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Determination of Plutonium in Highly Radioactive Liquid Waste by Spectrophotometry Using Neodymium as an Internal Standard for Safeguards Analysis

-Japan Support Program for Agency Safeguards (JASPAS) JC-19-

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A spectrophotometric determination using neodymium as an internal standard was developed for safeguards verification analysis of plutonium in highly radioactive liquid waste which is produced by the reprocessing of spent nuclear fuel. The internal standard is used as a means to analyze plutonium and also to authenticate the instrument conditions. The method offers reduced sample preparation and analysis time compared to isotope dilution mass spectrometry. The sample was mixed with a known amount of internal standard. Subsequently, plutonium was quantitatively oxidized to Pu(VI) by the addition of Ce(IV) for spectrophotometry. Plutonium concentration was calculated from a relation between Nd(III)/Pu(VI) molar extinction coefficient ratio and their absorbance ratio. The relative expanded uncertainty of the repeated analysis ($n = 5$) was 8.9% (coverage factor $k = 2$) for a highly radioactive liquid waste sample (173 mg L^{-1}). The determination limit was 6 mg L^{-1} (ten fold's the standard deviation). This method was validated through comparison experiments with isotope dilution mass spectrometry. The analytical results of plutonium in highly radioactive liquid waste using this method were agree well with values obtained using isotope dilution mass spectrometry. The proposed method can be applied to independent on-site safeguards analysis at the Tokai Reprocessing Plant.

Keywords: Spectrophotometry, Pu, Internal Standard, Nd, Highly Radioactive Liquid Waste, Safeguards

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+ Technology Development Department, Tokai Reprocessing Technology Development Center, Nuclear Fuel Cycle Engineering Laboratories.

保障措置のためのネオジムを内標準物質とした吸光光度法による高放射性廃液中のプルトニウムの定量

－日本の対 IAEA 保障措置技術開発支援計画（JASPAS）JC-19－

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使用済み核燃料を再処理する際に発生する高放射性廃液中に含まれる，プルトニウムの検認分析法として，ネオジムを内標準物質とした吸光光度法による定量手法を開発した。この方法は，内標準物質のネオジムを既知量添加した後，Ce(IV)によりプルトニウムを Pu(VI)に酸化し，Pu(VI)と Nd(III)の吸収ピークを対象として両ピークの吸光度比とモル吸光係数比の関係からプルトニウム濃度を算出するものである。この方法では、ネオジムを，プルトニウム濃度を算出するための内標準物質として用いる他，測定装置の健全性を確認する指標としても利用する。さらに本法は，同位体希釈質量分析法と比較して迅速な測定が可能であることから，保障措置上の適時性を確保できることが特徴である。プルトニウム濃度が 173 mg L^{-1} の高放射性廃液試料に対する評価として，相対拡張不確かさ($n = 5$)は 8.9 %(包含係数 $k = 2.0$)であり，定量下限は 6 mg L^{-1} (10 σ)であった。また，本法の妥当性を確認するため，同位体希釈質量分析法による比較分析を行った結果，両者の分析値は良好に一致し，東海再処理施設において査察側が現場で行う高放射性廃液中のプルトニウムの検認分析法として適用が可能であることを確認した。

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1 INTRODUCTION

Highly radioactive liquid waste (HALW), which is generated from reprocessing of spent nuclear fuel at the Tokai Reprocessing Plant (TRP), contains small amounts of plutonium at a low concentrations ($>10^{-4}$M). Since the 1990s, the International Atomic Energy Agency (IAEA) has attached particular importance to safeguards of the radioactive waste from nuclear facilities. As a result, the plutonium in HALW of TRP has been specifically examined as an object to be inspected. Since 1993, HALW samples have been taken for inspection and transported to IAEA's Safeguards Analytical Laboratory (IAEA-SAL) for verification analyses of plutonium in HALW. Sample preparation at the analytical laboratory of TRP, such as sampling, spiking and diluting, has been carried out for analyses by isotope dilution mass spectrometry (IDMS) 1)-3). The HALW sample is spiked with a plutonium standard, in which the isotope ratio differs from that of the sample. The spiked aliquot is diluted and dried in a vial for transportation, and is analyzed in the IAEA-SAL for independent measurement by inspector's own analytical equipment.

The IDMS has been considered as the most reliable analytical technique for accountability measurement of nuclear materials in spent fuel reprocessing plants. However, the IDMS requires a complicated procedure and skilled operator. This method is also time-consuming to obtain an analytical result because the sample must be transported for off-site analyses. Therefore, it takes a few months for an inspector to obtain analytical results. Thus, a rapid verification analysis of plutonium in HALW using conventional spectrophotometer was proposed to achieve on-site safeguards measurements to overcome these disadvantages.

Plutonium ions in acidic aqueous solution exist in trivalent, tetravalent and hexavalent oxidation states. In particular, Pu(VI) in HNO_3 medium is often used for spectrophotometry because of its sharp absorption peak at the wavelength of 830 nm; it has a larger molar extinction coefficient than either Pu(III) or Pu(IV) 4). These properties have been used for determination of plutonium 5), 6) in spent fuel reprocessing plants. Although the measurement precision of spectrophotometry is inferior to that of IDMS, the method we propose can be expected to be useful as a tool for timely on-site analysis and verification of plutonium in HALW.

Instruments and analytical schemes must be strictly checked for authentication to apply a method to verification analysis of a sample. An application of the internal standard method has been studied to meet requirements for the spectrophotometric technique. Consequently, an internal standard is used as a means to analyze plutonium in HALW and also to check instrument conditions. The internal standard is intended to be provided by the IAEA so that an inspector can control it independently. After spectrophotometric measurement, the inspector calculates the result from observed absorbance of the internal standard and compares it with a reference value that is not known by facility operators.

In addition, Nd(III) was chosen as an internal standard. The Nd(III) in HNO_3 medium exhibits its absorption maxima at around 795 nm 7) which is near the Pu(VI) absorption maximum (830 nm). That absorption maximum also presents the advantage that it has no other remarkable peaks that might inhibit Nd(III) measurements.

This report describes experimental results and acceptance test results performed with IAEA inspectors. Operating manual about Nd-internal standard method for verification analysis shows Appendix 1.

This study has been carried out as a part of Japan support program for agency safeguards (JASPAS JC-19) since 1999.

2 MEASUREMENT PRINCIPLE

In this work, plutonium concentration in an HALW sample was determined by spectrophotometry using Nd(III) as an internal standard (Nd-internal standard method). Pu(VI) absorption peak around 830 nm in the absorption spectrum has been used for the quantitative measurement of plutonium. Nd(III) has an absorption peak at 795 nm and can be utilized for the internal standard in this work. An absorption peak of around 795 nm was observed for HALW sample because it contains fission products including Nd(III). The absorption peak initially present in HALW is corrected with absorbance ratio at 830 nm and 795 nm of HALW sample. The plutonium concentration in HALW sample can be calculated using the Pu(VI)/Nd(III) molar extinction coefficient ratio, and the absorbance at 830 nm and 795 nm for HALW sample with and without Nd(III) as an internal standard. The equation of the plutonium concentration using Nd-internal standard method is lead as the following equation.

Pu(VI)/ Nd(III) absorbance ratio for HALW sample with Nd(III) as an internal standard is expressed in equation(1).

$$R_B = \frac{(A_{830})_X}{(A_{795})_X + (A_{795})_Y} \tag{1}$$

Here, R_B is the absorbance ratio for HALW sample with Nd(III) as an internal standard, $(A_{830})_X$ and $(A_{795})_X$ are absorbance at 830 nm and 795 nm for HALW sample without Nd(III) as an internal standard, respectively, and $(A_{795})_Y$ is absorbance at 795 nm of Nd(III) as an internal standard.

Pu(VI)/Nd(III) absorbance ratio for HALW sample is expressed in equation(2).

$$R_X = \frac{(A_{830})_X}{(A_{795})_X} \tag{2}$$

Here, R_X is the absorbance ratio for HALW sample, $(A_{830})_X$ and $(A_{795})_X$, are absorbance at 830 nm and 795 nm for HALW sample, respectively.

The equation (2) is substituted for the equation (1).

$$(A_{830})_X = \frac{R_X \cdot R_B}{R_X - R_B} \times (A_{795})_Y \tag{3}$$

Using Lambert-Beer's law, Nd(III) absorbance and Pu(VI) absorbance are shown in equation (4) and (5), respectively.

$$(A_{795})_Y = \varepsilon_{Nd} \times \frac{C_{Nd}}{A.W_{Nd}} \times L \tag{4}$$

$$(A_{830})_X = \varepsilon_{Pu} \times \frac{C_{Pu}}{A.W_{Pu}} \times L \tag{5}$$

Here, ε_{Nd} and ε_{Pu} are the molar extinction coefficients of Nd(III) and Pu(VI), respectively; C_{Nd} and C_{Pu} are the concentrations of the Nd(III) as an internal standard and Pu(VI) in the HALW sample, respectively; $A.W_{Nd}$ and $A.W_{Pu}$ are the atomic weight of neodymium and plutonium, respectively; L is the optical path length.

The equation (4) is expressed as the following equation.

$$(A_{795})_Y = \varepsilon_{Nd} \times \frac{M_{Nd}}{V_S \cdot A.W_{Nd}} \tag{6}$$

Here, M_{Nd} is the weight of the Nd(III) as an internal standard, and V_S is the sampling volume for HALW.

The equation (5) and the equation (6) are substituted for the equation (3). Therefore, the plutonium concentration is calculated from the following equation.

$$C_{Pu} = \frac{R_X \cdot R_B}{R_X - R_B} \times \frac{A.W_{Pu}}{A.W_{Nd}} \times \frac{\varepsilon_{Nd}}{\varepsilon_{Pu}} \times \frac{M_{Nd}}{V_S} \tag{7}$$

3. EXPERIMENTAL

3.1 Reagents

- Plutonium metal CRM 126:
  New Brunswick Laboratory (NBL) 99.962 ± 0.018wt%
- Uranium metal JAERI –U4:
  Japan Atomic Energy Research Institute (JAERI) 99.90%
- Uranium metal NBL CRM 116:
  New Brunswick Laboratory (NBL) 99.97%
- Cerium(IV) diammonium nitrate ($Ce(NH_4)_2(NO_3)_6$):
  Kanto Reagents, Analytical grade >95%
- Hydrofluoric acid (HF):
  Kanto Reagents, Analytical grade 46%-48%
- Nitric acid (HNO_3):
  Kanto Reagents, Analytical grade 60-61%
- Neodymium oxide (Nd_2O_3):
  Kanto Reagents, ＞99%
- Neodymium oxide (Nd_2O_3) (Using acceptance test):
  FLUKA
- Iron(II) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$):
  Kanto Reagents, Analytical grade 99.0%-102.0%

- Amidosulfuric acid ($HOSO_2NH_2$):
  Kanto Reagents, Analytical grade >99.5%
- Sodium nitrate ($NaNO_2$):
  Kanto Reagents, Analytical grade >98.5%
- L- Ascorbic acid:
  Kanto Reagents
- TEVA extraction chromatographic resin:
  Eichrom Technologies, Inc (U.S.A)
- ^{242}Pu spike:
  Provided by IAEA, ^{242}Pu abundance 99.9057%, $^{242}Pu/^{239}Pu$ atomic ratio 1210.0 ± 5.9

3.2 Preparation

A plutonium stock solution was prepared by dissolving plutonium metal in mixture of 8 M HNO_3 and 0.01 M HF. The plutonium stock solution was adjusted to 200 mgPu L^{-1}. Working standard solutions of plutonium were prepared by diluting the stock solution with 3 M HNO_3. A uranium stock solution for determining the molar extinction coefficient was prepared by dissolving uranium metal in 8 M HNO_3. A uranium stock solution for other experiments was prepared by dissolving uranium trioxide, which was reprocessed at TRP, in 8 M HNO_3. The uranium stock solution was adjusted to 4 gU L^{-1}. Working standard solutions of uranium were prepared by diluting the stock solution with 3 M HNO_3. A cerium solution as an oxidation reagents was prepared by dissolving 130 g of $Ce(NH_4)_2(NO_3)_6$ in 500 mL of 3 M HNO_3. A neodymium stock solution was prepared by dissolving 1.173 g of Nd_2O_3 in 3 M HNO_3. The neodymium stock solution was adjusted to concentration of 10 g L^{-1}.

A neodymium stock solution used in acceptance test was prepared by dissolving about 2 g of Nd_2O_3 in 1 M HNO_3. This stock solution has been adjusted to concentration of 25 mgNd g^{-1} solution. The aliquots were evaporated to dryness at 120 ℃ in flask.

TEVA extraction chromatographic resin (0.5 mL) was preconditioned with 3 M HNO_3 over 10 mL. A ^{242}Pu spike was used for IDMS. Reagent grade chemicals were used in all tests. Deionized water for dilution of the stock solution was used throughout the experiments.

3.3 HALW sample

HALW samples used were taken from some storage vessels at TRP, and collected in flasks in a shielded cell of analytical laboratory. Sample taking from the flask was then carried out by the use of sampling device immediately after stirring the solution to obtain a homogeneous sample including sludge.

3.4 Apparatus

3.4.1 Spectrophotometer

3.4.1.(1) System configuration

A schematic diagram of a UV VIS spectrophotometric system used in this work is shown in Figure 3-1. This system consists of a light source, monochromator, a detector, a sample cell, a reference cell and a computer for spectrophotometer control. A sample cell was installed in a shielded cell because the HALW sample is highly radioactive. Light flux from a light source was adjusted using mirrors. The light flux was transmitted to the sample cell using optical fibers. The details about spectrophotomtry system show the appendix 3.

3.4.1.(2) Specifications of the spectrophotometer

Specifications of the spectrophotometer are shown in Table 3-1.

3.4.1.(3) Specifications of the optical fiber

Specifications of the optical fiber are shown in Table 3-2.

3.4.1.(4) Sample cell

A schematic diagram of the sample cell is shown in Figure 3-2. The sample cell was sealed completely to keep moisture from entering. Optical fiber was connected from the side direction to the sample cell to obtain higher light intensity and to lessen a loss. The optical cell housing was stainless steel, which prevents corrosion by nitric acid. The sample cell was a flow type to replace a sample solution easily in remote control. The optical cell and stainless steel pipe were connected and sealed using an O-ring, which protected the connection from unexpected vibrations and shocks. The condensing lens and optical cell (optical pathway = 10 mm) were made of quartz.

3.4.2 Other apparatus

Sampling was performed by sampling device TD-2S (Tsunakawa Engineering). Reagents were added by the automatic burette Dosimat 665 (METROHM-SIBATA). The results of calibration for the sampling device and the automatic burette were shown in Appendix 2.

Isotope abundance ratio measurements on prepared sample for validation analysis were performed by thermal ionization mass spectrometry (TIMS) using Finnigan MAT262.

3.5 Sample preparation

3.5.1 Nd-internal standard method

The fraction of an HALW sample containing sludge was taken into a 50 mL Erlenmeyer flask containing a known amount of Nd(III) as an internal standard. For dissolving the plutonium in sludge, 2.0 mL of a mixed acid of 8 M HNO_3 and 0.025 M HF were added into the flask, and the sample was then heated at 150 ℃ on a hot plate to near-dryness. The dried sample was maintained at room temperature for 15 minutes, and was re-dissolved in 3 M HNO_3 of 14 mL. Another sample without neodymium standard was treated in the same procedure. Plutonium in the HALW sample was oxidized to hexavalent state by Ce(IV). An oxidant of 2 mL of 0.5 M Ce(IV) was added into each flask, and the sample solution was stirred. The sample was maintained for 5 minutes to oxidize the plutonium quantitatively to Pu(VI). The diluted solution was filtered through a paper filter (ADVANTEC No.5C) to remove insoluble fine residues. All of the reagents were added by burette from the outside of the shielded cell.

The prepared sample solution of 16 mL was introduced into the sample cell. After prepared sample solution was thermostated at room temperature, spectrophotometric measurement was made. Analytical procedure and measurement conditions of spectrophotometer were shown in Table 3-3 and Figure 3-3, respectively.

3.5.2 IDMS

Two aliquot of a sample solution was taken from HALW using a pipette, and spiked with ^{242}Pu. For dissolving the sludge, 3 mL of a mixed acid of 8 M HNO_3 and 0.01 M HF was added into each vial, and the sample was then heated at 150 ℃ on a hot plate to near-dryness. The dried sample was maintained at room temperature, and was re-dissolved in 3 M HNO_3.

The re-dissolved solution was stirred for 1 minute by a magnetic stirrer. The solution was filtered with a pre-filter of chromatographic column (Eichrom) to remove insoluble residues. The diluted solution of 1 mL was diluted to 40 mL with 3 M HNO_3, and stirred for 1 minute by a magnetic stirrer, 0.1 mL of this sample solution was taken to a vial. Plutonium in samples was completely adjusted to tetra-valence with 0.2 mL of 0.2 M sulfamic acid - 0.5 M Iron(II) sulfate solution and 0.5 mL of 1 M sodium nitrate solution, and they were dissolved again in 1 mL of 3 M HNO_3 after heating them at 95 ℃ to near-dryness. Each sample was individually passed through an extraction chromatography column (TEVA-Resin) adjusted with 3 M HNO_3, and fission products were separated from HALW by rinsing with 16 mL (2 mL × 8) of 3 M HNO_3. Plutonium was eluted with 1.5 mL of 5.7×10^{-2} M ascorbic acid solution (0.5 mL × 3). A solution of 13.1 M HNO_3 was added to the solution to decompose the ascorbic acid. They were dissolved in 1 M HNO_3 after heating them to near-dryness. For mass spectrometry, 1 μ Lof these solutions were coated on a filament by passing at 2.0 A electricity after evaporating the solution by passing at 0.7 A electricity. Another aliquot was also taken from the sample solution for isotopic analysis, and was treated same procedure as spiked samples. The relative isotopic ratio was determined for spiked and un-spiked.

