118.24	119.33	118.79	0.65	1.3978	1.3824	1.3901	0.78
19	TK2-397	156.50	156.53	156.52	0.01	121.82	120.36	121.09	0.85	1.2847	1.3005	1.2926	0.87
20	TK2-398	169.82	170.25	170.04	0.18	128.21	127.55	127.88	0.36	1.3245	1.3348	1.3297	0.54
21	TK2-399	169.69	169.43	169.56	0.11	120.60	120.13	120.37	0.28	1.4070	1.4104	1.4087	0.17
22	TK2-400	178.48	178.94	178.71	0.18	121.20	119.22	120.21	1.16	1.4726	1.5009	1.4868	1.35
23	TK2-401	156.46	156.73	156.60	0.12	121.19	120.48	120.84	0.42	1.2910	1.3009	1.2960	0.54
24	TK2-402	171.15	170.10	170.63	0.44	122.41	122.77	122.59	0.21	1.3982	1.3855	1.3918	0.64
25	TK2-403	161.21	160.93	161.07	0.12	120.06	119.79	119.93	0.16	1.3427	1.3434	1.3431	0.04
26	TK2-404	175.76	176.23	176.00	0.19	119.38	121.52	120.45	1.26	1.4723	1.4502	1.4612	1.07
27	TK2-405	168.07	167.63	167.85	0.19	121.14	118.85	120.00	1.35	1.3874	1.4104	1.3989	1.16
28	TK2-406	166.56	167.26	166.91	0.30	120.14	120.19	120.17	0.03	1.3864	1.3916	1.3890	0.27
				Ave.	0.20			Ave.	0.73			Ave.	0.74

4-3. Comparison of H-KED and DA

In order to evaluate the performance on the actual samples measured as the above precision test, several sets of parallel with DA measurements were made. Table 3 shows the results of the 28 H-KED measurements compared with the declared values by operator DA. The summary is shown below;

H-KED/DA(PNC)	*Average Difference (% rel.)*	*Standard Deviation (%)*
U conc. (Non-extrapolated)	-0.13	0.45
U conc. (Extrapolated)	0.20	0.53
U/Pu ratio	-2.59	1.09
Pu conc.	2.54	1.19

The relative standard deviation of the difference between U measurements (NDA-Non-extrapolated and DA) was around 0.5 %, whereas that of Pu measurement was around 1.2 %. These values include errors due to the H-KED measurement, sample handling and uncertainties in the DA. The difference on the Pu measurement precision between the results of the above mentioned reproducibility test and this performance test may also be due to the concetration difference of Pu used (Average of Pu for NDA/DA comparison is 1.3 g/L, whereas, 1.7 g/L for the reproducibility test). Therefore, it would be said that the precision found in this NDA/DA comparison does not give any problems referring to the requirement of the NDA verification of U and Pu in the input solutions.

Although the average difference of Pu seems to have significant bias, it is able to be adjusted by a correction factor.

Table 3. Results of HYBRID KED/XRF parallel test at TRP

No.	Batch No.	U Concentration					U/Pu Ratio			Pu Concentration		
		(Non-extrapolated)			(Extrapolated)							
		H-KED	IDMS (PNC)	H-KED/IDMS (RD %)	H-KED	H-KED/IDMS (RD %)	H-KED	IDMS (PNC)	H-KED/IDMS (RD %)	H-KED	IDMS (PNC)	H-KED/IDMS (RD %)
1	SH1-223	150.67	151.42	-0.50	152.13	0.47	137.53	141.59	-2.87	1.096	1.069	2.44
2	SH1-224	167.29	168.10	-0.48	167.70	-0.24	126.93	130.24	-2.54	1.318	1.291	2.11
3	SH1-225	185.54	185.24	0.16	184.52	-0.39	130.90	134.51	-2.69	1.417	1.377	2.93
4	SH1-226	166.32	167.15	-0.50	165.80	-0.81	134.72	137.43	-1.97	1.235	1.216	1.50
5	SH1-227	176.97	177.65	-0.38	176.61	-0.58	133.06	136.96	-2.84	1.330	1.297	2.53
6	SH1-228	163.57	163.77	-0.12	163.71	-0.04	126.76	130.72	-3.03	1.290	1.253	3.00
7	SH1-229	174.12	173.21	0.53	174.90	0.98	128.53	129.27	-0.57	1.355	1.340	1.10
8	SH1-230	170.01	169.69	0.19	169.78	0.05	131.03	132.33	-0.98	1.297	1.282	1.18
9	SH1-231	177.18	176.32	0.48	177.41	0.62	135.90	141.74	-4.12	1.304	1.244	4.80
10	SH1-232	166.79	166.73	0.04	167.81	0.65	130.16	133.04	-2.17	1.281	1.253	2.26
11	SH1-233	171.84	171.20	0.38	172.64	0.84	126.26	128.94	-2.08	1.361	1.328	2.51
12	SH1-234	163.68	163.85	-0.11	163.88	0.02	131.28	134.05	-2.07	1.247	1.222	2.01
13	TK2-391	149.78	151.09	-0.86	150.99	-0.06	125.67	126.88	-0.96	1.192	1.191	0.09
14	TK2-392	161.92	162.18	-0.16	162.95	0.48	116.18	117.84	-1.41	1.394	1.376	1.27
15	TK2-393	153.44	154.77	-0.86	154.26	-0.33	124.08	128.09	-3.13	1.237	1.208	2.34
16	TK2-394	153.76	154.13	-0.24	155.41	0.83	126.11	128.84	-2.12	1.219	1.196	1.93
17	TK2-395	145.50	147.53	-1.38	146.59	-0.64	121.26	126.61	-4.22	1.200	1.165	2.97
18	TK2-396	165.12	165.22	-0.06	165.44	0.13	118.79	122.21	-2.81	1.390	1.352	2.82
19	TK2-397	156.52	156.41	0.07	157.84	0.92	121.09	122.40	-1.07	1.293	1.278	1.15
20	TK2-398	170.04	170.01	0.02	170.50	0.29	127.88	129.43	-1.20	1.330	1.313	1.23
21	TK2-399	169.56	169.18	0.23	170.45	0.75	120.37	125.35	-3.98	1.409	1.350	4.38
22	TK2-400	178.71	177.63	0.61	178.98	0.76	120.21	125.38	-4.13	1.487	1.417	4.94
23	TK2-401	156.60	157.10	-0.32	157.52	0.27	120.84	125.54	-3.75	1.296	1.251	3.56
24	TK2-402	170.63	169.96	0.39	171.31	0.79	122.59	127.06	-3.52	1.392	1.338	4.05
25	TK2-403	161.07	161.33	-0.16	161.89	0.35	119.93	123.49	-2.88	1.343	1.306	2.80
26	TK2-404	176.00	176.30	-0.17	175.76	-0.31	120.45	124.97	-3.61	1.461	1.411	3.57
27	TK2-405	167.85	167.77	0.05	168.45	0.41	120.00	122.36	-1.93	1.399	1.371	2.02
28	TK2-406	166.91	167.51	-0.36	166.56	-0.57	120.17	125.09	-3.94	1.389	1.339	3.73
			Ave.	-0.13	Ave.	0.20		Ave.	-2.59		Ave.	2.54
			STD	0.45	STD	0.53		STD	1.09		STD	1.19

Based on the 28 DA results, the H-KED shows negatively biased values, particularly U/Pu ratio. The presently used calibration constants for KED and XRF should be updated as follows. However, the calibration constants should be applied the grand mean difference between the H-KED and DA results of the three laboratories (JNSB, IAEA and PNC).

	Presently used	*Updated to*
KED U-conc. (Non-extrapolated)	3.20694	3.20277
KED U-conc. (Extrapolated)	3.63525	3.64252
XRF U/Pu ratio	0.99548	1.02126

5. CONCLUSION

A NDA instrument which can allow more precise and rapid assay has been developed to quantify and verify nuclear materials of the input solutions at the TRP.

The performance for uranium determination with KED was confirmed to be excellent; an error of around 0.2% for 1000 sec. counting, while that for plutonium by the combination of KED and XRF spectrometer was also found to be satisfactory with an error of around 1% for 1000 sec. counting. It can be concluded that this system, H-KED, is sufficiently applied to in-situ determination of uranium/plutonium in a LWR fuel reprocessing input dissolver solutions for the safeguards verification purpose. Hereafter, the system will be implemented for the routine inspection at the Tokai Reprocessing Plant.

6. REFERENCES

[1] H. Ottmar and H. Eberle, "The Hybrid K-Edge/ K-XRF Densitometer: Principles/Design/Performance," KfK-4590 Kernforschungszentrum Karlsruhe report (1991).

[2] A. Kurosawa, K. Abe, O. Kitagawa, Y. Kuno, and J. Masui, "Richman's Densitometer," in Proceedings of the 15th Annual ESARDA Symposium, Rome, Italy, (1993) 255-260.

[3] K. Abe, A. Kurosawa, N. Surugaya, Y. Kuno, and J. Masui, "Further Improvement of Richman's Densitometer," in Proceedings of the IAEA Symposium on International Safeguards, Vienna, 14-18 March 1994.

[4] N. Surugaya, K. Abe, A. Kurosawa, M. Hinoda, H. Ikeda, and Y. Kuno, "Implementation of Non-Destructive Assay Systems for Efficient Verification of Nuclear Materials in Reprocessing Plant - Hybrid K-edge/XRF Densitometer and Advanced K-edge Densitometer -" in Proceedings of the INNM 37th Annual Meeting, Naples, Florida, 28 July - 1 August, 1996.

[5] R. Gunnink, W. D. Ruther, UCRL-52917 (1980).

TOKAI HYBRID K-EDGE/XRF DENSITOMETER (H-KED)

USER MANUAL

Tokai reprocessing plant
Power Reactor & Nuclear Fuel
Development Corporation

Tokai Hybrid K-edge/XRF Densitometer

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HYBRID K-EDGE/XRF DENSITOMETER (H-KED)

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Development Corporation

ABSTRACT

As a part of JASPAS programme, a non-destructive assay system for the accountability of uranium and plutonium in input dissolver solutions of a spent fuel reprocessing plant, "Hybrid KED/XRF Densitometer (H-KED)" has been developed at the Tokai Reprocessing plant (TRP) since 1991. The system is made up of the combination of a K-edge densitometer (KED) and an X-ray fluorescence (XRF) spectrometer. The KED is used to determine the uranium concentration, while XRF analysis is used to determine U/Pu weight ratio. The plutonium concentration can be calculated from both the measurement values. The measurement precision of an assay depends on the counting time chosen, but can be within 0.5% for uranium (approx. 180 g/L, n=10, LT:1000s.) and within 1.0% for plutonium (approx. 2 g/L, n=10, LT:1000s.). This manual describes the operation of the instrument, including software and hardware information.

INTRODUCTION

GENERAL

This document describes the operation of the application software of the "TOKAI HYBRID KED/XRF Densitometer" (H-KED). Other related manuals are the H-KED hardware and software information, also including available components information.

This system comprises of an X-ray generation tube and two HpGe semiconductor detectors. The system is made up of the combination of a K-edge densitometer (KED) and an X-ray fluorescence (XRF) spectrometer. The KED is used to determine the uranium concentration, while the XRF analysis is used to determine U/Pu weight ratio. The plutonium concentration can be calculated from both the measurement values. It uses an active non-destructive assay technique to measure the uranium and the plutonium concentration of the reprocessing input solution.

MEASUREMENT TECHNIQUE

The system shown in Fig.1 consists of an X-ray generation tube (COMET, MXR-160, 0.4-3.0, MAX. 160 kV, 19 mA), two Ge detectors (EG&G, LOAX type) and two sample holders (cells) in measurement cell system. The sample cell for KED has a 25 mm pass length and that for XRF is a 10 mm diameter of cylindrical cell. They are connected each other with stainless steel tube to be simultaneously filled with less than 8 ml of sample solution. The instrument was designed to be installed compactly (1m width, 1m height, 0.6m deep) into a small hot cave of lead shielding. The electronics, controller and CPU have been installed outside the shielded cell.

Primary X-ray from the generation tube is separated into the KED section and the XRF one by collimator. Each X-ray irradiates the sample holders filled with sample solution. The beam collimated less than 1 mm diameter, is introduced into the measurement cell to analyze the K-edge absorption at the KED section. The XRF spectrum induced by the primary X-ray is measured by another detector.

The characteristics of the system are as follows:

- The flow type cell system is employed to facilitate the remote operation of sample loading.

- The equipment is worked up compactly to install into the limited space.
- Energy calibration for the KED is performed with X-ray fluorescence of Pb (75.0 keV) from lead shield and K-edge absorption point of uranium (115.6 keV). X-ray fluorescence of uranium, Kα-1, Kα-2 and Kβ-1 are used for energy calibration for XRF analysis
- Uranium foil (approx. 0.3 mm in thickness) for KED, whereas a solidified MOX standard composed of uranium, JEARI U-4, and plutonium, NBL CRM-126, for XRF, are applied for quality control of the H-KED system. These reference samples are installed on a slide table in the measurement cell system and can remotely set at the appropriate position when MC-Bias measurement is conducted.
  The flow type cell can be observed from the outside by using CCD camera to confirm whether or not input solution is properly filled with.

Fig.2 shows the spectrum of typical input solution obtained with the KED, where the X-ray instrument was operated at fixed power of 150 kV/12 mA. The discontinuity observed is the K-edge point of the uranium, 115.6 keV.

The uranium concentration should be in promotion to the ratio of the detector counting at the upper energy (Eu) side to that at the lower energy (El) side in the KED, as given in the following equation.

$$U\,(g/L) = 1/(\Delta\mu \cdot d) \cdot \ln(C(En)/C(El))$$

where d is the sample thickness, 25 mm, and $\Delta\mu$ denotes difference of mass absorption coefficients for the energies Eu and El. As shown in Fig.2 the uranium concentration can be determined with the counting ratio of C(Eu) and C(El) at the channel of the K-edge where C(Eu) and C(El) are obtained from the counting values observed in the windows, whereas, K-edge point is determined by the differential curve of spectrum. Background counting arising from gamma ray, compton scatter, pile-up and so on due to the dissolver solution itself is subtracted from the spectrum of KED.

The XRF spectrum is shown in Fig. 3 where the peaks of U-Kα1 and Pu-Kα2 are 98.44 keV and 103.76 keV, respectively. The weight ratio of U/Pu is determined by the equation shown below. The factor, F, based on the overall relative detection efficiency, is

calculated, which should be determined by an initial calibration since F is dependent on the characteristic of detector and the shape of measurement cell.

$$\frac{U}{Pu} = \frac{A(U)}{A(Pu)} \frac{P(U\text{-}K\alpha 1)}{P(Pu\text{-}K\alpha 1)} \frac{F}{R_{U/Pu}}$$

where A: atomic weight, P: net peak area of the Kα1 X-rays, F: overall factor, $R_{U/Pu}$: ratio of excitation probabilities for emission of U-Kα1 and Pu-Kα1 in the primary X-ray beam.

MEASUREMENT PROCEDURE

The option of MC-Bias measurement should be carried out for quality control before every assay measurement. An assay consists of two independent concentration measurements. The results must be consistent or else the sample is checked for bubbles and then a third time.

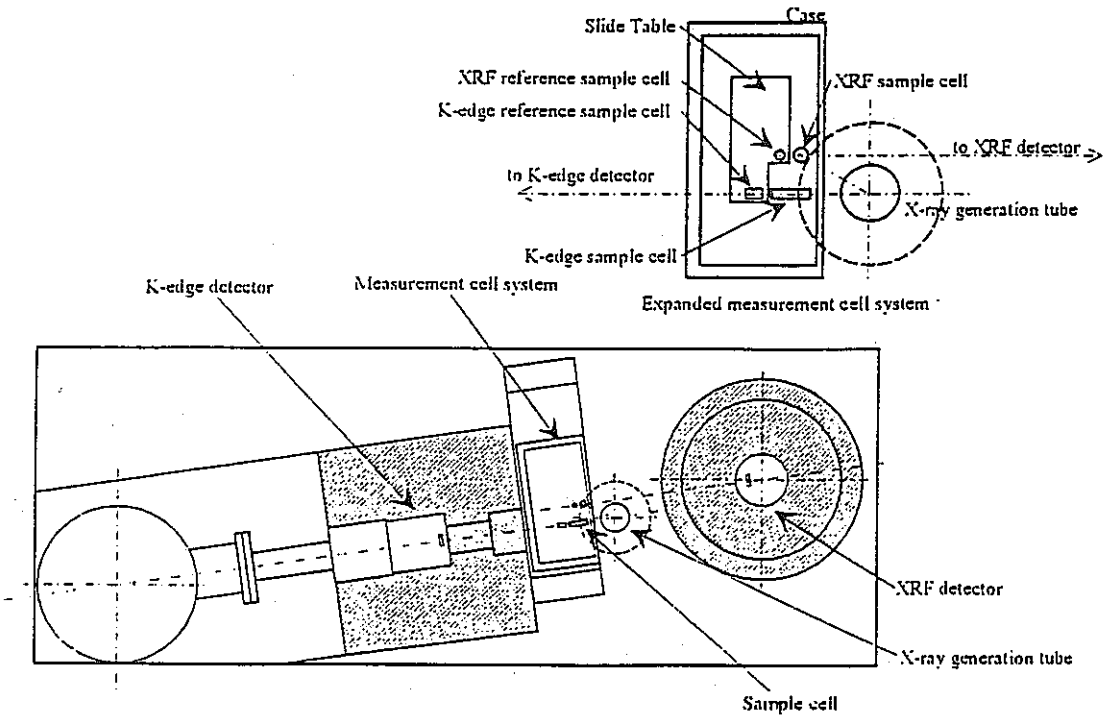


Fig. 1 The measurement geometry of Hybrid K-edge/XRF densitometer

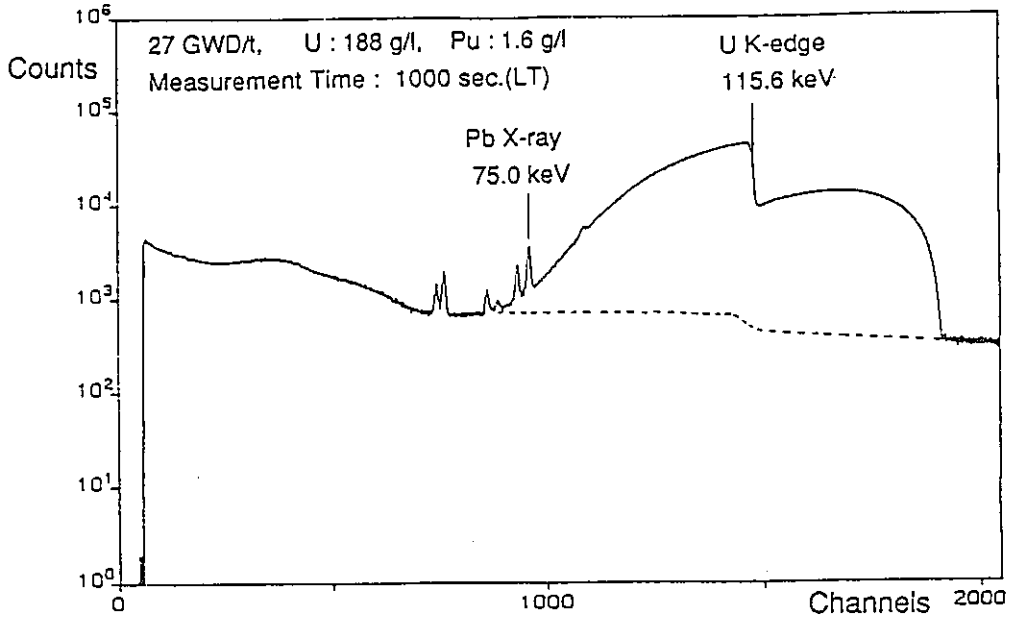


Fig.2 Typical K-edge spectrum of spent-fuel dissolver solution

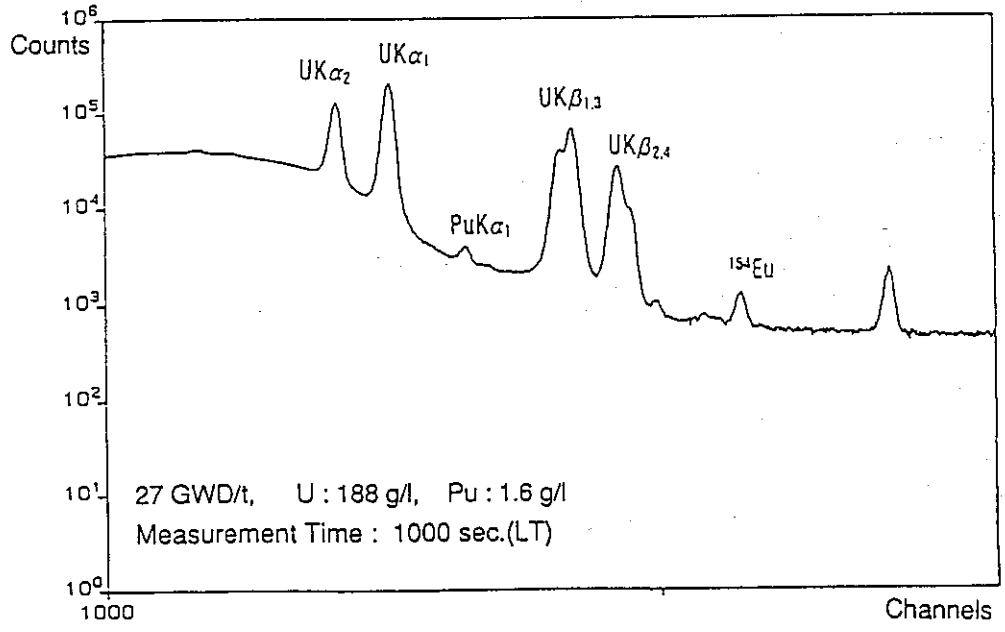


Fig. 3 Typical XRF spectrum of spent-fuel dissolver solution

1.Outline

(1)Software Configuration

This Program has the following menus:

1. Measurement

1)KED/XRF Assay

To conduct automatic measurement and analysis of KED/XRF (bubble test is also possible).

2)KED U Assay

To conduct automatic measurement and analysis of KED of U (bubble test is also possible).

3)KED Pu Assay

To conduct automatic analysis of KED of Pu (bubble test is also possible).

4)MC Bias

To confirm MC bias by means of the reference value.

5)MC Precision

To confirm MC precision by means of the reference value, using the χ^2 (chi-square) test.

6)Autocycle

To conduct automatic measurement and analysis of KED/XRF of a given number of times.

2. Archives

1)Copy Spectrum Files

To copy spectrum files.

2)Format New Diskette

To format floppy disks.

3)List Log

This option allow the user to output the measurement results.

3. Parameters

1)Miscellaneous

To set data relevant to the printer and the disk.

2)Count Times

To set data relevant to spectrum measurement time and others.

3)Edit parameters for KED (U)

To set the parameters for KED (U) analysis.

4)Edit parameters for KED (Pu)

To set the parameters for KED (Pu) analysis.

5)Edit parameters for XRF

To set the parameters for XRF analysis.

6)Edit parameters for MC Bias

To set the parameters for MC-Bias evaluation.

4. Analysis

1)Manual KED/XRF Assay

To repeat analysis of recorded KED/XRF spectra.

2)Manual KED U Assay

To repeat analysis of recorded KED spectra for U.

3)Manual KED Pu Assay

To repeat analysis of recorded KED spectra for Pu.

4)Manual XRF Assay

To repeat analysis of recorded XRF spectra.

5. Energy Calibration

To obtain formula showing the relation between the energy and the channel.

6. MCA Emulation (MCA Control)

This is the standard SEIKO EG&G "MCA Emulation" program. In this program, control operations of MCA and spectrum operations can be conducted by a PC.

7. MS-DOS Command

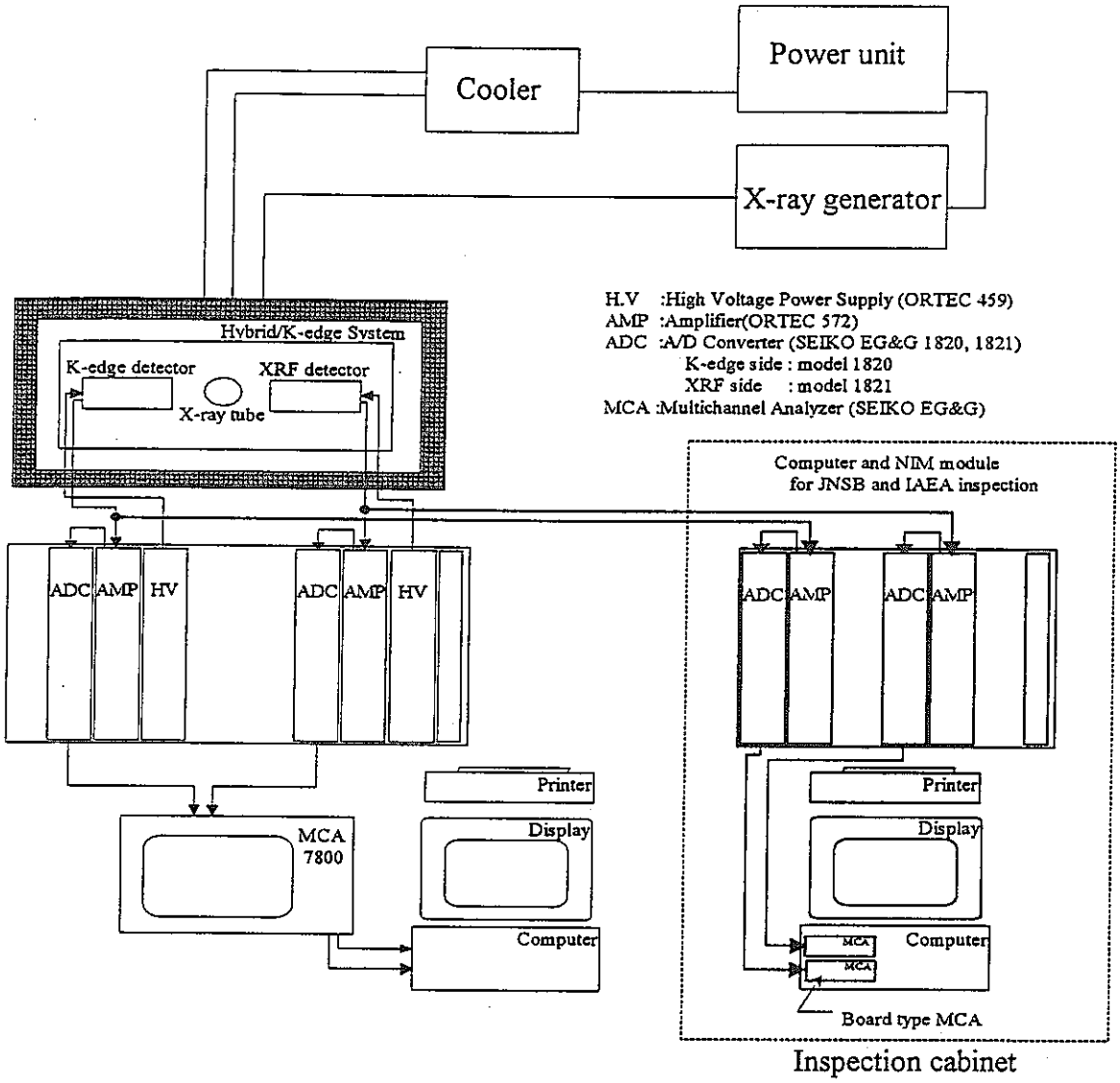
The MS-DOS commands can be executed without ending this Program.

(2)Hardware Configuration

The following is the hardware configuration of this System:

X-ray system	
X-ray tube	: COMET MXR-160/0.4-3.0
X-ray generator	: PHILIPS MG 164
Ge Detector	
KED	: EG&G ORTEC GLP-16195/10-S
XRF	: EG&G ORTEC GLP-16195/10-S
Electronics module	
Multichannel Analyzer (MCA)	: SEIKO EG&G 4200
Amplifier (AMP)	: ORTEC 572
A/D Converter (ADC)	: SEIKO EG&G 1820, 1821 K-edge side 1820 XRF side 1821
High Voltage Power Supply (H.V)	: ORTEC 459
Data Processing Unit	
Personal Computer	: NEC PC 9801BX2 FD(3.5inch. 2HD×2), HD(240MByte)
Display	: NEC Analog RGB
Printer	: NEC 80 characters/line

<Block diagram of Tokai Hybrid K-edge/XRF Densitometer>



2. Starting and Ending of the Program

(1)Starting this Program

This Program is started in the following procedure:

1. First start the electronic units for measurement and conduct adjustment if necessary.
   As to the starting and adjustment method of each module, refer to its operating manual.

2. Turn on the peripheral units of the personal computer.

3. Turn on the machines and devices controlled by the personal computer.

4. Turn on the personal computer.

5. The computer will automatically start the software when it is powered up.

(2)Ending this Program

This Program will be ended in the following procedure:

1. Press the Function Key 1 (f1) " Quit " in the Full menu and then press "Y" to confirm.

2. Turn off the personal computer.

3. Turn off the peripheral units of the personal computer.

4. Turn off the machines and devices controlled by the personal computer.

(3)Before Executing this Program

If the "Measurement" menu of this Program is executed, measurement of spectrum will be started, MCA being controlled by presetting done beforehand. Be sure to adjust the electronic devices for measurement (power source for high voltage, AMP, ADC, etc.) before executing the "Measurement".

* Soon after high voltage is applied, the field of the detector is not sufficiently stable. Keep running the detector for about several tens of minutes before it is applied high voltage.

*When adjustment of keV/ch is necessary, be sure to adjust AMP (gain) by means of the reference spectrum source (the reference test sample) before starting the measurement, so that the peak appears at a determined spot(ch).

As to the detailed adjustment method of the electronic devices for measurement, refer to their operation manuals.

Before executing the analysis program (KED/XRF) of this Program, the following data files must be available. Be sure to prepare and proofread them in advance.

<KED (K-edge analysis)>


<########. CHN> : Spectrum for K-edge measurement


<########. ROI> : ROI files for calculation of K-edge point and determination of Pb peak


<########. KEU> : K-edge Analysis Result Files for the K-edge spectra in corresponding pair

* K-edge analysis results are classified into the following two categories, depending upon the target element:
########. KEU : KEU: K-edge analysis result of U
########. KEP : KEP: K-edge analysis result of Pu
Among the above two, K-edge analysis result (KEU) is used for XRF analysis.

<XRF (X-ray Fluorescence Analysis)>

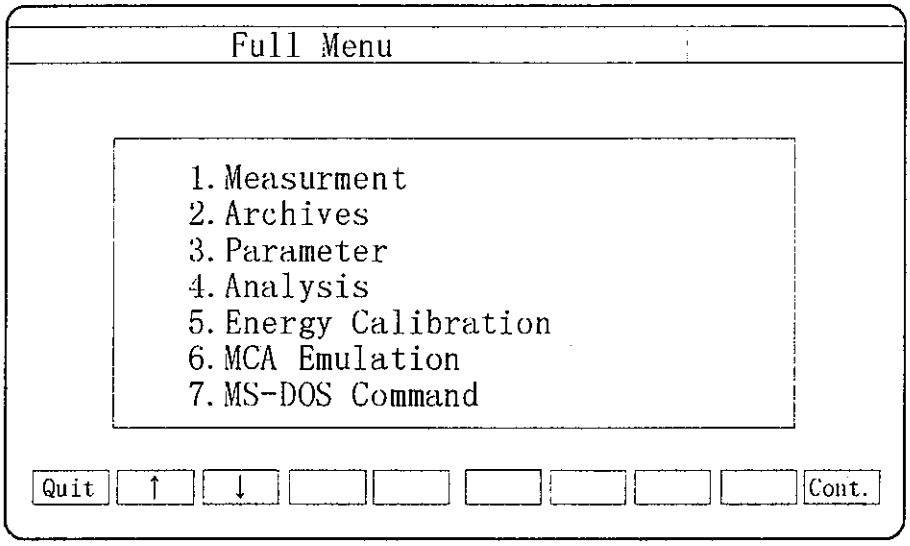

<########. CHN> : Spectrum for XRF measurement


<########. ROI> : ROI files for XRF Peak Analysis


<########. ENE> : Energy calibration Files for XRF

3.Operation

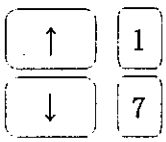
If this Program is started normally, the "**Full Menu**" shown below will appear on the display.



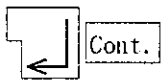
<Screen 1>

Position the cursor on the item to be executed, press the "**RET**" key or the "**Cont.**" key (**F/10** key) for execution.

If you want to execute using a mouse, position the mouse cursor on the desired item and click the left button of the mouse.



The cursor is moved by the arrow keys and/or the ten keys.