4 RESULTS AND DISCUSSIONS

4.1 Performance for spectrophotometer and determination of optimum conditions for measurement

4.1.1 Baseline noise

Baseline noise is one of an important factors affecting accuracy. In our spectrophotometric system, the noise in the absorption spectrum is greater than that of a conventional spectrophotometer because optical fiber was used. Therefore, the baseline noise was measured for checking the performance of this spectrophotometric system.

For checking the baseline noise, a solution of 3 M HNO_3 was passed through the sample cell. Its absorbance was measured ranging from 0 s to 120 s at the measuring wavelength of 830 nm (Figure 4-1). The difference between the mid-point and the maximum and minimum values was ±0.0003 Abs. Then, the baseline was corrected using a solution of 3 M HNO_3 as a blank, and the spectrum was stored in the computer's memory. Subsequently, the absorption spectrum of 3 M HNO_3 was measured at slit width of 5.0 nm ranging from 850 nm to 780 nm (Figure 4-2). The difference between the mid-point and the maximum and minimum values was ±0.0005 Abs.

No significant difference in noise was found compared with that of a conventional spectrophotometer.

4.1.2 Replacement volume of the solution in flow type optical cell

The volume needed to replace the sample solution in flow type optical cell was examined. The replacement volume in the cell was determined from the recovery of a solution of Nd(III). First, 30 mL of 3 M HNO_3 was added to the cell to replace the solution with 3 M HNO_3 completely. After the Nd(III) solution was added to the cell, absorbance at 795 nm was measured. Figure 4-3 shows a relationship between the recovery and added volume of the Nd(III) solution. As the figure, the solution of Nd(III) was recovered more than 99% by adding 14 mL. Therefore, the volume necessary to replace the sample solution was determined as more than 14 mL.

4.1.3 Optimization of slit width

In our spectrophotometric system, the noise in the absorption spectrum increases when compared with that of a spectrophotometer without optical fiber, because optical fiber was used. The noise affects the repeatability of the measurements. Thus, it is necessary to optimize the measurement conditions to lower the noise. The slit width of the spectrophotometer is one of the most important factors in a spectral measurement. The slit width was optimized by absorbance measurements using an HALW sample. Absorbance measurements of Pu(VI) and Nd(III) were carried out with different slit widths of 0.2 nm, 1.0 nm, and 5.0 nm. Table 4-1 shows the relationship between the relative standard deviation (RSD%) of the absorbance measurement and the slit width. When the slit width was 5.0 nm, absorbance of Pu(VI) and Nd(III) resulted in good reproducibility of 1.2% and 5.4%, respectively. Therefore, absorbance measurements were carried out using the 5.0 nm slit width in all the experiments.

4.2 Pre-preparation conditions

4.2.1 Amount of hydrofluoric acid for dissolving plutonium in an HALW sample containing sludge.

Plutonium in the sludge was dissolved to determine the total amount of plutonium in the HALW sample with sludge. For dissolving the plutonium, hydrofluoric acid(HF) was added to the HALW sample and the sample was heated in 6.3 M HNO_3 with the HF. Figure 4-4 shows the relationship between the HF concentration and plutonium concentration in the HALW sample. The plutonium concentration in the HALW sample without sludge was 30.6 mg L^{-1}. The plutonium concentration in HALW sample containing the sludge was 32.8 mg L^{-1}, and the concentration was increased about 20% by heating in 6.3 M HNO_3. The plutonium concentration was 40.0 mg L^{-1}; it showed a constant value when HF was adjusted to greater than 5×10^{-7} M. Therefore, a concentration greater than 5×10^{-7} M of HF was inferred to be sufficient for dissolving the plutonium in the HALW sample containing sludge.

4.2.2 Effect of uranium on plutonium measurement with hydrofluoric acid.

An HALW sample solution was heated on a hot plate to near-dryness after addition of HF to dissolve plutonium in the sludge. Influence of the HF was tested by determining plutonium in the standard solution with HF. Plutonium standard solutions were measured after the same pre-preparation for dissolving the plutonium in the sludge. The found value of the standard solution was 40% lower than the value of the solution. The remaining HF in the sample solution after heating is inferred to causes an error in determination of plutonium because of complexation with HF 8). Figure 4-5 shows the effect of uranium on plutonium with HF 8). The found values agreed well with the taken values when the concentration of uranium was adjusted to greater than 5.3×10^{-5} M. Therefore, it is confirmed that the remaining HF in the solution after heating has no influence on the plutonium concentration by coexisting uranium of same degree amount of plutonium. In this work, HF had no influence on the plutonium concentration because HALW contains more than 2×10^{-3} M uranium.

4.2.3 Amount of cerium(IV) as an oxidant

Oxidation from Pu(III), Pu(IV) to Pu(VI) is necessary before spectrophotometric measurements. This oxidation is accomplished by the addition of cerium(IV)(Ce(IV)). Figure 4-6 shows the relationship between the amount of Ce(IV) and absorbance of Pu(VI). The absorbance showed a constant value when 6.7×10^{-7} mol of Ce(IV) or more was added. The amount of Ce(IV) determined as optimum was determined as 1.5 times the equivalent molarity to the plutonium in HALW. Therefore, the amount of Ce(IV) was determined as 6.7×10^{-7} mol, corresponding to more than 1.5 times moles of plutonium at least.

4.2.4 Stability of hexavalent state of plutonium in HALW

The stability of the hexavalent state of plutonium in the HALW sample solution with Ce(IV) was examined. Figure 4-7 shows the relationship between absorbance of Pu(VI) at 830 nm after adding Ce(IV), along with elapsed time. The relative standard deviation of the measured absorbance was within 1.1% during 10—140 min. In this work, the HALW sample is measured within 30 min after adding Ce(IV). Therefore, the absorbance of Pu(VI) at 830 nm was not influenced by other elements contained in HALW.

4.2.5 Determination of the baseline and the peak heights of plutonium(VI) and neodymium(III)

Absorption spectra of HALW and Pu(VI) standard solution with Nd(III) are shown in Figure 4-8. The absorption spectrum of HALW is apparently different from that of the standard solution because the HALW sample is suspended as a result of remaining insoluble residues, which cause light scattering and background increasing. In addition, HALW contains other elements, e.g. Am(III), that have a certain absorbance. Therefore, the baseline determination to characterize the peaks of Nd(III) and Pu(VI) in HALW should be made in consideration of those effects. Regarding proper assignment of an established baseline, the following approaches were attempted for the absorption spectrum of HALW. Figure 4-9 shows modeling of baseline and peak height determination using two different means. The three-point method uses upper side point (838 nm) and lower side point (825 nm) bracketing Pu(VI) peaks (830 nm) to produce a baseline that has points determined with the inflection points obtained using the second-order derivative spectrum of Pu(VI) in 3 M HNO_3 medium. On the other hand, the two-point method uses a horizontal line extrapolated from the upper side point (838 nm) against the Pu(VI) peak. Both methods use the same extrapolated baseline to determine the Nd(III) peak height.

The applicability of these methods was investigated for baseline determination of the

Pu(VI) peak height. Plutonium concentrations were calculated using a calibration curve prepared with Pu(VI) standard solutions. They were compared with that of IDMS. As a result, the analytical value (86.3 ± 5.8 g L^{-1}) by two-point method agree well with that of IDMS (83.0 ± 0.2 g L^{-1}), whereas the three-point method gave a 15 % lower result (72.9 ± 3.0 g L^{-1}) than that of IDMS. Difference in analytical results are explainable by comparison of absorption spectra of HALW and Pu(VI) standard solution with Nd(III). As shown in Figure 4-8, absorptions between Nd(III) and Pu(VI) peak were observed in HALW spectra. Accordingly, overlapping of the absorbance was also observed at 825 nm, which is used for making a baseline. This would indicate that the three-point method evaluates the peak height of Pu(VI) as lower than the expected value. It is therefore possible that absorption is affected by other elements, e.g. Am(III) having an absorption peak at 811 nm [9], or that the increase of absorbance is caused by the scattering of light from fine particles in the measured sample. On the other hand, such influences at the upper side point of 838 nm and top of Pu(VI) peak (around 830 nm) can be of limited significance in the two-point method. This argument was based on an experimental result obtained from independent measurements of Am(III) at the wavelength. Therefore, the two-point method was chosen for baseline determination in peak evaluation of the HALW sample.

4.2.6 Molar extinction coefficient ratio

The experimental molar extinction coefficient ratio of Pu(VI) to Nd(III), $\varepsilon_{Pu}/\varepsilon_{Nd}$, needs to be determined in advance because the plutonium in the HALW sample is calculated from equation (7). The molar extinction coefficient ratio can be calculated from the relationship between the Pu(VI)/Nd(III) concentration ratio and the Pu(VI)/Nd(III) absorbance ratio. Figure 4-10 shows the relationship between the Pu(VI)/Nd(III) concentration ratio and the Pu(VI)/Nd(III) absorbance ratio, which was obtained from a set of measurements of Pu(VI) and Nd(III) standard solutions. A linear relationship exists (correlation coefficient: >0.999), and the calculated molar extinction coefficient ratio was 34.3 ± 3.1% (n = 6).

4.3 Plutonium measurement by Nd-internal standard method

4.3.1 Amount of neodymium(III) as an internal standard

Plutonium in an HALW was analyzed to confirm the effects of added amounts of Nd(III) as an internal standard. The Nd(III) was added to the HALW sample at 15−50 mg. The analytical results are shown in Table 4-2. The relative standard deviation (RSD%) for three different analyses of the HALW sample was within 3.8%. No significant difference was detected between the mean values for respective amounts of Nd(III). It is noteworthy that an inspector can control the amount of Nd(III) independently as an internal standard because it can be changed to any value of 15−50 mg. Consequently, the facility operator can be blinded to the amount of the internal standard. Using this method, an inspector can use the internal standard as an index for the authentication of the analytical scheme and the inspection procedure by adding neodymium as an internal standard and evaluating whether the results of absorbance measurements of the Nd(III) correspond to the amount of the added Nd(III).

4.3.2 Analytical results for standard solutions

A series of standard solutions ranging from 3.67 to 18.36 mg L^{-1} was prepared to demonstrate the proposed method. The samples were analyzed using the Nd-internal standard method and the analytical results are summarized in Table 4-3. Although the difference between the found and taken values on the sample with the lowest concentration was larger than others, this appears to result from the measurement uncertainty on spectrophotometry for such a sample with low concentration. Therefore, the fundamental performance of the method was proven for plutonium measurement in HNO_3.

4.3.3 Analytical results for HALW

Plutonium in the HALW sample was analyzed to confirm the performance of the Nd-internal standard method. The results were compared with the calibration curve method, which is a general spectrophotometric method. Table 4-4 shows the analytical results for concentration of plutonium in HALW obtained using the Nd-internal-standard method and the calibration curve method. The relative standard deviation (RSD %) for five repeated analyses of the HALW sample was 2.9%. No significant difference was found in precision between methods. The determination limit of 10 times the standard deviation was 6 mg L^{-1}. The analysis time required for an analyses was about 4 h.

4.3.4 Comparison of Nd-internal standard method and IDMS

Validation analyses were carried out using HALW samples taken at TRP. The analytical results obtained using the proposed method were compared with those from IDMS. Figure 4-11 shows a comparison of IDMS and the proposed method for plutonium. The error bars on the plots were determined from the expanded uncertainty 10), 11) (coverage factor $k = 2.0$). The plutonium in HALW obtained using the proposed method agreed well with values obtained by IDMS to within ± 10%.

4.4 Acceptance test

Two HALW samples were measured as a part of acceptance test performed on 2003 in order to evaluate the validity in this method by IAEA and JSGO. In addition to the two-point method, the results evaluated by the three-point method were also shown as reference.

4.4.1 Molar extinction coefficient ratio

Molar extinction coefficient ratio of Pu(VI) to Nd(III), $\varepsilon_{Pu} / \varepsilon_{Nd}$, was determined using a neodymium standard and plutonium standard prepared by IAEA. The relation between Pu(VI)/Nd(III) absorbance ratio and concentration ratio was shown in Figure 4-12. The correlation coefficient was resulted in 0.9985. The calculated coefficient ratio, $\varepsilon_{Pu} / \varepsilon_{Nd}$, was 36.989.

4.4.2 Analytical results for HALW

Validation analyses were carried out using HALW samples having different concentrations of plutonium. The analytical results obtained using the proposed method were compared with those from IDMS. The comparisons of IDMS and proposed method for plutonium were shown in Table 4-5 and Table 4-6. Plutonium concentrations using spectrophotometry shown in Table 4-5 and Table 4-6 were evaluated by two-point method and three-point method, respectively. The plutonium concentrations in HALW calculated by three-point method were lower than those of IDMS. On the other hand, the plutonium concentrations calculated by two-point method were agreed well with values obtained by IDMS within the range from

-8.9% to 6.3%.

5. CONCLUSIONS

An analytical method using a conventional spectrophotometer was developed for safeguards on-site verification to determine the plutonium in HALW at TRP. Validation of the proposed method in this work was carried out using IDMS with an actual HALW sample collected from TRP. The mean values obtained using the proposed method show good agreement with that of IDMS within ± 10%. The relative expanded uncertainty was found to be 8.9% (coverage factor $k = 2.0$) for a typical HALW sample (173 mg L^{-1}). The uncertainty values were lager than that of IDMS, but it appears that the value was within HALW analysis criteria. The determination limit was then calculated to be 6 mg L^{-1} from 10 times the standard deviation. This determination limit indicated that the proposed method can be used reasonably for determination of plutonium in HALW from TRP, for which the concentration was higher than about 40 mg L^{-1}.

This method offers easy and rapid determination of plutonium in HALW, requiring neither complicated analytical procedures nor skilled operators. The proposed method greatly simplifies the process of inspection activities without the necessity for transport of nuclear materials for off-site analyses. This approach greatly reduces the complexity and cost of analyses compared with IDMS. It requires no complicated procedures or expensive equipment. The proposed method with an authentication measure using a controlled standard of neodymium is applicable for rapid determination and allows implementation of independent safeguards analysis at TRP. Implementation of this method as part of inspection activity is currently being examined, as well as the declaration way from operator for accountability of plutonium in HALW at TRP.

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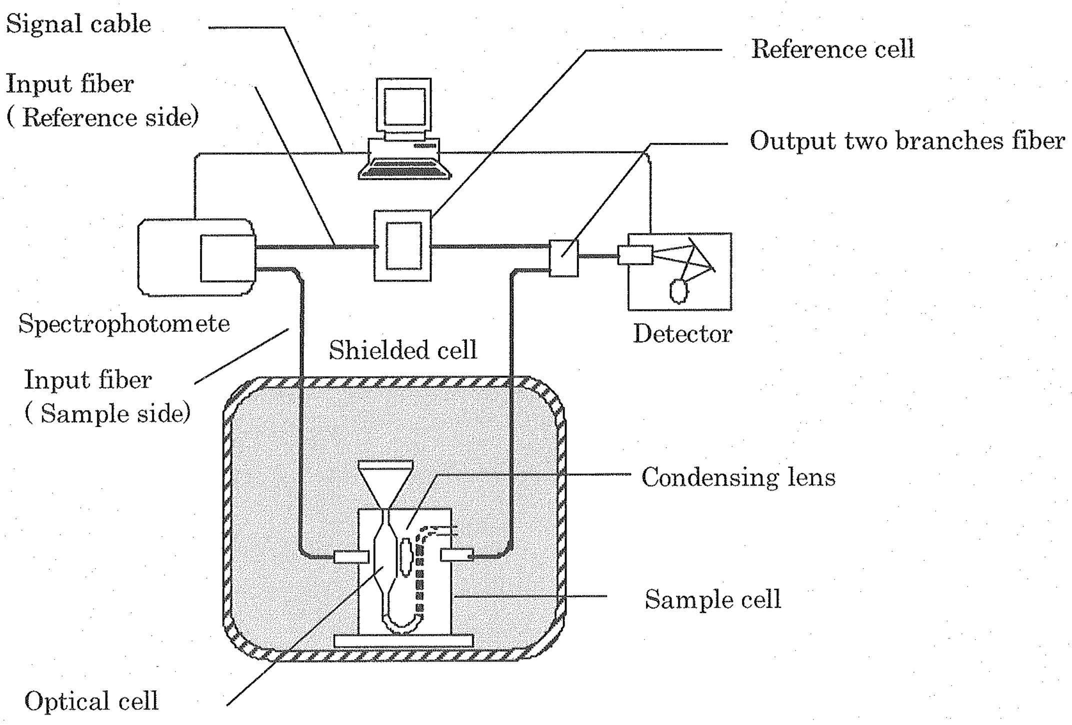


Fig.3-1 Schematic diagram of spectrophotometric system installed in shielded cell

Table 3-1 Specifications of spectrophotometer

Model	V550 UV/VIS Spectrophotometer (Jasco)
Optical system	Single monochromator double beam type UV/VIS region: 1200 lines/mm plane grating Czerny-Turner mount
Resolution	0.1 nm
Light source	Deuterium lamp: 190 to 350 nm Halogen lamp: 330 to 900 nm
Light source changeover wavelength	Any wavelength between 330 and 350 nm can be selected
Wavelength range	190 nm～900 nm
Wavelength repeatability	± 0.1 nm (at a spectral bandwidth of 0.5 nm)
Wavelength accuracy	± 0.3 nm (at a spectral bandwidth of 0.5 nm)
Spectral bandwidth	0.1, 0.2, 0.5, 1, 2, 5, 10 nm L2, L5, L10 nm (low stray-light mode)

Response Stray light	0.015%T (220 nm: NaI 10 g/L aqueous solution) (340 nm: $NaNO_2$ 50 g/L aqueous solution)
Photometric mode	Abs, %T, %R, Sam, Ref
Photometric range	-2～3 Abs 0～200 %T
Photometric repeatability	± 0.001 Abs. (0 to 0.5 Abs.)
Photometric accuracy	± 0.002 Abs. (0.5 to 1 Abs.), ± 0.002 Abs (0 to 0.5 Abs.), ± 0.004 Abs. (0.5 to 1 Abs.), ± 0.3%T
Response	Quick, Fast, Medium, Slow
Wavelength scanning	4000, 2000, 1000, 400, 200, 100, 40, 20, 10 nm/min
Wavelength moving speed	8000 nm/min
Data pitch	0.025, 0.05, 0.1, 0.2, 0.5, 1, 2, 5, 10 nm/data
Baseline flatness	± 0.001 Abs. (value obtained after baseline correction when temperature valiation is within 5 ℃, wavelength: 200 to 850 nm, response: Medium, spectra band width: 2 nm, and wavelength scanning: 100 nm/min
Baseline stability	± 0.004 Abs/hour (value obtained more than one hour after turning ON the power when temperature variation is within 5 ℃, wavelength: 250 nm, response: Slow, and spectral bandwidth: 2 nm)
Detector	Photomultiplier tube

Table3-2 Specifications of optical fiber

Model	Quartz optical fiber (MITUBISHI CABLE INDUSTRIS)
Optical fiber	ST230D Core diameter: 230μm, Fiber diameter: 250μ m N A: 0.2 ± 0.02
Rate of disconnection	Within 2%
Heat resistance	A section of sleeve: 120 ℃ Others: 80 ℃
Permission curve R	Over 80 mm
The number of fiber core	Sample cell → Detector Reference cell → Detector 164 core Spectrophotometer → Sample cell Spectrophotometer → Reference cell 108 core

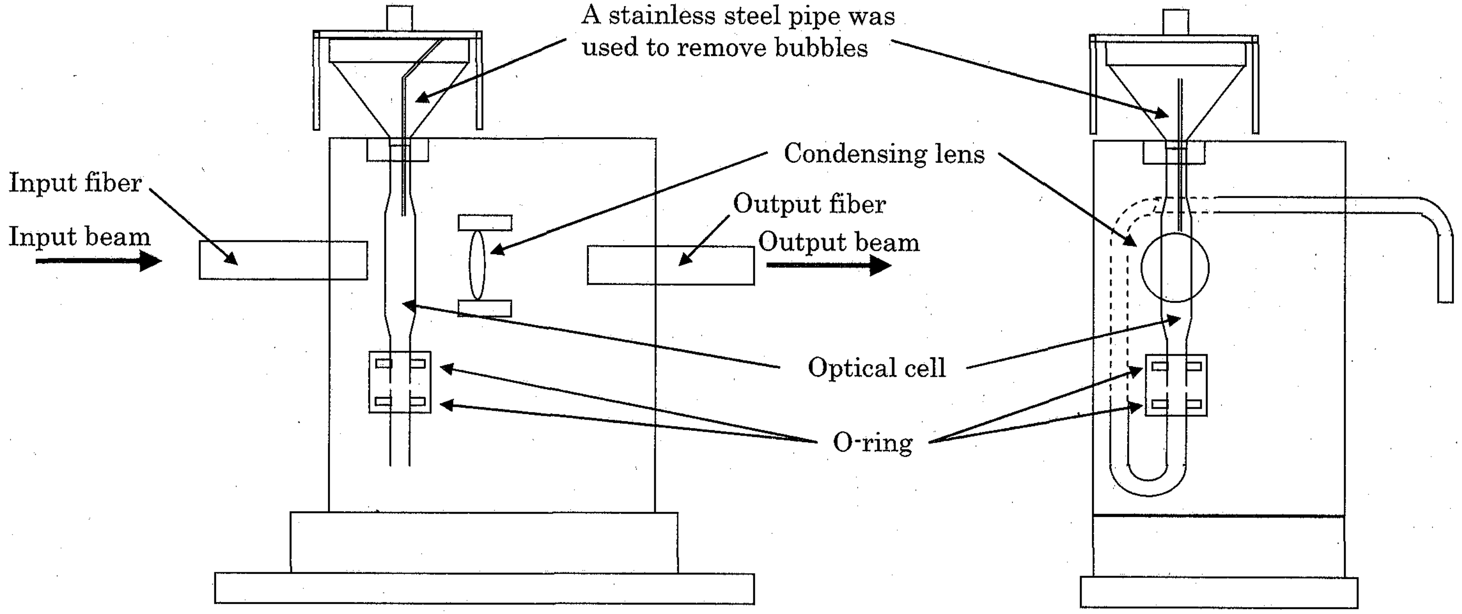


Fig. 3-2 Schematic diagram of sample cell

Table 3-3 Measurement conditions of spectrophotometer

Model	JASCO V-550
Band width	5.0 nm
Response	Medium
Measurement range	850 - 780 nm
Data pitch	0.2 nm
Scanning speed	40 nm min^{-1}
No. of cycle	1
Reference	3 M HNO_3

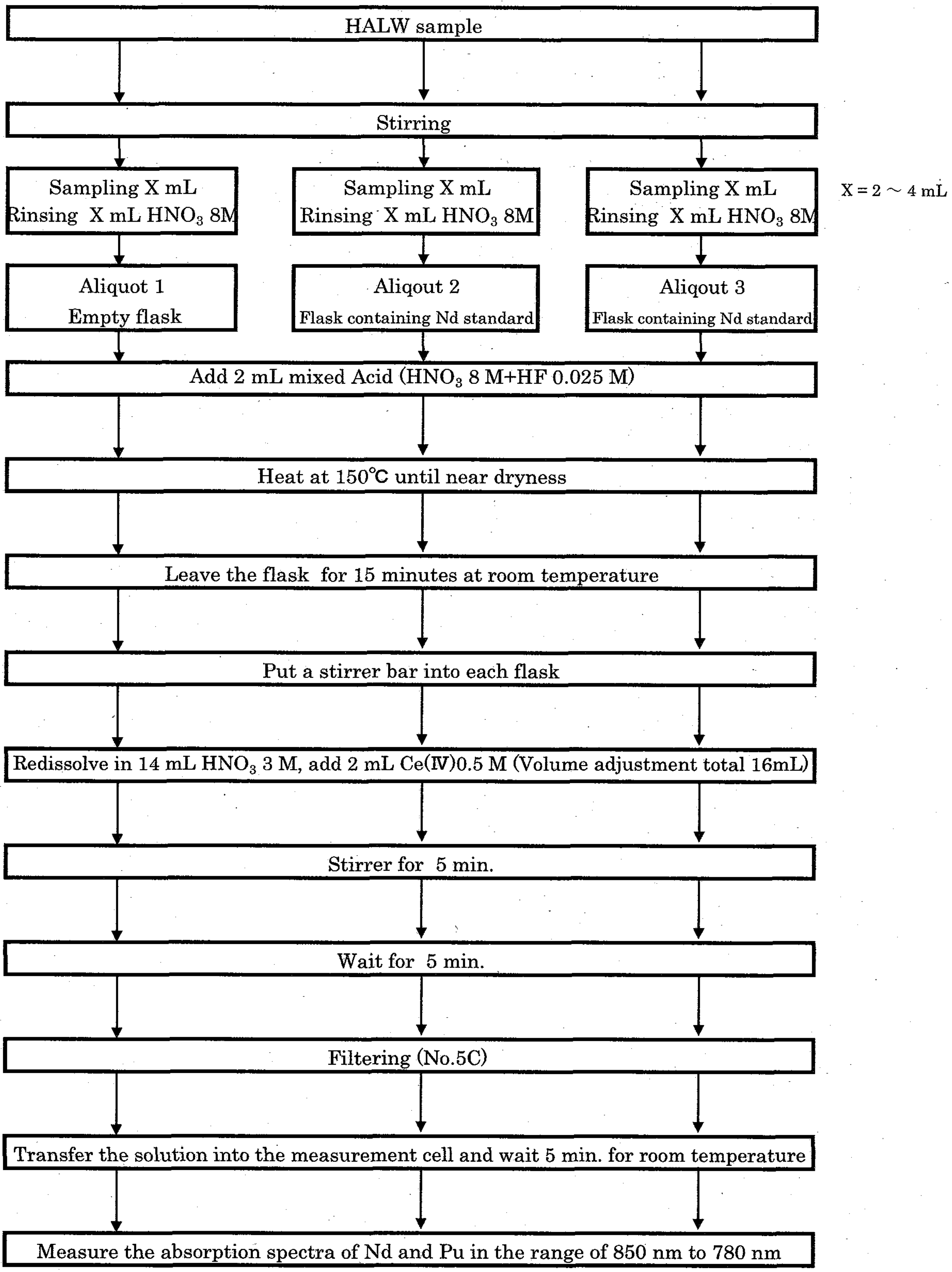


Fig. 3-3 Analytical procedure of plutonium in HALW using Nd-internal standard method

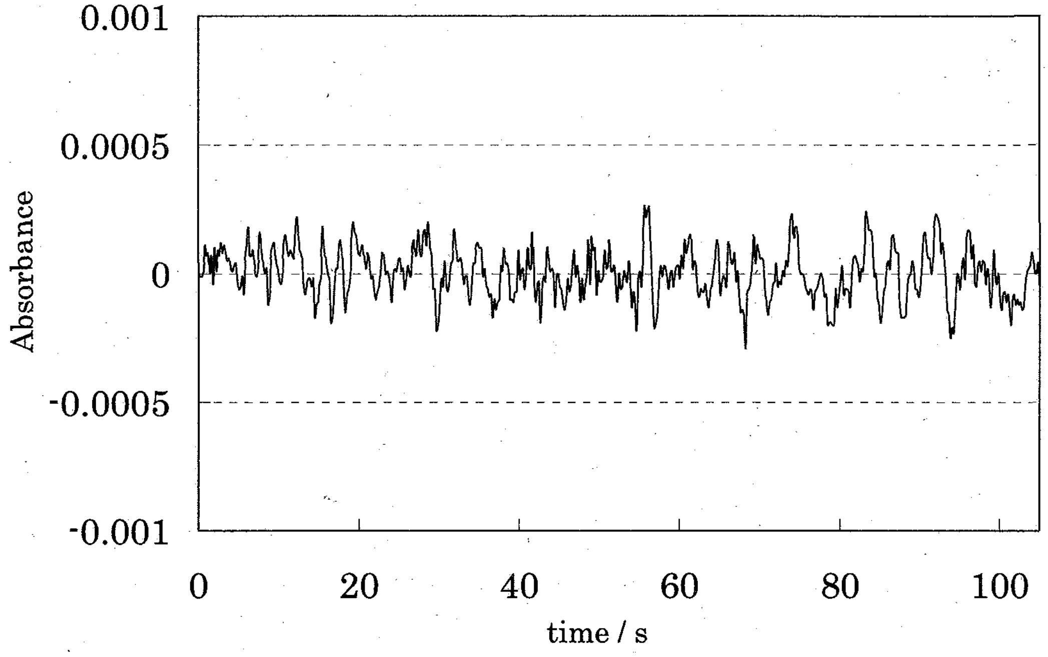


Fig. 4-1 Absorption spectrum of blank for 3 M HNO_3 ranging from 0 s to 100 s at 830 nm. Slit width: 5.0 nm, Data interval: 0.2 s.

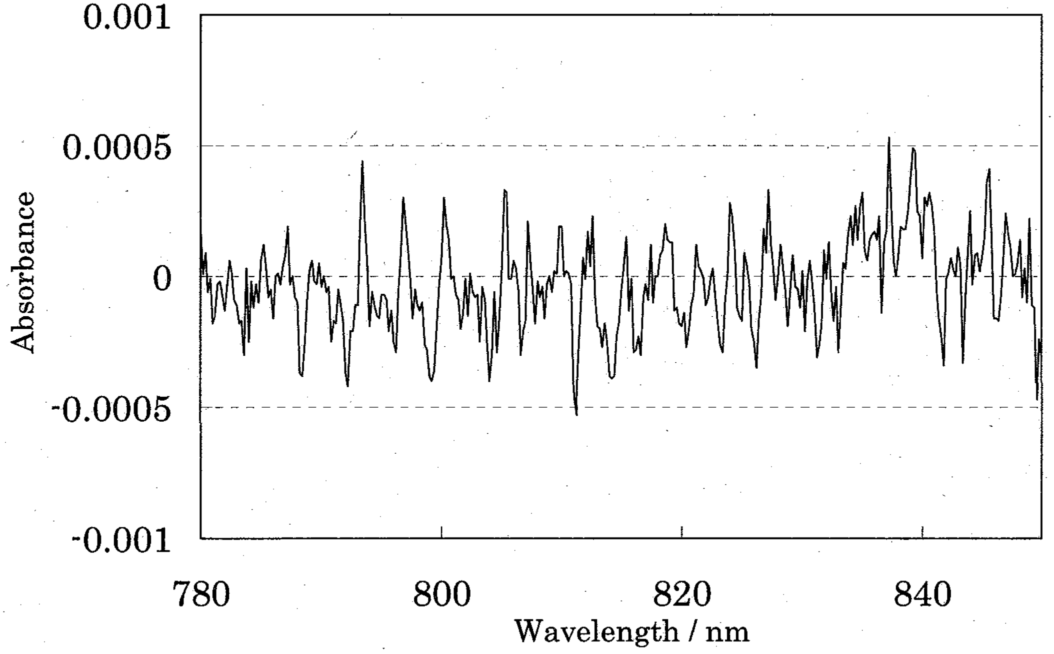


Fig. 4-2 Absorption spectrum of blank for 3 M HNO_3 ranging from 850 nm to 780 nm. Slit width: 5.0 nm, Scanning speed: 40 nm min^{-1}.

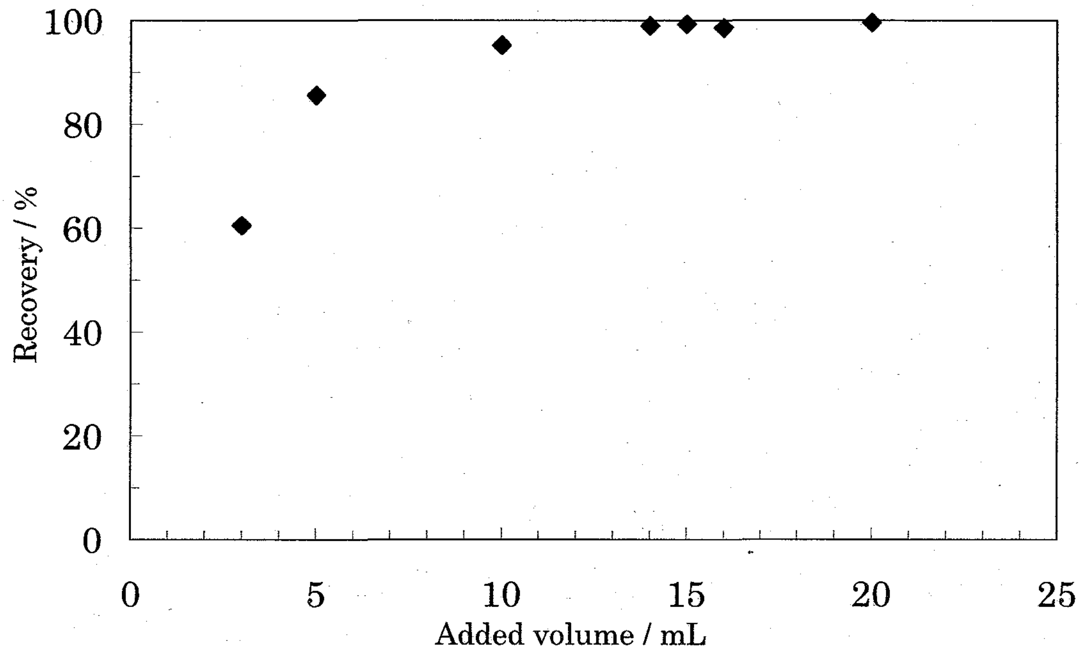


Fig. 4-3 Recovery of neodymium(III) solution in flow type optical cell. Recovery/% = [Measured absorbance at 795 nm when a certain volume of neodymium(III) solution was added to the sample cell] / [Measured absorbance at 795 nm when 30 mL of neodymium(III) solution was added to the sample cell] × 100. Neodymium(III) solution was 2.5 g L^{-1}.