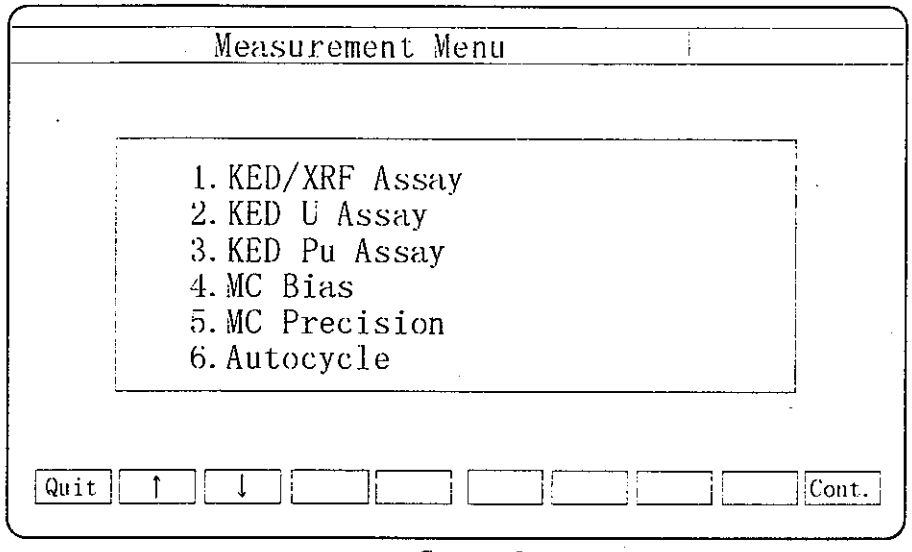


Pressing the "**RET**" key or the **F/10** key will execute the item on which the cursor stays.

To go back to the MS-DOS, press the **ESC** key or the **Quit** key "**F/1** key", then you will get it after confirmation.

(1)Measurement

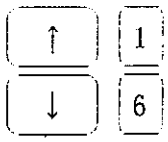
If the "**Measurement**" is executed, the Measurement Menu shown below will appear. Select the item you want to execute.



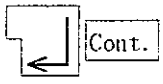
<Screen 2>

Position the cursor on the item to be executed, press the "**RET**" key or the "**Cont.**" key (F/10 key) for execution.

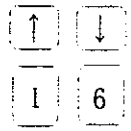
If you want to execute using a mouse, position the mouse cursor on the desired item and click the left button of the mouse.



The cursor is moved by the arrow keys and/or the ten keys.



Pressing the "**RET**" key or the **F/10** key will execute the item on which the cursor stays.



1 : Measurement

1 : KED/XRF Assay

To conduct automatic measurement and analysis of KED/XRF (bubble test is also possible).

2 : KED U Assay

To conduct automatic measurement and analysis of KED for U (bubble test is also possible).

3 : KED Pu Assay

To conduct automatic analysis of KED for Pu (bubble test is also possible).

4 : MC Bias

To confirm MC bias by means of the reference value.

5 : MC Precision

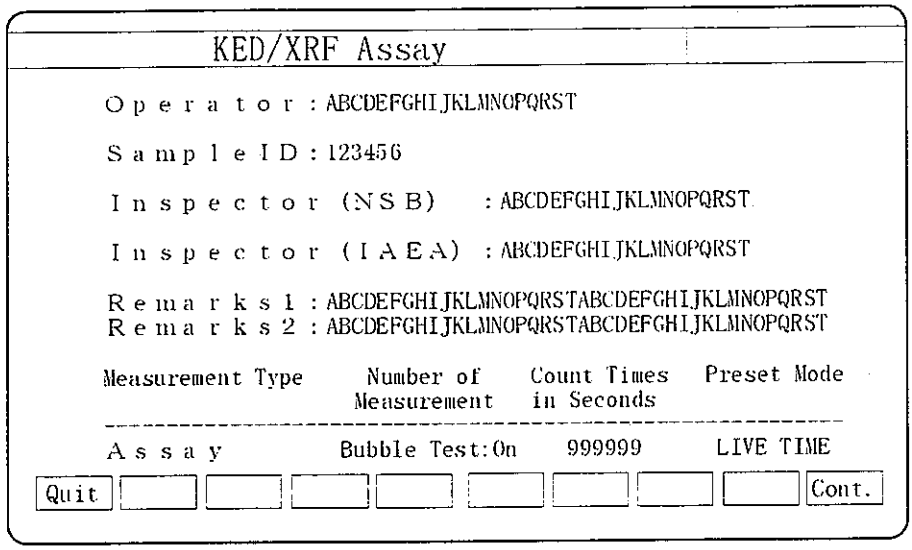
To confirm MC precision by means of the reference value, using the c^2 (chi square) test.

6 : Autocycle

To conduct automatic measurement of KED/XRF of a given number of runs.

(1-1)KED/XRF Assay

After measurement of the both spectra for KED/XRF is conducted at the specified conditions, the spectrum file will be stored at any name you want. Then KED analysis (for U conc.) and the XRF spectrum analysis (for Pu conc.) will automatically be started.



<Screen 3>

Enter the following items for KED/XRF automatic measurement and analysis.

Operator : Enter the name of the staff in charge of the KED/XRF automatic measurement and analysis. Limit: 20 characters

Sample ID : Enter the test sample ID. Limit: 6 characters

*The test sample ID entered here will make the spectrum file name.

Inspector(NSB) : Enter the inspector name (NSB). Limit:20 characters

Inspector(IAEA) : Enter the inspector name (IAEA). Limit:20 characters

Remarks1 : Enter the comments in two lines, each separately. Limit:40 characters

Remarks2 :

At this moment, the measurement conditions set by the "Parameter" are displayed at the lower part of the screen.

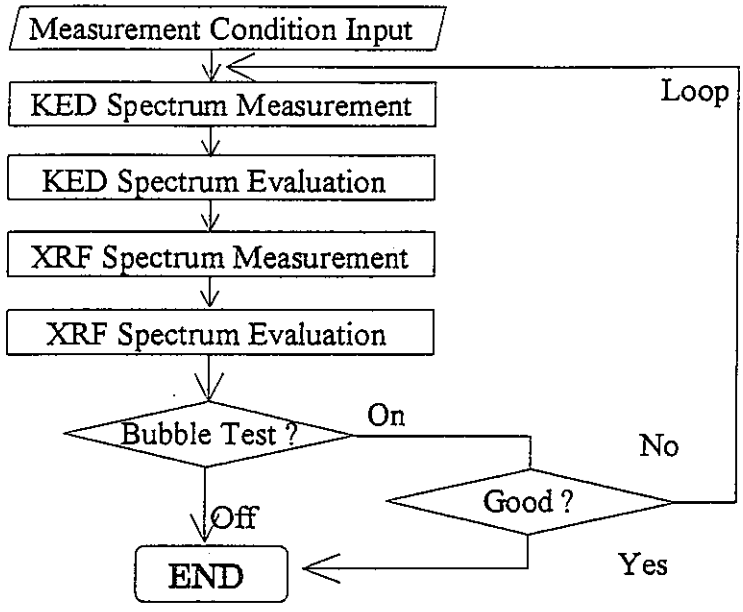
*The measurement conditions is not changed here.

After the necessary entries are made, the drive capacity for the spectrum registration and the same file name are checked by pressing the "Cont." key (F/10 key).

If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "**Miscellaneous**".

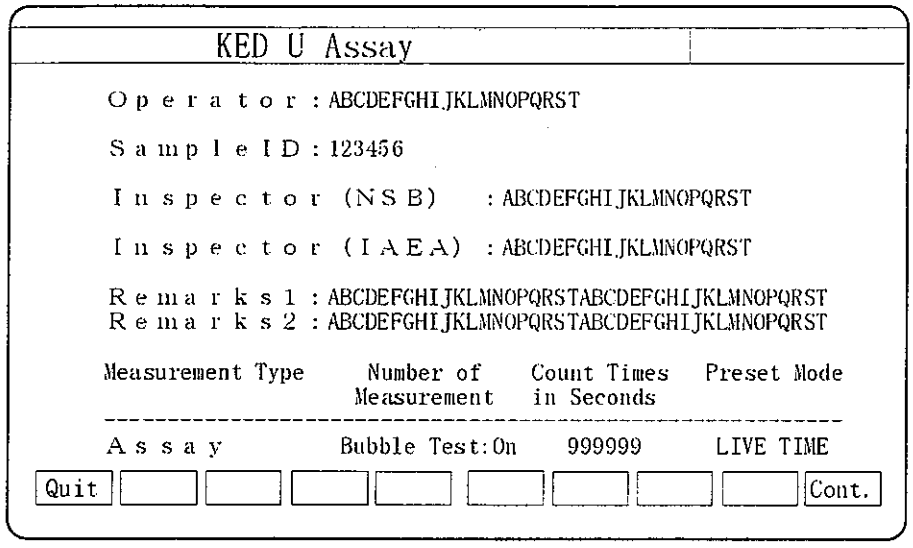
If no abnormality is found with the specified parameters, execute the "**KED/XRF Assay**".

< Process Rough Chart >



(1-2)KED U Assay

After measurement of KED spectrum is finished at the specified conditions, the spectrum file will be stored with a name you want. Then KED analysis (for U conc.) will automatically be started.



<Screen 4>

Enter the following items for KED(U) automatic measurement and analysis.

Operator : Enter the name of the staff in charge of the KED U Assay automatic measurement and analysis. Limit: 20 characters

Sample ID : Enter the test sample ID. Limit: 6 characters

*The test sample ID entered here will make the spectrum file name.

Inspector(NSB) : Enter the inspector name (NSB). Limit:20 characters

Inspector(IAEA) : Enter the inspector name (IAEA). Limit:20 characters

Remarks1 : Enter the comments in two lines, each separately. Limit:40 characters
Remarks2 :

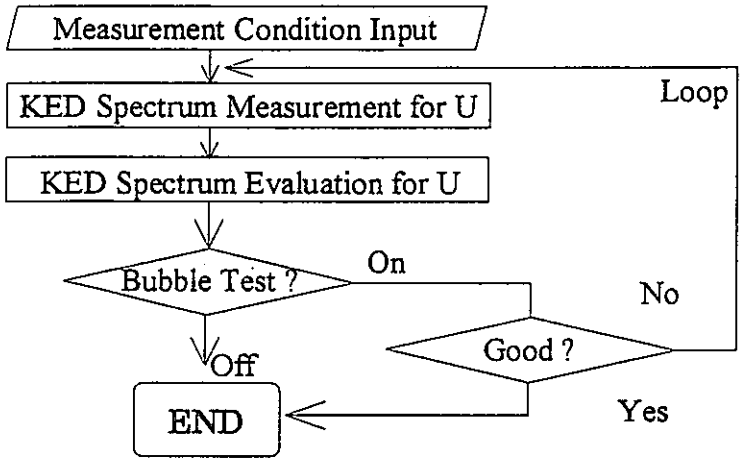
At this moment, the measurement conditions set by the "**Parameter**" are displayed at the lower part of the screen.

*The measurement conditions is not changed here.

After the necessary entries are made, the drive capacity for the spectrum registration and the same file name are checked by pressing the "Cont." key (F/10 key). If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "Miscellaneous".

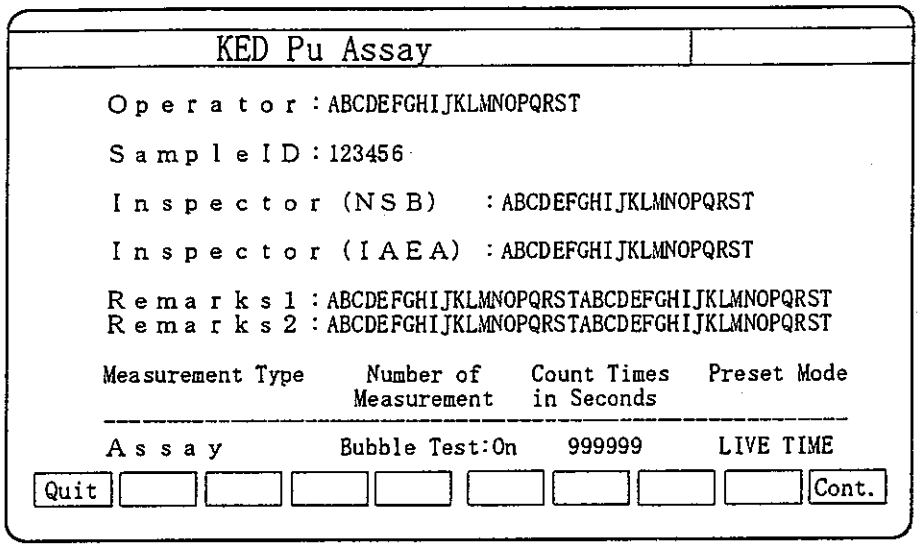
If no abnormality is found with the specified parameters, execute the "**KED U Assay**".

< Process Rough Chart >



(1-3)KED Pu Assay

After measurement of KED spectrum is finished at the specified conditions, the spectrum file will be stored with a name you want. Then KED analysis (for Pu conc.) will automatically be started.



<Screen 5>

Enter the following items for KED(Pu) automatic measurement and analysis.

Operator : Enter the name of the staff in charge of the KED Pu Assay automatic measurement and analysis. Limit: 20 characters

Sample ID : Enter the test sample ID. Limit: 6 characters
*The test sample ID entered here will make the spectrum file name.

Inspector(NSB) : Enter the inspector name (NSB). Limit:20 characters

Inspector(IAEA) : Enter the inspector name (IAEA). Limit:20 characters

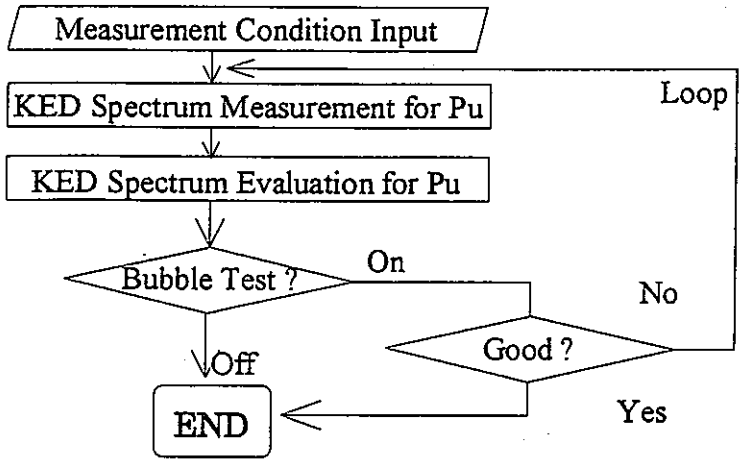
Remarks1 : Enter the comments in two lines, each separately. Limit:40 characters
Remarks2 :

At this moment, the measurement conditions set by the "**Parameter**" are displayed at the lower part of the screen.
*The measurement conditions cannot be changed here.

After the necessary entries are made, the drive capacity for the spectrum registration and the same file name are checked by pressing the "**Cont.**" key (**F/10** key). If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "**Miscellaneous**".

If no abnormality is found with the specified parameters, execute the "**KED Pu Assay**".

< Process Rough Chart >



The spectrum data measured by the **KED/XRF Assay**, or the **KED U Assay** or the **KED Pu Assay** is filed in accordance with the rules shown below:

SSSSSSMN.XXX

File extension
.CHN : spectrum file
.KEU : file for analysis result of U K-edge
.KEP : file for analysis result of Pu K-edge
.XRF : file for analysis result of XRF

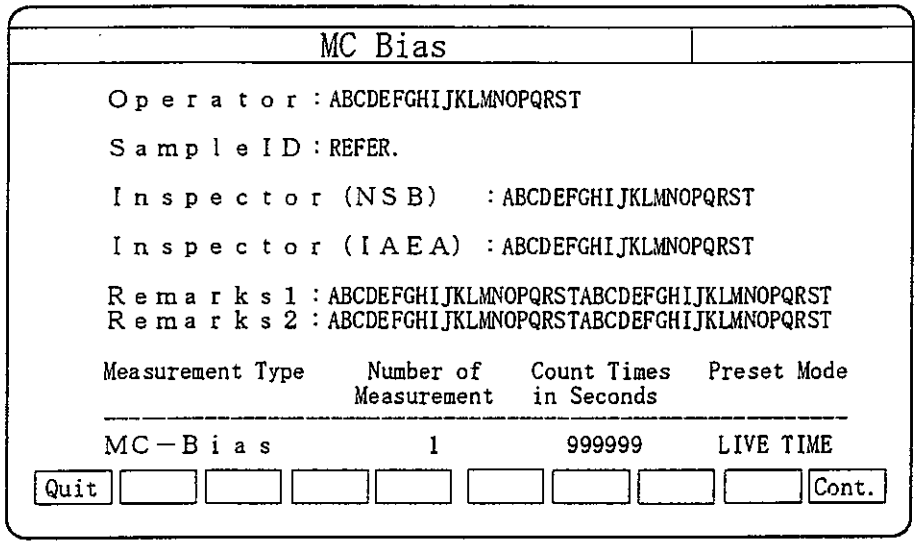
Number of repetitive measurement
(1,2,.....,9,A,B,C,D,E,F)

Type of spectrum for measurement
K : K-edge spectrum
X : XRF spectrum

Sample ID

(1-4)MC-Bias

After measurement of the both spectra for KED/XRF is conducted at the specified conditions, the spectrum file will be stored at any name you want. Then KED analysis (for U conc.) and the XRF analysis (for U/Pu Ratio) will automatically be started, and the MC Bias evaluation will follow.



<Screen 6>

Enter the conditions for KED/XRF automatic measurement and analysis for the MC Bias evaluation.

Operator : Enter the name of the staff in charge of execution of the MC Bias. Limit: 20 characters

Reference Sample ID : Enter the reference sample ID. (as to the initial display, "***REFER.***") Limit: 6 characters

Inspector(NSB) : Enter the inspector name (NSB). Limit:20 characters

Inspector(IAEA) : Enter the inspector name (IAEA). Limit:20 characters

Remarks1 : Enter the comments in two lines, each separately. Limit:40 characters
Remarks2 :

At this moment, the measurement conditions set by the "**Parameter**" are displayed at the lower part of the screen.

*The measurement conditions cannot be changed here.

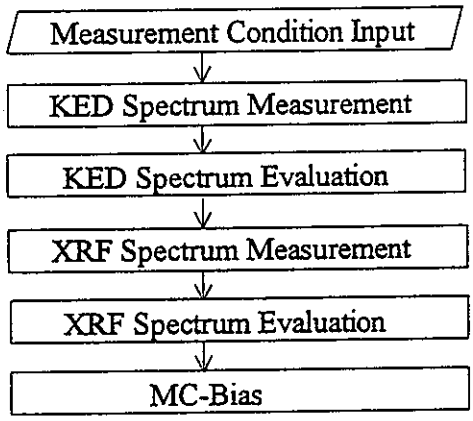
After the necessary entries are made, the spectrum file name is created and displayed in accordance with the rule, by pressing the "**Cont.**" key (**F/10** key). Press the key "Y" for confirmation.

Here, the last one character of the file name can be altered. Once the file name is confirmed, the drive capacity for the spectrum registration is cheked and confirmed whether or not there is any other file with the same name. If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "**Miscellaneous**".

If no abnormality is found with the specified parameters, execute the "**MC Bias**".

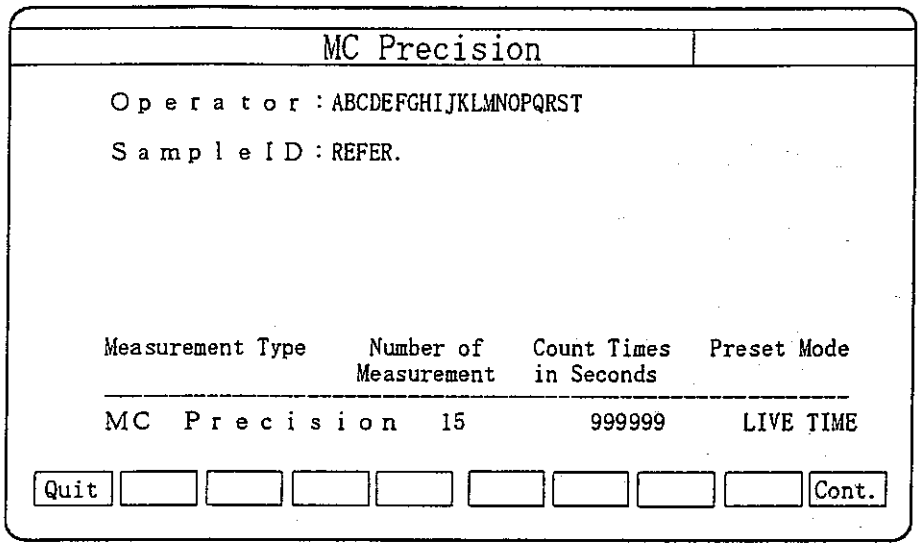
*When files are created in a same directory within a day, the file will be named with a single incrementing sequential digit on the end, like 1, 2, 3..... This last digit can be changed, if you want.

< Process Rough Chart >



(1-5)MC-Precision

After measurement of the both spectra for KED/XRF is conducted at the specified conditions, the spectrum file is stored with any name you want. Then KED analysis (for U conc.) and the XRF spectrum analysis (for U/Pu Ratio) will automatically be started, and the evaluation of the MC precision by the χ^2 test will follow.



<Screen 7>

Enter the operator name and sample ID for KED/XRF automatic measurement and analysis for the MC precision evaluation.

Operator : Enter the name of the staff in charge of the execution of the MC precision. Limit: 20 characters

Reference Sample ID : Enter the reference sample ID. Limit: 6 characters
(as to the initial display, "*REFER.*")

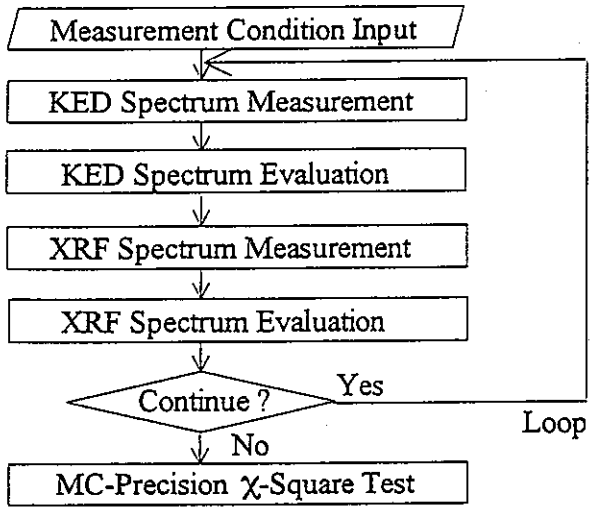
At this moment, the measurement conditions set by the "**Parameter**" are displayed at the lower part of the screen.

*The measurement parameters cannot be changed here.

After the necessary entries are made, the drive capacity for the spectrum registration will be confirmed by pressing the "**Cont.**" key (**F/10** key) and be confirmed whether or not there is any other file with the same name. If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "**Miscellaneous**".

If no abnormality is found with the specified parameters, the "**MC precision**" will be executed.

< Process Rough Chart >



*The spectra data measured by **MC Bias** is filed in accordance with the rules shown below:

MBMMDDMN.XXX

File extension
.CHN : spectrum file
.KEU : file for analysis result of U K-edge
.XRF : file for analysis result of XRF

Number of repetitive measurement
(1,2,.....,9,A,B,C,D,E,F)

Type of spectrum for measurement
K : K-edge spectrum
X : XRF spectrum

The day of measurement

The month of measurement

*The spectra data measured by **MC Precision** is filed in accordance with the rules shown below:

MPMMDDMN.XXX

File extension
.CHN : spectrum file
.KEU : file for analysis result of U K-edge
.XRF : file for analysis result of XRF

Number of repetitive measurement
(1,2,.....,9,A,B,C,D,E,F)

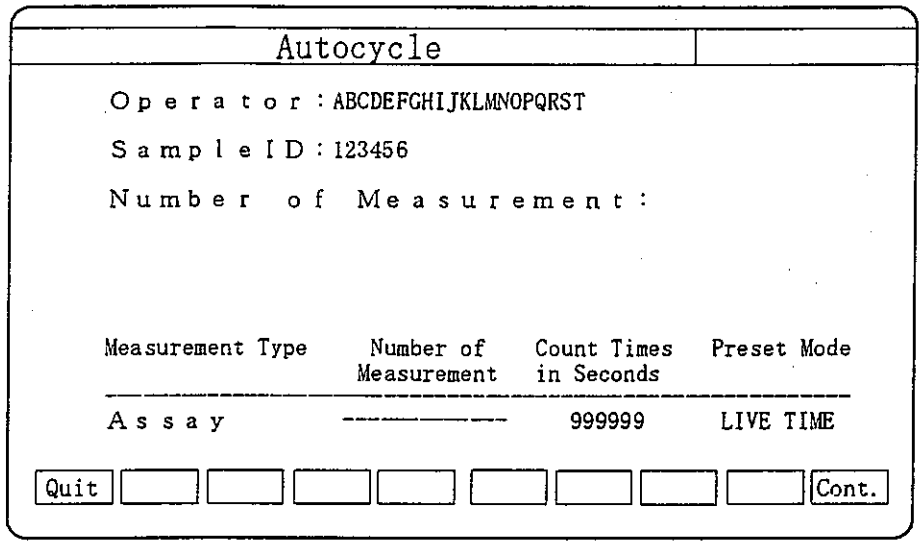
Type of spectrum for measurement
K : K-edge spectrum
X : XRF spectrum

The day of measurement

The month of measurement

(1-6)Autocycle

After measurement of the both spectra of KED/XRF are finished at the specified conditions, the spectra files are stored with a name you want.



<Screen 8>

Enter the conditions for KED/XRF automatic measurement and analysis.

Operator : Enter the name of the staff in charge of the KED/XRF automatic measurement and analysis. Limit: 20 characters

Sample ID : Enter the test sample ID. Limit: 6 characters or digits

*The test sample ID entered here will make the spectrum file name.

Number of Measurement : Enter the number of repetition of the measurement. Range: 1 - 15

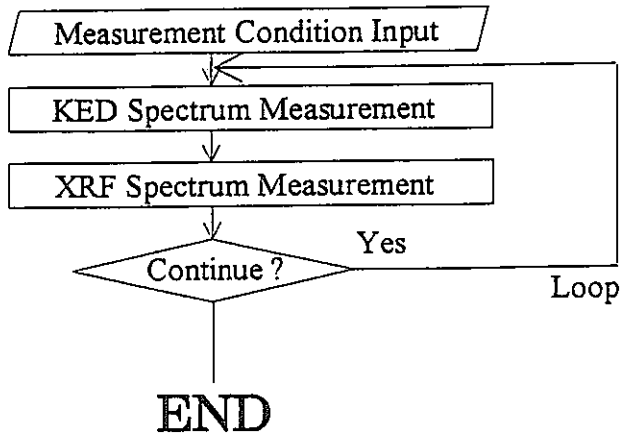
At this moment, the measurement conditions set by the "**Parameter**" are displayed at the lower part of the screen.

*The measurement conditions cannot be changed here.

After the necessary entries are made, the drive capacity for the spectrum registration is confirmed by pressing the **cont.** key "**F/10** key" and be confirmed whether or not there is any file with the same name. If these confirmations are not sufficiently done, make necessary changes such as storing drive change by the Parameters "**Miscellaneous**".

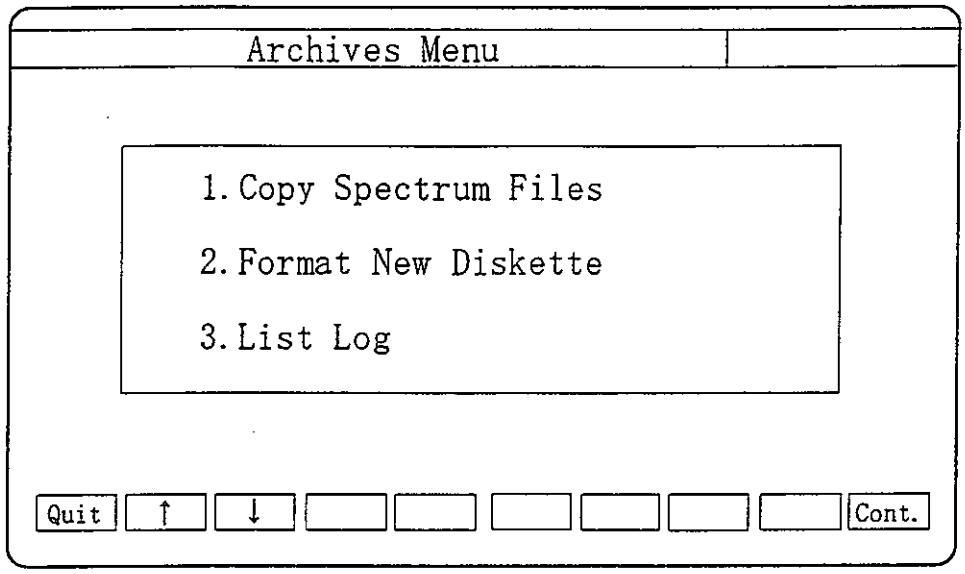
If no abnormality is found with the specified parameter, the "**KED/XRF Assay**" option will be executed.

< Process Rough Chart >



(2)Archives

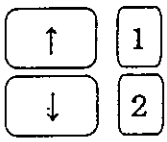
If the "**Archives**" are executed, the Archive Menu shown below will appear on the display. Select the item you want to execute.



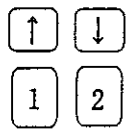
<Screen 9>

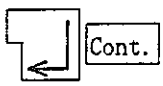
Position the cursor on the item to be executed, press the "**RET**" key or the "**Cont.**" key (**F/10** key) for execution.

If you want to execute using a mouse, position the mouse cursor on the desired item and click the left button of the mouse.



The cursor can be moved by the arrow keys and/or the ten keys.





Pressing the "**RET**" key or the **F/10** key will execute the item on which the cursor stays.

Archives Menu

1 : Copy Spectrum Files

To copy spectrum files.

2 : Format New Diskette

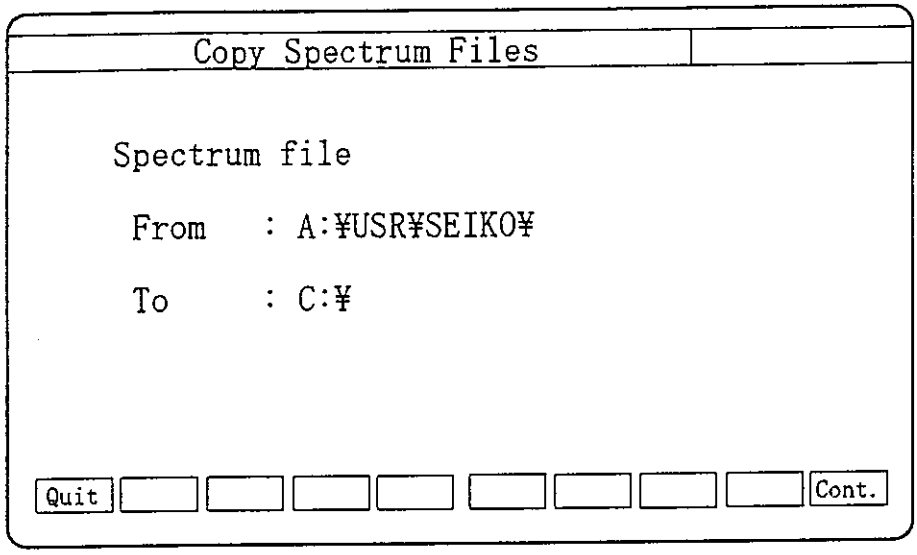
To format new diskettes.

3 : List Log

This option allow the user to printout the measurement results.

(2-1)Copy Spectrum Files

Any file (files only with a CHN extension) can be copied successively from the specified path.



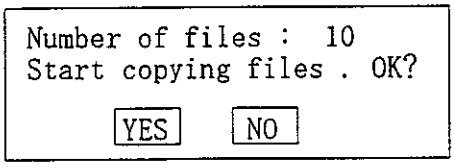
<Screen 10>

Enter the parameter to successively copy the specified files.

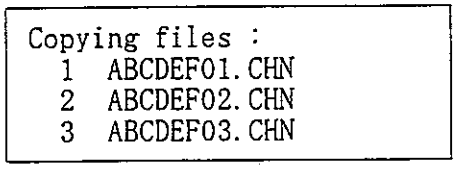
From : Enter the sending file names with drive and directory name. Limit: 30 characters (including the extension .CHN)
If you want to designate a specific file, be sure to put the extension (.CHN). on the file name end.

To : Enter the receiving file names with drive and directory name. Limit: 30 characters

Designate the file sending drive and the file receiving drive in this order, and press the "**Cont.**" key , then the number of the files to be copied will be displayed.



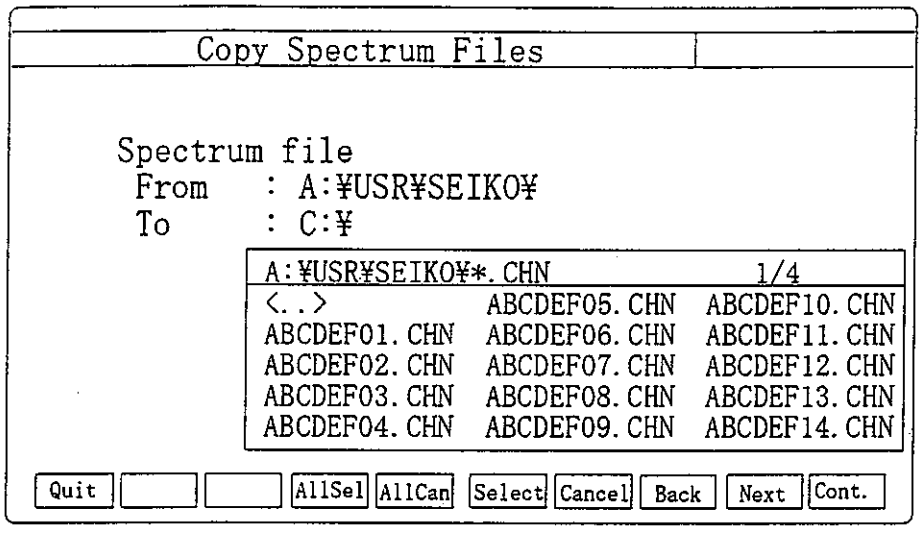
Here, select **YES** and copying will start.



The names of the files being copied will be displayed in the coping order. When all copies are finished, the message "**All copied. Hit any key.**" will appear.

Hit any key and the Screen 10 will come back.

*Designation examples in the column "**From**" :
(1)When a specific file name is specified----" A:¥ABCDEF01.CHN "
The specified file will be copied.
(2)When a specific directory name is specified----" A:¥USR¥SEIKO¥ "
All the files in the directory will be copied.
(3)When a wild card is specified----" A:¥USR¥SEIKO¥ *.CHN "
The corresponding files will be displayed as shown below. Select whatever you want.



<Screen 11>

The Screen 10 is for selecting file(s). Except for the indications with the marks of [ ] and < >, which are drive names and directory names respectively.
all the indications are the names of the existing files.

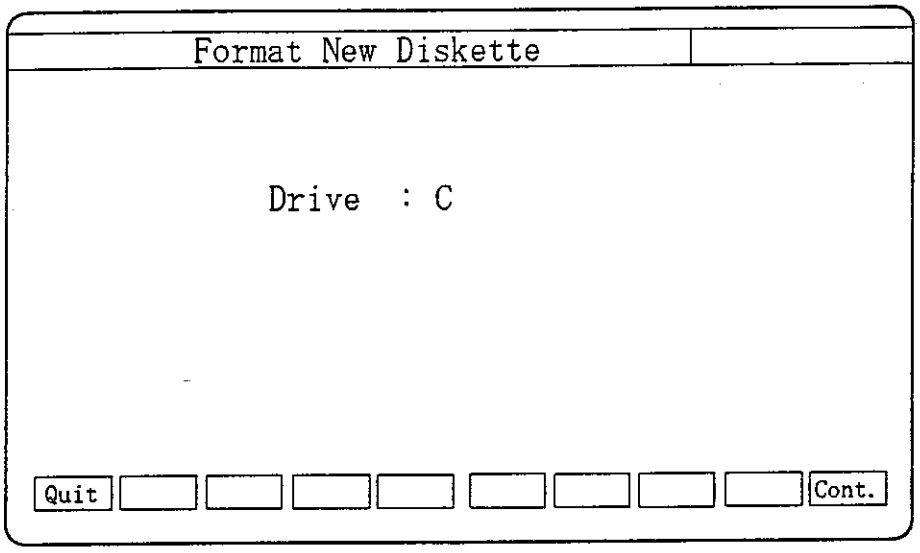
Position the cursor on the name of the files you want to copy by the arrow keys and press the **Select** key "**F/6** key". Then the selected file will be displayed in red color. When file selection is finished, press the **Cont.** key "**F/10** key" and the Screen 10 will come back.

The functions allocated for other keys are shown below:

Quit "**F/1**" : Operation will be interrupted and the screen will go back to the state where entry in the "From" column is expected.
AllSel "**F/4**" : All the files in the directory will be selected.
AllCan "**F/5**" : All the files selected will be canceled.
Cancel "**F/7**" : The files selected on the cursor will be canceled.
Next "**F/9**" : To be pressed when the existing files exceed the screen capacity. When hit, the next page of the file list will appear.
Back "**F/8**" : When hit, the previous page of the file list will appear.

(2-2)Format New Diskette

The floppy disk inserted in the specified drive will be formatted.



<Screen 12>

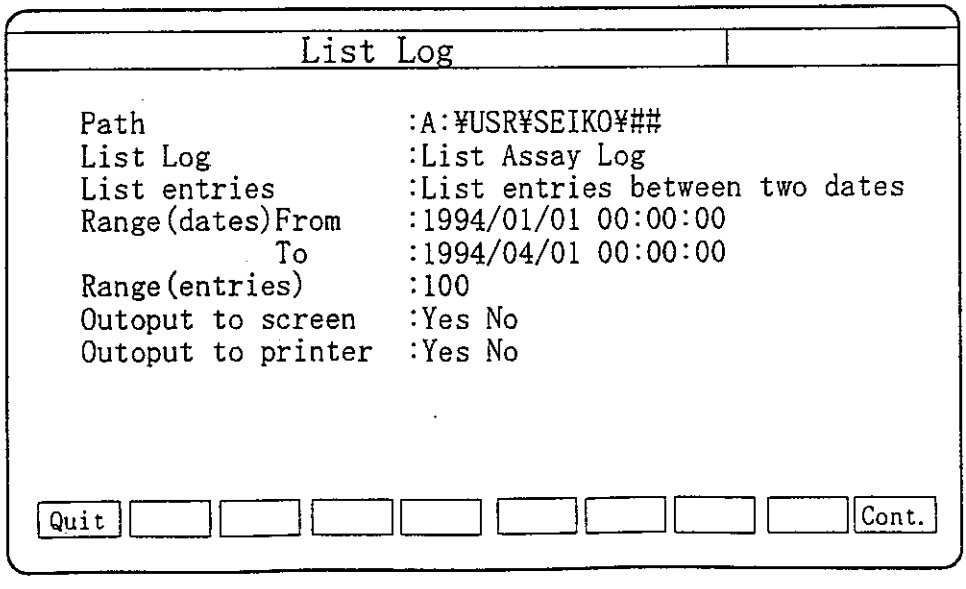
Enter the name of the drive where the floppy disk to be formatted is set.

Press the **RET** key or the **Cont.** key "**F/10** key" , and after confirmation to go ahead is given, formatting will start.

*Formatting is done for 1.25MB (3.5in.).
*In this screen, formatting of a hard disk cannot be done.

(2-3)List Log

The list of measurement results will be shown in screen or printed out. If this option is selected, the screen shown bellow will appear. Enter the appropriate parameters.



Path: Enter the name of drive and directory.
List Log: Select the option for the list format.
1 List Assay Log : KED/XRF, KED-U, KED-Pu
2 List MC Log : MC-Bias, MC-Precision
3 List Measurement Log : All measurement
List entries: Select the list range in following options.
1 List entries between two dates : Date range
2 List n entries : Currently n entries
3 List all entries : Currently All entries
Range (dates) From :
To :
Range (entries) :
Output to screen : Yes or No
Output to Printer : Yes or No

The desired list will be shown in bellow after Pressing "f-10" key to continue.

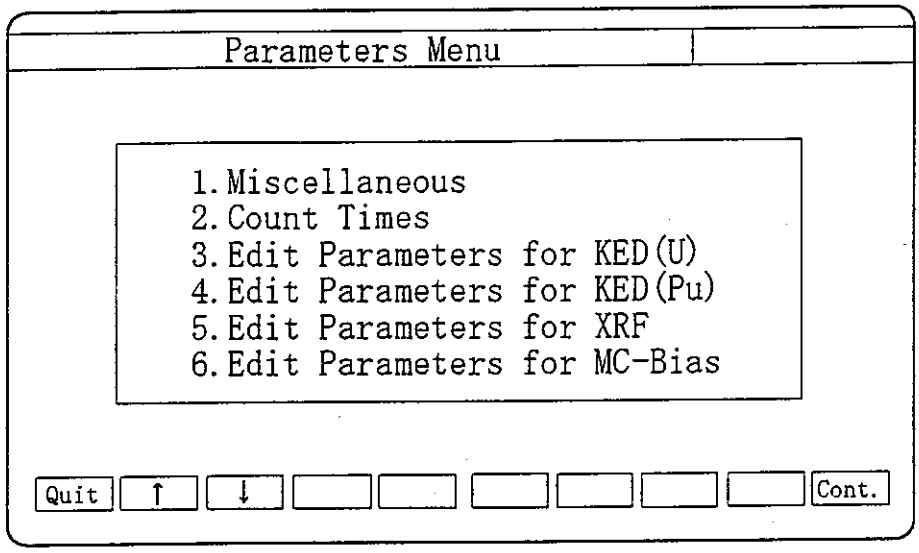
List Measurement Log all entries 95/01/11 11:15
Directory is C:\usr\seiko

Type	Sample ID	KED U (g/l)	XRF ratio	Pu-conc. (g/l)	Quality	Cycle	Date
A	MK1004	227.53	110.65	2.0564	Passed	Ave.	95/01/01
A	MK1004	227.53	110.65	2.0564		2/2	95/01/01
A	MK1004	227.53	110.65	2.0654		1/2	95/02/01
MC-B	Refer.	200.00	100.00	2.000	Passed	1/1	94/12/24
MC-P	Refer.	200.00	100.00	2.000	Passed	Ave	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	5/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	4/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	3/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	2/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	1/5	94/12/10

Press F/1 key to quit. (to printout) Use arrow keys to move the screen.

(3)Parameters

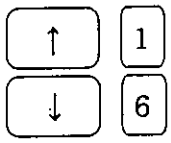
If the "**Parameters**" is executed, the Parameters Menu shown in below will appear. Select the item you want to execute.



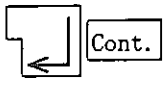
<Screen 13>

Position the cursor on the item to be executed, press the "**RET**" key or the "**Cont.**" key (**F/10** key) for execution.

If you want to execute using a mouse, position the mouse cursor on the desired item and click the left button of the mouse.



The cursor can be moved by the arrow keys and/or the ten keys.



Pressing the "**RET**" key or the **F/10** key will execute the item on which the cursor stays.

3 : Parameters

1 : Miscellaneous

To set the data related to the disk.

2 : Count Times

To set the time for spectrum measurement and others.

3 : Edit Parameters for KED(U)

To set the conditions for KED (U) analysis.

4 : Edit Parameters for KED(Pu)

To set the conditions for KED (Pu) analysis.

5 : Edit Parameters for XRF

To set the conditions for XRF analysis.

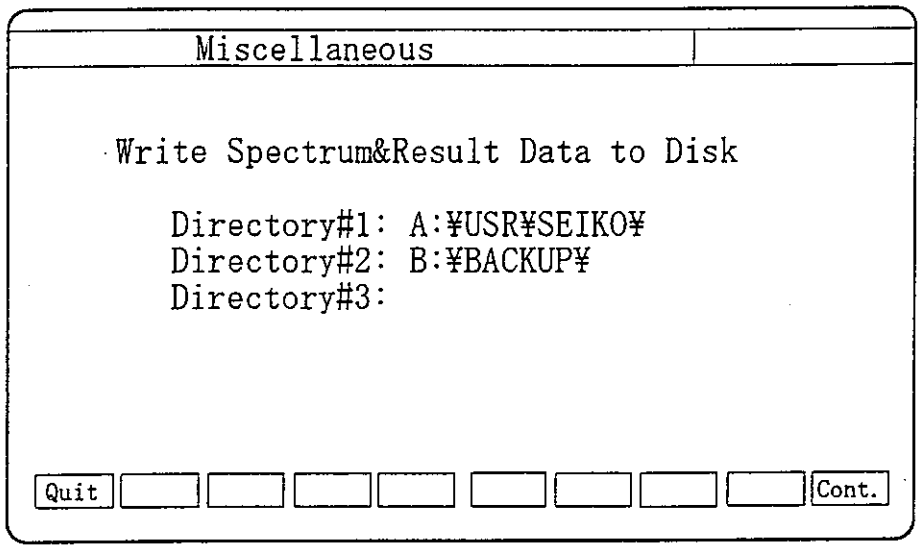
6 : Edit Parameters for MC bias

To set the conditions for MC Bias evaluation.

As to the operations of 3 to 6, the set parameters will automatically be output to the printer.

(3-1)Miscellaneous

The disk for storage is set here.



<Screen 14>

Directory#1:
Directory#2:
Directory#3:

Here, the names of the drive and the directory, where the spectra data and the spectra analysis results data are stored after the KED/XRF automatic measurement and analysis, are set.

Limit: 30 characters

Storage of up to three different files is possible. If you do not want to store in double or triple copies, just do not enter anything in the columns "**Directory #2** and **#3**"

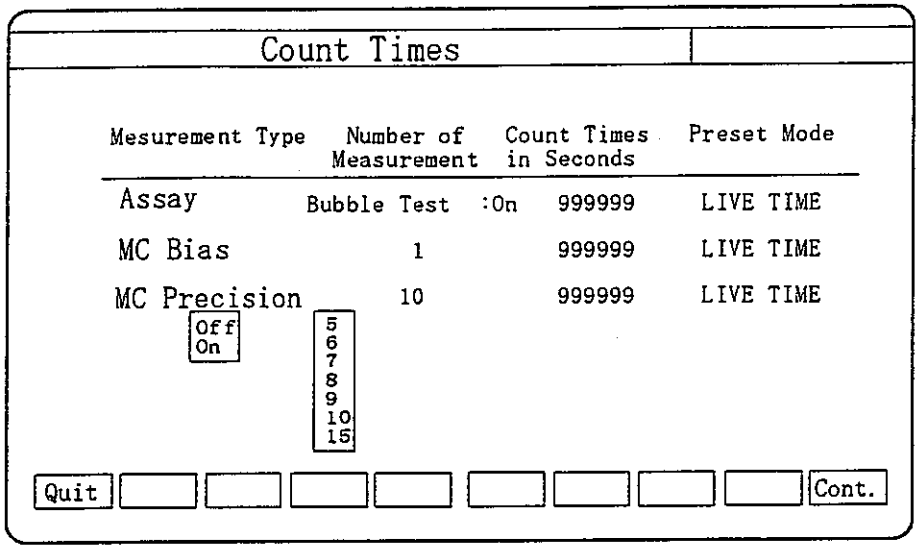
An example of the entry format is shown below:
(Example)

A:¥USR¥SEIKO¥

When proper entry is not made in the **Directory #1**, measurement and analysis will not start, even if some entry is made in the **Directory #2** and/or **#3**.

(3-2)Count Times

The time for measurement and the number of repetition are set here.



<Screen 15>

The time, mode and number of repetition for each measurement are set here.

Number of Measurement : The number of repetition is specified.

Assay : Whether or not the bubble test is conducted determines the number of repetition.

off : once

on : twice; if there is any abnormality. three times.

MC Bias : Measurement is made just once, therefore no entry is necessary.

MC Precision : Select one out of 5.6.7.8.9.10 and 15.

Select one out of two alternatives

Select one out of 7 alternatives

Count Times in Seconds : Enter measurement time in seconds.

Range 60 - 999999

Preset Mode : Select measurement time mode.

01:LIVE TIME
02:REAL TIME

Select one out of two alternatives

(3-3)Edit Parameters for KED(U)

The parameters for the KED analysis conditions to calculate the U concentration are set here.

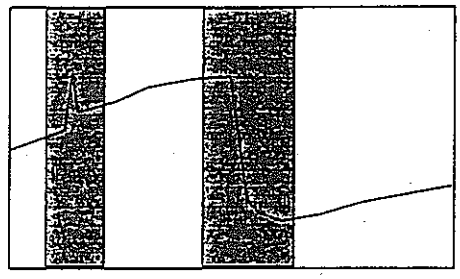
```
Edit Parameters for KED(U)

ROI data file                :A:¥USR¥SEIKO¥K-EDGE.ROI

5point smoothing             :YES    NO
No. of smoothing             :12
Pb-Kα1 energy(keV)           :123.456
K-edge  energy(keV)          :123.456
C(El)   region(keV)    Start:123.45 ~  End:123.45
C(Eu)   region(keV)    Start:123.45 ~  End:123.45
BG Window Low (keV)    Start:123.45 ~  End:123.45
          High(keV)    Start:123.45 ~  End:123.45
K-edge factor
   Extrapoleted (cm2/mg)     :1234.5678
   Non-extrapo. (cm2/mg)     :1234.5678
Cell path length     (mm)    :12.3
[Quit] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [Cont.]
```

<Screen 16>

ROI data file

Limit: 30 characters

Designate the ROI files (the files with the extension .ROI) that store the two ROIs set in advance as Pb-Kα1 and K-edge center calculation areas by the MCA emulation.



*Too large Pb-Kα1 value and K-edge value may cause evaluation of wrong peaks. The maximum width of the both areas is 200 ch.

5 point smoothing

Selection of YES/NO

To designate whether or not 5 points smoothing is conducted before obtaining the K-edge center.

No. of smoothing

Range 1 - 99

To designate the number of times for 5 points smoothing. If smoothing is not opted in the previous screen, the entry will be invalid.

Pb-Kα1 energy(keV)

Range 70.0 - 80.0

To designate precise energy value of the Pb-Kα1 peak in the keV unit.

K-edge energy(keV)

Range 110.0 - 130.0

To designate precise energy value of the U K-edge in the keV unit.

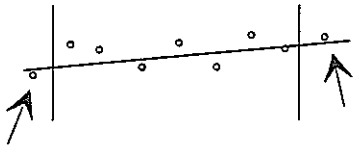
*After the analysis, the relation (a simple equation) between the energies and the channels is obtained from the two points, or the Pb-Kα1 centroid (ch) and its centroid energy, and the K-edge center (ch) and its energy.

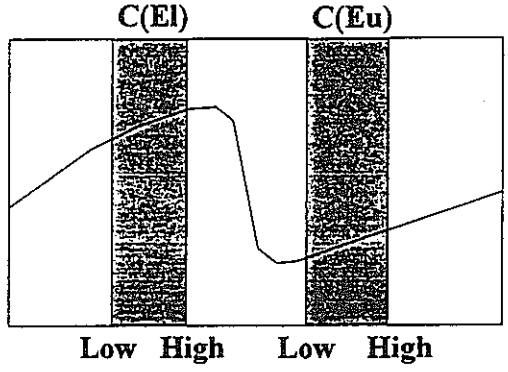
C(El) region (keV) Start End
C(Eu) region (keV) Start End

Range 80.0 - 150.0

Designate the range for regression of C(E1) and C(Eu). The actual range to be used in the regression is the one converted from the energies specified here to the channel by the energy conversion formula.

*When the converted values (ch, decimal fractions) are rounded to integers, the values on the lower energy end are rounded up and the values on the higher energy end are rounded down.

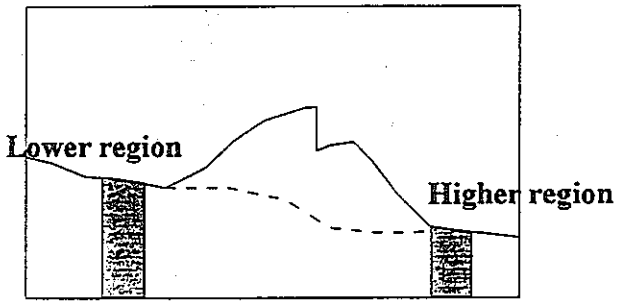




BG Windows Low (keV) Start End
〃 〃 High (keV) Start End

Range 0.0 - 999..99

Enter the range for calculating the lower and higher regions, that are used for BG adjustment.



K-edge factor (Difference of mass attenuation coefficients : $\triangle\mu$)
Extrapolated (cm²/mg)
Non-extrapo. (cm²/mg)

Range 0.00001 - 1000.0

Designate the K-edge factor in cm²/mg that is to be used for U concentration conversion by the extrapolate and non-extrapolate.

Cell path length (mm)

Range 0.1 - 99.9

Designate the cell path length (d) in mm that is used for U concentration conversion.

(3-4)Edit Parameters for KED(Pu)

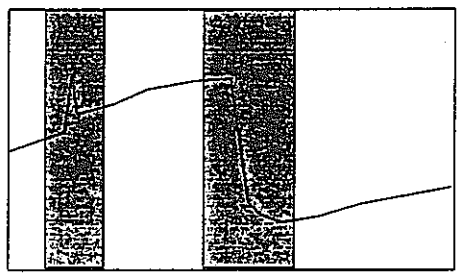
The parameters for the KED analysis conditions to calculate the Pu concentration are set here.

```
Edit Parameters for KED(Pu)

ROI data file              :A:¥USR¥SEIKO¥K-EDGE.ROI

5point smoothing           :YES    NO
No. of smoothing           :12
Pb-Kα1 energy(keV)         :123.456
K-edge energy(keV)         :123.456
C(El)   region(keV)   Start:123.45 ~  End:123.45
C(Eu)   region(keV)   Start:123.45 ~  End:123.45
BG Window Low (keV)   Start:123.45 ~  End:123.45
          High(keV)   Start:123.45 ~  End:123.45
K-edge factor
   Extrapoleted (cm2/mg)   :1234.5678
   Non-extrapo. (cm2/mg)   :1234.5678
Cell path length    (mm)   :12.3

[Quit] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [Cont.]
```

<Screen 17>

ROI data file

Designate the ROI files (the files with the extension .ROI) that store the two ROIs set in advance as Pb-Kα1 and K-edge center calculation areas by the MCA emulation.



*Too large Pb-Kα1 value and K-edge value may cause evaluation of wrong peaks. The maximum width of the both areas is 200 ch.

Limit: 30 characters

5 point smoothing

To designate whether or not 5 points smoothing is conducted before obtaining the K-edge center.

Selection of YES/NO

No. of smoothing

To designate the number of times for 5 points smoothing. If smoothing is not opted in the previous screen, the entry will be invalid.

Range 1 - 99

Pb-Kα1 energy(keV)

To designate precise energy value of the Pb-Kα1 peak in the keV unit.

Range 70.0 - 80.0

K-edge energy(keV)

To designate precise energy value of the Pu K-edge in the keV unit.

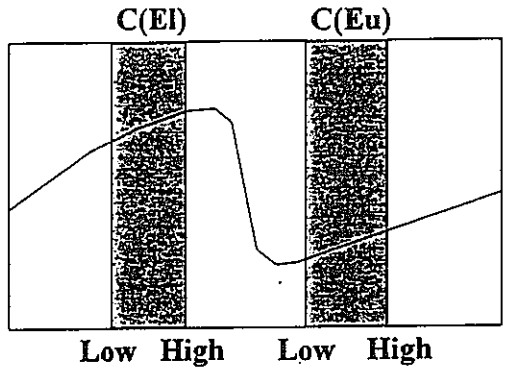
Range 110.0 - 130.0

*After the analysis, the relation (a simple equation) between the energies and the channels is obtained from the two points, or the Pb-Kα1 centroid (ch) and its centroid energy, and the K-edge center (ch) and its energy.

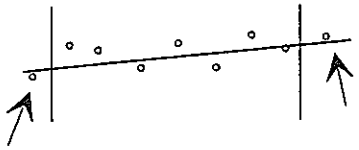
C(El) region (keV) Start End
C(Eu) region (keV) Start End

Range 80.0 - 150.0

Designate the range for regression of C(E1) and C(Eu). The actual range to be used in the regression is the one converted from the energies specified here to the channel by the energy conversion formula.



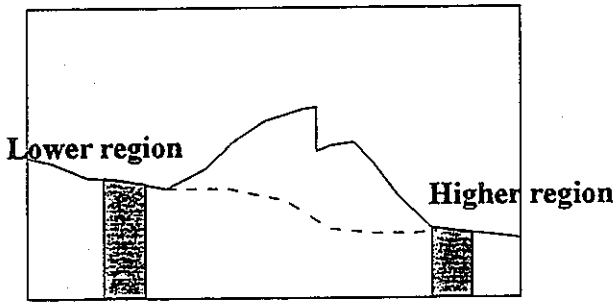
*When the converted values (ch, decimal fractions) are rounded to integers, the values on the lower energy end are rounded up and the values on the higher energy end are rounded down.



BG Windows Low (keV) Start End
〃 〃 High (keV) Start End

Range 0.0 - 999..99

Enter the range for calculating the lower and higher regions, that are used for BG adjustment.



K-edge factor (Difference of mass attenuation coefficients : $\Delta\mu$)
Extrapolated (cm²/mg)
Non-extrapo. (cm²/mg)

Range 0.00001 - 1000.0

Designate the K-edge factor in cm²/mg that is to be used for Pu concentration conversion by the extrapolate and non-extrapolate.

Cell path length (mm)

Range 0.1 - 99.9

Designate the cell path length (d) in mm that is used for Pu concentration conversion.

(3-5)Edit Parameters for XRF

The parameters for XRF analysis conditions to calculate the Pu concentration are set here.

```
Edit Parameters for XRF

ROI data file              :A:¥USR¥SEIKO¥K_EDGE.ROI
XRF energy cal. file       :A:¥USR¥SEIKO¥X_RF.ENE

Factors of R(U/Pu)        A: 1.0000E+004   B:-1.0000E+004
BG Windows Low    (keV)Start:123.45 ～  End:123.45
           Middle(keV)Start:123.45 ～  End:123.45
           High   (keV)Start:123.45 ～  End:123.45
Sensitivity               :1
Region to Identify(keV)   :12.3
U-Kα1  energy(keV)        :123.456
           Peak Width Lower:1.23 * FWHM   Upper:1.23 * FWHM
Pu-Kα1 energy(keV)        :123.456
           Peak Width Lower:1.23 * FWHM   Upper:1.23 * FWHM
U-Kα2  energy(keV)        :123.456
U-Kβ1  energy(keV)        :123.456
Atomic Weight          U :238.08    Pu:239.50
Factor                    :1.23456E+123
First Limit               :1.95
Second Limit              :3.18
[Quit] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [Cont.]
```

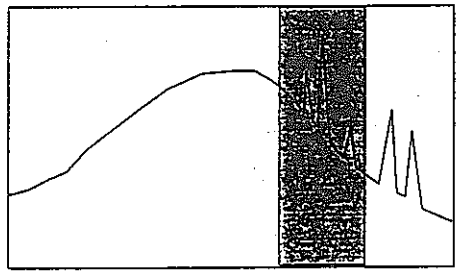
<Screen 18>

ROI data file

Designate the ROI files (the files with the extension .ROI) that store the ROI set in advance as XRF peak analysis region by the MCA emulation option.

Limit: 30 characters

*The maximum analysis region is 400ch.



XRF energy cal. file

Designate the energy correction files to be used for the qualitative analysis to obtain the peaks of U-Kα1, U-Kα2 and U-Kβ1.

Limit:30 characters
(with extension .ENE)

Factors of R(U/Pu) A B

Designate the coefficient A and B in the correction formula to obtain the excitation probability of fluorescent X-ray (U/Pu) shown below:

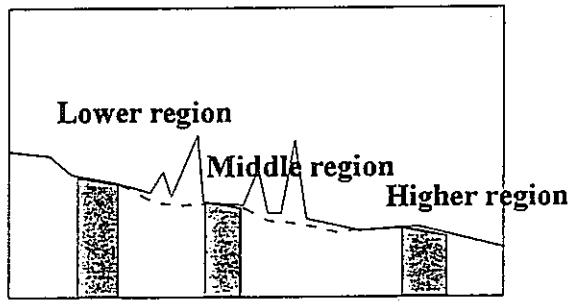
Range
-1.00000E-5～
1.00000E + 5

$$R(U/Pu)=A \times \exp(B \times U)$$

Here: U is U conc. (g/l)

BG Window Low (keV) Start End Range 0.0 - 999.99
〃 〃 Middle (keV) Start End
〃 〃 High (keV) Start End

Enter, in keV, the range for calculating the lower, middle and higher region areas that are used for BG adjustment.

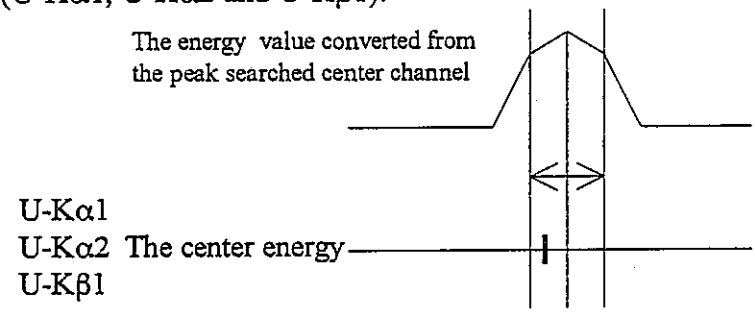


Sensitivity Range 1 - 3

Designate the peak search sensitivity.

Region to Identify(keV) Range 1.0 - 50.0

Designate, in keV, the permissible energy range (ambiguity) measured from the centroid energy as shown below, at the time of the qualitative analysis (U-Kα1, U-Kα2 and U-Kβ1).



U-Kα1 energy(keV)
Pu-Kα1 energy(keV)

Designate the centroid energy of the peak used for Pu concentration evaluation. Range 90.0 - 120.0

U-Kα1 Peak Width Lower * FWHM Upper * FWHM
Pu-Kα1 Peak Width Lower * FWHM Upper * FWHM Range 1.0 - 2.0

To obtain the peak areas, enter the cumulative region channels as coefficient to FWHM for both the lower and higher energy end, and the peak area is obtained by the formula shown below:

$$Peak\ \ Area = \sum_{i=sta}^{end} Count(i)$$

sta = the peak centroid channel - the lower energy end factor × FWHM
end = the peak centroid channel - the Higher energy end factor × FWHM

Atomic Weight U , Pu

Designate U and Pu mass number for the concentration conversion. Range 0.01 - 1000.00

Factor

Designate overall factor for U concentration conversion.

Range 1.00000E-7 - 1.00000E+7

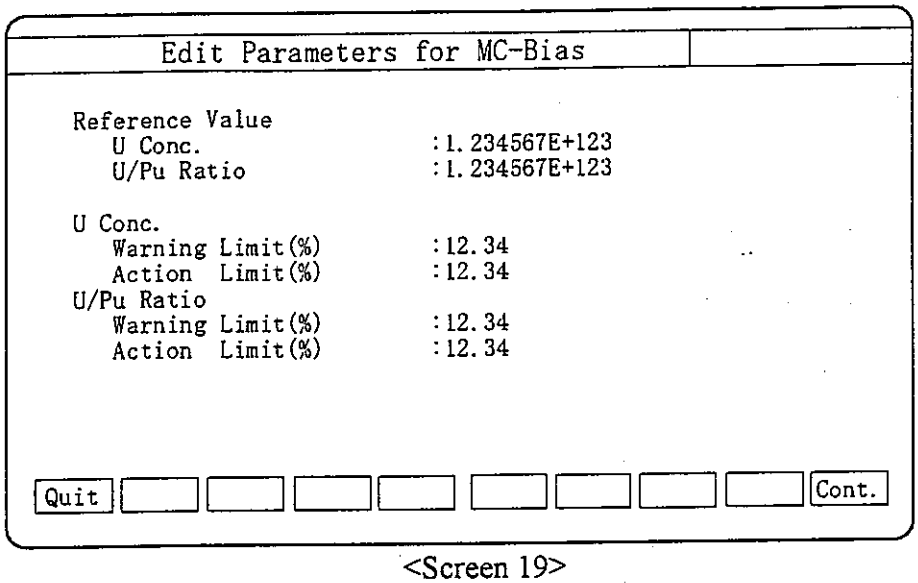
First Limit
Second Limit

Designate the boundary values for the bubble test. The initial values are 3.18 and 1.95, respectively.

Range 1.00 - 9.99

(3-4)Edit Parameters for MC-Bias

The parameters for MC-Bias are set here.



<Screen 19>

Reference Value

U Conc.

Designate the K-edge reference value.

Range 1.000000E+0 - 1.000000E+3

U/Pu ratio

Designate the XRF reference value.

Range 1.000000E+0 - 1.000000E+3

U Conc.

Warning Limit(%)

Action Limit(%)

Designate the boundary values for alarm issuance and error recognition on the K-edge evaluation results.

Range 0.01 - 99.9

U/Pu Ratio

Warning Limit(%)

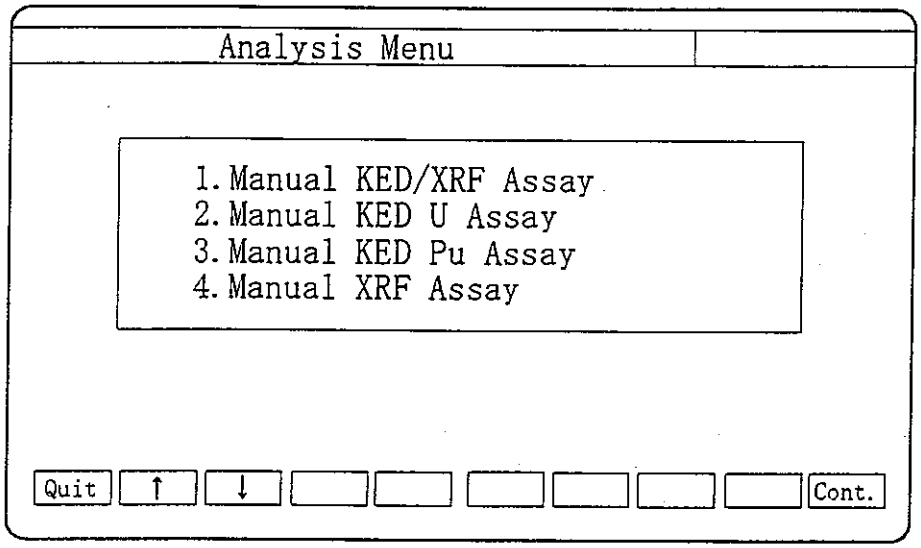
Action Limit(%)

Designate the boundary values for alarm issuance and error recognition on the XRF evaluation results.