Table 4-1 Effect of slit width on plutonium(VI) and neodymium(III) absorbance in HALW

Slit width	0.2 nm	1.0 nm	5.0 nm
Absorbance of Pu(VI)	0.0110 ± 0.0001	0.0076 ± 0.0007	0.0067 ± 0.0001
RSD % 1), 2)	(24.7 %)	(15.4 %)	(1.2 %)
Absorbance of Nd(III)	0.0115 ± 0.0018	0.0049 ± 0.0004	0.0042 ± 0.0002
RSD % 1), 2)	(8.6 %)	(8.0 %)	(5.4 %)

1) Relative standard deviation; 2) n = 3

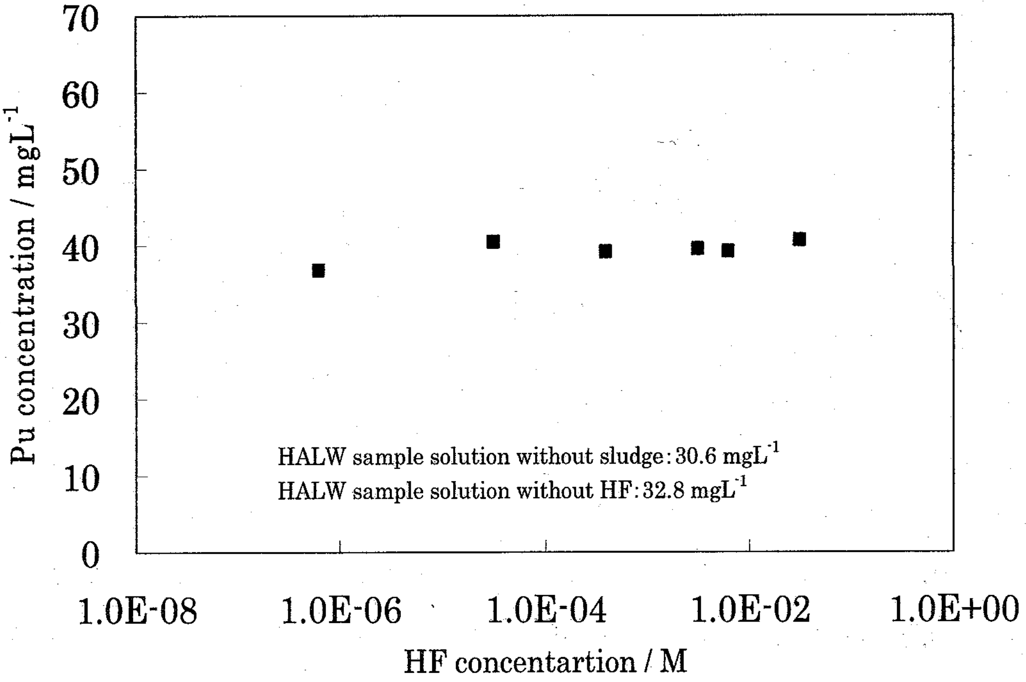


Fig. 4-4 Relation between plutonium concentration and hydrofluoric acid concentration. Nitric acid concentration was 6.3 M. The sample was heated at 150 ℃ to near-dryness after addition of hydrofluoric acid.

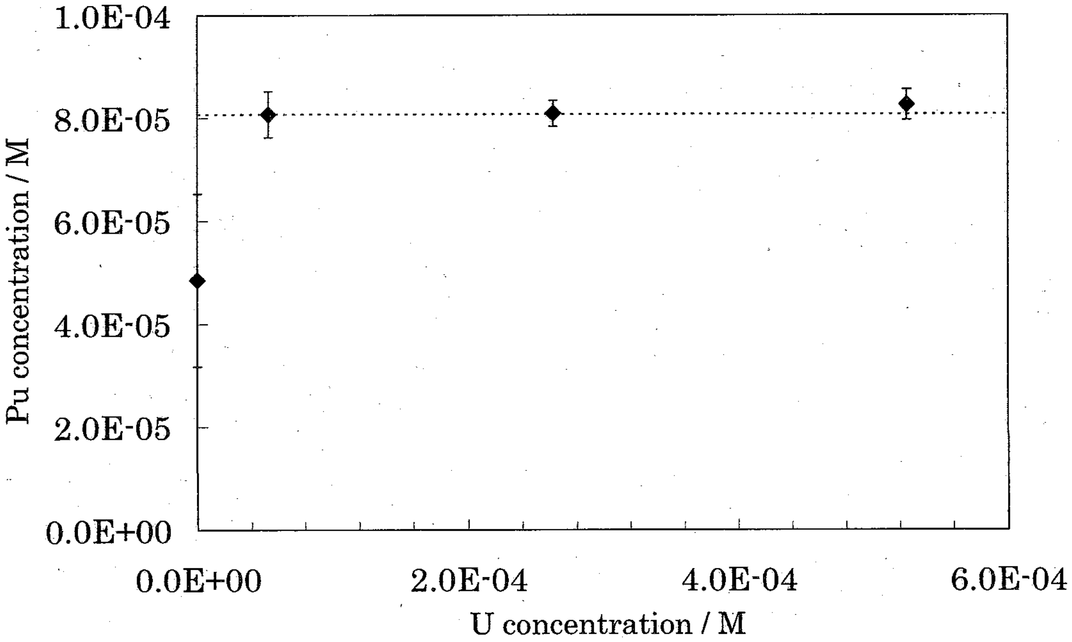


Fig. 4-5 Effect of uranium addition on plutonium measurement with hydrofluoric acid. The dotted line shows the taken value of plutonium concentration. The sample solution was heated to near-dryness after the hydrofluoric acid concentration was adjusted to 3×10^{-3} M.

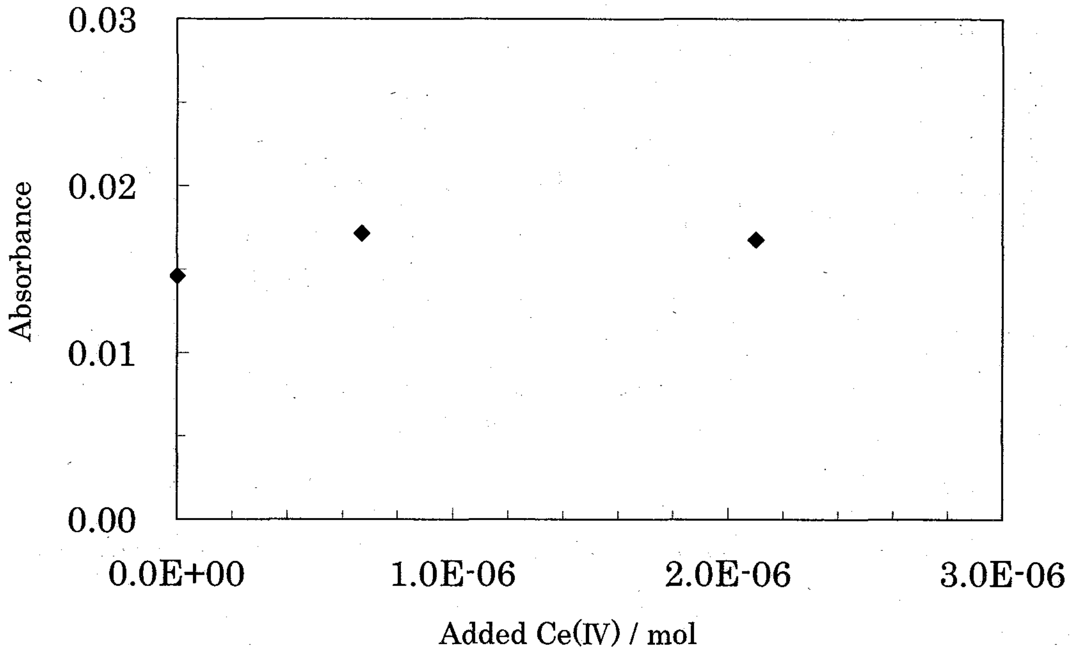


Fig.4-6 Effect of cerium(IV) on the oxidation of plutonium in HALW. Measurement wavelength was 830 nm.

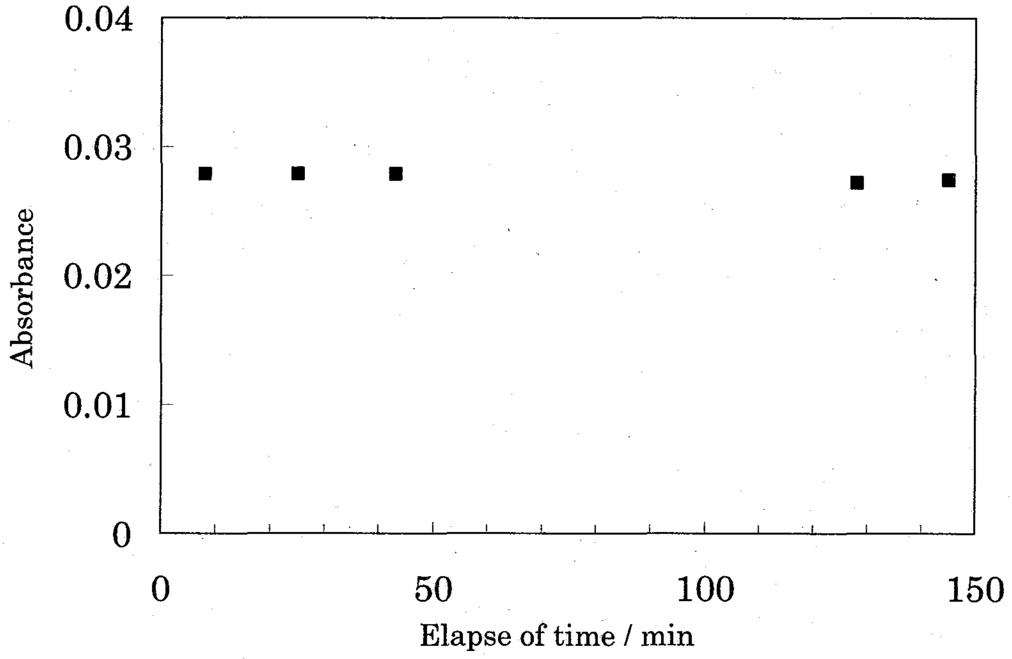


Fig.4-7 Stability of the hexavalent state of plutonium in HALW with cerium(IV).

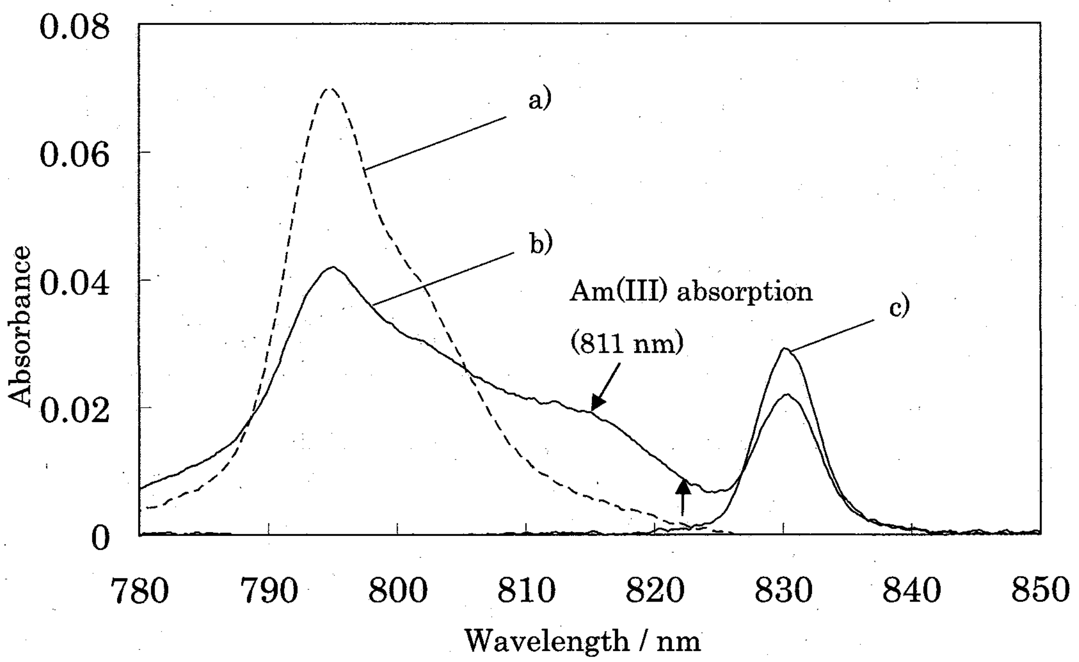
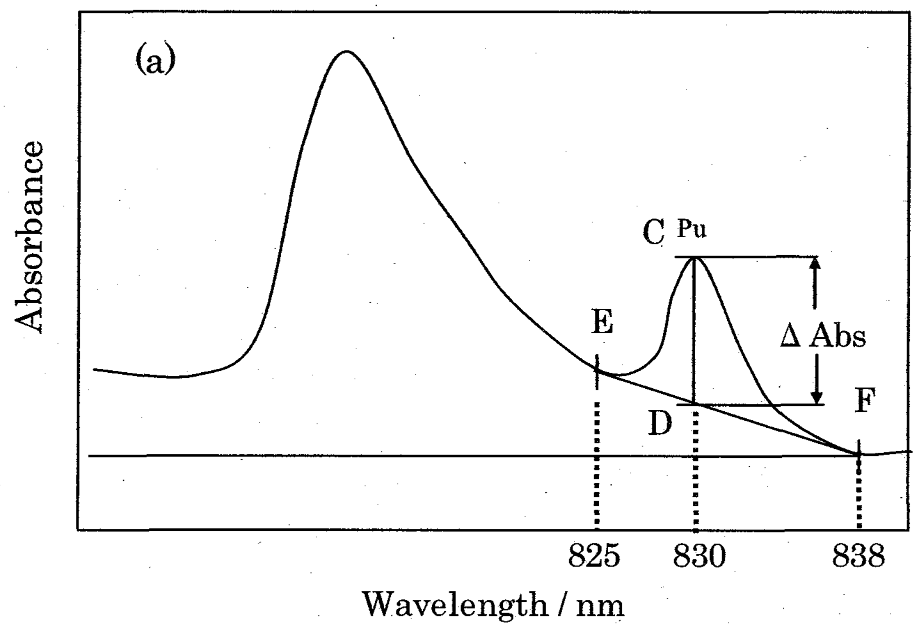


Fig.4-8 Comparison of absorption spectra of a) 1.3 g L^{-1} neodymium(III), b) HALW with cerium(IV), c) 0.025 g L^{-1} plutonium(VI) in 3 M HNO_3



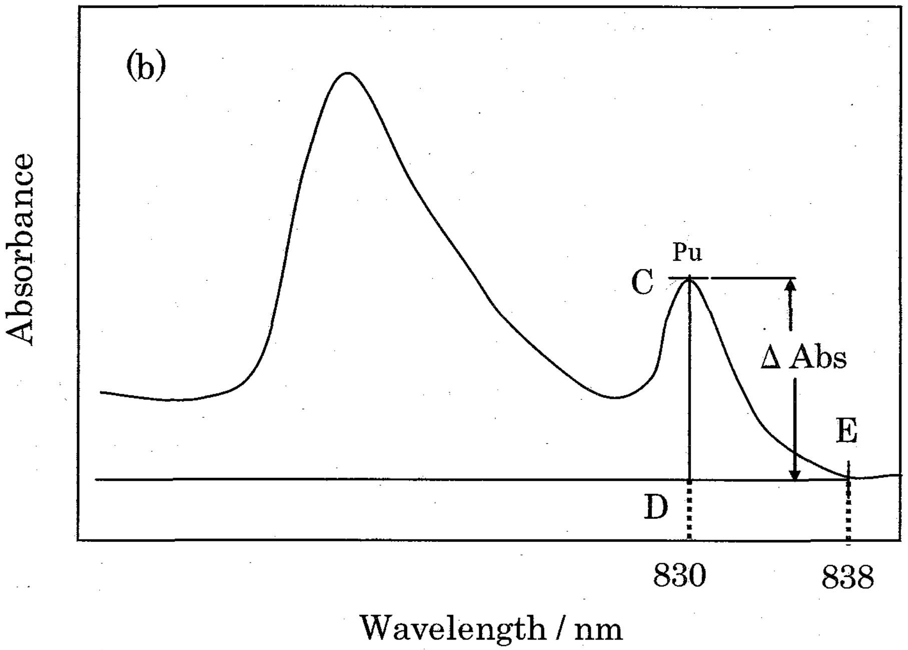


Fig.4-9 Determination of plutonium(VI) absorbance and neodymium(III) absorbance using (a) three-point method and (b) two-point method.

Three-point method designates 3 points to determine the baseline and two-point method designates 2 points to determine the baseline.

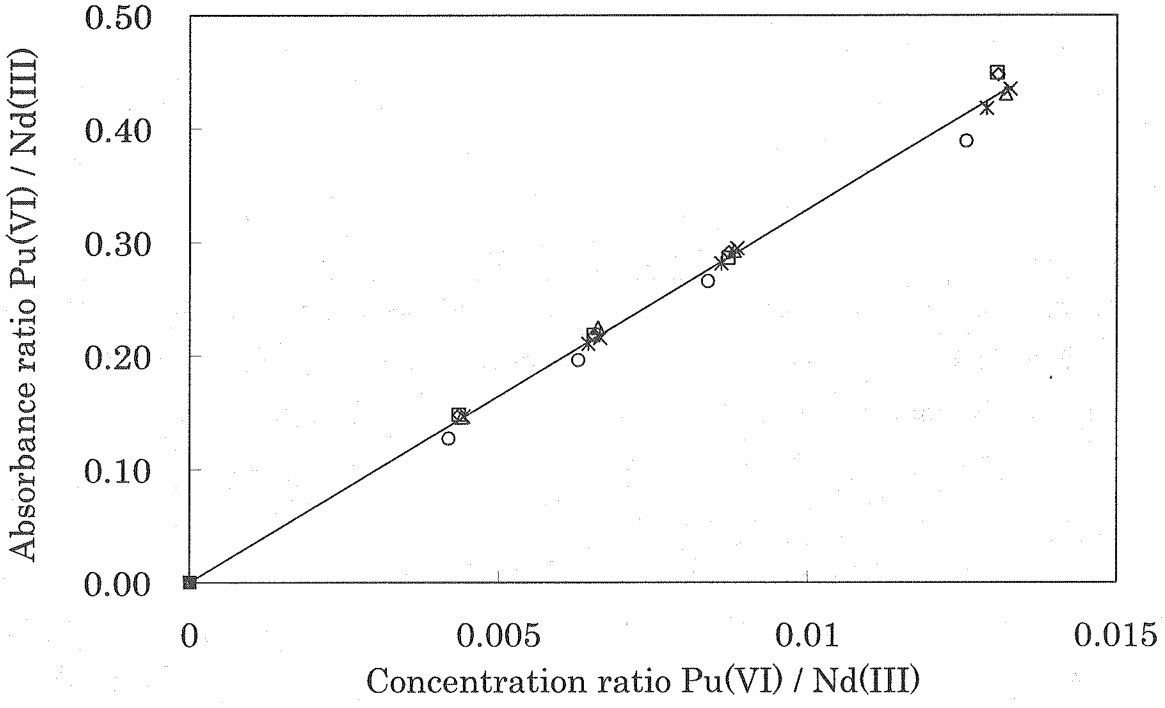


Fig.4-10 Relation between plutonium(VI) / neodymium(III) concentration ratio and plutonium(VI) / neodymium(III) absorbance ratio.

Table 4-2 Influence of added amount of neodymium(III) as an internal standard on plutonium concentration in HALW.

Taken neodymium(III) / mg	Found plutonium [1] / mg L^{-1}	SD [2] / mg L^{-1}	RSD [3] / %
15	13.3	0.51	3.8
30	13.0	0.02	0.1
50	13.3	0.17	1.3

1) n=3; 2) Standard deviation; 3) Relative standard deviation

Table 4-3 Analytical results for standard solution using Nd-internal standard method.

Sample No.	Taken plutonium 1) / mg L-1	Found plutonium 2), 3) / mg L-1	Difference 4) / %
1	3.67	3.96	7.6
2	9.18	9.00	2.0
3	18.36	18.00	2.0

1) Neodymium(III) concentration was 0.01 M in each samples; 2) n=2; 3) neodymium(III) as an internal standard concentration was 0.01 M; 4) Difference%= | (Found)-(Taken) | / (Taken) × 100

Table 4-4 Analytical results of plutonium for HALW using Nd-internal standard method and calibration curve method.

Sample No	Determination method	
	Calibration curve method	Nd-internal standard method 1)
n = 1	19.6	22.1
n = 2	21.2	22.3
n = 3	20.8	21.3
n = 4	20.2	20.9
n = 5	20.4	21.1
Average / mg L-1	20.6	21.5
SD 2) / mg L-1	1.0	0.6
RSD 3) / %	4.7	2.9

1) Amount of neodymium(III) as an internal standard was 20 mg; 2) Standard deviation; 3) Relative standard deviation.

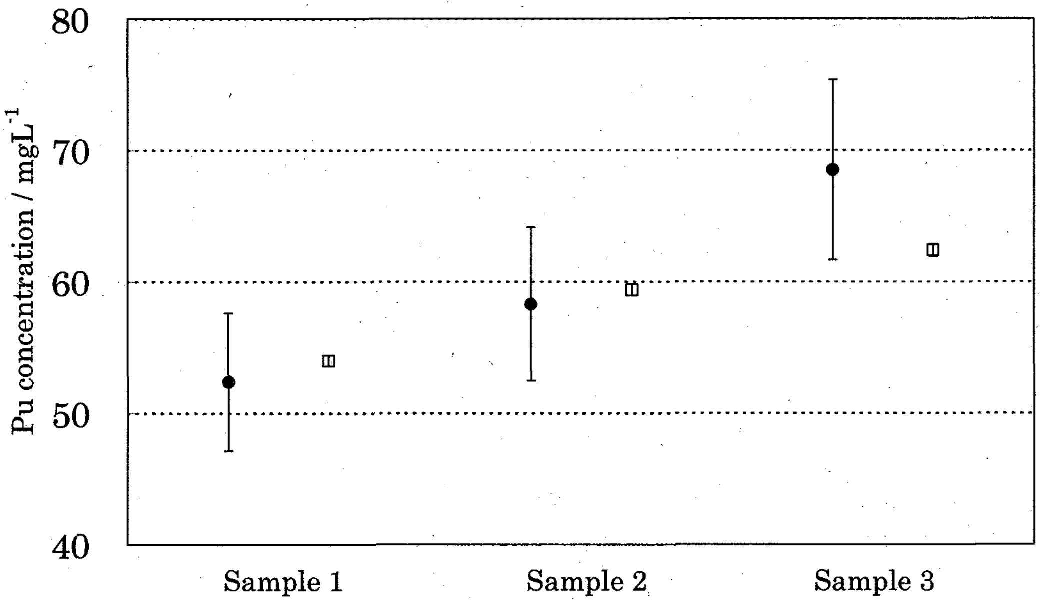


Fig.4-11 Comparison of Nd-internal standard method and IDMS. ●: Spectrophotometry using Nd as an internal standard, □: IDMS; Error bar shows expanded uncertainty (Coverage factor $k = 2$).

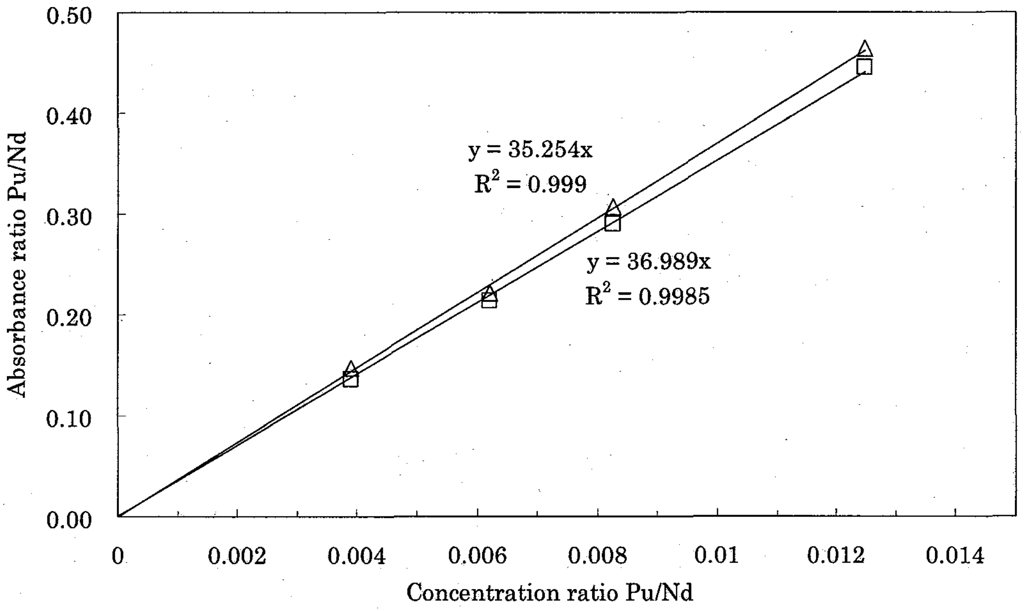


Fig.4-12 Relation between plutonium(VI) / neodymium(III) concentration ratio and plutonium(VI) / neodymium(III) absorbance ratio. Peak heights of plutonium(VI) and neodymium(III), △ : Two-point-method, □ : Three-point method

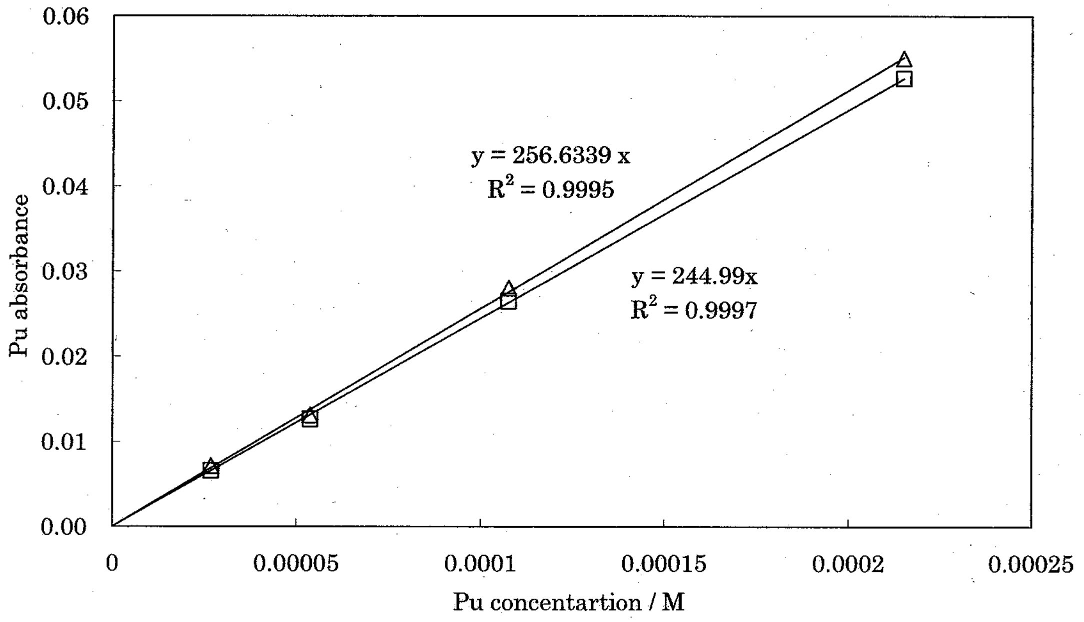


Fig. 4-13 Relation between plutonium(VI) concentration and plutonium(VI) absorbance. Peak heights of plutonium(VI) and neodymium(III): △ : Two-point-method, □ : Three-point method

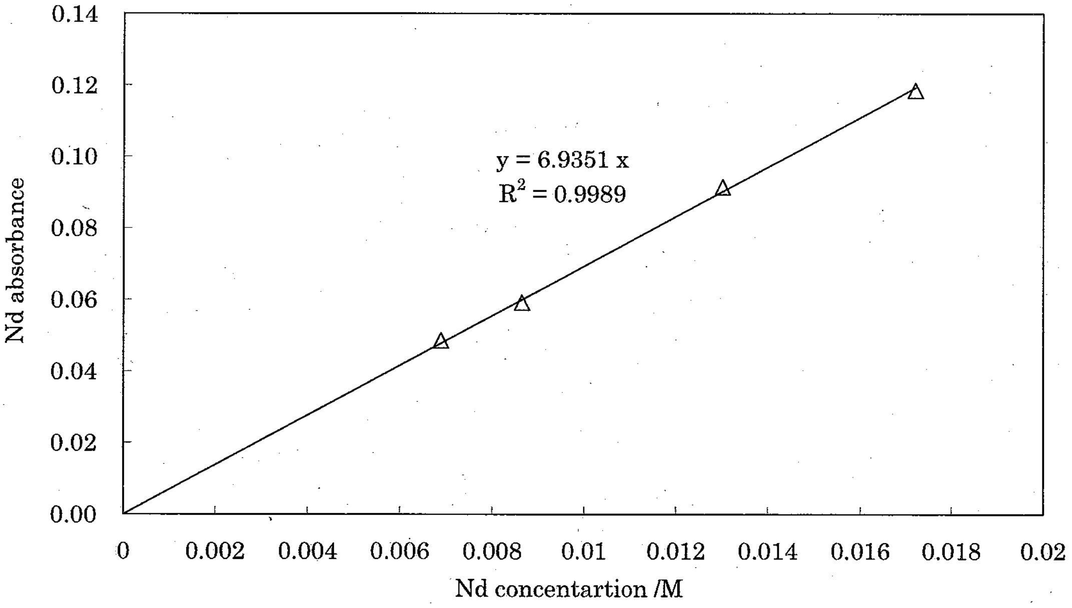


Fig. 4-14 Relation between neodymium(III) concentration and neodymium(III) absorbance . Peak heights of plutonium(VI) and neodymium(III): Two-point method

Table 4-5 Analytical results for HALW using Nd-internal standard method by two-point method at acceptance test

Peak height : Two-point method

Sample-1

Method	Lab.		Quantification	Average / mg L^{-1}	SD [1] / mg L^{-1}	RSD [2] / %	Difference / mg L^{-1}		Difference / (X) / %
	JNC	(X)		152.0	0.6	0.4			
IDMS	NMCC	(Y)		154.2			(X)-(Y)	-2.2	-1.4
	SAL	(Z)		152.9	1.5	1.0	(X)-(Z)	-0.9	-0.6
		(A)	Nd-intrnal standard method	161.5	4.1	2.5	(A)-(X)	9.5	6.3
Spectrophotometry	JNC	(B)	Calibration curve method [3]	158.1	5.7	3.6	(A)-(Y)	6.1	4.0
		(C)	Calibration curve method [4]	159.1	2.1	1.3	(A)-(Z)	7.1	4.7

Sample-2

Method	Lab.		Quantification	Average / mg L^{-1}	SD [1] / mg L^{-1}	RSD [2] / %	Difference / mg L^{-1}		Difference / (X) / %
	JNC	(X)		379.1	2.2	0.6			
IDMS	NMCC	(Y)		386.2			(X)-(Y)	-7.1	-1.9
	SAL	(Z)		369.3	17.7	4.8	(X)-(Z)	9.8	2.6
		(A)	Nd-intrnal standard method	345.5	0.9	0.2	(A)-(X)	-34	-8.9
Spectrophotometry	JNC	(B)	Calibration curve method [3]	345.4	11.0	3.2	(A)-(Y)	-34	-8.9
		(C)	Calibration curve method [4]	335.1	5.0	1.5	(A)-(Z)	-44	-12

1) Standard deviation; 2) Relative standard deviation; 3) HALW sample with Nd standard; 4) HALW sample without Nd standard

Table 4-6 Analytical results for HALW using Nd-internal standard method by three-point method at acceptance test

Peak height : Three-point method

Sample-1

Method	Lab.		Quantification	Average / $\mathrm{mg\ L^{-1}}$	SD 1) / $\mathrm{mg\ L^{-1}}$	RSD 2) / %	Difference / $\mathrm{mg\ L^{-1}}$		Difference / (X) / %
IDMS	JNC	(X)		152.0	0.6	0.4			
	NMCC	(Y)		154.2			(X)-(Y)	-2.2	-1.4
	SAL	(Z)		152.9	1.5	1.0	(X)-(Z)	-0.9	-0.6
Spectrophotometry	JNC	(A)	Nd-intrnal standard method	136.0	7.3	5.3	(A)-(X)	-16	-11
		(B)	Calibration curve method 3)	139.0	3.8	2.7	(A)-(Y)	-13	-8.6
		(C)	Calibration curve method 4)	136.2	1.8	1.3	(A)-(Z)	-16	-10

Sample-2

Method	Lab.		Quantification	Average / $\mathrm{mg\ L^{-1}}$	SD 1) / $\mathrm{mg\ L^{-1}}$	RSD 2) / %	Difference / $\mathrm{mg\ L^{-1}}$		Difference / (X) / %
IDMS	JNC	(X)		379.1	2.2	0.6			
	NMCC	(Y)		386.2			(X)-(Y)	-7.1	-1.9
	SAL	(Z)		369.3	17.7	4.8	(X)-(Z)	9.8	2.6
Spectrophotometry	JNC	(A)	Nd-intrnal standard method	319.3	2.0	0.6	(A)-(X)	-60	-16
		(B)	Calibration curve method 3)	323.9	12.4	3.8	(A)-(Y)	-55	-15
		(C)	Calibration curve method 4)	311.8	3.4	1.1	(A)-(Z)	-67	-18

1) Standard deviation; 2) Relative standard deviation; 3) HALW sample with Nd standard; 4) HALW sample without Nd standard

Appendix 1

Verification analysis using Nd-internal standard method

-Operating Manual-

Tokai Reprocessing Plant
JAEA

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CONTENTS

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1. General

1.1 Scope

This procedure describes the determination of the plutonium concentration in HALW by spectrophotometry.

The procedure applies to the analysis of solutions containing less than 400 mg/L and more than 50 mg/L of plutonium.

1.2 Principle and Outline of the Procedure

Plutonium concentration in HALW sample is determined using Nd as an internal standard, so called Nd-internal standard method. Sludge in HALW sample is dissolved in mixture of HNO_3 and HF. Subsequently plutonium is quantitatively oxidized to plutonium (VI) in 3M HNO_3 using a Ce(IV) nitrate solution before spectrophotometric measurement. The plutonium concentration in HALW can be calculated using the molar extinction coefficient ratio of Nd (III) and Pu (VI), $\varepsilon_{Nd}/\varepsilon_{Pu}$, and the absorbance ratio of Pu (VI) and Nd (III), $(A_{Pu})_Y/(A_{Nd})_Y$ in HALW sample that a known quantity of Nd is being added. The amount of Nd initially existing in HALW is corrected with the absorbance ratio, $(A_{Pu})_X/(A_{Nd})_X$, in HALW sample without Nd spike.