Range 0.01 - 99.99

(4)Analysis

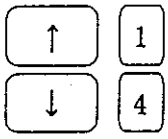
If the "**Analysis**" is executed, the Analysis Menu shown below will appear on the display. Select the item you want to execute.



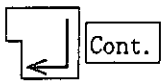
<Screen 20>

Position the cursor on the item to be executed, press the "**RET**" key or the "**Cont.**" key (**F/10** key) for execution.

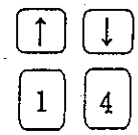
If you want to execute using a mouse, position the mouse cursor on the desired item and click the left button of the mouse.



The cursor can be moved by the arrow keys and/or the ten keys.



Pressing the "**RET**" key or the **F/10** key will execute the item on which the cursor stays.



4 : Analysis

1 : Manual KED/XRF Assay

To read the files storing the spectra and to conduct the KED/XRF analysis.

2 : Manual KED U Assay

To read the files storing the spectra and to conduct the KED analysis of U.

3 : Manual KED Pu Assay

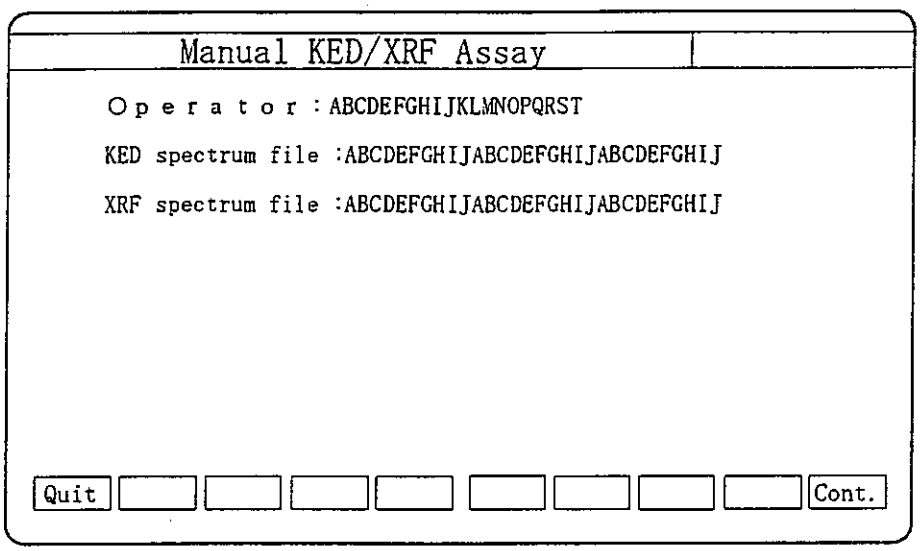
To read the files storing the spectra and to conduct the KED analysis of Pu.

4 : Manual XRF Assay

To read the files storing the spectra and to conduct the XRF analysis.

(4-1)Manual KED/XRF Assay

The KED analysis (for U conc.) and XRF spectra analysis (for Pu conc.) of the KED/XRF spectra data stored in the disks are continued.



<Screen 21>

Enter the conditions for KED/XRF manual analysis.

Measurement of the spectra by MCA is not conducted here. The operation here are only for analysis/re-analysis of the KED/XRF spectra previously measured and stored.

Operator : Enter the name of the staff in charge of the KED/XRF manual analysis. Limit: 20 characters

KED Spectrum file : Designate the file names of the spectra measured for KED analysis. Limit: 30 characters

*Designate by full path name.

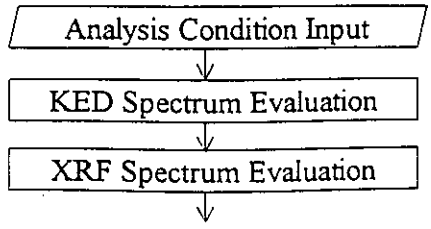
XRF Spectrum file : Designate the file names of the spectra measured for XRF analysis. Limit: 30 characters

*Designate by full path name.

If no abnormality is found with the specified parameters, the "**Manual KED/XRF Assay**" is executed.

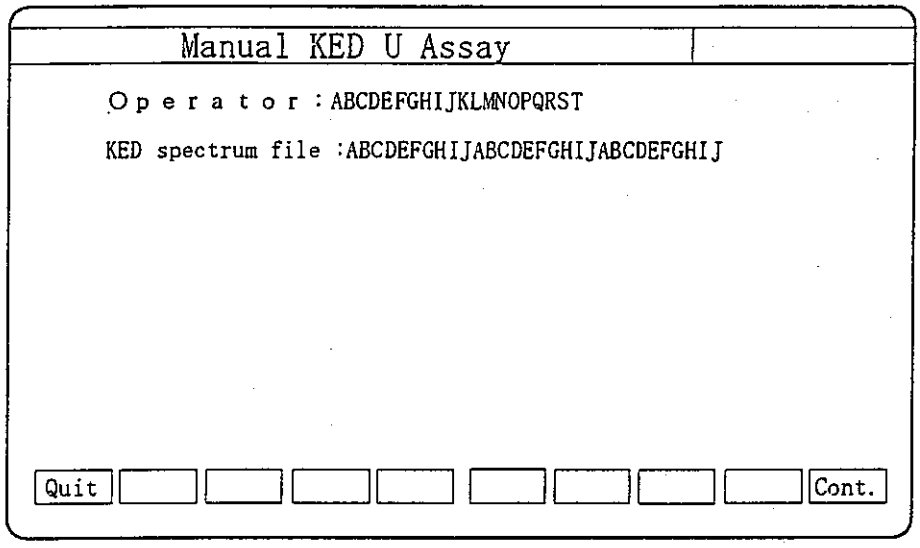
*The KED and XRF analysis results files are registered with the extension names changed to (KEU) and (XRF), respectively.

< Process Rough Chart >



(4-2)Manual KED U Assay

The KED analysis (for U conc.) of the KED spectra data stored in the disk is conducted.



<Screen 22>

Enter the conditions for KED manual analysis.

Measurement of the spectra by MCA is not conducted here. The operation here are only for analysis/re-analysis of the KED spectra previously measured and stored.

Operator : Enter the name of the staff in charge of the KED manual analysis. Limit: 20 characters

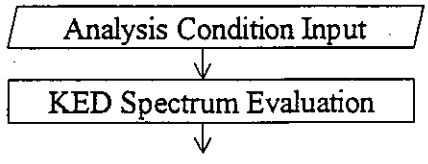
KED spectrum file : Designate the file names of the spectra measured for KED analysis. Limit: 30 characters

*Designate by full path name.

If no abnormality is found with the specified parameters, the "**Manual KED U Assay**" is executed.

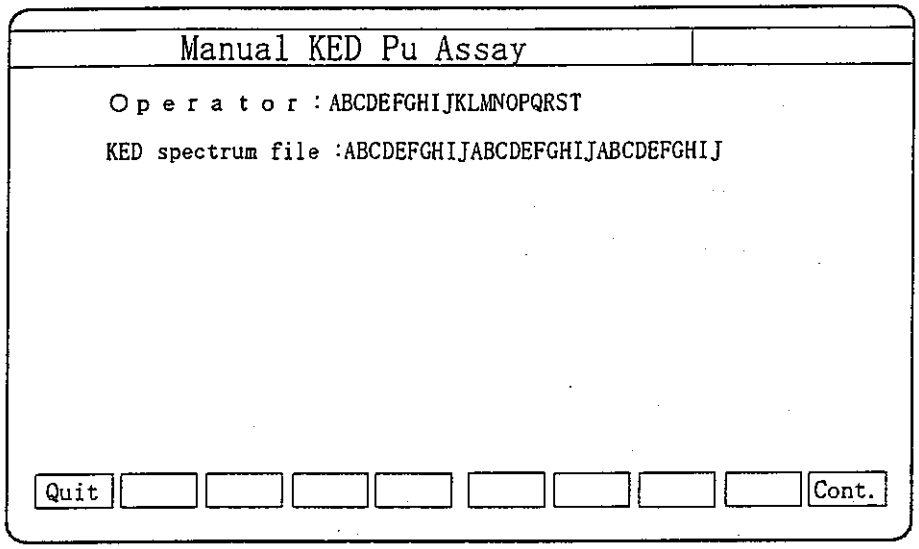
*The KED analysis result files are registered with the extension names changed to (.KEU).

< Process Rough Chart >



(4-3)Manual KED Pu Assay

The KED analysis (for Pu conc.) of the KED spectra data stored in the disk is conducted.



<Screen 23>

Enter the conditions for KED manual analysis.

Measurement of the spectra by MCA is not conducted here. The operation here are only for analysis/re-analysis of the KED spectra previously measured and stored.

Operator : Enter the name of the staff in charge of the KED manual analysis. Limit: 20 characters

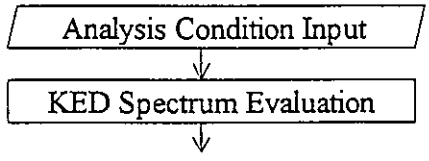
KED spectrum file : Designate the file names of the spectra measured for KED analysis. Limit: 30 characters

*Designate by full path name.

If no abnormality is found with the specified parameters, the "**Manual KED Pu Assay**" is executed.

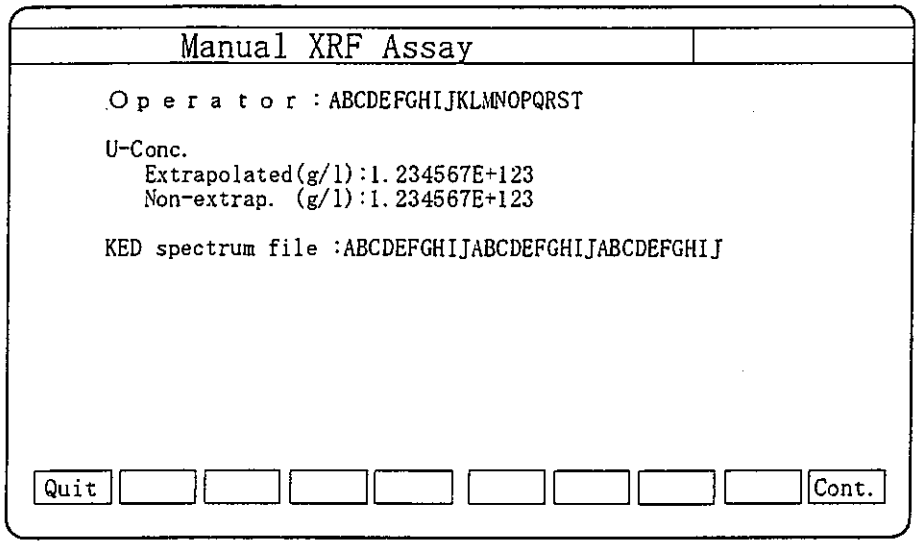
*The KED analysis result files are registered with the extension names changed to (.KEP).

< Process Rough Chart >



(4-4)Manual XRF Assay for U/Pu ratio

The XRF spectra analysis (for Pu conc.) of the XRF spectra data stored in the disks are conducted.



<Screen 24>

Enter the conditions for the XRF manual analysis.

Measurement of the spectra by MCA is not conducted here. The operation here are only for analysis/re-analysis of the XRF spectra previously measured and stored.

Operator : Enter the name of the staff in charge of the XRF manual analysis. Limit: 20 characters

U-Conc.
Extrapolated(g/l) :
Non-extrapo. (g/l) :

Enter the U concentration data separately for extrapolate and non-extrapolate analysis of XRF. Limit: 1.0E001 - 1.0E+003

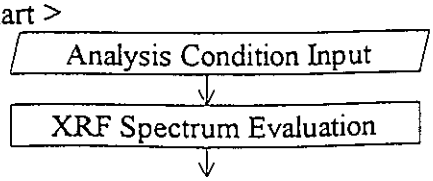
XRF spectrum file : Designate the file names of the spectra measured for XRF analysis. Limit: 30 characters

*Designate by full path name.

If no abnormality is found with the specified parameters, the "**Manual XRF Assay**" is executed.

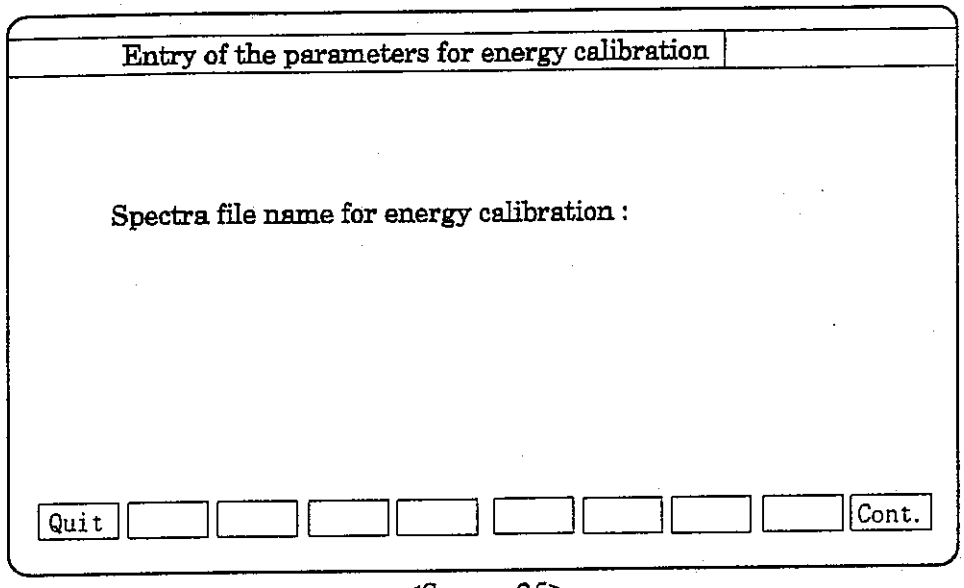
*The XRF analysis result files are registered with the extension names changed to (XRF).

< Process Rough Chart >



(5)Energy Calibration

The relation between the channels and the energies is obtained in the form of primary and secondary function, using the spectra for the measurement of the standard beam source for energy calibration, and is stored for later energy calibration.



<Screen 25>

Spectra File Name for Energy Calibration

Limit: 30 characters

Designate the file name (with extensions of .CHN/.TXT) of the spectra measured for energy calibration as well as the drive name and the directory name.

*Designation by wild card (*) is permitted. As to the detail of the wild card, refer to "Selection of file by wild card" in the Appendix.

(Example) B:¥USR¥SEIKO¥TEST01.CHN

< What is the spectra file for energy calibration ? >

"The spectra file for energy calibration" is the data on the spectra used to measure the standard beam source for energy calibration. In other words, it is the data on the spectra used for the measurement of data with a known peak energy.

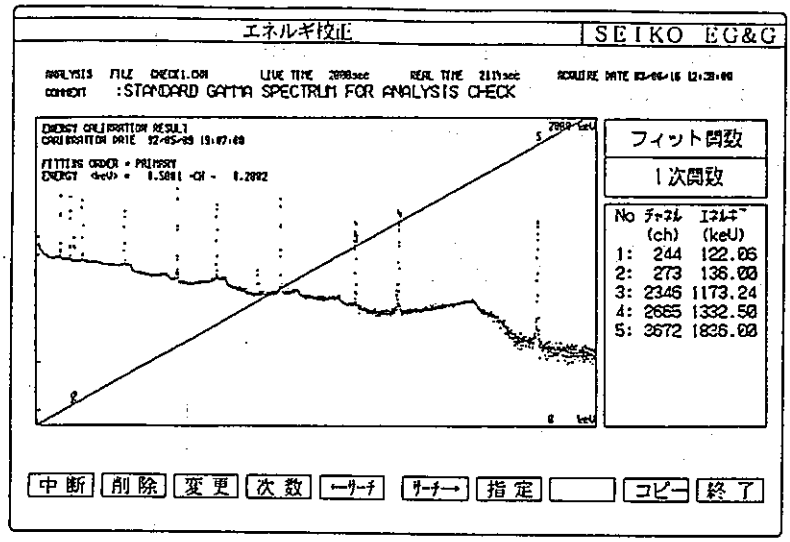
< Conditions to measure the spectra for energy calibration >

Existence of necessary number of the peaks to obtain the approximation functions (primary/secondary function).

→ When calibration is conducted by a primary function, at least two peaks are required.

→ When calibration is conducted by a secondary function, at least three peaks are required.

If the spectra file name for energy calibration is specified, the screen shown in the next page will appear.



<Screen 26>

Energy calibration

Fit function
Primary function
No. Channel Energy

Designate here the data for energy calibration (the peak centroid channel and its energy), looking at the spectra on the screen.

The functions of the keys are shown below:

← →

To move the cursor on the spectrum from left to right and from right to left. Use these keys to move the cursor to the target peak centroid channel.

↑ ↓

To move the cursor upward or downward on the data entered for approximate calculation of the energy calibration. Use these keys to "delete" or "change" the existent data pair.

中断

Interruption :

To interrupt the execution of the energy calibration program.

削除

Deletion:

To delete data where the cursor is located out of the data entered for energy calibration.

変更

Change:

To change the energy value of the entry data where the cursor is located in the screen shown in below.

Enter the peak energy (keV)
channel energy ####.## KeV
Range(0.0KeV ～ 3000.0KeV)

次数
Order :

To select the order of the approximation function for energy calibration. If this key is pressed , the order of the function rolls over between primary and secondary in the right upper corner of the screen.

←サーチ
← Search:

To search the spectrum from the channel where the cursor is currently located to the lower energy end. If a peak is not identified, the cursor will sit in the 0 channel.

→サーチ
→ Search:

To search the spectrum from the channel where the cursor is currently located to the higher end. The cursor will stay at the first channel identified. If the peak is not identified, the cursor will sit at the last channel.

指定
Designation:

Enter the channel of the spectrum where the cursor is currently located. If the channel is entered, the screen for entering the energy of the channel will appear. Enter the energy in keV.

Enter the peak energy (keV) #### channel energy ####.## KeV Range(0.0KeV ～ 3000.0KeV)

If more than two data are entered, the approximation function will automatically be obtained by the least square method (the initial degree being primary). The calibration (approximation) result will be indicated in the spectrum graph after the calculation is finished.

Check the appended graph, and if the approximation is not satisfactory edit the data using the functions such as "delete", "change", "degree" and "designation".

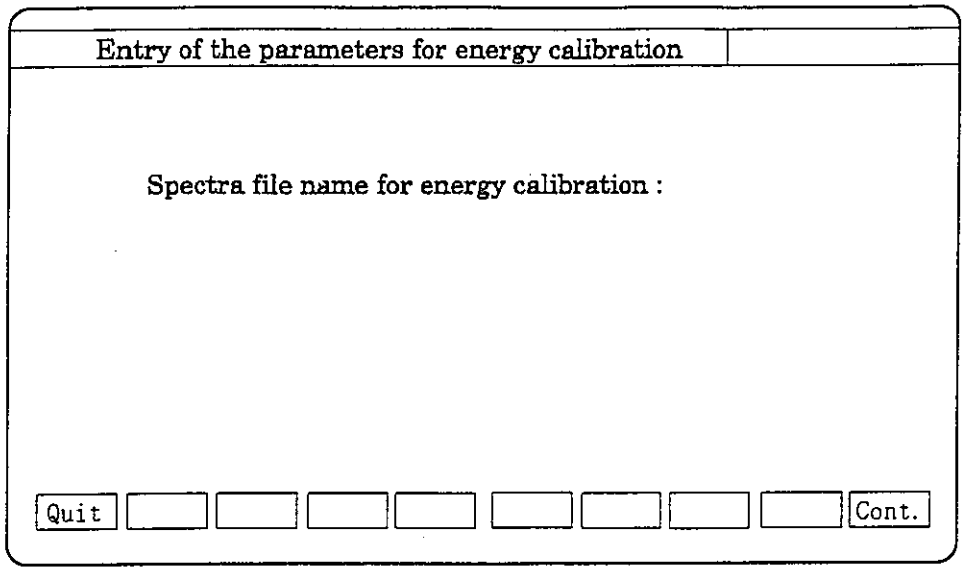
コピー
Copy:

This key is for making a graphic hard copy of the screen.

If this key is pressed , the screen for copy confirmation will appear. Once the confirmation is made, soon starts copying. Prepare a printer before confirmation.

終了
Quit:

After calibration is finished and the calibration result is checked, press this key to quit the program. Then, the screen for designating the data file name storing the calibration data will appear. Enter the energy calibration file name, and the calibration result is automatically registered.



<Screen 27>

<Energy Calibration Data File Name >

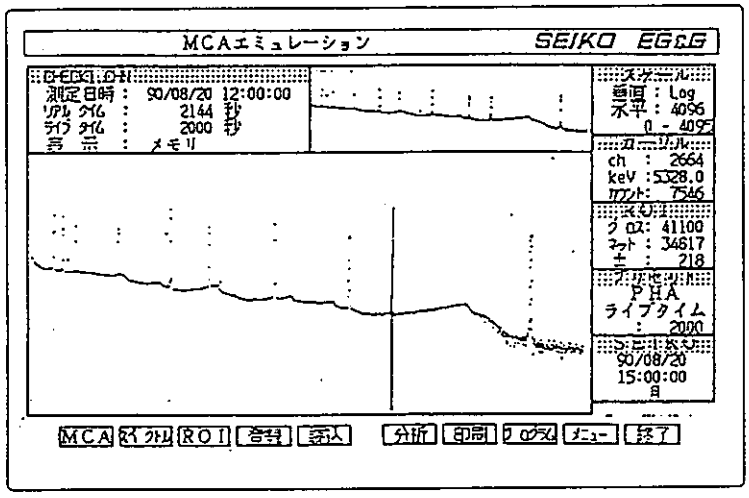
Designate the name (with extension .ENE) of the file to store the energy calibration data as well as the drive name and the directory name.

*The calibration result will be made in text file with the specified name. The data to be stored is shown below:

	<symbol name>
(1)File name of the spectrum used for energy calibration	SPECTRUM
(2)Date of calibration (YY/MM:DD hh:mm:ss)	CLB-DATE
(3)Fit order (1 or 2)	FT-ORDER
(4)Approximation function; secondary coefficient	ANS-2
(5)Approximation function; first coefficient	ANS-1
(6)Approximation function; nil order coefficient	ANS-0

(6)MCA Emulation

This is the standard "MCA Emulation" program of SEIKO EG&G. In this program control and operations of MCA can be by means of a PC.



<Screen 28>

As to the detail of the operation, refer to the "MCA Emulation Operation Manual".

Printed by Japanese

In MCA emulation, operations such as spectrum calculation and format printing can be made in addition to the MCA basic operations.

MCA Basic Operations:

START	To start MCA measurement.
STOP	To stop MCA measurement.
ERASE	To erase the MCA data.
PRESET	To preset MCA.
DATA TRANSFER	To transfer data between MCA and the PC.

Spectrum Calculation Function:

PEAK SEARCH	To search the peak of the primary differentiation for the smoothing.
5 POINTS SMOOTHING	To conduct 5 points smoothing.
2 POINTS ENERGY CALIBRATION	To calibrate the energy using the primary function.

Format Printing:

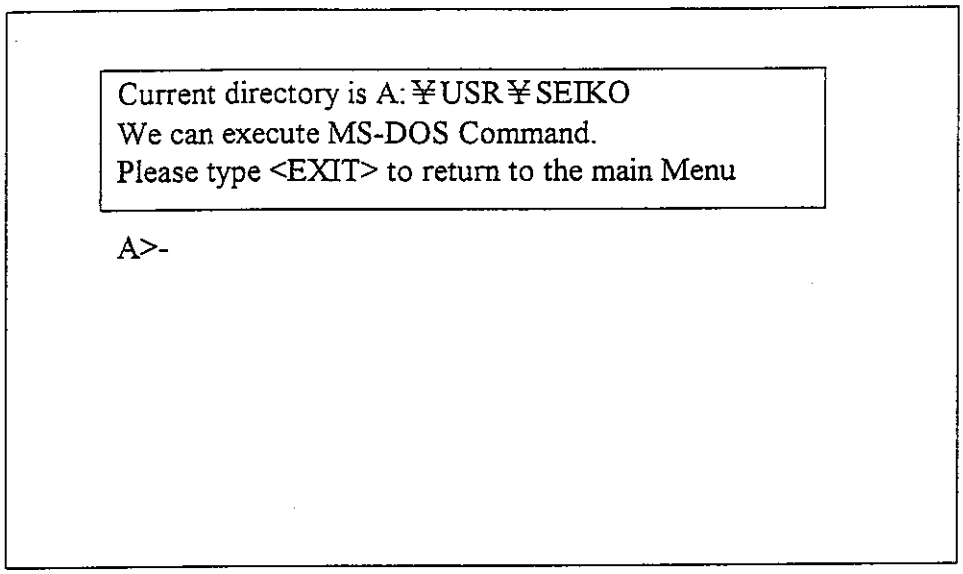
Printer output of all the available data.
Printer output of the specified data of ROI.
Printer output of the ROI information.
Graphic hard copy of the spectrum.

(7)MS-DOS Command

The MS-DOS commands can be executed without quitting this Program.

The MS-DOS commands are conducted in the sub process. Therefore, programs such as word processor that require a large execution size are not executable. The external commands that are not registered in this System cannot be executed either.

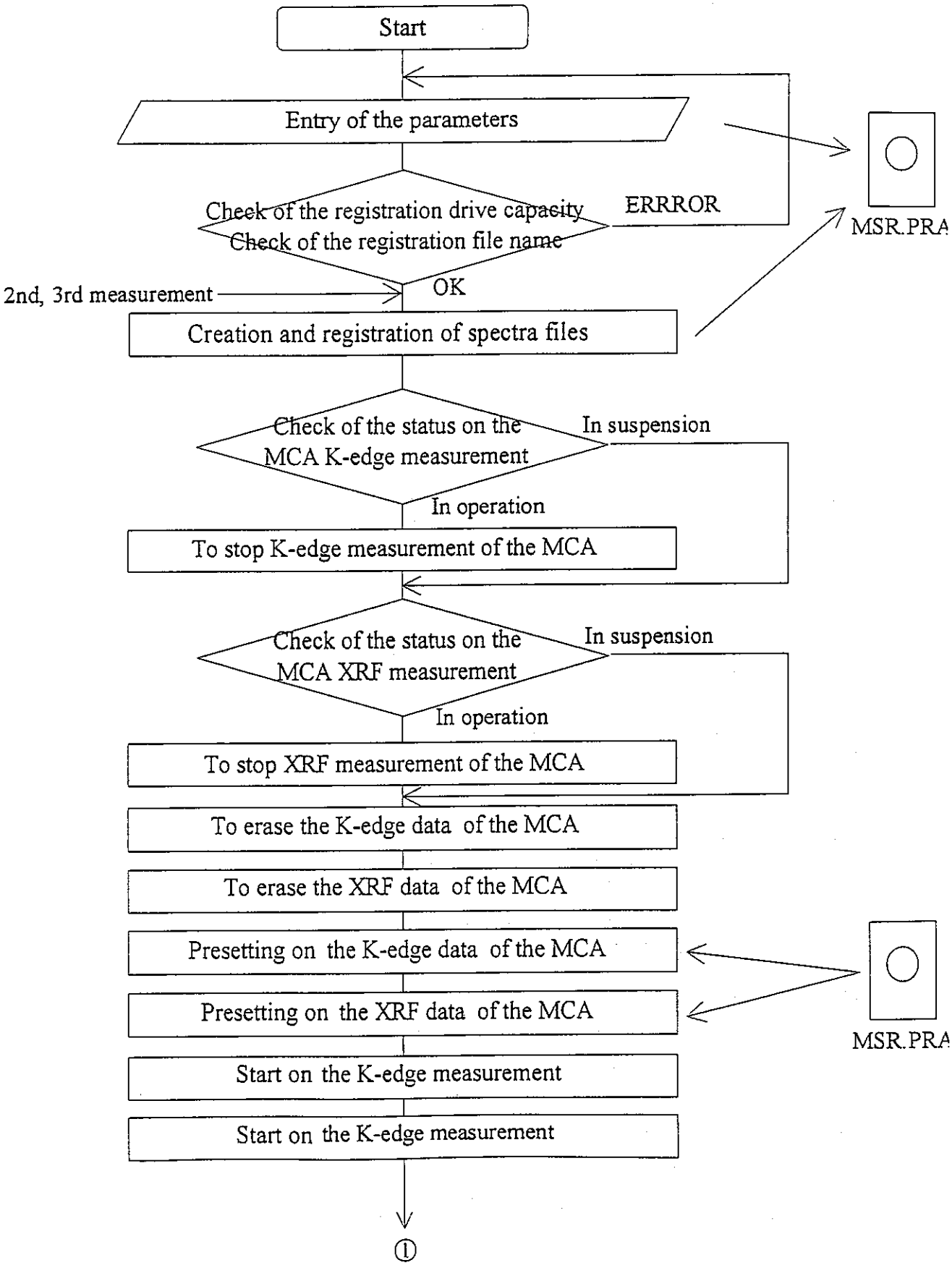
Type "EXIT" to go back to the main menu.