2 Apparatus and reagents

2.1 Instruments

- Spectrophotometer – Jasco V-550 single monochromator
- LAP TOP Computer
- Evaluation Software—Jasco V series spectra manager Ver.1.53N
- Measurement cell, flow type, quartz, 1cm
- Bundle optical fibers (core diameter 230 μm, cladding diameter 250 μm)
- Thermometer
- 50 mL Erlenmeyer flask
- Automatic burette
- Sampling device to take HALW aliquot
- Hot plate
- Filter paper (No.5C) and funnel

2.2 Reagents

- A plutonium stock solution ($0.2\ gL^{-1}$) prepared by dissolving a large size dry spike (LSD) containing 2 mgPu in 3 M HNO_3
- 0.5 M Cerium nitrate - $Ce(NH_4)_2(NO_3)_6$ in 3 M HNO_3
- 3 M HNO_3

- 8 M HNO_3
- Nd_2O_3
- 8 M HNO_3 + 0.025 M HF

2.3 Measurement Conditions

- Band width : 5.0 nm
- Scanning speed : 40 nm/min
- Data pitch : 0.2 nm
- Scanning wavelength- range : 780 -850 nm
- No. of cycle : 1

3 Sample Preparation

3.1 Standard solutions containing known amount of Nd and Pu for the determining the calibration factor

Operator (JNC) activities	Inspector (IAEA, JSGO) activities	Location
1) Write down LSD spike No. and Nd spike flasks No.	Prepare a LSD spike and a set of four Nd spikes.	
2) Introduce the LSD spike into G.Box No.1 (Bag in). Introduce the Nd spike flasks into hot cell No.5C (bag in).	Check the LSD spike No. Check the Nd spike No.	G105 No.1 G.Box G105 No.5C Cell
3) Transfer the Nd spikes flasks to No.4.		G105 No.5C – No.4
4) Remove aluminum cap from the LSD spike.	I/O (Inspector Observation)	G105 No.1 G.Box
5) Transfer the LSD spike by pneumatic tube to No.5C and transfer to No.5B.		G105 No.1 – No.5C – No.5B
6) Remove rubber cap from the LSD vial.		G105 No.5B
7) Put a stirrer bar into the LSD vial.		G105 No.5B
8) Transfer the LSD vial to No.4.		G105 No.5B – No.4
9) Add 10 ml 3 M HNO_3 by dispenser (Automatic burette). (Red Line).		G105 No.4
10) Transfer the LSD vial to No. 5A.		G105 No.4 – No.5A
11) Stir for 5 min. to completely dissolve the LSD spike.	Check the completeness of the dissolution	G105 No.5A
12) Transfer the LSD spike solution to No.4.	I/O	G105 No.5A – No.4
13) Add 0.5 mL, 1.0 mL, 2.0 mL and 4.0 mL of the solution to each Nd spike flask by sampling device (White line).		G105 No.4

14) Add 2 mL of HNO_3 8 M + HF 0.025 M using dispenser. (Green Line).		
15) Heat them on a hot plate at 150^0 C until near dryness.	Check the temperature > 150 ^{0} C.	
16) Stop heating and leave the flasks for a few minutes.	Check the dryness.	G105 No.4
17) Put a stirrer bar into the flask.	I/O	
18) Add 14mL HNO_3 3M by dispenser (Automatic burette). (Red Line).	Check the dissolution.	
19) Add 2 mL $Ce(NH_4)_2(NO_3)_6$ 0.5 M by dispenser. (Yellow Line).	I/O	
20) Stir for 5 min.		G105 No.4 – No.5A
21) Wait for 5min.		G105 No.5a – No.4
22) Filter the solution through a filter paper (No.5C).	Check the flask No.	
23) Transfer the filtered solutions in flask to Cell No.5A for spectrophotometric measurement. Pu peak top (around 830 nm) Nd peak top (around 795 nm)	I/O	G105 No.4 – No.5A

3.2 HALW sample

Operator (JNC) activities	Inspector (IAEA, JSGO) activities	Location
1) Transfer the flask containing HALW sample to No.4.	Check the sample No.	G105 No.1 – No.4
2) Take HALW sample 2 to 4 mL (Sampling volume depends on the sample conditions such as Pu concentration.) into a flask containing Nd spike (n=2) by sampling device. Take HALW sample 2 to 4 mL into an empty flask (n=1) by sampling device. (White Line). Taken immediately after mixing.	Check the flask No. I/O (Inspector Observation)	G105 No.4
3) Add 2 mL of HNO_3 8 M + HF 0.025 M by dispenser. (Green Line).		
4) Heat them on a hot plate at 150^0 C until near dryness.	Check the temperature >150 ^{0} C.	
5) Stop heating and leave the flasks for 15 minutes.	Check the dryness.	G105 No.4
6) Put a stirrer bar into each flask.	I/O	
7) Add 14mL HNO_3 3M by dispenser (Automatic burette). (Red Line).	Check the dissolution.	
8) Add 2 mL $Ce(NH_4)_2(NO_3)_6$ 0.5 M by dispenser. (Yellow Line).		
9) Stir for 5 min.	I/O	
10) Wait for 5 min.		
11) Filter the solution through a filter paper (No.5C) to remove particles.	Check the flask No.	
12) Transfer the filtered solutions	I/O	G105 No.4 – No.5A

in flask to cell No.5A for spectrophotometric measurement. Pu peak top (around 830 nm) Nd peak top (around 795 nm)		

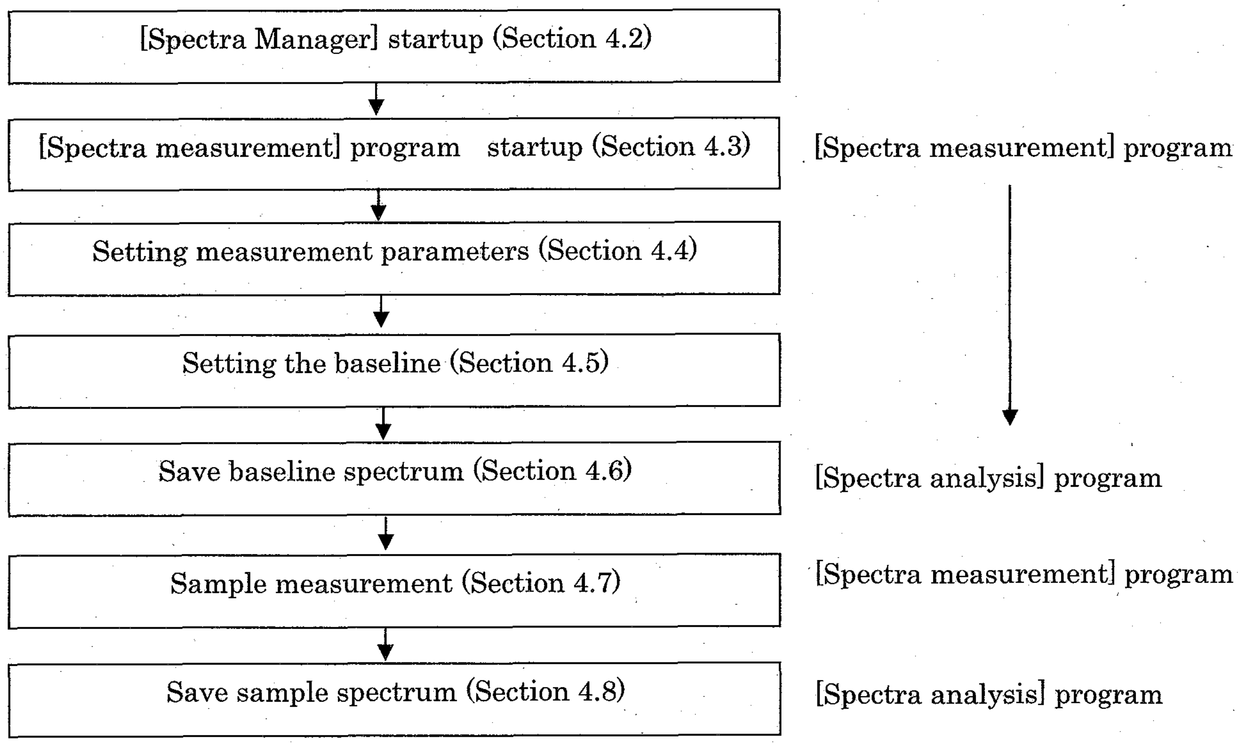
4. Spectrum Measurement

This section describes the procedures for starting the “Spectrum Measurement program”, measuring standard samples, saving measured spectra.

4.1 Procedural overview

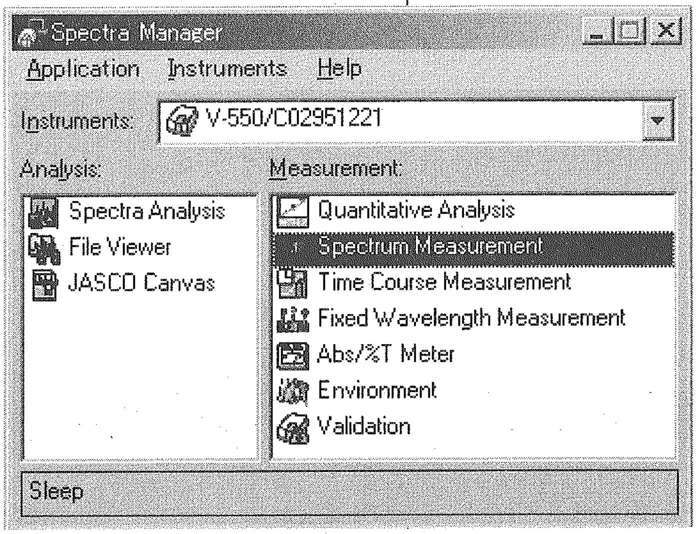
The [Spectrum measurement] program measures sample spectra for a set of measurement parameters. It also does baseline measurement for correcting sample spectra. Spectra cannot be printed or saved in the [Spectrum Measurement] program. [Spectrum measurement] automatically starts the [Spectra Analysis] program and the spectra are displayed in the active view. Spectra can be saved and printed in the [Spectra Analysis] program.

<Procedural overview>



4.2 [Spectra Manager] startup

1) Select [Jasco]-[Spectra Manager] (or double-click)	The [Spectra Manager] dialog box appears.



Instruments: V-550/C02951221

Sleep

Fig. 4.1 [Spectra Manager] dialog box

4.3 [Spectrum measurement] program startup

1) Double-click [Spectrum Measurement] in the [Spectra Manager] dialog box.	The program starts and the following dialog box appears.

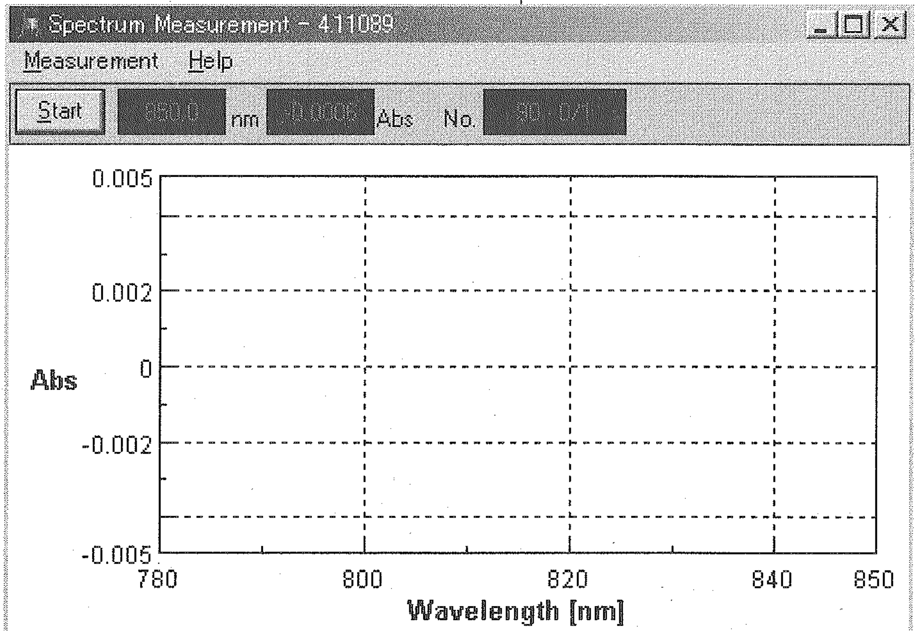
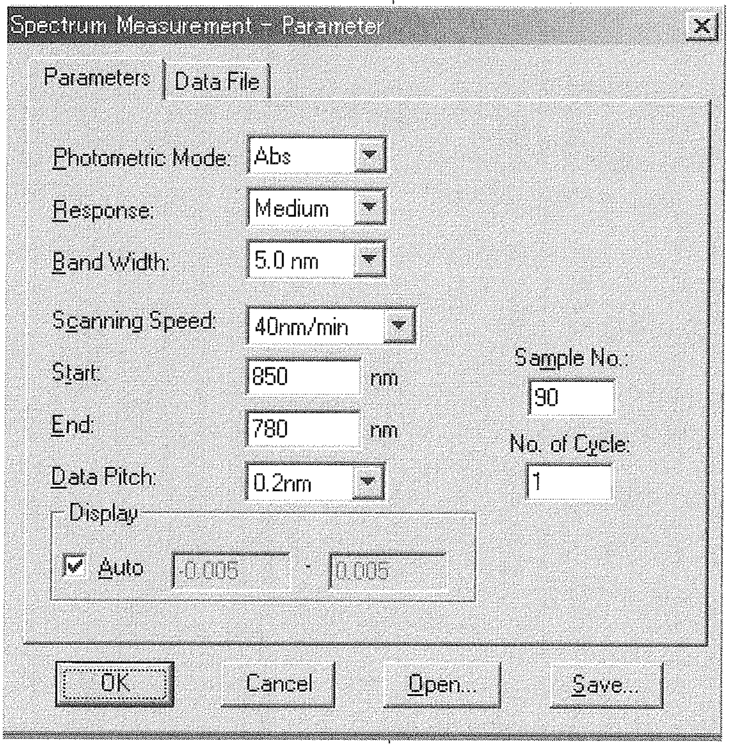


Fig. 4.2 [Spectra Measurement] dialog box

4.4 Setting measurement parameters

1) Select [Measurement]-[Parameters] in the [Spectra Measurement] dialog box.

The following dialog box appears.



Spectrum Measurement - Parameter

Parameters | Data File

Photometric Mode: Abs
Response: Medium
Band Width: 5.0 nm
Scanning Speed: 40nm/min
Start: 850 nm
End: 780 nm
Data Pitch: 0.2nm
Display
☑ Auto -0.005 - 0.005
Sample No.: 90
No. of Cycle: 1

Fig. 4.3 [Spectra Measurement-Parameter] dialog box

2) Input parameters (①) or reload parameters from parameters list (②).

① Input following measurement Parameters in the [Spectrum measurement-Parameter] dialog box.

[Parameters]

Photometric Mode :Abs.
Response :Medium
Band Width : 5.0 nm
Scanning Speed : 40 nm/min
Start : 850 nm
End : 780 nm
Data Pitch : 0.2 nm
No. of Cycle : 1
Display : Auto

② Reload the parameters from parameter list in the [Parameters-Open] dialog box.	The parameters in the [Parameters-Open] dialog box was inputted and saved in advance.
a) Click <Open> in the [Spectra Measurement-Parameter] dialog box.	The following dialog box appears.

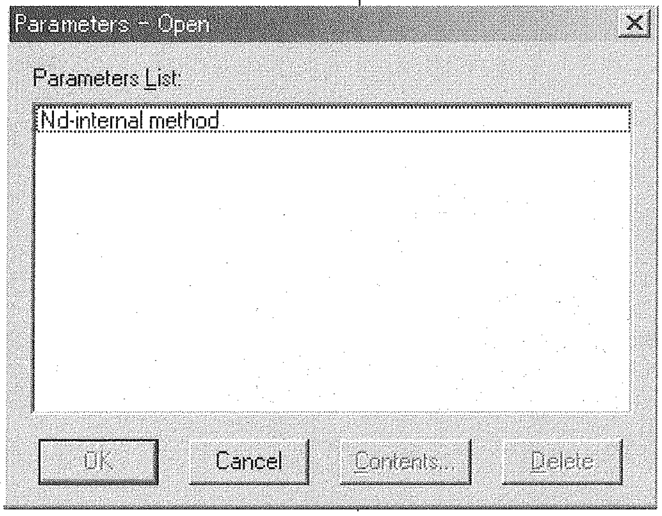
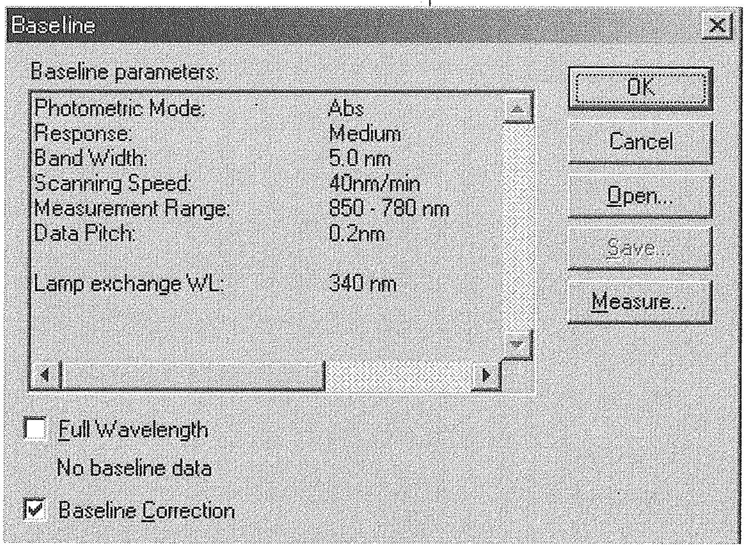


Fig. 4.4 [Parameters-Open] dialog box

b) Select parameter name (e.g. [Nd-internal method]) from "Parameters List" in the [Parameters-Open] dialog box.	
c) Click <OK> in the [Parameters-Open] dialog box.	Closes the [Parameters-Open] dialog box.
3) Click <OK> in the [Spectra Measurement-Parameter] dialog box.	The parameters transfer to the spectrophotometer.