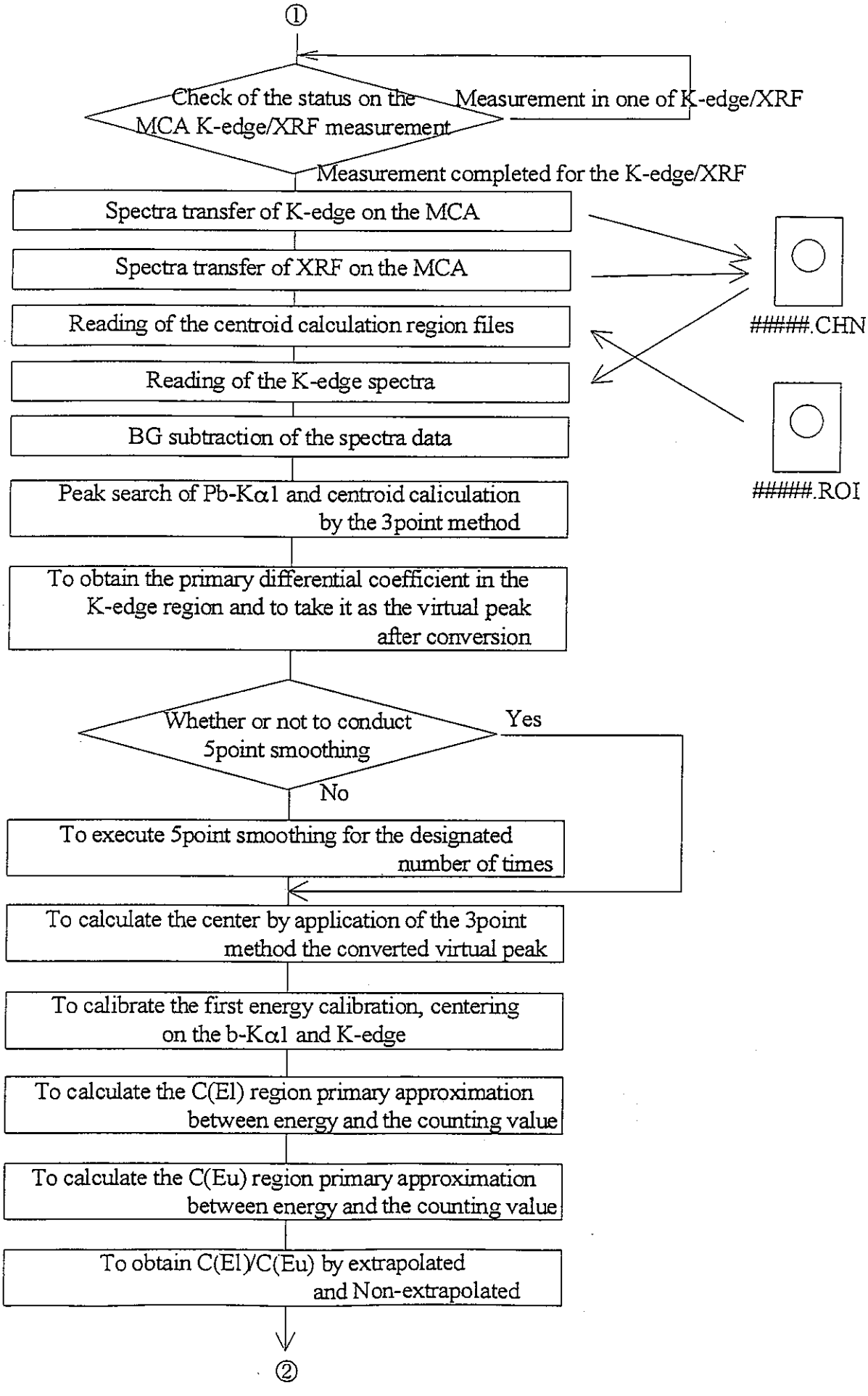


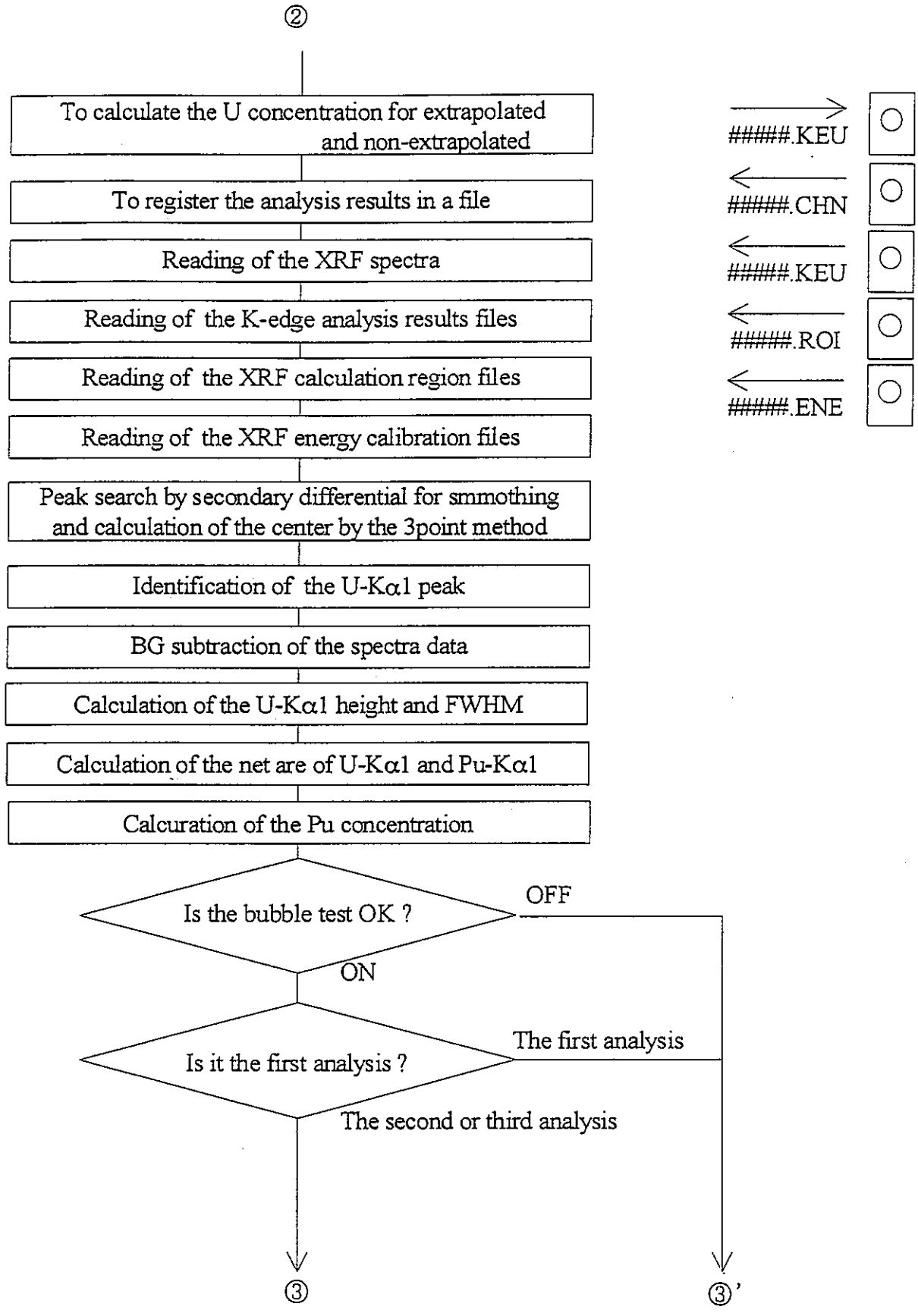
<Screen 29>

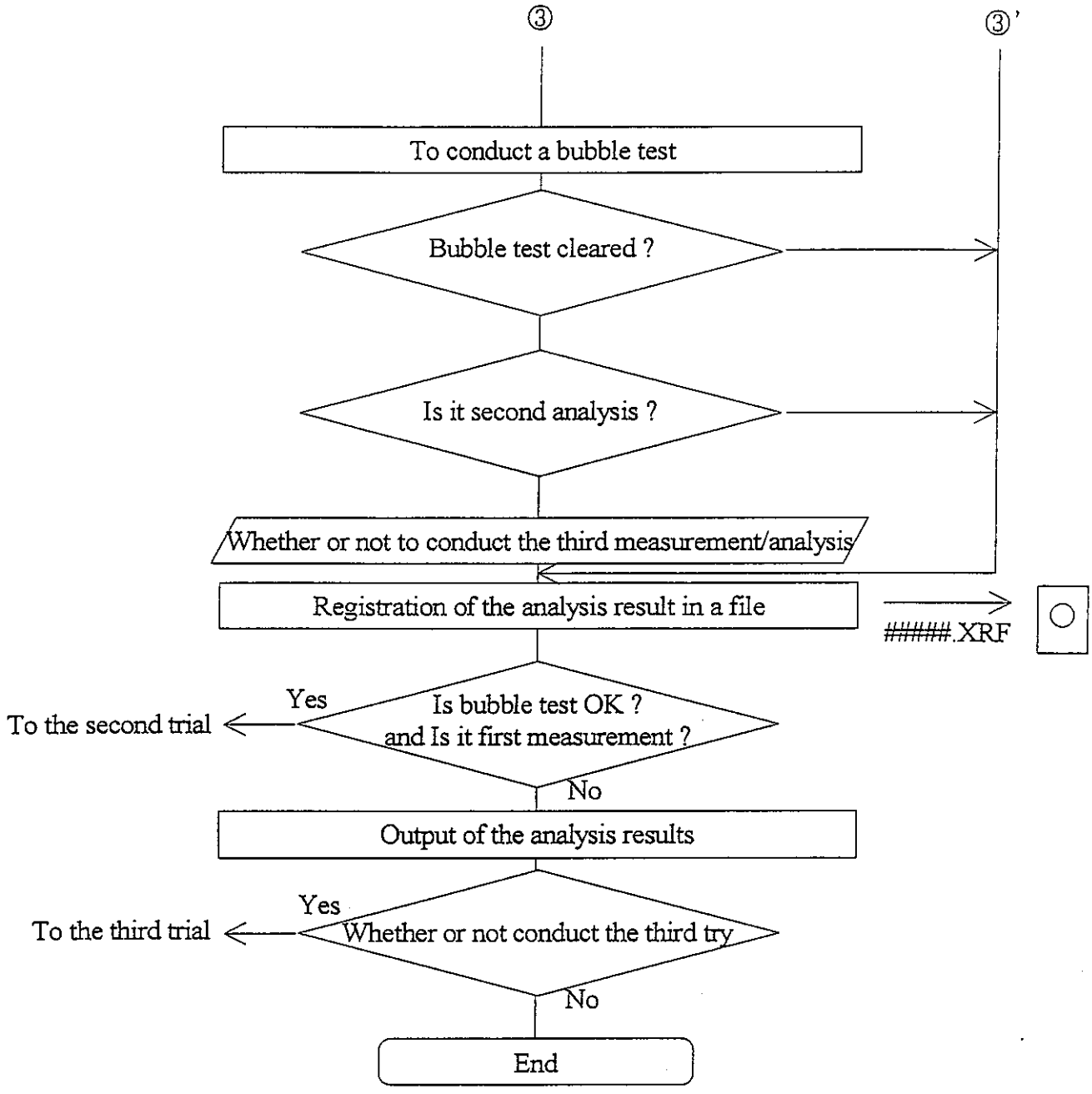
4. General Description of the Analysis

(1)Flow Chart of "K-edge/XRF Automatic Analysis" Program Start




①
Check of the status on the MCA K-edge/XRF measurement
Measurement in one of K-edge/XRF
Measurement completed for the K-edge/XRF
Spectra transfer of K-edge on the MCA
Spectra transfer of XRF on the MCA
Reading of the centroid calculation region files
Reading of the K-edge spectra
BG subtraction of the spectra data
Peak search of Pb-Kα1 and centroid caliculation by the 3point method
To obtain the primary differential coefficient in the K-edge region and to take it as the virtual peak after conversion
Whether or not to conduct 5point smoothing
Yes
No
To execute 5point smoothing for the designated number of times
To calculate the center by application of the 3point method the converted virtual peak
To calibrate the first energy calibration, centering on the b-Kα1 and K-edge
To calculate the C(El) region primary approximation between energy and the counting value
To calculate the C(Eu) region primary approximation between energy and the counting value
To obtain C(El)/C(Eu) by extrapolated and Non-extrapolated
②
#####.CHN
#####.ROI


②
To calculate the U concentration for extrapolated and non-extrapolated
To register the analysis results in a file
Reading of the XRF spectra
Reading of the K-edge analysis results files
Reading of the XRF calculation region files
Reading of the XRF energy calibration files
Peak search by secondary differential for smmothing and calculation of the center by the 3point method
Identification of the U-Kα1 peak
BG subtraction of the spectra data
Calculation of the U-Kα1 height and FWHM
Calculation of the net are of U-Kα1 and Pu-Kα1
Calcuration of the Pu concentration
Is the bubble test OK ?
OFF
ON
Is it the first analysis ?
The first analysis
The second or third analysis
③
③'
#####.KEU
#####.CHN
#####.KEU
#####.ROI
#####.ENE


③
③'
To conduct a bubble test
Bubble test cleared ?
Is it second analysis ?
Whether or not to conduct the third measurement/analysis
Registration of the analysis result in a file
######.XRF
Yes
To the second trial
Is bubble test OK ?
and Is it first measurement ?
No
Output of the analysis results
Yes
To the third trial
Whether or not conduct the third try
No
End

(2)Evaluation and Calculation

--K-edge Spectra Analysis--

① BG is subtracted from the measured spectra in accordance with the formula shown below:

$$Bi = bn + (bm - bn)\frac{\sum_{j=n}^{i} Yj}{\sum_{k=n}^{m} Yk}$$

Here,

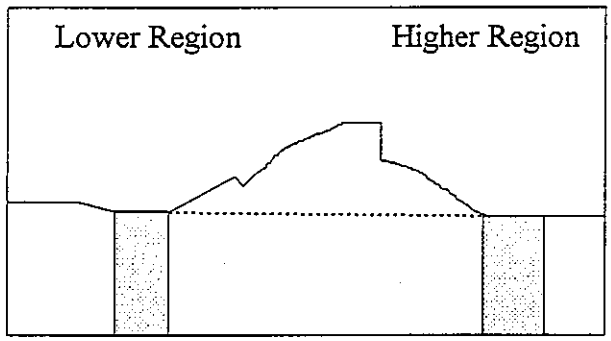
Bi = BG counts at the channel i

Yi = counts at the channel i

bn = BG level on the lower energy end (average counts at the lower region area calculation range)

bm = BG level on the higher energy end (average counts at the higher region area calculation range)

< The lower and higher region area calculation range to be used for BG adjustment >

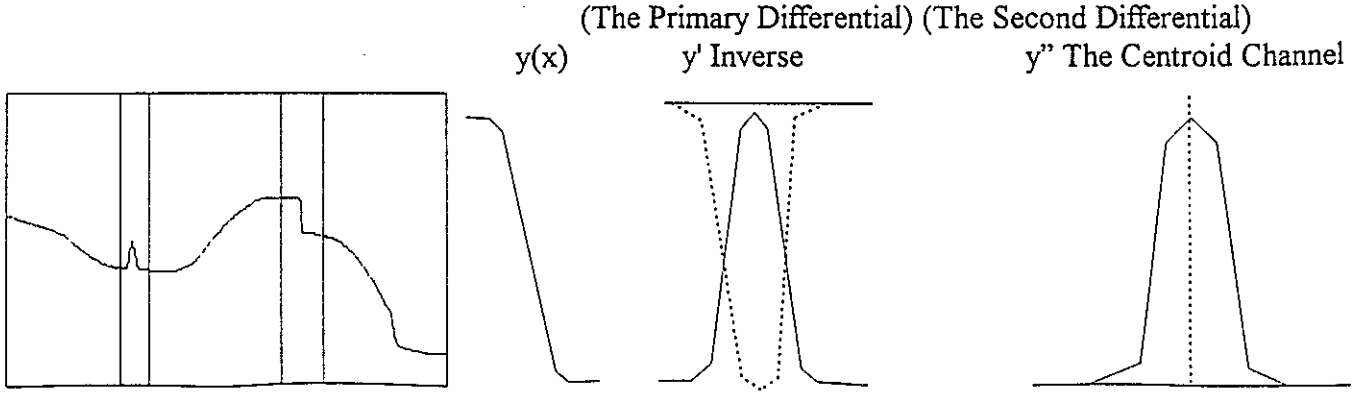


② Peak search is conducted by the primary differential method, smoothing of the Pb-Kα1 calculation region (ROI) set in advance in the K-edge spectra, and the peak centroid of the Pb-Kα1 is obtained.

*In case that plural peaks are obtained in the calculation region, the program is failed. Be sure not to designate unnecessarily large width (ROI), since it may cause a wrong search.

③ The primary differential coefficient is obtained in the K-edge center calculation region (ROI) set in advance in the K-edge spectra. The obtained polarity of y' (x) is inverted and is adopted as the virtual peak in the + direction. If application of the 5 points smoothing is specified here, smoothing is applied to y' (x). The centroid channel is calculated from the virtual peak obtained as above, using the 3 points method.

*The number of smoothing is specified by parameter.



④ The primary energy calibration formula is obtained by use of the centroid channel and energy of Pb-Kα1 and K-edge as shown is ② and ③ .

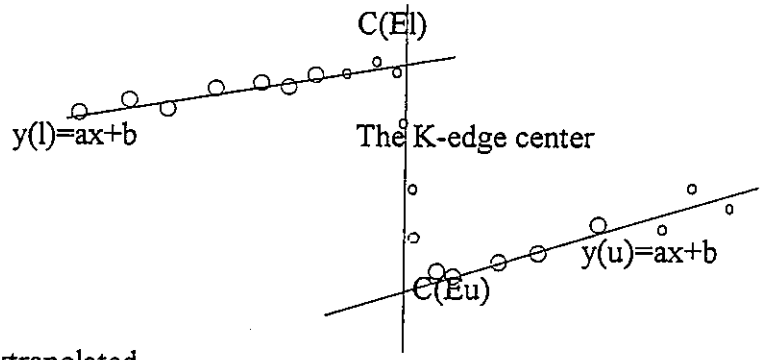
$$ene(keV)=A \cdot ch + B$$

⑤ The C(El) lower and higher end energies, and the C(Eu) lower and higher end energies are inverted by the energy calibration formula obtained as shown in <4> and the channels corresponding to the specified energies are taken as the regression region of the lower and higher end, respectively. When the energies are converted to channels, fractions are rounded down to integer for the lower end and rounded up for the higher end.

(Example) When the value for the lower end is 100.4ch and the value for the higher end is 115.2ch, the region is (100 - 116).

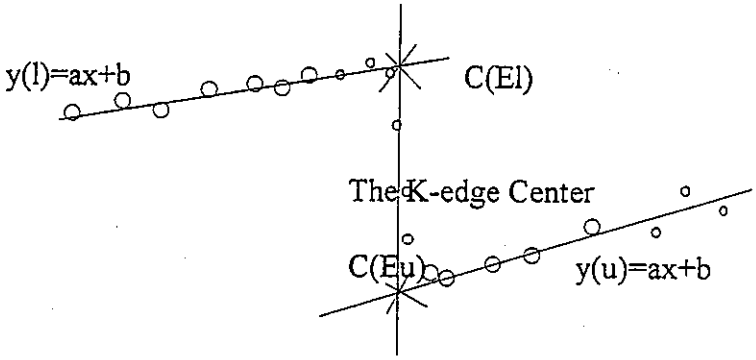
*The regression width within 50 channels will be valid. If the regression width converted by the energy calibration exceeds 50 channels, it is considered to be an error.

A first regression is conducted by the least squares method in the C(El) and C(Eu) regions already obtained.



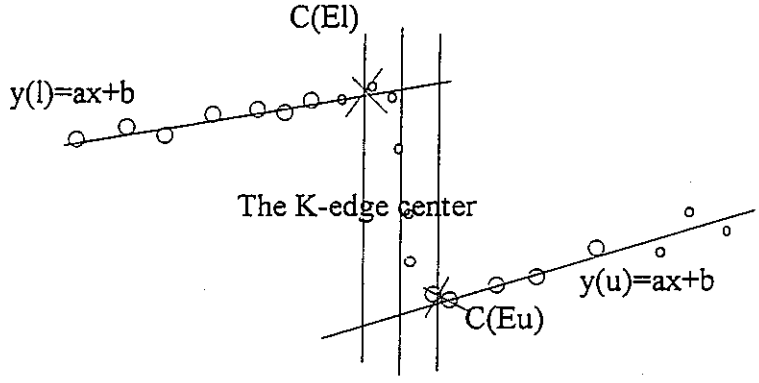
Calculation by Extrapolated

The K-edge center channel obtained as shown in ② is substituted for the equations of (y(l) = ax + b) and (y(u) = ax + b) (each equation is extrapolated) and the counts values C(El) and C(Eu) in the K-edge center channel are obtained.



Calculation by non-extrapolated

The C(El) higher end energy and the C(El) lower end energy, both specified here, are converted to channels and the results (with fractions) are substituted for the equations of (y(l) = ax + b) and (y(u) = ax + b). The obtained counts values will make C(El) and C(Eu).



⑥ The U concentration (g/l) is obtained from the net counts C(El) and C(Eu) obtained as above by use of the U concentration conversion formula shown below:

$$U = \frac{1}{\Delta\mu \times d \times 0.1} \times \ell n \frac{C(El)}{C(Eu)} \times 1000$$

Here,

U = U conc. (g/l)

$\Delta\mu$ = K-edge factor (cm^2/mg) *Difference of the mass attenuation coefficients

d = cell length (mm)

*In case the extrapolate is adopted for the calculation;
C(El):Net counts at the K-edge center on the lower energy end
C(Eu):Net counts at the K-edge center on the higher energy end

*In case the non-extrapolate is adopted for the calculation;
C(El):Net counts on the higher energy end of C(El) region
C(Eu):Net counts on the lower energy end of C(Eu) region

*$\Delta\mu$ (K-edge factor) will have a different value for extrapolate and non-extrapolate.

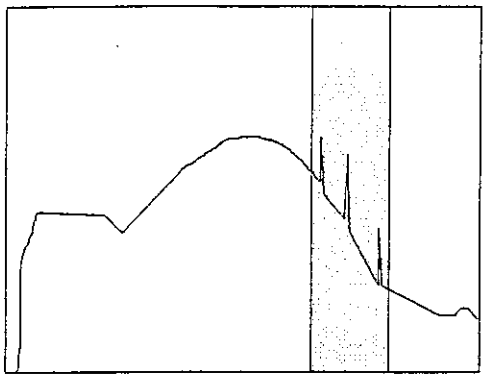
⑦ The X-ray tube output energy (tube voltage)

The channel for the counts value equivalent to the 10 times of the bm shown in ① is obtained from the spectra data before BG adjustment and then is converted to channel by use of the energy calibration formula. The result will make the output energy of X-ray tube.

If the obtained energy is X(keV), the output energy of the X-ray tube (kV) is X, since 1eV equals to 1V.

--XRF Spectra Analysis--

① Peak search of U-Kα1 is conducted in the peak analysis region (ROI) set in advance in the XRF spectra.



Peak search is conducted in the peak analysis region by use of the secondary differential method for smoothing and the peak centroid is obtained by the 3 points method. The obtained peak centroid is converted to energy value using the energy calibration formula for XRF.

The search width is expanded up and down from the centroid energy (keV) of the U-Kα1 set beforehand for the peak identification width, and a peak is searched among the centroid energies converted as above.
(Peak search and peak identification)

*The peak search sensitivity and the peak identification width are specified by parameters.

② BG is subtracted from the measured spectra by the formula shown below:

$$Bi = bn + (bm - bn)\frac{\sum_{j=n}^{i} Yj}{\sum_{k=n}^{m} Yk}$$

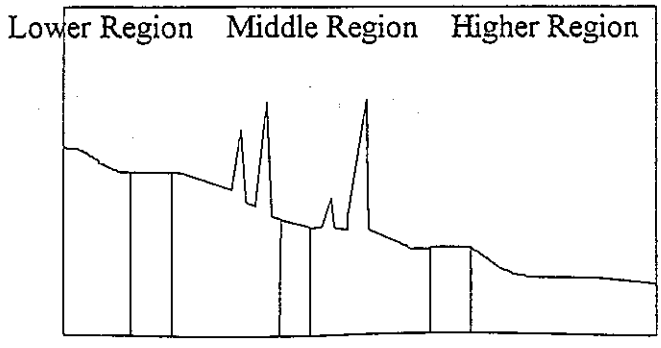
Here,

Bi = BG counts at the channel i
Yi = counts at the channel i
bn = BG level on the lower energy end (average counts at the lower region area calculation range)
bm = BG level on the higher energy end (average counts at the middle region area calculation range)

or,

bn = BG level on the middle energy end (average counts at the lower region area calculation range)
bm = BG level on the higher energy end (average counts at the higher region area calculation range)

<The lower, middle and higher region area calculation range to be used for BG adjustment >

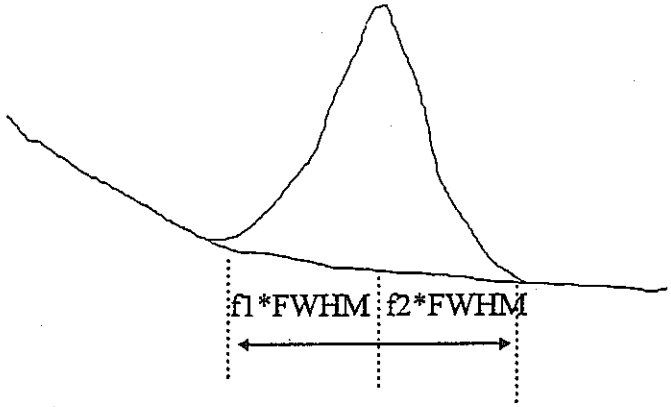


③ The peak centroid height of the U-Kα1 is obtained from the spectra with the BG adjustment and the FWHM is calculated by the non-extrapolate method.

The FWHM of the Pu-Kα1 is calculated as being equal to the FWHM of U-Kα1.

④ The peak region is obtained, by accumulating the counts values for the regions obtained, for one thing, deducting the lower energy region factor multiplied by the FWHM, and for another, adding the higher energy region factor multiplied by the FWHM.

*The lower and higher energy region factors are specified by parameters.



f1 : Lower Energy Region Factor
f2 : Higher Energy Region Factor

⑤ R(U/Pu) is obtained, by substituting the U concentration obtained in the K-edge peak search for the excitation probability ratio calibration formula.

$$R(U/Pu) = A \times \exp(B \times U(g/\ell))$$

R(U/Pu) are obtained for both U values by the extrapolate and non-extrapolate.

⑥ U/Pu is obtained by the U/Pu ratio conversion formula shown below, using the net areas of U-Kα1 and Pu-Kα1, and the excitation probability ratio, each already obtained.

$$\frac{U}{Pu} = \frac{A(U)}{A(Pu)} \times \frac{P(UK\alpha 1)}{P(PuK\alpha 1)} \times \frac{F}{R(U/Pu)} = \Gamma$$

$$Pu = \frac{U}{\Gamma}$$

Here,

A (U)	mass number of U
A (Pu)	mass number of Pu
P (U-Kα1)	net area counts of U
P (Pu-Kα1)	net area counts of Pu
F	efficiency rate of all measured values
R (U/Pu)	excitation probability ratio of Kα1 fluorescent X-ray

--Error Estimation--

① K-edge measurement

(Non-extrapolated)

$$U\,conc. = \frac{1}{\Delta\mu \times x \times 0.1} \times \ell n \frac{C(E\ell)}{C(Eu)} \times 1000$$

and the formula for the uncertainty is

$$\text{Uncertainty in U conc.} = U\,conc. \times \sqrt{\left(\frac{\sigma \text{ of } C(E\ell)}{C(E\ell)}\right)^2 + \left(\frac{\sigma \text{ of } C(Eu)}{C(Eu)}\right)^2}$$

σ of C(Eℓ) is estimated by

$$\sigma\ of\ C(E\ell) = \sqrt{\left\{\frac{1}{N} + \frac{(X-\overline{X})^2}{\sum_{i=1}^{n}(X_i-\overline{X})}\right\}} \times Ve$$

where N: Number of sample

Ve: Error distribution from regression line

$$Ve = \frac{\sum_{i=1}^{N}(Yi-(A\cdot Xi+B))^2}{N-2}$$

X: Established energy of $C(E\ell)$ region

Non-extrapolated : 113.3keV

Extrapolated : 115.6keV

$\overline{X}$: Average of $C(E\ell)$ region

$$\overline{X} = \frac{\sum_{i=1}^{N} Xi}{N}$$

Xi: Energy of the channel i

Yi: Counts of the cannel i

σ of $C(Eu)$ is omitted.

(Extrapolated)

This method is omitted.

② XRF measurement

$$U/Pu\ ratio = \frac{A(U)}{A(Pu)} \times \frac{P(UK\alpha 1)}{P(PuK\alpha 1)} \times \frac{F}{R_{(U/PU)}}$$

Then,

$$Uncertainty\ in\ U/Pu\ ratio = U/Pu\ ratio \times \sqrt{\left(\frac{\sqrt{G_U + B_U}}{P(UK\alpha 1)}\right)^2 + \left(\frac{\sqrt{G_{Pu} + B_{Pu}}}{P(PuK\alpha 1)}\right)^2}$$

where,

G_U : Gross counts of UKα1 peak
B_U : B.G counts of UKα1 peak
G_{Pu} : Gross counts of PuKα1 peak
B_{pu} : B.G counts of PuKα1 peak

③ Pu concentration

$$Pu\ conc. = \frac{U\ conc.}{U/Pu\ ratio}$$

so that

$$Uncertainty\ in\ Pu\ conc. = Pu\ conc. \times \sqrt{\left(\frac{Uncertainty\ in\ U\ conc.}{U\ conc.}\right)^2 + \left(\frac{Uncertainty\ in\ U/Pu\ ratio}{U/Pu\ ratio}\right)^2}$$

--Bubble Test--

The objective of the bubble test is to check whether or not there is any bubble generation in the cell during the measurement. The test is conducted for U concentration (g/l), Pu (g/l) and U/Pu ratio.

① An assumption is made that the two measurement and their analysis had the following results.

The first measurement result: $X1 \pm S1$

The second measurement result: $X2 \pm S2$

The average: $\bar{x} = (X1 + X2)/2$

The test parameter: $Z1 = (|X1 - X2|)/S1$

*Here, Sn means the standard deviation of the n time.

If Z1 is smaller than 3.18, the test is considered to be successful, and the result will be as follows:

$answer = \bar{x} \pm (S1/1.414)$ (Z1<3.18)

End of assay !

② If Z1 is equal to 3.18 or larger, the third measurement and analysis should be conducted.

The third measurement result: $X3 \pm S1$

The average: $\bar{x} = (X1 + X2 + X3)/3$

If the value which is most far from the average $\underline{x}$ is x*, the test parameter will be as follows:

The test parameter: $Z2 = (|\underline{x} - X^*|)/S1$

If $Z2 < 1.95$, the test is considered to be successful, and the result will be as follows:

$answer = \bar{x} \pm (S1/1.732)$ (Z2<1.95)

End of assay!

If $1.95 \leq Z2 < 3.18$, the value x* will be eliminated out of the calculation, and the result will be as follows:

$ans = (X_i + X_{i+1})/2 \pm (S1/1.414)$

If $3.18 \leq Z2$, there is a possibility that there is bubble intrusion.

The sample is out of control and should be performed DA.

*The reference values of 3.18 and 1.95 used in the above calculations are specified by the parameter option.

--MC-Bias Test--

This test should be done regularly as part of the measurement control program. The measurement can be done with reference material, such as MOX powder including U and Pu standards, and uranium foil. After the measurement, the assay results are compared with reference values stored in the parameter file. The test is for the U concentration (g/l) and the U/Pu ratio.

① An assumption is made that the following results are obtained after a measurement and analysis:

U conc. (g/l)	non-extrapolated	$X1 \pm S1$
U/Pu ratio		$X2 \pm S2$

*Here, Sn is the standard deviation.

The measured reference values of the reference material are as follows:

U conc. (g/l)	R1
U/Pu ratio	R2

Then, the test parameters will be as follows:

U conc. (g/l)	non-extrapolated	$Z1 = (X1 - R1) \times 100 / R1$
U/Pu Ratio		$Z2 = (X2 - R2) \times 100 / R2$

② The boundary values for the test parameters are set as follows:

	Warning	Error
U conc. (g/l)	W1 (%)	E1 (%)
U/Pu ratio	W2 (%)	E2 (%)

Here, Wn < En.

Then, the test result for U concentration (g/l) by non-extrapolate will be as follows:

$\|Z1\| < W1$	→	good
$W1 \leq \|Z1\| < E1$	→	warning
$E1 \leq \|Z1\|$	→	error

and the test result for U/Pu ratio will be as follows:

$\|Z2\| < W2$	→	good
$W2 \leq \|Z2\| < E2$	→	warning
$E2 \leq \|Z2\|$	→	error

If all the test results are good, the test will be successful.
If the test results have some warning but no error, the test will end as warning.
If there is any error in the test results, the test will end as error.

--MC-Precision Test--

The objective of the test is to conduct χ^2 verification of the results obtained through measurements of the specified times (5～10, and 15) and the resultant KED/XRF analyses. The test is for the U concentration (g/l) and the U/Pu ratio.

① Measurements and analyses are conducted for n times, and the average x and the standard deviation s are calculated as shown below:

	U-Conc. Non-extrapo.	U/Pu Ratio
1st	###.## +- #.##	###.## +- #.##
2nd	###.## +- #.##	###.## +- #.##
3rd	###.## +- #.##	###.## +- #.##
.	.	.
.	.	.
.	.	.
n th	###.## +- #.##	###.## +- #.##
Average	###.##	###.##
Stand. Dev.	#.##	#.##
Chi-square	#.##	#.##

$$\text{Average } x = (\sum Xi) / n$$

$$\text{Standard deviation } s = \sqrt{\sum (Xi - \underline{x})^2 / (n-1)}$$

② χ^2 is calculated as shown below:

$$\chi^2 = \frac{(S.D.\ of\ sample)}{(Est.\ S.D.)}$$

where

$$S.D.\ \text{of sample} = \sqrt{\frac{\sum_{i=1}^{n} (xi - \bar{x})^2}{n-1}}$$

"Est. S.D." is the uncertainty from the last measurement

Example

n	U conc.	S.D.
1	190.43	0.84
2	190.43	0.69
3	190.64	0.78
4	189.89	0.63
5	191.20	0.62
Ave.	190.52	
S.D.	0.47	

$$\chi^2 = \frac{(0.47)^2}{(0.62)^2}$$

$$= 0.57$$

$0.12<0.57<2.79$ and $0.05<0.57<3.72$ → End of Assay

The boundary values for the verification are shown below:

Number of measurement n	(degree of freedom) n-1	Warning Limit 95% confidence	Action Limit 99% confidence
5	4	0.12 - 2.79	0.05 - 3.72
6	5	0.17 - 2.57	0.08 - 3.35
7	6	0.21 - 2.41	0.11 - 3.09
8	7	0.24 - 2.29	0.14 - 2.90
9	8	0.27 - 2.19	0.17 - 2.75
10	9	0.30 - 2.11	0.19 - 2.62
15	14	0.40 - 1.87	0.29 - 2.24

If the ratio, χ^2/degrees of freedom, stays out of the warning limit and the action limit, the test result will be WARNING or ERROR, respectively. The boundary values are considered not to be in the confidence.

If all the test results are good for the U concentration and the U/Pu ratio, the test will be successful.

If the test results have some warning but no error, the test will end as warning. If there is any error in the test results, the test will end as error.

(3)Printout

1)KED/XRF Assay

【 PRT1-1 】 KED / XRF Printout results (without bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage: __________kV               Temperature     : _________℃
Remarks1: ########################################
Remarks2: ########################################

                                      --- Parameters ---
K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg        Non-extrapo.  : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV
XRF Parameter
  U-Kal  energy : ###.###keV
  Pu-Kal energy : ###.###keV
  Atomic Weight  U : ###.##             Pu : ###.##
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    Middle           ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV

                                   --- Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : ####  (###.##)      ####  (###.##)
    Upper energy side : ####  (###.##)      ####  (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                  C(Eu)                  ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l

XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.              Real Time : ######sec.
  R(U/Pu)  Extrapolated : #.####E+## Non-Extrapo. : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak          Energy      Centroid    FWHM     Peak area
    U-Kal       ###.##keV   ####.##ch   #.###    #######.## +- ####.##
    Pu Kal      ###.##keV   ####.##ch   #.###    #######.## +- ####.##
  U/Pu Ratio Extrapolated: #.### +- #.##  Non Extrapo. : #.### +- #.##
  Pu Conc.
    Extrapolated : ###.#### +- ##.## g/l
    Non-extrapo. : ###.#### +- ##.## g/l
```

【 PRT1-2-1/5 】 KED / XRF Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 1

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:          kV                Temperature      :          ℃
Remarks1: ########################################
Remarks2: ########################################

                                      --- Parameters ---
K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg          Non-extrapo. : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                 End
    Low              ###.##keV   ~         ###.##keV
    High             ###.##keV   ~         ###.##keV
XRF Parameter
  U-Kal  energy : ###.###keV
  Pu-Kal energy : ###.###keV
  Atomic Weight  U : ###.##               Pu : ###.##
  BG Window          Start                 End
    Low              ###.##keV   ~         ###.##keV
    Middle           ###.##keV   ~         ###.##keV
    High             ###.##keV   ~         ###.##keV

                                  --- 1st Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge    : ###.##keV (####.##ch)
  Pb-Kal      : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                  C(El)                 C(Eu)                 ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l

XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.              Real Time : ######sec.
  R(U/Pu)   : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak          Energy      Centroid    FWHM    Peak area
    U-Kal       ###.##keV   ####.##ch   #.###   ########.## +- ####.##
    Pu Kal      ###.##keV   ####.##ch   #.###   ########.## +- ####.##
  U/Pu Ratio : #.### +- #.##
```

【 PRT1-2-2/5 】 KED / XRF Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 2

                                --- 2nd Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                   C(Eu)                   ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l

XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.              Real Time : ######sec.
  R(U/Pu)    : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak          Energy      Centroid    FWHM    Peak area
    U-Kal       ###.##keV   ####.##ch   #.###   #######.## +- ####.##
    Pu Kal      ###.##keV   ####.##ch   #.###   #######.## +- ####.##
  U/Pu Ratio : #.### +- #.##
```

【 PRT1-2-3/5 】 KED / XRF Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 3

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage: _____kV                    Temperature     : _____℃
Remarks1: ########################################
Remarks2: ########################################
K-edge Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV
Last Data File : ########.KEU             ########.XRF
Live Time      : ######sec.
KED Real Time  : ######sec.               XRF Real Time : ######sec.

1st Results:  U-Conc.                Pu-Conc.                U/Pu Ratio
 Extrapolated ###.## +- #.##         ###.## +- #.##          ###.## +- #.##
 Non-extrapo. ###.## +- #.##         ###.## +- #.##          ###.## +- #.##

2nd Results:  U-Conc.                Pu-Conc.                U/Pu Ratio
 Extrapolated ###.## +- #.##         ###.## +- #.##          ###.## +- #.##
 Non-extrapo. ###.## +- #.##         ###.## +- #.##          ###.## +- #.##

Average(Z1):  U-Conc.                Pu-Conc.                U/Pu Ratio
 Extrapolated ###.## +- #.##         ###.## +- #.##          ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)   ###.## +- #.##(#.##)

                                     --- Assay Option FAILED ---
Reload sample solution,
Perform third measurement.
```

*If the bubble test is successful, the printout will be as follows:

```
Average(Z1):  U-Conc.                Pu-Conc.                U/Pu Ratio
 Extrapolated ###.## +- #.##         ###.## +- #.##          ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)   ###.## +- #.##(#.##)

                                     --- Assay Option PASSED ---
End of assay !
```

【 PRT1-2-4/5 】 KED / XRF Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 4

                                --- 3rd Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge    : ###.##keV (####.##ch)
  Pb-Kal      : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                   C(Eu)                  ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l

XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.              Real Time : ######sec.
  R(U/Pu)   : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak        Energy      Centroid    FWHM    Peak area
    U-Kal     ###.##keV   ####.##ch   #.###   #######.## +- ####.##
    Pu Kal    ###.##keV   ####.##ch   #.###   #######.## +- ####.##
  U/Pu Ratio : #.### +- #.##
```

【 PRT1-2-5/5 】 KED / XRF Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 5

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:          kV                Temperature      :          ℃
Remarks1: ########################################
Remarks2: ########################################
K-edge Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ～       ###.##keV
    High             ###.##keV   ～       ###.##keV
Last Data File : ########.KEU             ########.XRF
Live Time      : ######sec.
KED Real Time  : ######sec.               XRF Real Time : ######sec.

1st Results:  U-Conc.              Pu-Conc.              U/Pu Ratio
 Extrapolated ###.## +- #.##       ###.## +- #.##       ###.## +- #.##
 Non-extrapo. ###.## +- #.##       ###.## +- #.##       ###.## +- #.##

2nd Results:  U-Conc.              Pu-Conc.              U/Pu Ratio
 Extrapolated ###.## +- #.##       ###.## +- #.##       ###.## +- #.##
 Non-extrapo. ###.## +- #.##       ###.## +- #.##       ###.## +- #.##

3rd Results:  U-Conc.              Pu-Conc.              U/Pu Ratio
 Extrapolated ###.## +- #.##       ###.## +- #.##       ###.## +- #.##
 Non-extrapo. ###.## +- #.##       ###.## +- #.##       ###.## +- #.##

Average(Z2):  U-Conc.              Pu-Conc.              U/Pu Ratio
 Extrapolated ###.## +- #.##       ###.## +- #.##       ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##) ###.## +- #.##(#.##)

                                   --- Assay Option FAILED ---

Z2>#.##, Sample is out of control
   must Perform DA on this sample
```

*If the bubble test is successful, the printout will be as follows:

```
Average(Z2):  U-Conc.              Pu-Conc.              U/Pu Ratio
 Extrapolated ###.## +- #.##       ###.## +- #.##       ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##) ###.## +- #.##(#.##)

                                   --- Assay Option PASSED ---

End of assay !
```

2)KED U (or Pu) Assay

【 PRT2-1 】 KED U Printout results (without bubble test)

```
Tokai Hybrid/K-edge Measurement Results

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:            kV              Temperature      :          ℃
Remarks1: ########################################
Remarks2: ########################################

                                      --- Parameters ---
K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg         Non-extrapo. : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ～       ###.##keV
    High             ###.##keV   ～       ###.##keV

                                   --- Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                   C(Eu)                   ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l
```

【 PRT2-2-1/5 】 KED U Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results                              Page 1

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:  _____kV                   Temperature      :  _____℃
Remarks1: ########################################
Remarks2: ########################################

                                    --- Parameters ---

K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg        Non-extrapo. : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV

                                --- 1st Measurement Results ---

K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : ########cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)      End Ch(keV)
    Lower energy side : #### (###.##)      #### (###.##)
    Upper energy side : #### (###.##)      #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                                                                 ln[(C(El)/C(Eu)]
                  C(El)                  C(Eu)
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l
```

【 PRT2-2-2/5 】 KED U Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results                                  Page 2

                                  --- 2nd Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                  C(Eu)                  ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l
```

【 PRT2-2-3/5 】 KED U Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results                          Page 3

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:           kV               Temperature      :          ℃
Remarks1: ########################################
Remarks2: ########################################
K-edge Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ～       ###.##keV
    High             ###.##keV   ～       ###.##keV
Last Data File : ########.KEU             ########.XRF
Live Time      : ######sec.
KED Real Time  : ######sec.

1st Results:  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##

2nd Results:  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##

Average(Z1):  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)

                                  --- Assay Option FAILED ---
Reload sample solution,
Perform third measurement.
```

*If the bubble test is successful, the printout will be as follows:

```
Average(Z1):  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)

                                  --- Assay Option PASSED ---
End of assay !
```

【 PRT2-2-4/5 】 KED U Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results                                  Page 4

                                --- 3rd Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : ########cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                   C(Eu)                    ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l
```

【 PRT2-2-5/5 】 KED U Printout results (with bubble test)

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)                    Page 5

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage: _____kV                    Temperature     : _____℃
Remarks1: ########################################
Remarks2: ########################################
K-edge Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV
Last Data File : ########.KEU             ########.XRF
Live Time      : ######sec.
KED Real Time  : ######sec.

1st Results:  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##

2nd Results:  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##

3rd Results:  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##

Average(Z2):  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)

                                  --- Assay Option FAILED ---
Z2>#.##, Sample is out of control
   must Perform DA on this sample
```

*If the bubble test is successful, the printout will be as follows:

```
Average(Z2):  U-Conc.
 Extrapolated ###.## +- #.##
 Non-extrapo. ###.## +- #.##(#.##)

                                  --- Assay Option PASSED ---
End of assay !
```

3)Manual KED/XRF Assay

【 PRT3 】

```
Tokai Hybrid/K-edge Measurement Results(K-edge/XRF)

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                          Measurement Type: Assay
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:            kV                Temperature     :          ℃
Remarks1: ########################################
Remarks2: ########################################

                                        --- Parameters ---
K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg          Non-extrapo.  : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                 End
    Low              ###.##keV   ～        ###.##keV
    High             ###.##keV   ～        ###.##keV
XRF Parameter
  U-Kal  energy : ###.###keV
  Pu-Kal energy : ###.###keV
  Atomic Weight  U : ###.##               Pu : ###.##
  BG Window          Start                 End
    Low              ###.##keV   ～        ###.##keV
    Middle           ###.##keV   ～        ###.##keV
    High             ###.##keV   ～        ###.##keV

                                    --- Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.                Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : #######cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(E1) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(E1) = #.####E+## * energy(keV) + #.####E+##
                  C(E1)                   C(Eu)                  ln[(C(E1)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l

XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.                Real Time : ######sec.
  R(U/Pu)    : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak          Energy      Centroid    FWHM    Peak area
    U-Kal       ###.##keV   ####.##ch   #.###   #######.## +- ####.##
    Pu Kal      ###.##keV   ####.##ch   #.###   #######.## +- ####.##
  U/Pu Ratio : #.### +- #.##
  Pu Conc.
    Extrapolated : ###.#### +- ##.## g/l
    Non-extrapo. : ###.#### +- ##.## g/l
```

4)Manual KED U (or Pu) Assay

【 PRT4 】

```
Tokai Hybrid/K-edge Measurement Results(K-edge(U))

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage: _____kV                    Temperature     : _____℃
Remarks1: ########################################
Remarks2: ########################################

                                    --- Parameters ---
K-edge Parameter
  K-edge factor
    Extrapolated : #.#####cm2/mg        Non-extrapo. : #.#####cm2/mg
  Cell path length : ##.#mm
  BG Window          Start                End
    Low              ###.##keV   ~        ###.##keV
    High             ###.##keV   ~        ###.##keV

                                  --- Measurement Results ---
K-edge(U-Conc.) Measurement
  Data File : ########.KEU
  Live Time : ######sec.              Real Time : ######sec.
  Calculated cut-off energy : ###.##kV
  Total count rate : ########cps
  Detector resolution(Pb-Kal FWHM) : #.###keV
  Fitting Area          Start Ch(keV)       End Ch(keV)
    Lower energy side : #### (###.##)       #### (###.##)
    Upper energy side : #### (###.##)       #### (###.##)
  U K-edge     : ###.##keV (####.##ch)
  Pb-Kal       : ###.##keV (####.##ch)
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Correlation Formula
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
    Lower energy side : Count(El) = #.####E+## * energy(keV) + #.####E+##
                    C(El)                   C(Eu)                   ln[(C(El)/C(Eu)]
    Extrapolated : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
    Non-extrapo. : #.####E+##(###.#keV)  #.####E+##(###.#keV)  #.####E+##
  U-Conc.
    Extrapolated : ###.## +- #.## g/l
    Non-extrapo. : ###.## +- #.## g/l
```

5)Manual XRF Assay

【 PRT5 】

```
Tokai Hybrid/K-edge Measurement Results(XRF)

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: ######                        Measurement Type: Assay
Operator: ####################          Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:          kV                Temperature     :          ℃
Remarks1: ########################################
Remarks2: ########################################

                                      ---- Parameters ----
XRF Parameter
  U-Kal  energy : ###.###keV
  Pu-Kal energy : ###.###keV
  Atomic Weight  U : ###.##              Pu : ###.##
  BG Window          Start               End
    Low              ###.##keV   ~       ###.##keV
    Middle           ###.##keV   ~       ###.##keV
    High             ###.##keV   ~       ###.##keV

                                   ---- Measurement Results ----
XRF Measurement
  Data File : ########.XRF
  Live Time : ######sec.              Real Time : ######sec.
  R(U/Pu)   : #.####E+##
  Energy Cal.(keV) = #.####E-## * ch + #.####E+##
  Overall efficiency factor : #.####E+##
  Peak          Energy      Centroid    FWHM    Peak area
    U-Kal       ###.##keV   ####.##ch   #.###   #######.## +- ####.##
    Pu Kal      ###.##keV   ####.##ch   #.###   #######.## +- ####.##
  U/Pu Ratio Extrapolated: #.### +- #.## Non-extrapo. : #.### +- #.##
```

6)MC Bias

【 PRT6 】

```
Tokai Hybrid/K-edge Measurement Results

Inspector  :  NSB: ####################       IAEA: ####################

Sample ID: REFER.                        Measurement Type: MC-Bias
Operator: ####################           Measurement Date: DD-MM-YY   hh:mm:ss
Tube Voltage:           kV               Temperature      :          ℃
Remarks1: ########################################
Remarks2: ########################################
K-edge Cell path length : ##.#mm
Data File      : MB######.KEU            MB######.XRF
Live Time      : ######sec.
KED Real Time : ######sec.                XRF Real Time : ######sec.

Reference Value:
             U-Conc. (Non-extrapo.)                      U/Pu Ratio
             ###.##                                      ###.##

Measurement Results(Bias%):
             U-Conc. (Non-extrapo.)                      U/Pu Ratio
             ###.## +- #.##(##.##)                       ###.## +- #.##(##.##)

                                  --- MC-Bias Option PASSED ---
End of MC-Bias
```

*If the test results is a warning , the printout will be as follows:

```
                                  --- MC-Bias Option FAILED ---
WARNING:MC-Bias test result exceeds the Warning Limit
```

*If the test results is a error , the printout will be as follows:

```
                                  --- MC-Bias Option FAILED ---
ERROR:MC-Bias test result exceeds the Action Limit
```

7)MC Precision

【 PRT7 】

```
Tokai Hybrid/K-edge Measurement Results

Inspector  :  NSB: ####################      IAEA: ####################

Sample ID: REFER.                          Measurement Type: MC-Precision
Operator: ####################             Measurement Date: DD-MM-YY   hh:mm:ss
                                           Temperature      :  _________ ℃
K-edge Cell path length : ##.#mm
Last Data File: ########.KEU               ########.XRF
Live Time      : ######sec.
KED Real Time : ######sec.                  XRF Real Time : ######sec.

Measurement Results:
              U-Conc. (Non-extrapo.)                        U/Pu Ratio
  1st         ###.## +- #.##                                ###.## +- #.##
  2nd         ###.## +- #.##                                ###.## +- #.##
  3rd         ###.## +- #.##                                ###.## +- #.##
  4th         ###.## +- #.##                                ###.## +- #.##
  5th         ###.## +- #.##                                ###.## +- #.##
  6th         ###.## +- #.##                                ###.## +- #.##
-------------------------------------------------------------------------
 Average      ###.##                                        ###.##
 Stand.Dev      #.##                                          #.##
 Chi-square     #.##                                          #.##
 Analysis method tested:
    Error Limits Reduced Chi-square        >#.##  and  <#.##
    Warning Limit Reduced Chi-square       >#.##  and  <#.##

                              --- MC-Precision Option PASSED ---
End of MC-Precision
```

*If the test results is a warning , the printout will be as follows:

```
                              --- MC-Precision Option FAILED ---
WARNING:MC-Precision test Chi-square exceeds the Warning Limit #.## - #.##
```

*If the test results is a error , the printout will be as follows:

```
                              --- MC-Precision Option FAILED ---
ERROR:MC-Precision test Chi-square exceeds the Action Limit #.## - #.##
```

8)Parameters

【 PRT8-1 】 Edit Parameters for KED(U)

```
Edit Parameter (U) K-edge

ROI data file                        : ########.###
5point smoothing                     : YES
No. of smoothing                     : ##
Pb-Kal energy          (KeV)         : ###.###
K-edge energy          (KeV)         : ###.###
C(El)  region          (KeV)   Start: ###.##    ~    End: ###.##
C(Eu)  region          (KeV)   Start: ###.##    ~    End: ###.##
BG Window Low          (KeV)   Start: ###.##    ~    End: ###.##
          High         (KeV)   Start: ###.##    ~    End: ###.##
K-edge factor
  Extrapolated      (cm2/mg)         : ####.######
  Non-extrapo.      (cm2/mg)         : ####.######
Cell path length        (mm)         : ##.#
```

【 PRT8-2 】 Edit Parameters for XRF

```
Edit Parameter XRF

ROI data file                        : ########.ROI
Reference energy Cal. file           : ########.ENE
Factors of R(U/Pu)  A                : -#.######E+##
                    B                : -#.######E+##
BG Window Low          (KeV)   Start: ###.##    ~    End: ###.##
          Middle       (KeV)   Start: ###.##    ~    End: ###.##
          High         (KeV)   Start: ###.##    ~    End: ###.##
Sensitivity                           : #
Region to Identify     (KeV)          : ##.##
U-Ka1 energy           (KeV)          : ###.####
      Peak Width lower                : #.## * FWHM
      Peak Width upper                : #.## * FWHM
Pu-Ka1 energy          (KeV)          : ###.####
      Peak Width lower                : #.## * FWHM
      Peak Width upper                : #.## * FWHM
U-Ka2 energy           (KeV)          : ###.####
U-Kb1 energy           (KeV)          : ###.####
Atomic Weight U                       : ####.##
              Pu                      : ####.##
Factor                                : #.#####E+##
First  Limit                          :#.##
Second Limit                          :#.##
```

【 PRT8-3 】 Edit Parameters for MC-Bias

```
Edit Parameter MC-Bias

Reference Value  U Conc.              : #.######E+##
                 U/Pu Ratio          : #.######E+##
U Conc.      Warning Limit   (%)     : ##.##
             Action  Limit   (%)     : ##.##
U/Pu Ratio   Warning Limit   (%)     : ##.##
             Action  Limit   (%)     : ##.##
```

9) List log

【 PRT9 】

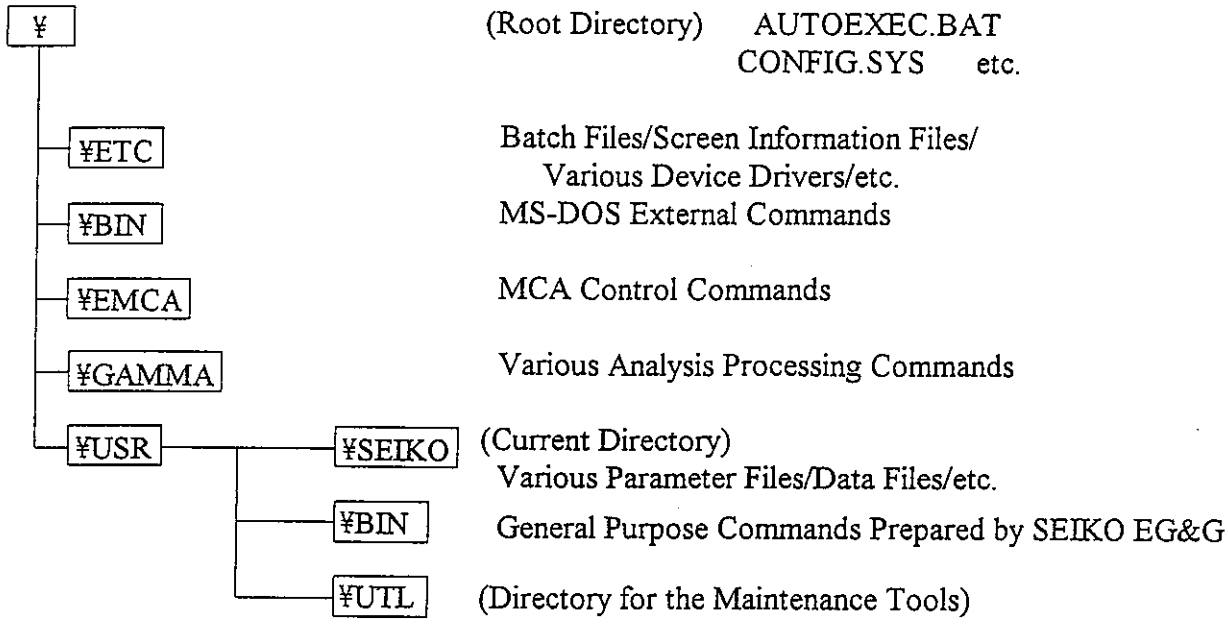
List Measurement Log all entries 95/01/11 11:15
Directory is C:\usr\seiko

Type	Sample ID	KED U (g/l)	XRF ratio	Pu-conc. (g/l)	Quality	Cycle	Date
A	MK1004	227.53	110.65	2.0564	Passed	Ave.	95/01/01
A	MK1004	227.53	110.65	2.0564		2/2	95/01/01
A	MK1004	227.53	110.65	2.0654		1/2	95/02/01
MC-B	Refer.	200.00	100.00	2.000	Passed	1/1	94/12/24
MC-P	Refer.	200.00	100.00	2.000	Passed	Ave	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	5/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	4/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	3/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	2/5	94/12/10
MC-P	Refer.	200.00	100.00	2.000	Passed	1/5	94/12/10

Press F/1 key to quit. (to printout) Use arrow keys to move the screen.

Appendix A

○ The directory structure of this Program is shown below:



*Root Directory: This is the root directory in the drive.
Current Directory: This is the directory this Program is currently executing.

○ File Selection by the Wild Card

When you do not recall the exact name of the file, file selection by the wild cards (*, ?) is available. If a wild card is specified, all the files satisfying the specification will appear in the sub window as shown below.

The example given below is for selection of a file in the directory ¥SPECT.

<Sub Window>

B: ¥SPECT¥*. *			1/1
<..>	DAT001	DAT002	DAT003
DAT004	DAT005	DAT006	DAT007
DAT008	DAT009	DAT010	DAT011
DAT012	DAT013	DAT014	DAT015

All the files satisfying the specification and the cursor to enter the target file are shown in the sub window.

<File Selection Method>

(1)Position the entry cursor on the target file name by the mouse or the arrow keys.

(2)Then, click the right button of the mouse or press the return key, and the target file is selected.

*When there are too many files satisfying the specification for a sub window, the files will appear in several sub windows. The page number of the sub window that is currently displayed is shown in the right upper corner of the screen (like 1/2, or 1/5).

The page number can be switched by the function keys, F/8 (for previous screen) or F/9 (for next screen).

If the symbol < .. > in the sub window is selected, the one grade upper directory will be searched. The root directories can also be searched by selecting the root name like < A: > or < C: >.

○ The data file structure used in this Program is shown below:
Data File Name : MSA.PRA
Location of Data File : ¥USR¥SEIKO
Purpose of Use : These data files store the parameter information to be used when "K_EDGE.EXE / X_RF.EXE" are in operation.
File Type : ASCII, Record Delimiter <CR><LF>

Symbol	Designation Limit	Description
OPERAT	20 characters	Operator name
SMPID	6 characters	Sample ID
NSB	20 characters	Inspector's name (NSB)
IAEA	20 characters	Inspector's name (IAEA)
REMK1	40 characters	Comment 1 for the analysis
REMK2	40 characters	Comment 2 for the analysis
KFIL	30 characters (including the path name)	Registration name of the K-edge spectra files (.CHN)
XFIL	30 characters (including the path name)	Registration name of the XRF spectra files (.CHN)
#CENT	30 characters (including the path name)	K-edge center calculation region file name (.ROI)
#SMFL	Y/N	Whether or not to designate the 5 point smoothing
#SMCN	1 - 99	Number of times of smoothing
#EDSG	1.0 - 50.0	K-edge virtual peak search sensitivity (no change allowed in the parameter entry screen).
#PBSG	1.0 - 10.0	Pb-Kα1 peak search sensitivity (no change allowed in the parameter entry screen).
#PCNT	70.0 - 80.0	Pb-Kα1 centroid energy (keV)
#KCNT	100.0 - 120.0	K-edge center energy (keV)
#LSTA	80.0 - 150.0	C(El) energy start (keV)
#LEND	80.0 - 150.0	C(El) energy end (keV)
#USTA	80.0 - 150 0	C(Eu) energy start (keV)
#UEND	80.0 - 150.0	C(Eu) energy end (keV)
#BLSTA	0.0-999.99	Start of lower energy region end with BG adjustment (keV)
#BLEND	0.0-999.99	End of lower energy region end with BG adjustment (keV)
#BHSTA	0.0-999.99	Start of higher energy region end with BG adjustment (keV)
#BHEND	0.0-999.99	End of higher energy region end with BG adjustment (keV)
#IMAT	1.0E-5 - 1.0E+2	Mass absorption coefficient (cm2/mg) for non-extrapolate
#OMAT	1.0E-5 - 1.0E+2	Mass absorption coefficient (cm2/mg) for extrapolate
#CLLN	1.0 - 1000.0	Cell length (mm)

*The mark # should be change to "U" or "P". U: KED(U), P: KED (Pu).

Symbol	Designation Limit	Description
URLT	30 characters (including the path name)	K-edge (U) analysis result file name (.KEU)
PRLT	30 characters (including the path name)	K-edge (Pu) analysis result file name (.KEP)
XRLT	30 characters (including the path name)	XRF analysis result file name (.XRF)
XANL	30 characters (including the path name)	Name of the files for XRF peak analysis region (.ROI)
XENE	30 characters (including the path name)	Name of the files of the energy calibration for XRF (.ENE)
XBLSTA	0.0-999.99	Start of lower energy (keV) region end with BG adjustment
XBLEND	0.0-999.99	End of lower energy (keV) region end with BG adjustment
XBMSTA	0.0-999.99	Start of middle energy (keV) region end with BG adjustment
XBMEND	0.0-999.99	End of middle energy (keV) region end with BG adjustment
XBHSTA	0.0-999.99	Start of higher energy (keV) region end with BG adjustment
XBHEND	0.0-999.99	End of higher energy (keV) region end with BG adjustment
SIGM	1 - 3	XRF peak search sensitivity
KVWT	1.0 - 50.0	Peak identification width (keV)
UKEV	90.0 - 120.0	U-Kα1 centroid energy (keV)
PKEV	90.0 - 120.0	Pu-Kα1 centroid energy (keV)
UKEV2	90.0 - 120.0	U-Kα2 centroid energy (keV)
UKEVB	90.0 - 120.0	U-Kβ1 centroid energy (keV)
UPKWDL	1.0 - 2.0	U-Kα1 peak region factor, lower end
UPKWDH	1.0 - 2.0	U-Kα1 peak region factor, higher end
PPKWDL	1.0 - 2.0	Pu-Kα1 peak region factor, lower end
PPKWDH	1.0 - 2.0	Pu-Kα1 peak region factor, higher end
UMAT	1.0E-5 - 1.0E+2	U mass value
PMAT	1.0E-5 - 1.0E+2	Pu mass value
FACTOR	1.0E-7 - 1.0E+7	Efficiency of all the measured figures
RFCT0	-1.0E-5 - 1.0E+5	Constant a for R(U/Pu)
RFCT1	1.0E-5 - 1.0E+2	Constant b for R(U/Pu)
MBUREF	1.0E-0 - 1.0E+3	U reference value for MC-Bias
MBPREF	1.0E-0 - 1.0E+3	U/Pu reference value for MC-Bias
MBUWNG	0.001 - 9.999	U Warning limit for MC-Bias
MBUACT	0.001 - 9.999	U Action limit for MC-Bias
MBPWNG	0.001 - 9.999	U/Pu warning limit for MC-Bias
MBPACT	0.001 - 9.999	U/Pu Action limit for MC-Bias

*The mark # should be change to "U" or "P". U: KED(U), P: KED (Pu).

Symbol	Designation Limit	Description
WIDTH	5 - 50 Energy calibration parameter	(Region for obtaining area) *1
SENSI	1 - 3 Energy calibration parameter	Sensitivity σ against base area *1
RENZO	Energy calibration parameter	Continuous number of significance recognition *1
BUBBLE	Y/N	Whether or not to have completed a bubble test
LIMIT1	1.0 - 5.0	The first limit for the bubble test
LIMIT2	1.0 - 5.0	The second limit of the bubble test
MPLNUM	5,6,7,8,9,10,15	Number of MC-Precision loops
ATMMD	LT/RT	Assay measurement time mode *2
BTMMD	LT/RT	MC-Bias measurement time mode *2
PTMMD	LT/RT	MC-Precision measurement time mode *2
APRTM	1 - 999999	Assay measurement time (sec.)
BPRTM	1 - 999999	MC-Bias measurement time (sec.)
PPRTM	1 - 999999	MC-Precision measurement time (sec.)
CPYSRC	30 characters (including the drive name)	Directory which the spectra file is copied from
CPYDST	30 characters (including the drive name)	Directory which the spectra file is written in
DRIVE1	30 characters (including the path name)	Drive and directory to store the spectra / analysis files
DRIVE2	30 characters (including the path name)	Drive and directory to store the spectra / analysis files (backup 1)
DRIVE3	30 characters (including the path name)	Drive and directory to store the spectra / analysis files (backup 2)
KCOM	60 characters	K-edge spectra comment *1
XCOM	60 characters	XRF spectra comment *1
AUTNUM	1 - 15	Number of Autocycle measurement times
WCONST	1.0 - 10.0	XRF peak search filter width *1
MENMOD	S/F	To distinguish the Short Menu and the Full Menu (set by a batch file)
LOOPNUM	1 - 15	Work variant at the time of repetitive measurement
BBLPNUM	3	Maximum number of times of the bubble test (fixed)
FW_ROI	1 - 10	FWHM calculation width (±ch) *1
CSIZE	8288	Spectra file size (fixed)
KSIZE	846	K-edge analysis result file size (fixed)
XSIZE	1178	XRF analysis result file size (fixed)

*1 No change permitted in the parameter entry screen

*2 LT: live time RT: real time

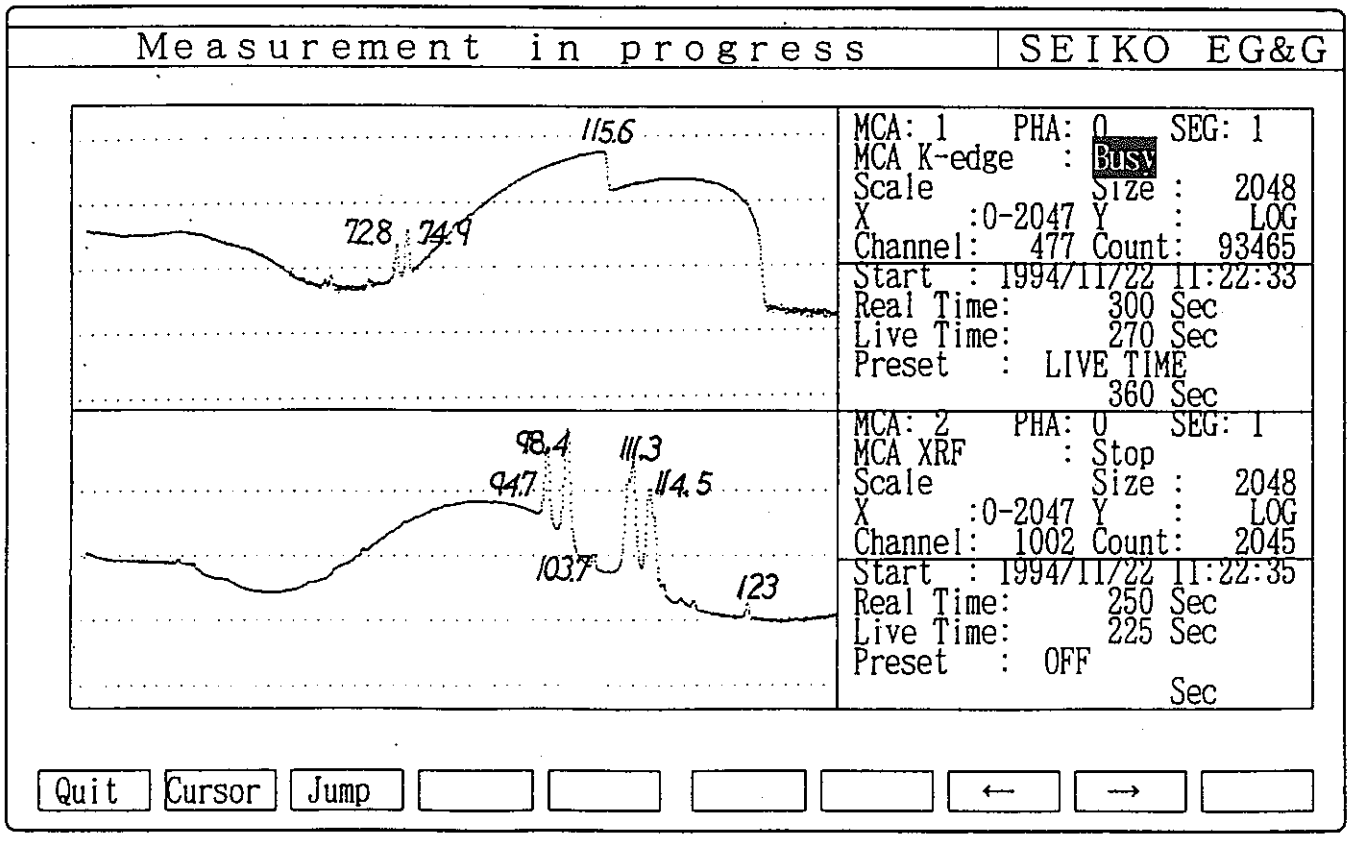
Data File Name : ########.ENE
Purpose of Use : Energy data file used when "X_RF.EXE" is in operation
File Type : ASCII, Record Delimiter <CR><LF>

Symbol	Designation Limit	Description
SPECTRUM	30 characters (including the path name)	Spectra file for energy calibration
CLD-DAATE	YY/MM/DD hh:mm:ss	Date of calibration
FT-ORDER	1/2	Fitting order
ANS-2	12.5E	Secondary coefficient
ANS-2	12.5E	Primary coefficient
ANS-0	12.5E	Nil order coefficient

APPENDIX B

·SPECTRAL DISPLAY

The MCA spectra display can be seen in KED/XRF measurements in progress, without exit the HYBRID KED/XRF program. The spectra from X-ray which passed through sample solution are shown below. K-edge of uranium can be seen at 115.6 keV in the KED spectrum. Please note that lead shields can fluoresce at 72.8 and 74.9 keV in the spectrum. While sample solution can fluoresce at 94.7keV (U-Ka2), 98.4 keV (U-Ka1), 103.7 keV (Pu-Ka1), 111.3 keV(U-K β 1,3), 114.5 keV(U-K β 2,4), in the XRF spectrum. Also passive γ -ray of Eu can be seen at 123 keV.



·TROUBLE SHOOTING

MALTICHANNEL ANALYZER

The MCA board is set in a computer. This system assumes the factory default. If the computer has problem finding the MCA, the software error will occur showing "PROGRAM ERROR" in the screen.
If the communications between the MCA and computer have a problem, reset the computer by turning off the power.

COMPUTER

The computer sometimes locks up. When the computer is turned on and locks up, press the switch to turn the power off. Then turn the power on and wait for the operating system to get started.

If the number of stored files in a diskette exceeds 192, the message "Program Error" will appear and any measurements do not start.

·DOS COMMANDS

To leave the program and enter the DOS environment, use MS-DOS Command option. To return the program, type "exit "or "autoexec".

To format a new diskette in drive B, insert a new high-density diskette into drive and type "format B:". The formatting process is required for most new diskettes and will destroy all previous information on the diskette.

To copy from one high-density diskette to another, type "diskcopy B: C:" and follow the instructions. This will destroy all previous information on the target diskette.

CALIBRATION

INTRODUCTION

It might seen desirable to have calibration solutions spanning the measurement range of the instrument. However, it is generally impractical to do so. Below about 100 gU/L and 0.5 gPu/L for KED/XRF measurement, the statistical precision becomes poor enough to require excessively long running times to obtain a meaningful calibration precision (0.1% RSD). Above 400 gU/L there can be problems with keeping the uranium in solution. The user should also note that extensive evaluations exist in the literature; not every facet of the instrument's performance requires frequent repetition.

The physics of the measurement dose not require the calibration materials to span the range of the instrument for every calibration attempt. Indeed, a single point dose provide a good calibration if the concentration of the standard is known accurately. It is recommended that a minimum of two solutions of different concentration be used to calibrate. This can provide a quality check on the accuracy of the concentration claimed for the standards.

A better calibration can often be obtained with more measurement and more standards.

DATA ACQUISITION

Measurement count time is needed at least 800 s live time for each measured sample. The concentration of calibration samples is around 200 gU/L, 1.5 gPu/L which is the typical input solution produced in TRP. It is recommended that the standard solutions are prepared more than 10 batches. Each sample should be measured by IDMS at a time. The results provide standard value for the KED/XRF. These results should be independent of sample, concentration, or operator.

After acquiring data from each calibration solution standard, calculate the ratio of KED/IDMS (uranium) and XRF/IDMS (uranium/plutonium) for the calibration constant. The KED and XRF factor can be computed by the average of ratios and changed in the parameters option "Edit parameters".

Plotting the data can illustrate if there are correlations or trends. The plot should include each result, not average results. Outliers indicate an incorrect standard value for the claimed concentration or a transcription error.

SAFETY EVALUATION

The part of measurement system is completely covered with lead shield to avoid leaking X-ray and γ -ray which can cause damage to health. The rate of X-ray passing through the shield at a distance of 30 cm from the surface of shield door is not significant. The evaluation value is around 4 μ Sv/h as same as that of back ground level at the room.

DOS/APPLICATION SOFTWARE INSTALLATION PROCEDURE

1. FORMAT THE HARD-DISK

1) With the computer turned off, set the floppy-disk of "SYSTEM#1" into Drive 1.
2) Turn on the computer. Screen shows "A>"
3) Type prompt "A>FORMAT", then press RETURN.
4) Highlight the lower right line(on screen#1),then press RETURN.

* Screen#1