4.5 Setting the baseline

The baseline defines the "0" absorbance level. The baseline value is subtracted from the measured data in order to determine the correct spectrum of sample. The baseline is inherent to each instrument. In order to maximize the accuracy of the spectra, the baseline must be measured under the same conditions as those used for measuring the spectra.

<table>
<tr><td>1) Select [Measurement]-[Baseline] to start the baseline measurement in the [Spectrum Measurement] dialog box.</td><td>The following dialog box appears.</td></tr>
<tr><td colspan="2">


Baseline parameters:
Photometric Mode: Abs
Response: Medium
Band Width: 5.0 nm
Scanning Speed: 40nm/min
Measurement Range: 850 - 780 nm
Data Pitch: 0.2nm
Lamp exchange WL: 340 nm
☐ Full Wavelength
No baseline data
☑ Baseline Correction

Fig. 4.5 [Baseline] dialog box
</td></tr>
<tr><td>2) Remove the check mark from [Full Wavelength] check box. Put a check mark in the [Baseline Correction] check box in the [Baseline] dialog box.</td><td></td></tr>
</table>

3) Click <Measure> in the [Baseline] dialog box.

The baseline measurement starts.

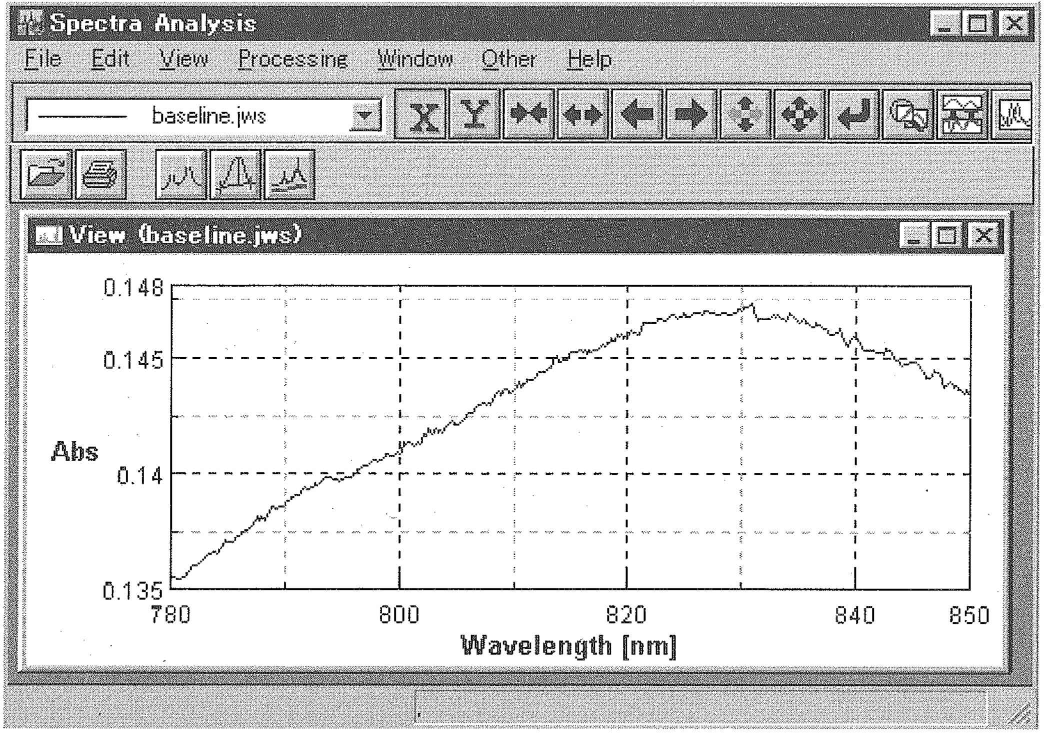


Fig. 4.6 Baseline spectrum

4) When measurement finished, the [Parameters-Save] dialog box opens. Input parameter name you want.

Baseline data is automatically transferred to the [Spectra Analysis] program and is displayed on the view.

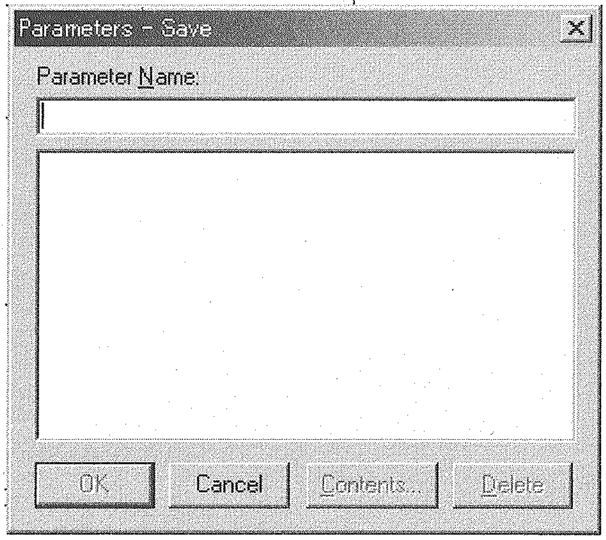


Fig. 4.7 [Parameters-Save] dialog box

5) Click <OK> in the [Parameters-Save] dialog box.	

4.6 Save baseline spectrum

When baseline measurement is finished, the [Spectra Analysis] program stars automatically and baseline spectrum is displayed in the active view.

1) Select [File]-[Save As] to display the [Save As] dialog box in the [Spectra Analysis] dialog box.	Saves the active spectrum in the active view under a new filename and location.
2) Select drive or directory from [save in] drop down list.	
3) Input the filename you want in the [File name] text box.	
4) Click <Save> to save spectrum.	

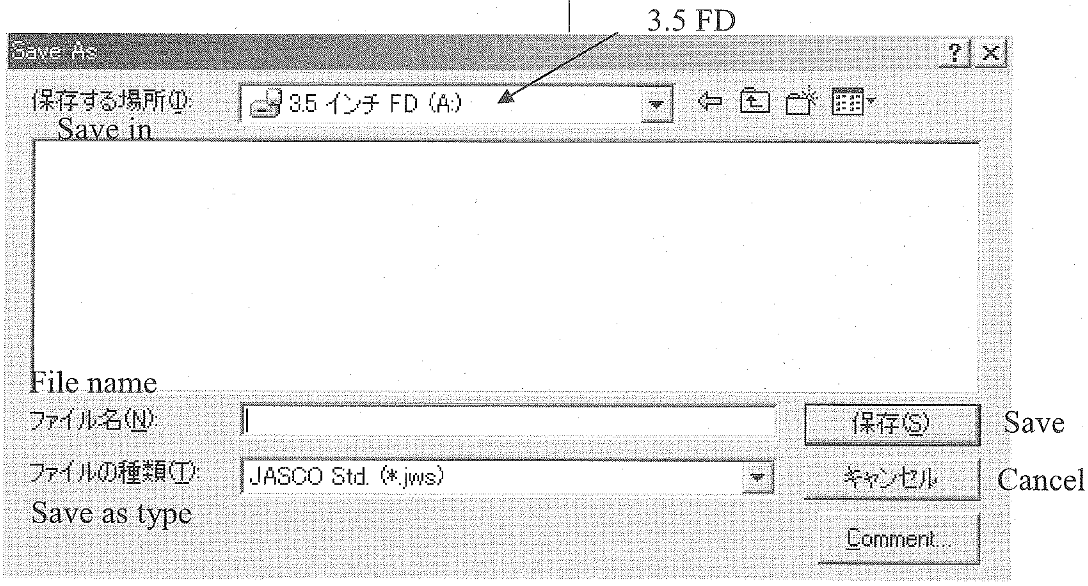


Fig. 4.8 [Save As] dialog box

4.7 Sample measurement

1) Place the sample in the measurement cell	
2) Select [Measurement]-[Auto Zero] in the [Spectra Measurement] dialog box.	Sets the observation value of the current wavelength to zero.
3) Click [Measurement] -[Start] (or click on the <start> button.)	The sample is measured and the measurement progression appears. When measurement is finished, the [Spectra Analysis] program stars automatically and spectrum is displayed in the active view.

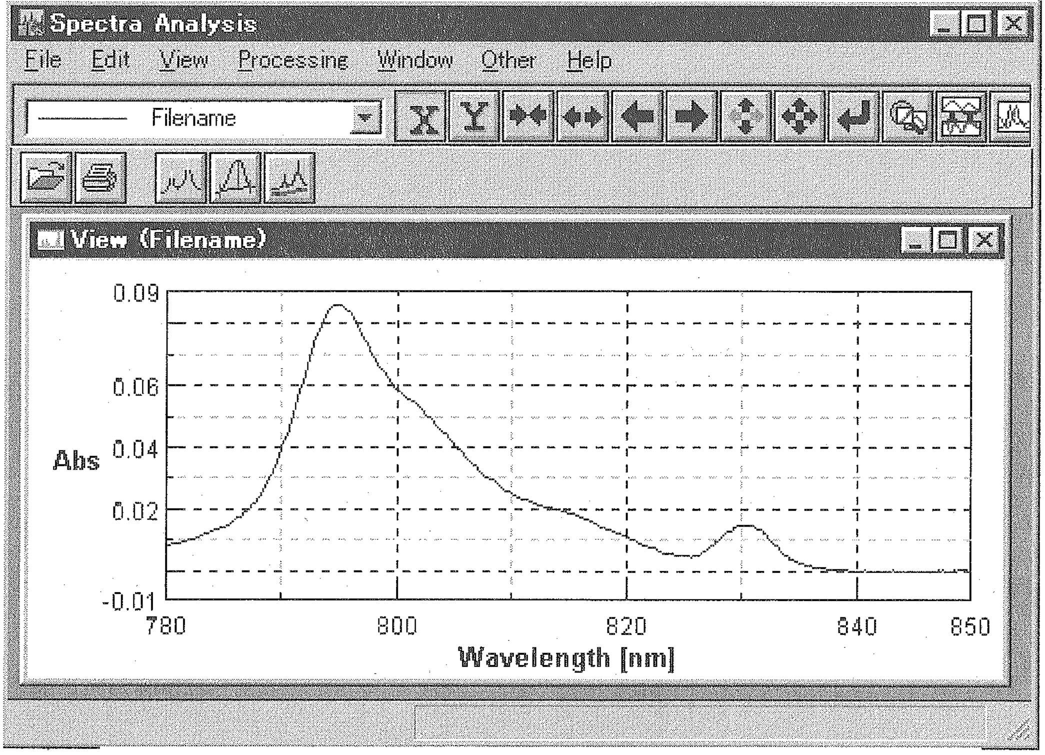


Fig. 4.9 [Spectra Analysis] dialog box (spectrum view)

4.8 Save sample spectrum

When sample measurement is finished, the [Spectra Analysis] program stars automatically and sample spectrum is displayed in the active view.

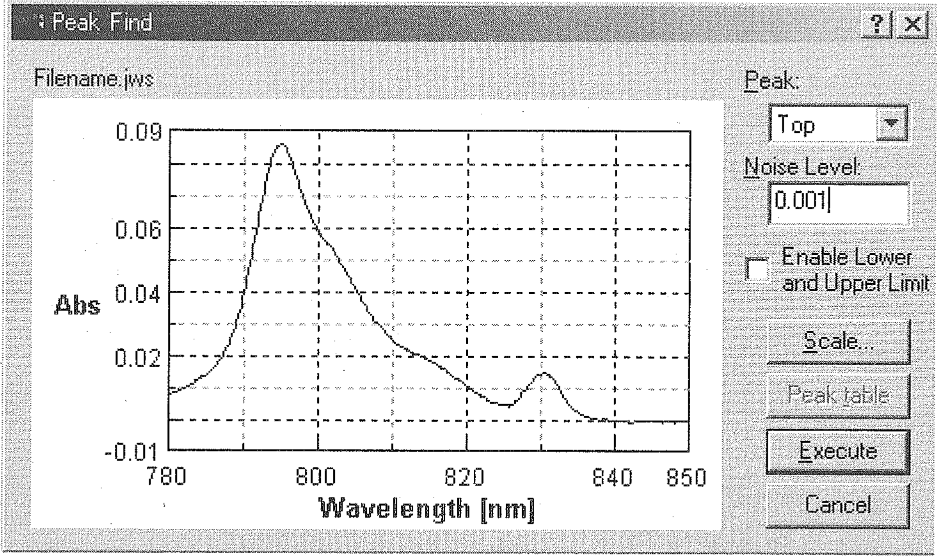
1) Repeat step 1)-4) in "4.6 Save baseline spectrum" .	

5. Spectra Analysis Program

5.1 Peak Find

This function finds spectrum peaks positions.

1) Select [Processing]-[Peak Process]-[Peak Find] (or click the button on the tool bar) to display the [Peak Find] dialog box in the [Spectra Analysis] dialog box.	Displays the [Peak Find] dialog box used to detect wavelength of spectrum peaks.



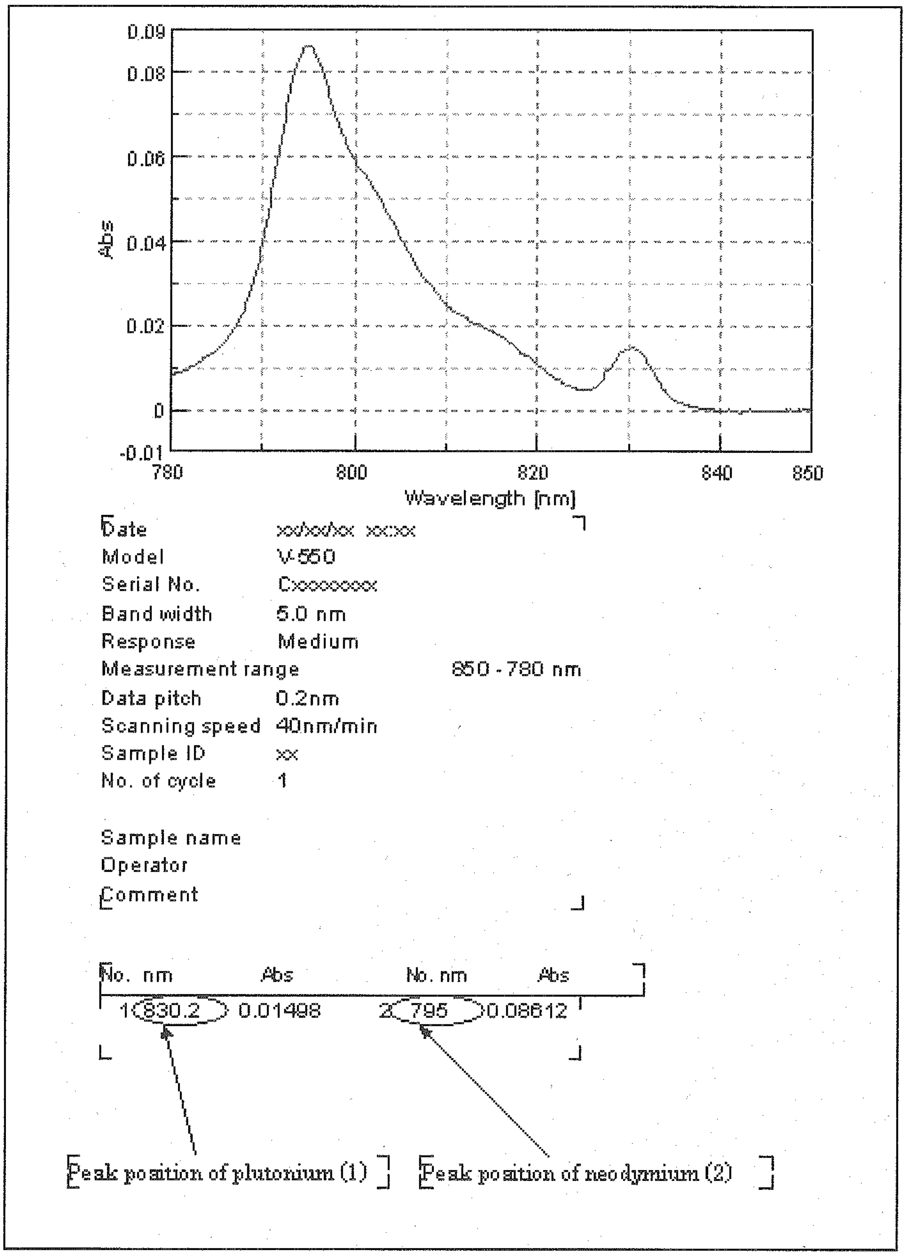
Peak: Top

Noise Level: 0.001

☐ Enable Lower and Upper Limit

Fig. 5.1 [Peak Find] dialog box

2) Select [Top] from peak drop-down list.	Lists available modes for detecting peaks/valleys.
3) Input a value 0.001 into [Noise Level] text box.	Input a threshold value used to recognize a peak. When the difference between the start of a peak to its apex does not exceed this value, the peak will not be recognized. The units are the same as those used in the active spectrum.
4) Click <Execute>.	Finds plutonium and neodymium peaks . The dialog box closes and the results of the search are displayed.
5) Click <Print>.	Prints out the peak table (Fig. 5.2).