```
(Command)
FORMATコマンド Ver. 5. 30 Copyright (C) NEC Corporation 1983,1993

装 置 名(Device)          フロッピディスク(Flopy disk)

フォーマットする装置名を指定してください(Select the device for format)
（ESCキーを押すと処理を中止することができます）
((Press "ESC"key to quite))

フロッピディスク(Flopy disk)      固定ディスク(Hard disk)
```

5) Highlight the sixth line from top (on screen#2),then press RETURN.

*Screen #2

```
(Command)
FORMATコマンド Ver. 5. 30 Copyright (C) NEC Corporation 1983,1993

装置番号(Disk No.)  1
フォーマット形式   拡張フォーマット              接続状況
(Type of format)   (Extention format)           (Information)
マップ     (Map)                                1：固定ディスク    #1
領域確保   (Area securing)                          (Hard disk)
領域解放   (Area emancipation)
初期化     (Initializing)
状態変更   (Condition change)
終了       (Quit)

矢印キーで項目を選択し、リターンキーを押してください(Select the above item you
want using [↑] [↓] keys,pressRETURN)
（ESCキーを押すと処理を中止することができます）
((Press "ESC" key to quite))
```

6) Highlight the lowest left line(on screen#3). then press RETURN.
Initializing starts (the disk indicator turns on).

Wait for about 7 minutes.

Screen#3

```
                    (Command)
ＦＯＲＭＡＴコマンド　Ｖｅｒ．５．３０ Copyright (C) NEC Corporation 1983,1993

        装置番号(Disk No.)  1
        フォーマット形式    拡張フォーマット
         (Type of format)    (Extention format)
    システム名      状態      FROM      TO(ｼﾘﾝﾀﾞ)    ｻｲｽﾞ      BOOT
     (System)     (Condition)                        (Size)
  MS-DOS 5.00     アクティブ 00001  ～  0387         0257       可
                   (Active)                                    (Good)

 装置全体を初期化します。よろしいですか (Are you sure that hard disk is formatted ?)
  （ＥＳＣキーを押すと処理を中止し、前画面に戻ることができます）
 ((Press "ESC" key to quite))

    [はい]      いいえ
    (Yes)       (No)
```

7) When initializing ends (the disk indicator turns off),then press any key.

8) Highlight the 4th line from top (on screen#4),then press RETURN.
*Screen#4

```
                    (Command)
ＦＯＲＭＡＴコマンド　Ｖｅｒ．５．３０ Copyright (C) NEC Corporation 1983,1993

    装置番号(Disk No.)  1
    フォーマット形式    拡張フォーマット
    (Type of format)    (Extention format)
    マップ      (Map)                    ---------- 接続状況 ----------
   [領域確保]   (Area securing)                    (Information)
    領域解放    (Area emancipation)       １：固定ディスク      #1
    初期化      (Initializing)                     (Hard disk)
    状態変更    (Condition change)       ------------------------------
    終了        (Quit)

 矢印キーで項目を選択し、リターンキーを押してください(Select the above item you
 want using[↑] [↓] keys,pressRETURN)
  （ＥＳＣキーを押すと処理を中止することができます）
 ((Press "ESC" key to quite))
```

9) Highlight the 7th line from top (on screen#5),wait until disk indicator turns off.

*Screen#5

```
                (Command)
ＦＯＲＭＡＴコマンド　Ｖｅｒ．５．３０ Copyright (C) NEC Corporation 1983,1993

    装置番号(Disk No.)          1
    フォーマット形式            拡張フォーマット
    (Type of format)            (Extention format)
    確保容量(Memory)            0257 MB
    先頭シリンダ(First cylinder)00001
    システム(System)    転送する(Forwarding)
    ボリュームラベル(Volume label)
    実行(Execute)


確保する領域は何メガバイトですか　HELPキーを押すとマップを表示します
確保可能な容量は1～128MBです。
 (How much MB do you want ?)
  (ESCキーを押すと処理を中止し、前画面に戻ることができます。)
        Press ESC key to interupt.
確保領域=
```

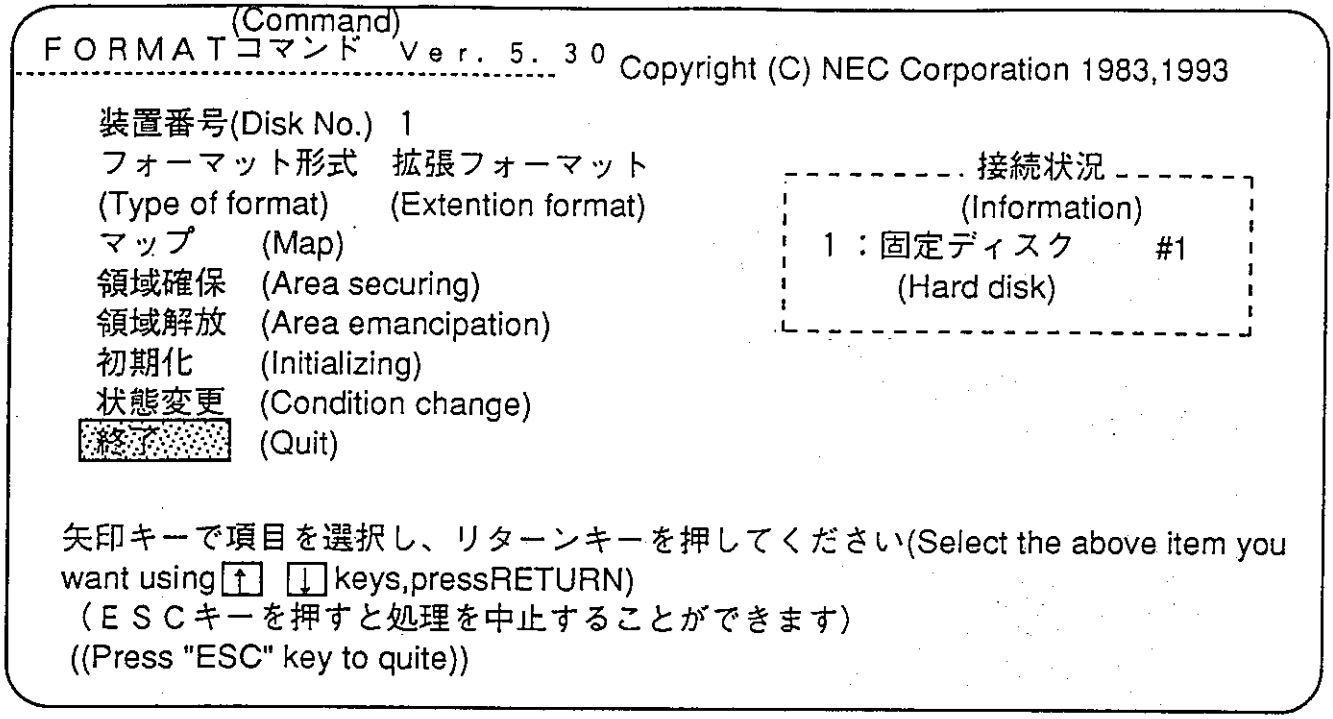
10) Highlight the lowest left line(on screen#6). then press RETURN. Area securing carries out.

*Screen#6

```
                (Command)
ＦＯＲＭＡＴコマンド　Ｖｅｒ．５．３０ Copyright (C) NEC Corporation 1983,1993

    装置番号(Disk No.)          1
    フォーマット形式            拡張フォーマット
    (Type of format)            (Extention format)
    確保容量(Memory)            0257 MB
    先頭シリンダ(First cylinder)00001
    システム(System)    転送する(Forwarding)
    ボリュームラベル(Volume label)
    実行　(Execute)

領域の確保を行います。準備はよろしいですか(Area securing will be carried out.
Are you sure ?)
 （はい：確保する　いいえ：確保しない）
 （ＥＳＣキーを押すと処理を中止し、前画面に戻ることができます）
 ((Press "ESC" key to quite))
 はい　　いいえ
 (Yes)     (N o)
```

11) Highlight the 8th line(on screen#7),then press RETURN.

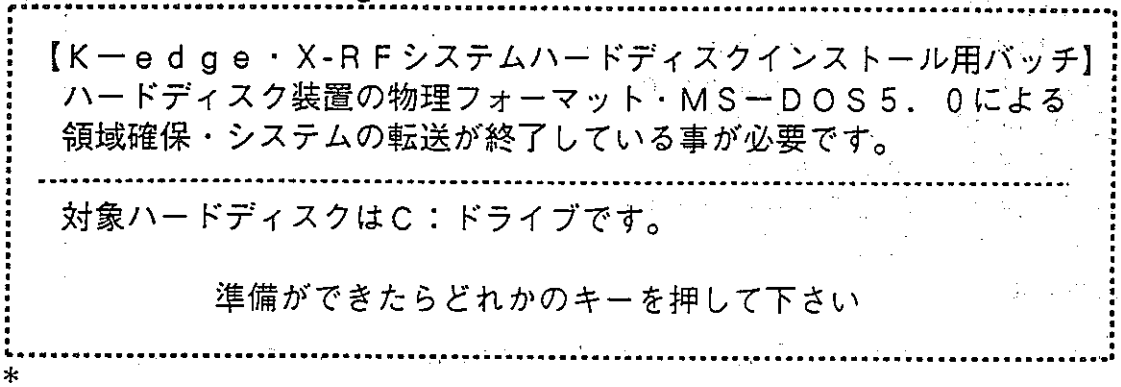
* Screen#7



End of Hard disk Initialization

2. INSTALL THE PROGRAM

1) Reset the computer seting the floppy-disk of "SYSTEM#1" into Drive 1.
2) Type on prompt A>ETC¥MKHD,press RETURN.
3) Screen shows message



*

Hard disk instllation Batch for K-edge/X-RF system

Format of hard disk , areasecuring and system forwarding are necessary before installation of hard disk.

Hard disk : Drive [c:]

Press any key to continue

Press any key

4) Screen shows message

ハードディスクのセットアップを行います。
ＳＹＳＴＥＭ＃１と刻印されたラベルのフロッピーがＡドライブに入っていることを確認して下さい。

ＳＹＳＴＥＭ＃１と刻印されたラベルのフロッピーがＡドライブに入っていない場合は、ＳＹＳＴＥＭ＃１と刻印されたラベルのフロッピーを上段のドライブに入れてリセットキーを押して下さい。
ＭＳ－ＤＯＳが立ち上がったらＳＴＯＰキーを押して下さい。

準備ができたらどれかのキーを押して下さい

*

Set up the Hard disk.
Road the "SYSTEM#1" disk sealed by label into Drive A.

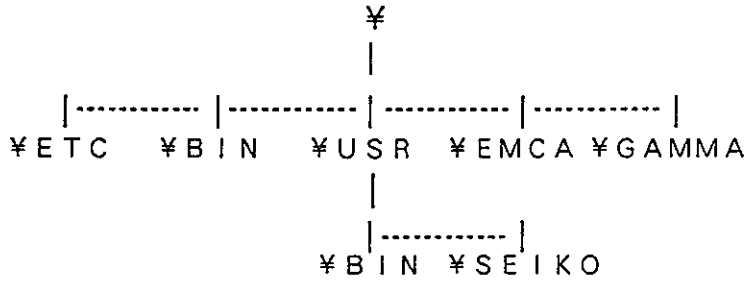
If "SYSTEM#1" disk don't set to Drive A , Press Reset key after putting disk into the upper Drive.
Press "STOP" key when MS-DOS starts.

Press any key to continue.

Press any key.

5) Screen shows message

Ｋ－ｅｄｇｅ・Ｘ－ＲＦシステムのディレクトリ構造
(Directory structure of K-edge/X-RF system)



準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Press any key to continue.

6) Screen shows message

Ａドライブから『ＳＹＳＴＥＭ＃１』をコピーします。
("SYSTEM#1 will be copied from "Drive A")
準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Press any key

7) Screen shows message

Ｂ：ドライブに『ＳＹＳＴＥＭ＃２』のメディアをいれてください。
(Road "SYSTEM#2" disk into "Drive B")
準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Insert SYSTEM#2 diskette in drive 2.
Press any key

8) Screen shows message

Ｂ：ドライブに『ＳＹＳＴＥＭ＃３』のメディアをいれてください。
(Road "SYSTEM#3" disk into "Drive B")
準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Take the SYSTEM#2 diskette out and Insert SYSTEM#3 diskette in drive 2.
Press any key

9) Screen shows message

B：ドライブに『ＳＹＳＴＥＭ＃4』のメディアをいれてください。
(Road "SYSTEM#4" disk into "Drive B")
準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Take the SYSTEM#3 diskette out and Insert "SYSTEM#4" disk into Drive 2.
Press any key

10) Screen shows message

B：ドライブに『ＳＹＳＴＥＭ＃5』のメディアをいれてください。
(Road "SYSTEM#5" disk into "Drive B")
準備ができたらどれかのキーを押して下さい
(Press any key to continue)

Take the SYSTEM#4 diskette out and Insert "SYSTEM#5" disk into Drive 2.
Press any key

11) Screen shows end message

ハードディスクへのインストールは全て終了しました

A：ドライブからフロッピィを抜き、一度リセットを押して
プログラムを立ち上げて下さい。

現在のディレクトリは　A：のルート　です。
◇ ◇ ◇　お疲れさまでした　◇ ◇ ◇

*

Installation has been finished.

Remove the flopy disk from Drive A and reset to run the program.

Present directory is "A".

Thanks for your efforts

Reset the power switch to run the program after removing all the floppy disks from Drive 1 and 2.

End of installation. HKEDG DOS/APPLICATION SOFTWARE can start.

Note:

The Personal Computer (PC-9801) is not compatible with IBM-PC. Please note following items.

1) Drive name

Boot drive is assigned as A drive.

If you boot-up the PC from internal hard disk, drive name will be assigned as follows.

A drive: Internal hard disk
B drive: Front upper floppy drive
C drive: Front lower floppy drive

If you boot-up the PC from front upper floppy drive, then

A drive: Front upper floppy drive
B drive: Front lower floppy drive
C drive: Internal hard disk

2) Path delimiter

Use the mark "¥" as path delimiter instead of back slash "\".

Root directory of A drive is shown as "A: ¥".

3) Disk format

Default floppy disk format is 1.2M Byte format for 2HD disk. This is not accepted by IBM PC. However, PC-9800 can use IBM-PC 1.44M Byte formatted disk too . If you want to use the data obtained by NEC computer on IBM-PC, you have to use disks which formatted by IBM-PC.

4) Disk Operating system (DOS)

The PC-9801 uses Microsoft Disk Operating System (MS-DOS) version 5. Although this DOS is almost same with be used by IBM-PC, only few DOS command is contained in the software. Please note that all messages from command are shown in Japanese.

HYBRID KED/XRF OPERATION PROCEDURE

1. MC-Bias Measurement

1-0. Turn on the computer. The "FULL MENU" screen appears.

1: Measurement
2: Archives
3: Parameters
4: Analysis
5: Energy Calibration
6: MCA Emuration
7: MS-DOS Command

1-1. Highlight the "1: Measurement" option and press "RETURN" key. The "Measurement" screen appears.

1: KED/XRF Assay
2: KED U Assay
3: KED Pu Assay
4: MC Bias
5: MC Precision
6: Autocycle

1-2. Highlight the "4: MC-Bias" option and press "RETURN" key. The input data screen appears.

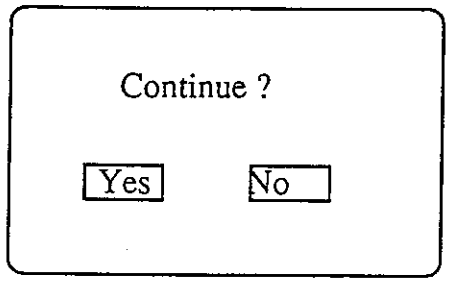
Operator:
Sample ID:
Inspector(NSB):
Inspector(IAEA):
Remarks 1:
Remarks 2:

Measurement Type	Number of Measurement	Count Time in Seconds	Present Mode
MC-Bias	1	1000	LIVE TIME

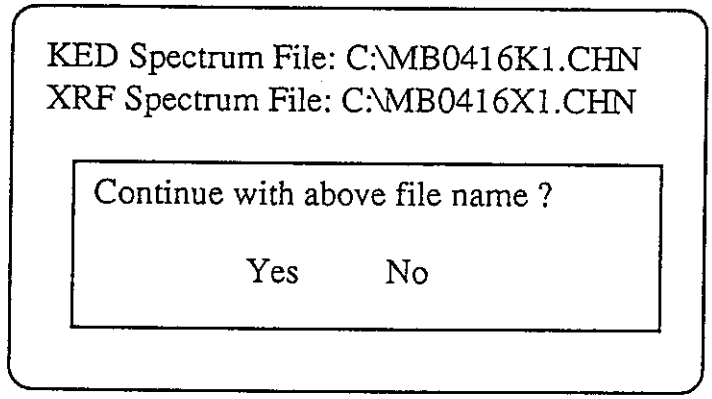
1-3. Highlight "Operator", press "RETURN" key, type operator name and press "RETURN" key.

1-4. Highlight "Sample ID", press "RETURN" key, type the reference sample ID and press "RETURN" key.

1-5. Highlight "Inspector(NSB)", press "RETURN" key, type JNSB inspector name, and press "RETURN" key.
1-6. Highlight "Inspector(IAEA)", press "RETURN" key, type IAEA inspector name, and press "RETURN" key.
1-7. Highlight "Remarks", press "RETURN" key, type temperature and other remarks, if any, and press "RETURN" key. (Checks the parameters of "Number of measurement" and "Count time in Seconds".)
1-8. Press the function key F10 (Cont.). The following screen appears. Insert a formatted 3.5 HD diskette into the drive (labeled 1 or B)



1-9. Press "Y". The following screen appears.



1-10. Move the reference samples ovserved on the measurement cell monitor to the measurement position.

1-11. Assure that the reference sample is in measurement position.

Press the "ON" button (black) on the Control unit of the X-ray system (MGC 30).

Wait until Voltage and current stabilize at 150kV and 15mA respectively.

Check that the blinking in the control unit panel is continuous.

1-12. Press "Y". The following screen appears.

Are you sure: Reference sample in position?
X-ray on?

[Y or N]

1-13. Press "Y". The measurement starts.

1-14. At the end measurement a printout is obtained. The following screen appears.

Please turn off the X-ray system
and press any key.

1-15. Press "OFF" button (red) on the Control unit of X-ray system, to stop the X-ray generation and press any key.

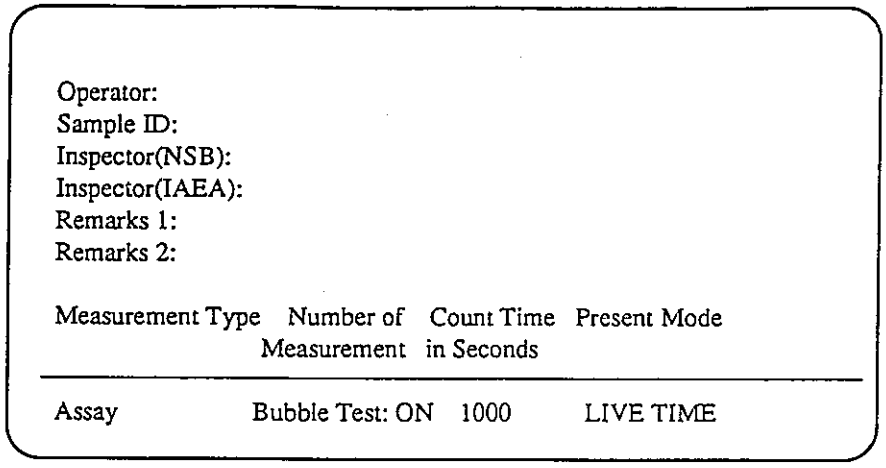
1-16. If the print out shows "MC-Bias Option Passed", proceed to the next stage.
If the print out shows "MC-Bias Option Failed", repeat measurement. If the second measurement results fails, DA sample should be prepared.

2. Assay Measurement

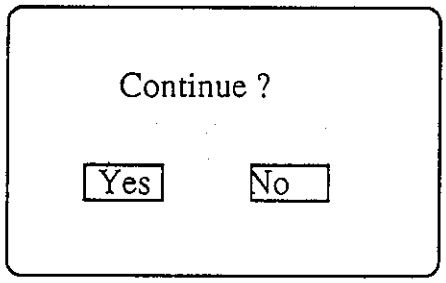
2-1. After sample rinsing and loading, Highlight the "1: KED/XRF Assay" option in the measurement menu and press "RETURN" key.

1: KED/XRF Assay
2: KED U Assay
3: KED Pu Assay
4: MC Bias
5: MC Precision
6: Autocycle

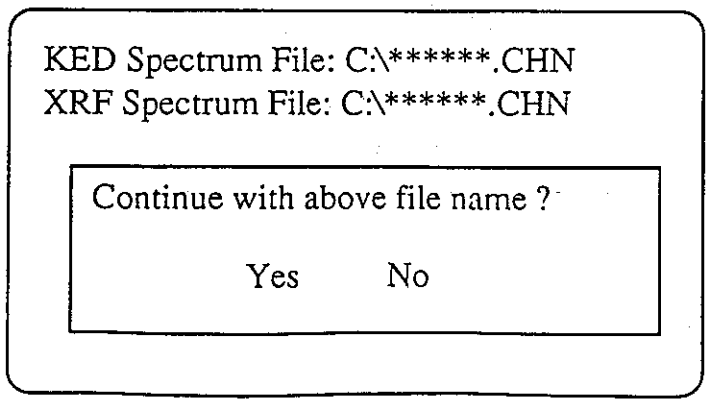
2-2. The input data screen appears.



2-3. Highlight "Operator", press "RETURN" key, type operator name and press "RETURN" key.
2-4. Highlight "Sample ID", press "RETURN" key, type the reference sample ID and press "RETURN" key.
2-5. Highlight "Inspector(NSB)", press "RETURN" key, type JNSB inspector name, and press "RETURN" key.
2-6. Highlight "Inspector(IAEA)", press "RETURN" key, type IAEA inspector name, and press "RETURN" key.
2-7. Highlight "Remarks", press "RETURN" key, type temperature and other remarks, if any, and press "RETURN" key. Checks the parameters of "Number of measurement" (Bubble test is on) and "Count time in Seconds" (1000).
2-8. Press the function key F10 (Cont.). The following screen appears.



2-9. Press "Y". The following screen appears.



2-10. Assure that the sample is in proper position.
Press the "ON" button (black) on the Control unit of the X-ray system.
Wait until Voltage and current stabilize at 150kV and 15mA respectively.
Check that the blinking in the control unit panel is continuous.

2-11. Press "Y". The following screen appears.

> Are you sure: Sample is loaded?
> X-ray on?
>
> [Y or N]

2-12. Press "Y". The measurement starts. (Two runs)

If the bubble test result shows "PASSED", proceed to next step. If the bubble test on the two runs shows "FAILED", perform third measurement. If the print out shows result passed, proceed to next step. If the print out shows result failed, DA preparation should be performed.

2-13. At the end measurement a printout is obtained. The following screen appears.

> Please turn off the X-ray system
> and press any key.

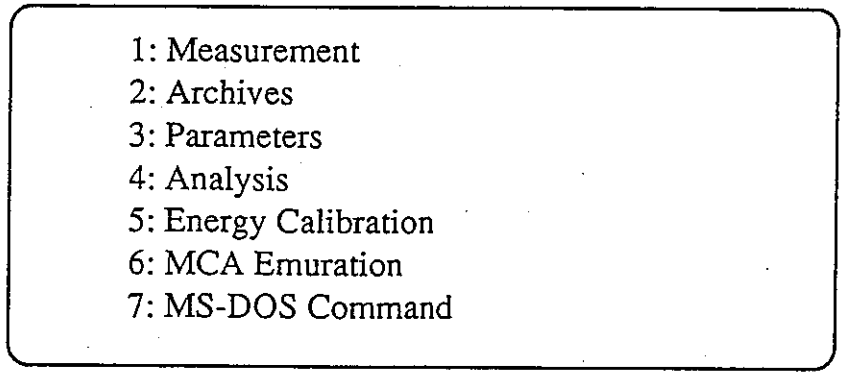
2-14. Press "OFF" button (red) on the Control unit of X-ray system, to stop the X-ray generation, and press any key.

END OF ASSAY

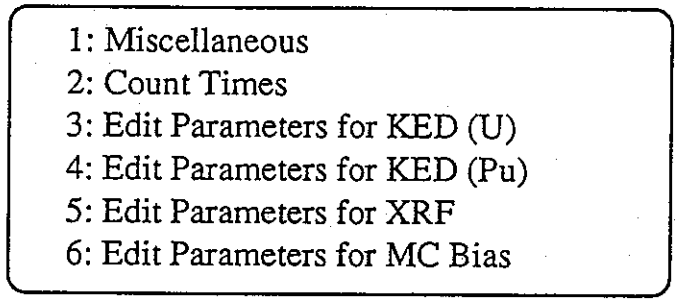
Edit Parameters for HYBRID KED/XRF

1. Miscellaneous

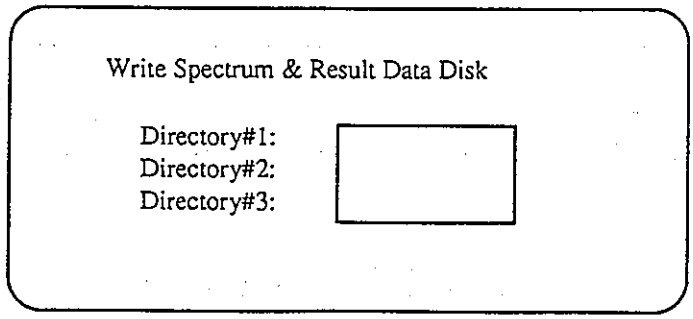
1-0. Turn on the computer. The "FULL MENU" screen appears.



1-1. Highlight the "3: Parameters" option and press "RETURN" key. The "Parameters Menu" screen appears.



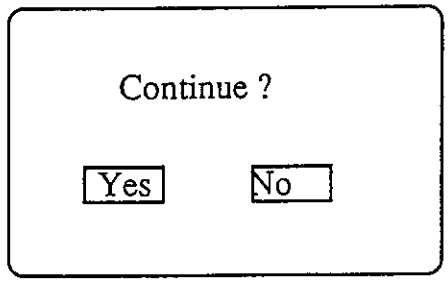
1-2. Highlight the "1: Miscellaneous" option and press "RETURN" key. The input data screen appears.



1-3. Highlight "Directory#1", press "RETURN" key, type directory name like "A:¥user¥IAEA" and press "RETURN" key.

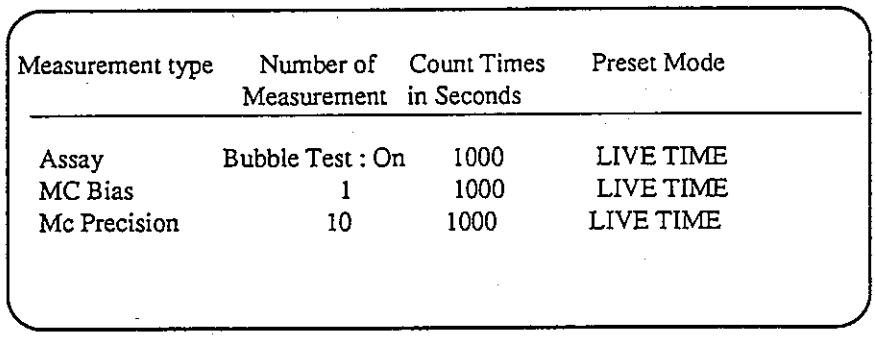
1-4. Highlight "Directory#2", press "RETURN" key, type directory name "B:" and press "RETURN" key.

1-5. Highlight "Directory#3", press "RETURN" key, type directory name "C:" and press "RETURN" key.
1-6. Press the function key F10 (Cont.). The following screen appears.



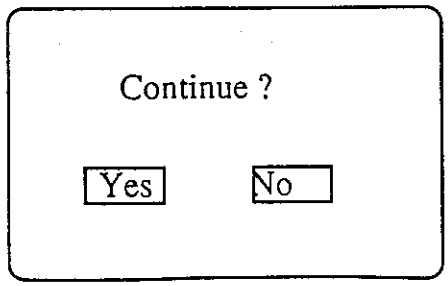
1-7. Press "Y". The Parameter Menu screen appears.

2. Count Times
2-1. Highlight the "2: Count Times" option and press "RETURN" key. The input data screen appears.



Measurement type	Number of Measurement	Count Times in Seconds	Preset Mode
Assay	Bubble Test : On	1000	LIVE TIME
MC Bias	1	1000	LIVE TIME
Mc Precision	10	1000	LIVE TIME

2-2. Highlight each Parameters in above screen, press "RETURN" key, put the appropriate parameter and press "RETURN" key, respectively.
2-3. When you finish editing, press the function key F10 (Cont.). The following screen appears.



2-4. Press "Y". The Parameter Menu screen appears.

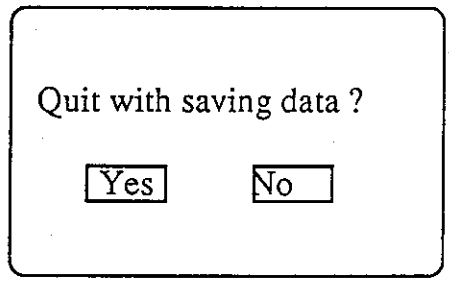
3. Edit Parameters for KED (U)

3-1. Highlight the "3: Edit parameters for KED (U)" option and press "RETURN" key. The input data screen appears.

ROI data file	: A:¥USR¥SEIKO¥KEDU.ROI
5point smoothing	: YES NO
No. of smothing	: 10
Pb-Ka1 energy (keV)	: 74.970
K-edge energy (keV)	: 115.6
C(EL) region (keV) start	:112.70-End: 118.30
C(EU) region (keV) start	:123.20-End: 124.60
BG Window Low (keV) start	: 61.80-End: 64.80
High(keV) start	:151.90-End: 158.40
K-edge factor	
Extrapolated(cm2/mg)	: 3.53170
Non-extrapo.(cm2/mg)	: 3.13802
Cell path length (mm)	: 25.0
First Limit : 3.18	
Second Limit : 1.95	

3-2. Highlight each Parameters in above screen, press "RETURN" key, put the appropriate parameter and press "RETURN" key, respectively.

3-3. When you finish editing, press the function key F10 (Cont.). The following screen appears.



3-4. Press "Y". The Parameter Menu screen appears.

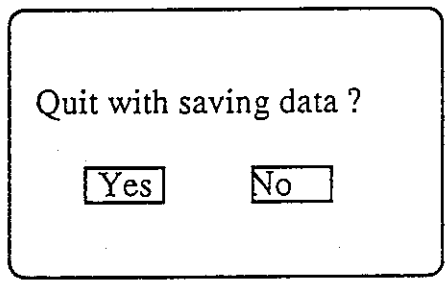
4. Edit Parameters for KED (Pu)

4-1. Highlight the "4: Edit parameters for KED (Pu)" option and press "RETURN" key. The input data screen appears.

ROI data file	: A:¥USR¥SEIKO¥KEDP.ROI
5point smoothing	: YES NO
No. of smothing	: 10
Pb-Ka1 energy (keV)	: 74.970
K-edge energy (keV)	: 121.3
C(EL) region (keV) start	:112.70-End: 118.30
C(EU) region (keV) start	:123.20-End: 124.60
BG Window Low (keV) start	: 61.80-End: 64.80
High(keV) start	:151.90-End: 158.40
K-edge factor	
Extrapolated(cm2/mg)	: 3.53170
Non-extrapo.(cm2/mg)	: 3.13802
Cell path length (mm)	: 25.0
First Limit : 3.18	
Second Limit : 1.95	

4-2. Highlight each Parameters in above screen, press "RETURN" key, put the appropriate parameter and press "RETURN" key, respectively.

4-3. When you finish editing, press the function key F10 (Cont.). The following screen appears.



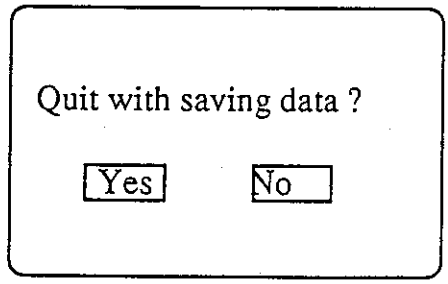
5. Edit Parameters for XRF

5-1. Highlight the "5: Edit parameters XRF" option and press "RETURN" key. The input data screen appears.

ROI data file	: A:YUSRYSEIKOYKED.ROI
XRF energy	: A:YUSRYSEIKOYXRF.ENE
Fuctors of R(U/Pu)	A: 1.0000E+004 B:-1.0000E+004
BG Windows Low (keV)start	:123.45-End:123.45
Middle(keV)start	:123.45-End:123.45
High(keV)start	:123.45-End:123.45
Sensitivity	:1
Region to Identify(keV)	:12.3
U-Ka1 energy(keV)	:123.456
Peak width Lower	:1.23*FWHM Upper:1.23*FWHM
Pu-Ka1 energy(keV)	:123.456
Peak width Lower	:1.23*FWHM Upper:1.23*FWHM
U-Ka2 energy(keV)	:123.45
U-KB1 energy(keV)	:123.45
Atomic weight	U:238.00 Pu: 239.60
Factor	:1.23456E+123
First Limit : 3.18	
Second Limit : 1.95	

5-2. Highlight each Parameters in above screen, press "RETURN" key, put the appropriate parameter and press "RETURN" key, respectively.

5-3. When you finish editing, press the function key F10 (Cont.). The following screen appears.



5-4. Press "Y". The Parameter Menu screen appears.

6. Edit parameters for MC-Bias

6-1. Highlight the "6: Edit parameters for MC-Bias" option and press "RETURN" key. The input data screen appears.

```
Reference Value
  U Conc.          : #.#####E+###
  U/Pu Ratio    :  #.#####E+###

U Conc.
  Warning Limit(%) : 2.000
  Action  Limit (%) : 5.000
U/Pu Ratio
  Warning Limit(%) : 2.000
  Action  Limit (%) : 5.000
```

6-2. Highlight each Parameters in above screen, press "RETURN" key, put the appropriate parameter and press "RETURN" key, respectively.

6-3. When you finish editing, press the function key F10 (Cont.). The following screen appears.

```
Quit with saving data ?

  Yes      No
```

6-4. Press "Y". The Parameter Menu screen appears. Press F1 key to quit and go back to the Full Menu screen.

HARDWARE INFORMATION

Tokai reprocessing plant
Power Reactor & Nuclear Fuel
Development Corporation

HARDWARE INFORMATION

The HYBRID KED/XRF Densitometer was installed by PNC in the TRP laboratory, in 1991-1994. The system was designed, based upon the information of the densitometer presented by Ottmar et al., KfK. Most of the mechanical and electric units used are domestic products, which enables easy maintenance.

MEASUREMENT CONDITION

The X-ray system requires filters to reduce the intensity of low energy X-rays. Filtering can be accomplished with 1-2cm iron (Fe) metal and 1-2mm cadmium (Cd) metal for the X-radiation.

REVIEW OF THE MECHANICAL

The mechanical section was installed in 1992 in the TRP laboratory (room G-104, No.1), as can be seen in Photo.1, that was compactly designed in the limited space. The drawing in Fig.1 represents a horizontal cross-sectional view of the mechanical set-up. A stainless steel cell for K-edge measurement (25 mm length) and a grass cell for XRF measurement (10 mm diameter) are set in the measurement cell system (Photo.2). Reference samples used for MC-Bias measurement are also mounted on a slide table which can remotely controlled by magnet. Sample solution is introduced into the cells, where the instrument is adapted, from the inner box in No.1 shielded cell. The CCD camera can observe the grass cell whether or not the solution is completely filled with. This flow type cell is easily washed with water or alcohol, and dried as shown in Fig.2. The sampling process is explained in detail in Fig.3. The manipulation mentioned above is performed by handy controller (Fig.4). The electronics module, the inspection cabinet including computer to manipulate this system and NIM module, and measurement cell monitor are located in the room G-105 (Photo.3).

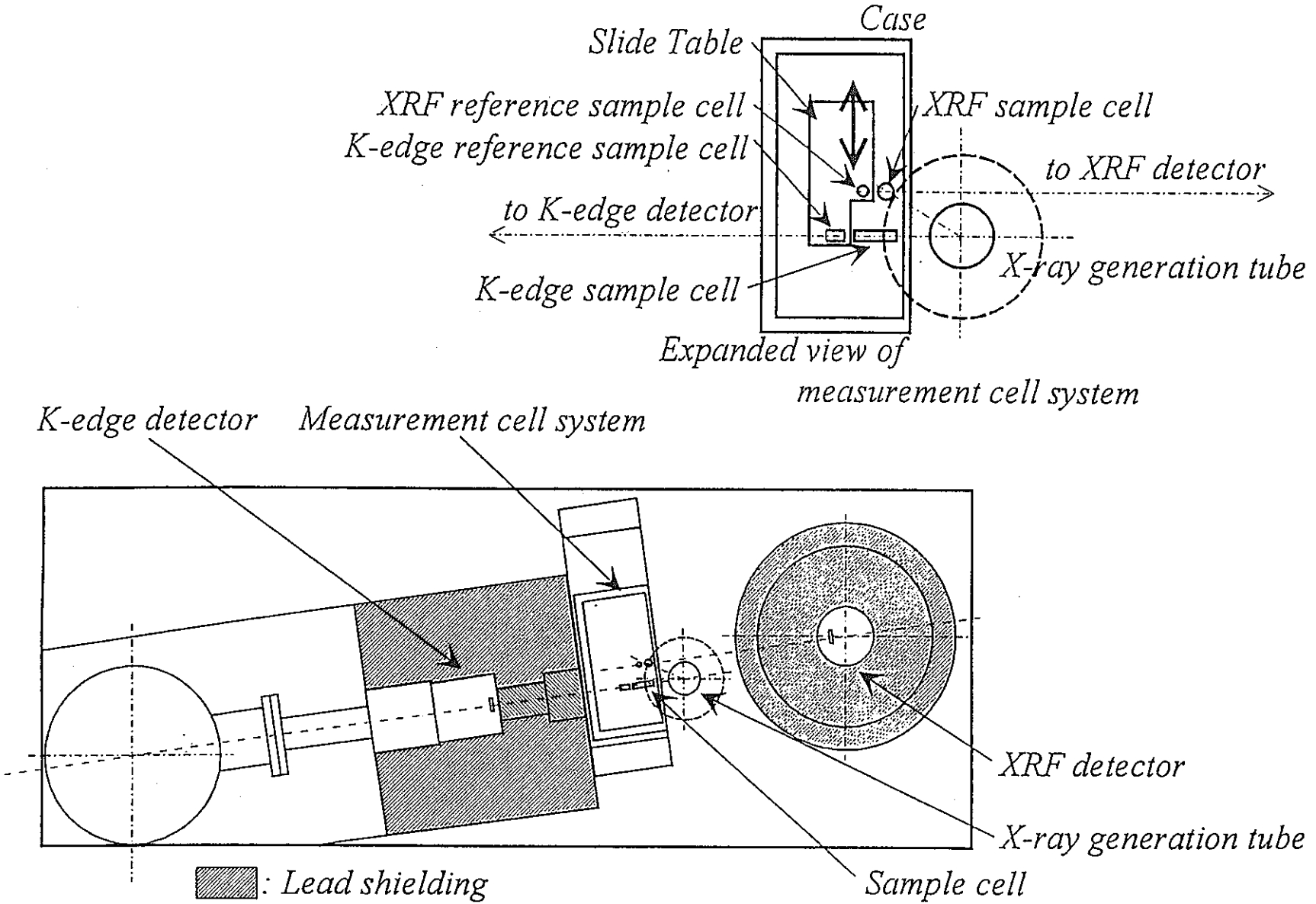


Fig. 1 Schematic diagram of instrument installed in shielded cell

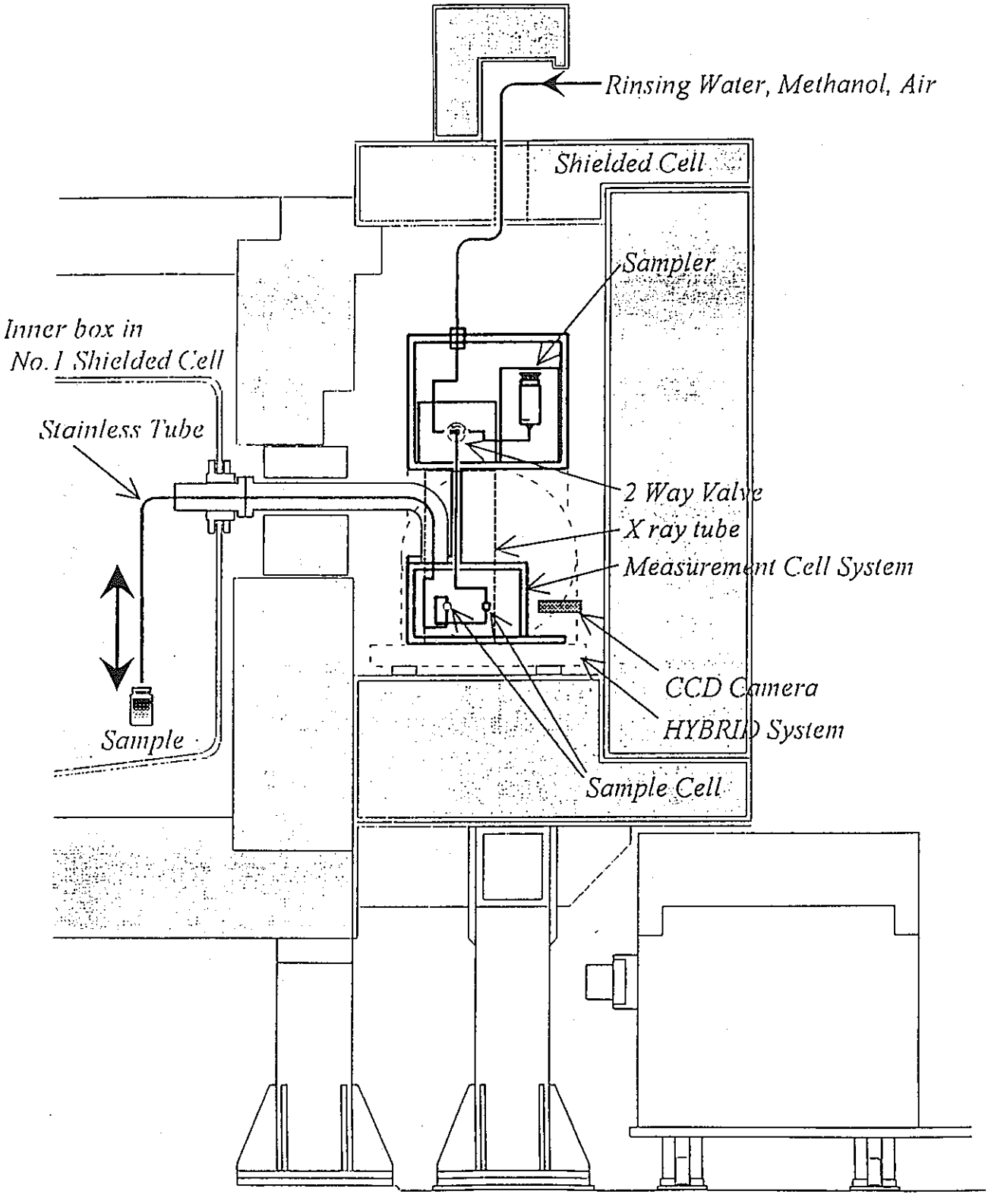


Fig. 2 Schematic diagram of measurement cell system in shielded cell

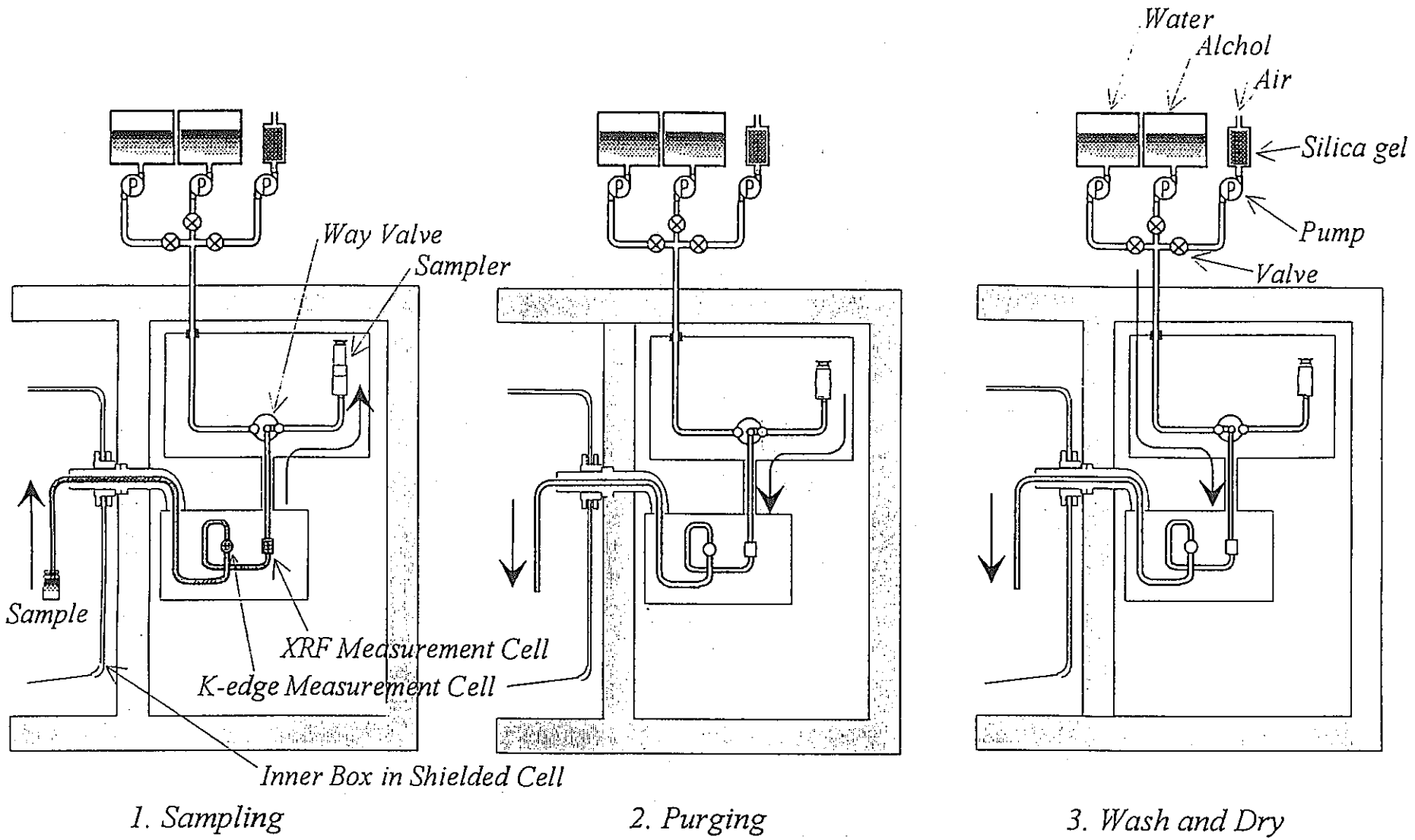


Fig. 3 Schematic digram of sampling process

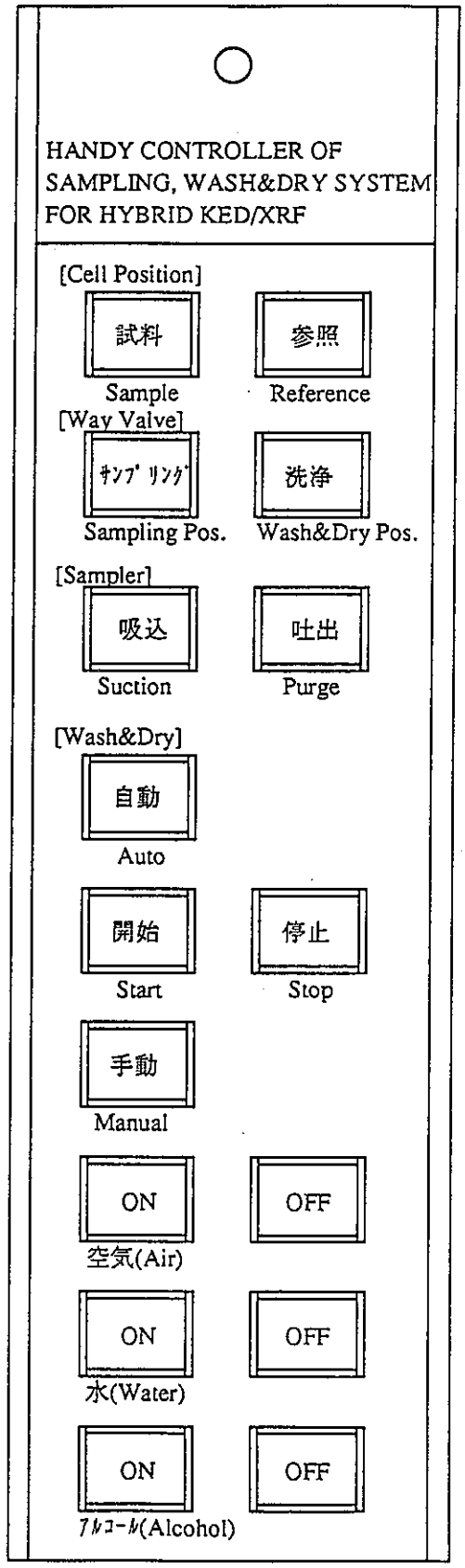


Fig.4 Controller

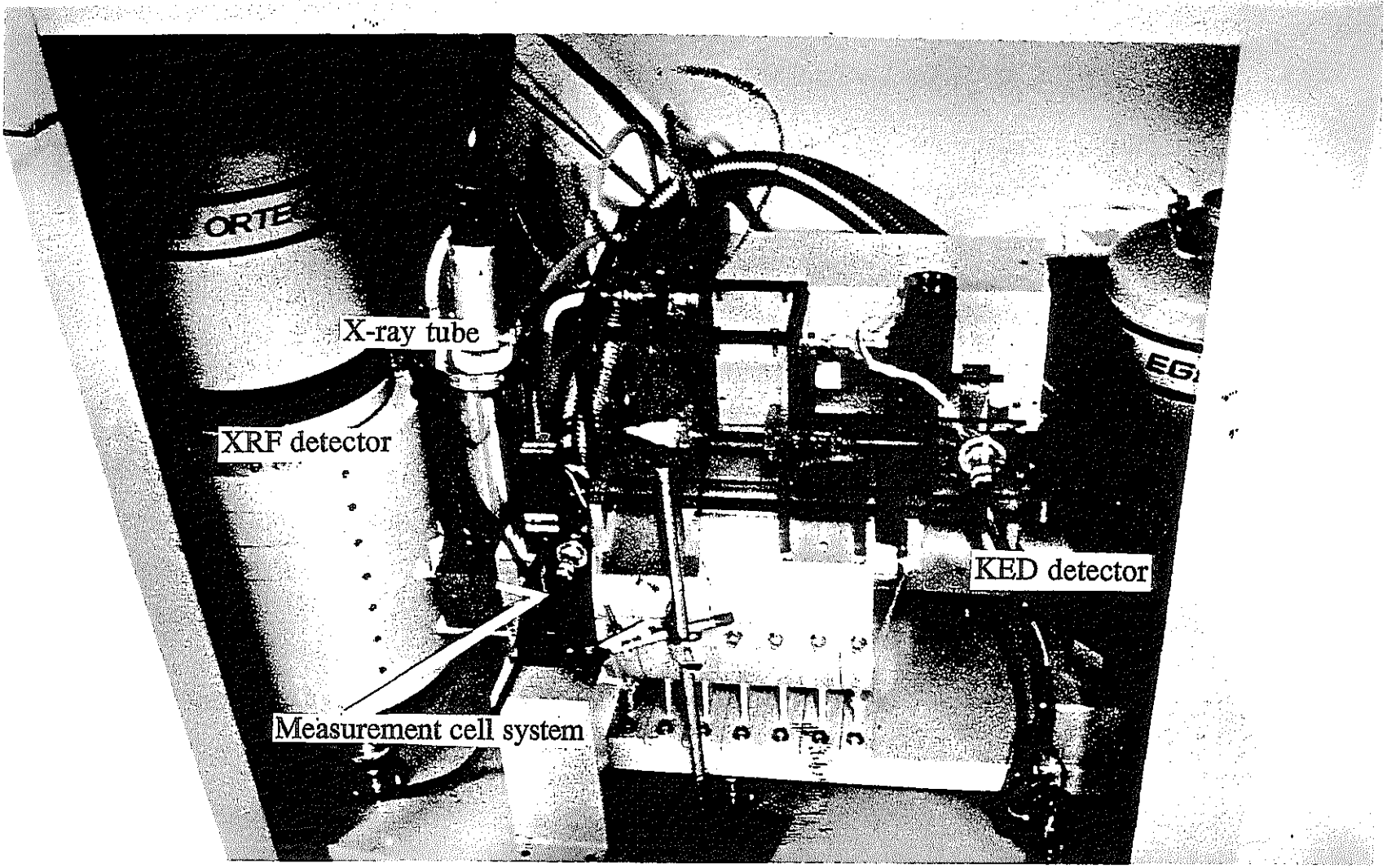


Photo.1 Mechanical set-up of the Hybrid KED/XRF instrument installed in the shielded cell.

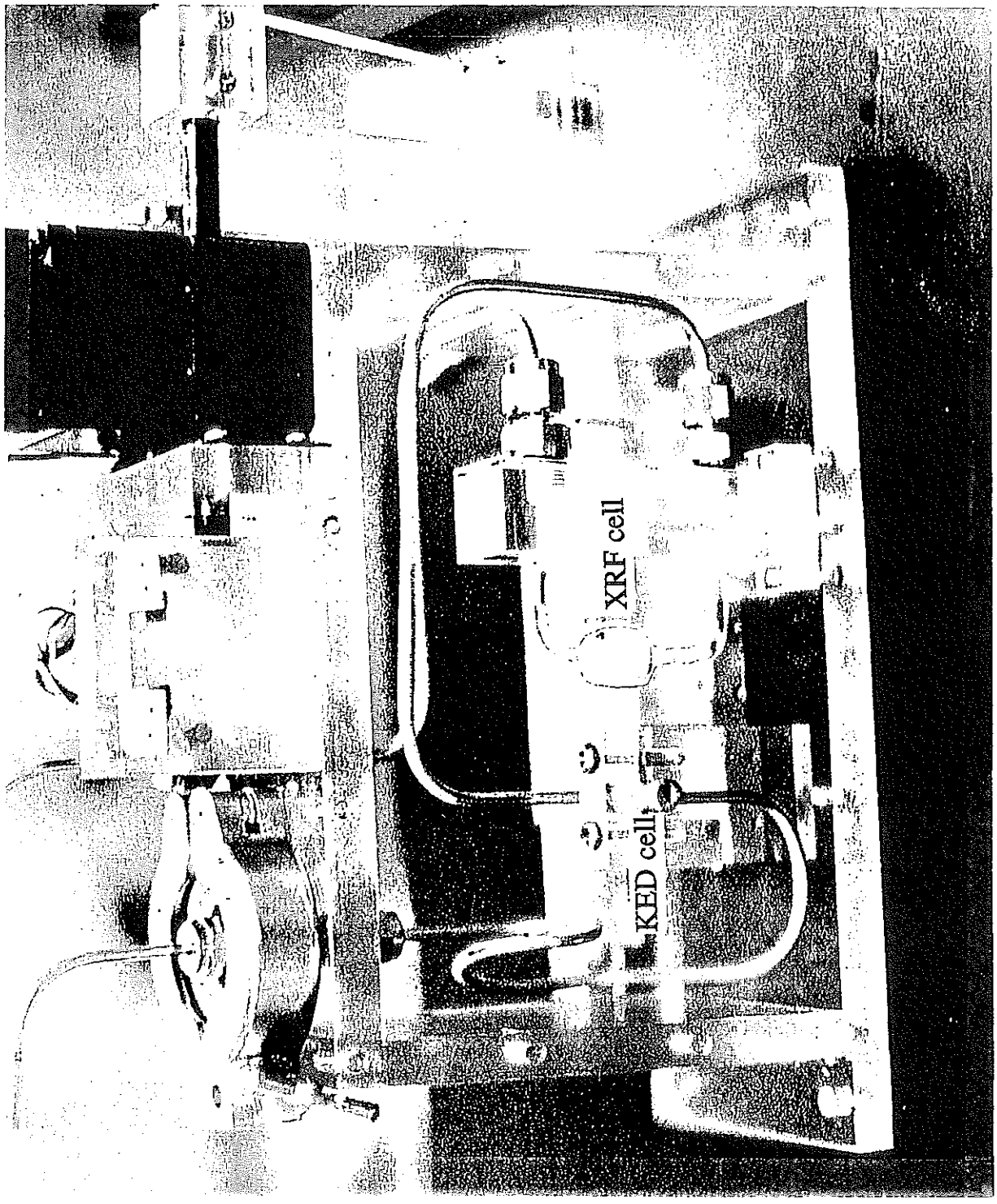


Photo.2 Measurement cell system.

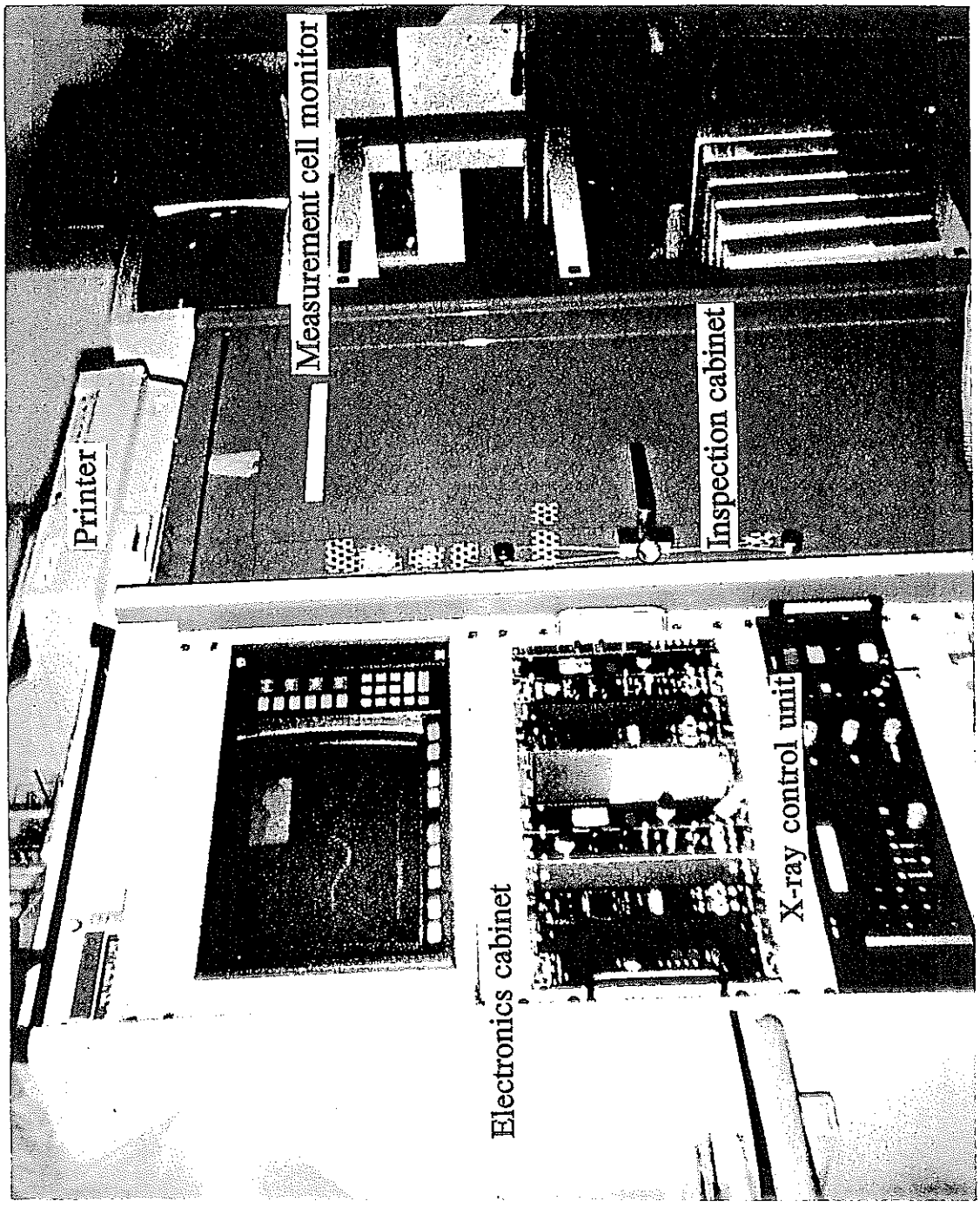


Photo.3 Equipment located in the room G-105.