Date	xx/xx/xx xx:xx
Model	V-550
Serial No.	Cxxxxxxxx
Band width	5.0 nm
Response	Medium
Measurement range	850 - 780 nm
Data pitch	0.2nm
Scanning speed	40nm/min
Sample ID	xx
No. of cycle	1
Sample name	
Operator	
Comment	

No.	nm	Abs	No.	nm	Abs
1	830.2	0.01498	2	795	0.08612

Peak position of plutonium (1)

Peak position of neodymium (2)

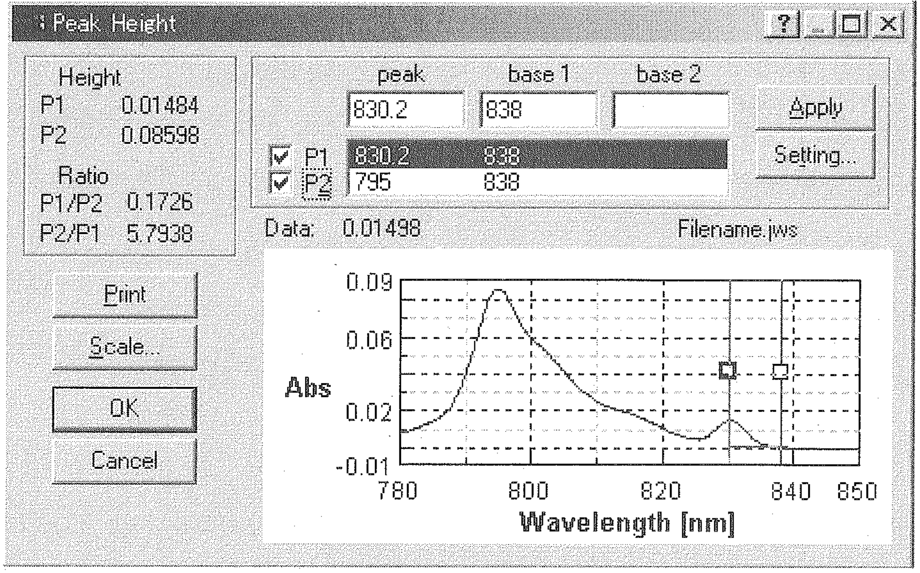
6) Click <OK>.	Closes the [Peak Find] dialog box.

5.2 Peak Height

This function detects the peak height and calculates the height ratio.

1) Select [Processing]-[Peak Process]-[Peak Height] (or click the button on the tool	

bar) to display the [Peak Height] dialog box in the [Spectra Analysis] dialog box.	



Height
P1 0.01484
P2 0.08598

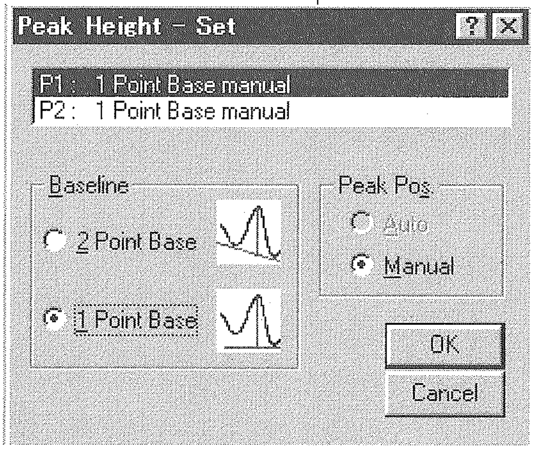
Ratio
P1/P2 0.1726
P2/P1 5.7938

	peak	base 1	base 2
	830.2	838	
☑ P1	830.2	838	
☑ P2	795	838	

Data: 0.01498
Filename.jws

Fig. 5.3 [Peak Height] dialog box

2) Check [P1] checkbox. Check [P2] checkbox.	Select the peak to calculate peak height. P1: Pu P2: Nd
3) Click <setting...>.	Open the [Peak Height-Set] dialog box. Set the peak height detecting parameters.



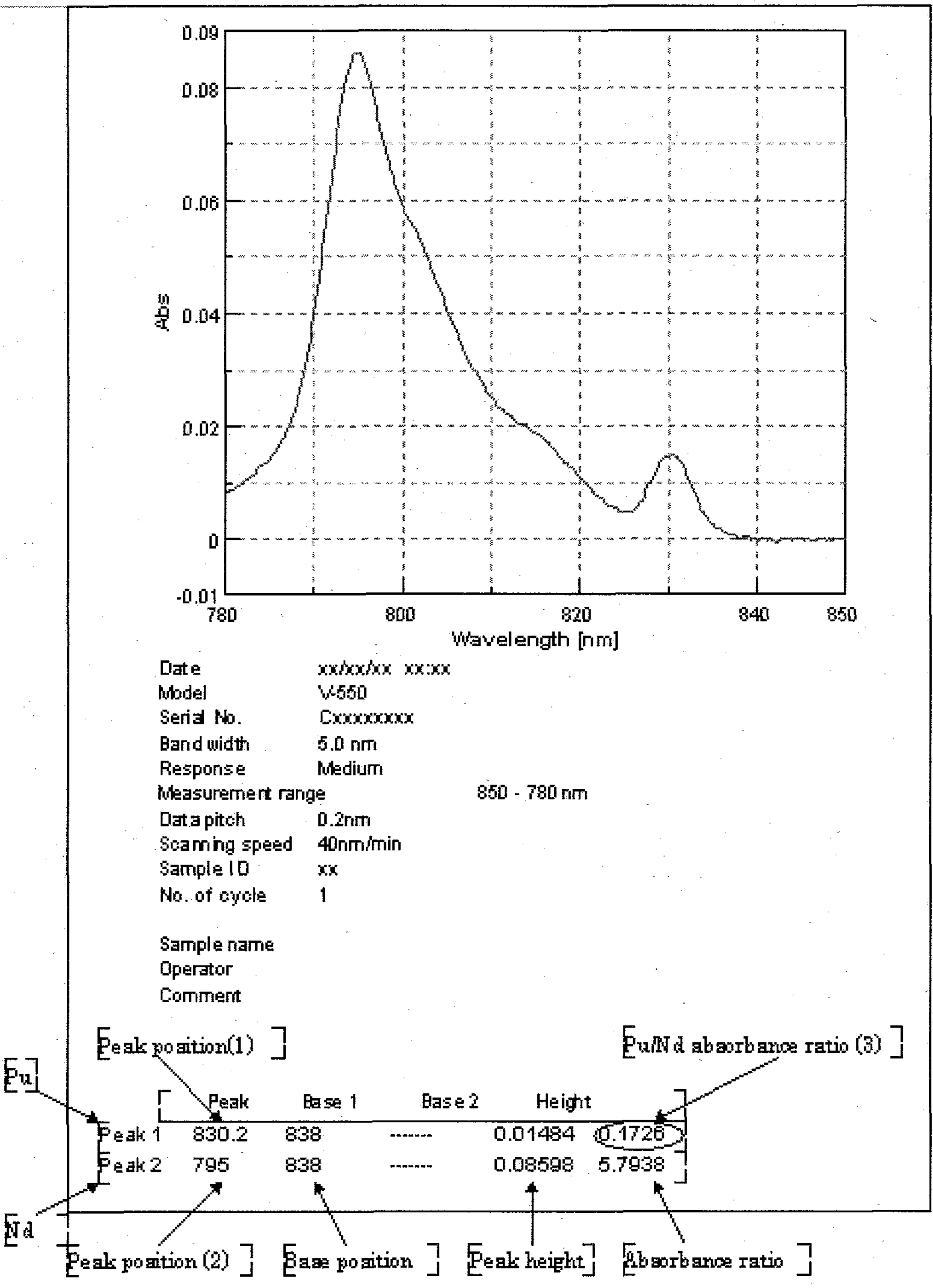
P1 : 1 Point Base manual
P2 : 1 Point Base manual

Baseline
○ 2 Point Base
● 1 Point Base

Peak Pos
○ Auto
● Manual

Fig. 5.4 [Peak Height-Set] dialog

4) Click [P1] in the parameter display field. Select [1 Point Base] option button in the [Baseline] group.	Set the parameter for [P1].
5) Click [P2] in the parameter display field. Select [1 Point Base] option button in the [Baseline] group.	Set the parameter for [P2].
6) Click <OK>.	Closes the [Peak Height -Set] dialog box.
7) Click row of numbers to the right of [P1] in the information display in the [Peak height] dialog box.	The wavelengths of peak [P1] and the base position are displayed in the wavelength setting fields, allowing the wavelength to be changed.
8) Input a value into text box in the wavelength setting fields. Input a plutonium peak position into the <peak> text box. Plutonium peak position is shown in the peak find table (1) (Fig. 5.5). Input 838 into the <base1> text box. Input a blank into the <base2> text box.	Set the base wavelength for Peak 1. The wavelength can be set by directly inputting a value into text box



Date xx/xx/xx xx:xx
Model V-550
Serial No. Cxxxxxxxx
Band width 5.0 nm
Response Medium
Measurement range 850 - 780 nm
Data pitch 0.2nm
Scanning speed 40nm/min
Sample ID xx
No. of cycle 1

Sample name
Operator
Comment

	Peak	Base 1	Base 2	Height	
Peak 1	830.2	838	-------	0.01484	0.1726
Peak 2	795	838	-------	0.08598	5.7938

Fig. 5.5 Peak Height Table

9) Click <Apply>.	Calculate the peak height of plutonium around 830 nm.
10) Click row of numbers to the right of [P2] in the information display.	The wavelengths of peak [P2] and the base position are displayed in the

	wavelength setting fields, allowing the wavelength to be changed.
11) Input a value into text box in the wavelength setting fields. Input a neodymium peak position into the <peak> text box. Neodymium peak position is shown in the peak find table ② (Fig.5.5). Input 838 into the <base1> text box. Input a blank into the <base2> text box.	Set the base wavelength for Peak 2. The wavelength can be set by directly inputting a value into text box.
12) Click <Apply>.	Calculate the peak height of neodymium around 794.6 nm.
13) Click <Print>.	Prints out spectra, peak and base positions, peak heights and peak ratios (Fig.5.5).
14) Click <OK>.	Closes the [Peak Height]-dialog box.

6. Calculation of calibration factor and Pu concentration

1) Input parameters and results of absorbance ratio to the EXCEL format to calculate calibration factor and Pu concentration.
2) After inputting parameters and results of absorbance ratio, print out this EXCEL format.

7. References

1) JASCO Corporation: "V-5 3 0/550/560/570 Spectrophotometer Instruction Manual V-500 for Windows"
2) JASCO Corporation: "Spectra Analysis Program Instruction Manual Jasco Spectrometers for Windows "

Appendix 2

Calibration of sampling device and automatic burette

Table 1 Calibration of sampling /diluting device

No.	Sampling volume (0.5 mL)		Sampling volume (1.0 mL)	
	Weight / g	Volume[*1] / mL	Weight / g	Volume[*2] / mL
1	0.4923	0.4936	1.0033	1.0068
2	0.5025	0.5039	1.0039	1.0074
3	0.4973	0.4986	1.0043	1.0078
4	0.4969	0.4982	1.0019	1.0054
5	0.4968	0.4981	1.0015	1.0050
6	0.4974	0.4987	1.0013	1.0048
7	0.4971	0.4984	1.0049	1.0084
8	0.4990	0.5004	1.0021	1.0056
9	0.4967	0.4980	1.0008	1.0043
10	0.4970	0.4983	1.0046	1.0081
11			1.0031	1.0066
12			1.0024	1.0059
13			1.0007	1.0042
14			1.0040	1.0075
Average	0.4973	0.4986	1.0028	1.0063
SD	0.0025	0.0025	0.0014	0.0014
RSD / %	0.50		0.14	

[*1] Density: 0.997296 g/cm^3 at 24.0℃

[*2] Density: 0.996512 g/cm^3 at 27.0℃

Table 2 Calibration of automatic burette

No.	Diluting volume (2.0 mL)		Diluting volume (14.0 mL)	
	Weight / g	Volume / mL	Weight / g	Volume / mL
1	1.9746	1.9800	13.9204	13.9581
2	1.9640	1.9693	13.9277	13.9655
3	1.9674	1.9727	13.9483	13.9861
4	1.9674	1.9727	13.9502	13.9880
5	1.9730	1.9783	13.9508	13.9886
6	1.9636	1.9689	13.9525	13.9903
7	1.9765	1.9819	13.9330	13.9708
8	1.9650	1.9703	13.9518	13.9896
9	1.9667	1.9720	13.9548	13.9926
10	1.9666	1.9719	13.9595	13.9973
Average	1.9685	1.9738	13.9449	13.9827
SD	0.0046	0.0046	0.0130	0.0131
RSD / %	0.23		0.09	

Density: 0.997296 g/cm^3 at 24.0℃

Appendix 3

Spectrophotometry system

Figure 1 shows a schematic diagram of the optical pathway for the spectrophotometer that was used in this work. Light was transmitted by optical fiber from both the reference and a sample cell sides to equilibrate the light intensities because the intensity of light on the sample and reference sides should ideally be equal.

The light should be transmitted to the same location of the photomultiplier because the photomultiplier has different sensitivity according to the location where the lights are transmitted. In this spectrophotometric system, the lights on the sample and reference sides were transmitted to the same location of the photomultiplier because the light is collected to a single optical fiber using two fiber branches and a single pathway. Consequently, the sensitivity differential of photomultiplierl, and the SN ratio are improved when the light on the sample and reference sides were transmitted to the photomultiplier.

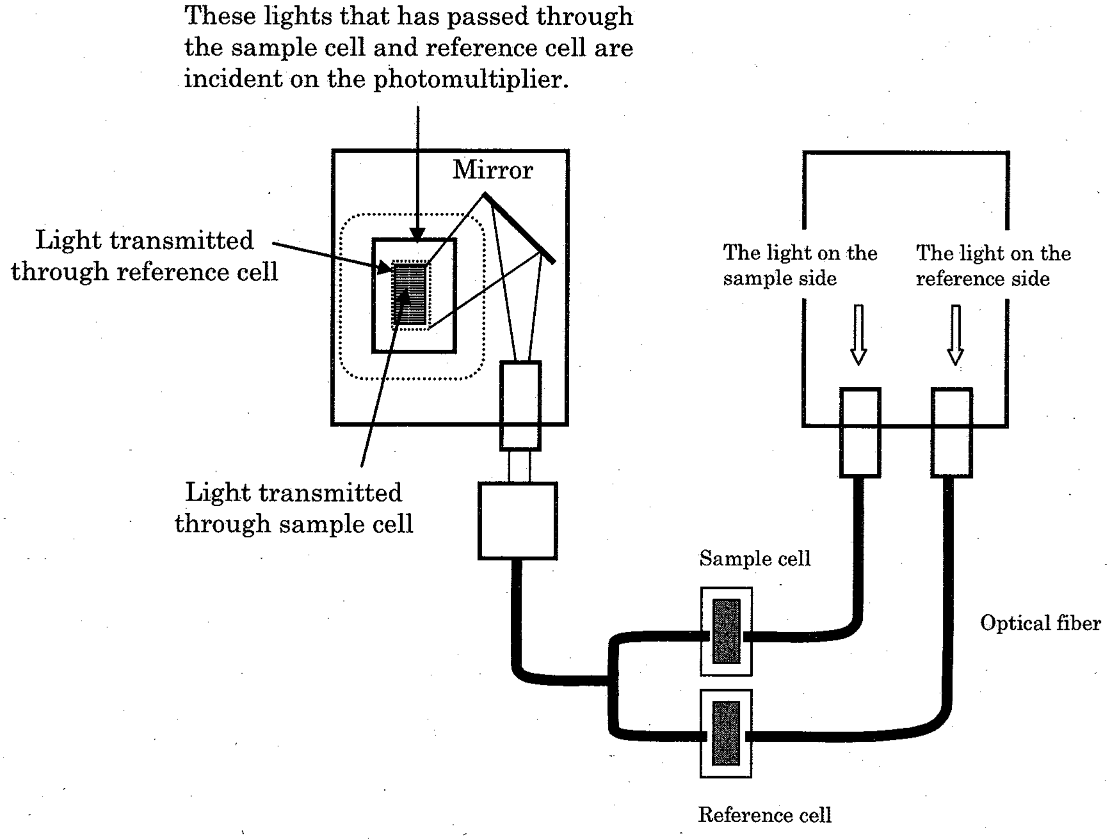


Fig. 1 Schematic diagram of the optical pathway for the spectrophotometer

国際単位系（SI）

表１．SI 基本単位

基本量	SI 基本単位	
	名称	記号
長さ	メートル	m
質量	キログラム	kg
時間	秒	s
電流	アンペア	A
熱力学温度	ケルビン	K
物質量	モル	mol
光度	カンデラ	cd

表２．基本単位を用いて表されるSI組立単位の例

組立量	SI 基本単位	
	名称	記号
面積	平方メートル	m^2
体積	立法メートル	m^3
速さ，速度	メートル毎秒	m/s
加速度	メートル毎秒毎秒	m/s^2
波数	毎メートル	m-1
密度（質量密度）	キログラム毎立法メートル	kg/m^3
質量体積（比体積）	立法メートル毎キログラム	m^3/kg
電流密度	アンペア毎平方メートル	A/m^2
磁界の強さ	アンペア毎メートル	A/m
（物質量の）濃度	モル毎立方メートル	mol/m^3
輝度	カンデラ毎平方メートル	cd/m^2
屈折率	（数の）1	1

表５．SI 接頭語

乗数	接頭語	記号	乗数	接頭語	記号
10^{24}	ヨタ	Y	10^{-1}	デシ	d
10^{21}	ゼタ	Z	10^{-2}	センチ	c
10^{18}	エクサ	E	10^{-3}	ミリ	m
10^{15}	ペタ	P	10^{-6}	マイクロ	µ
10^{12}	テラ	T	10^{-9}	ナノ	n
10^{9}	ギガ	G	10^{-12}	ピコ	p
10^{6}	メガ	M	10^{-15}	フェムト	f
10^{3}	キロ	k	10^{-18}	アト	a
10^{2}	ヘクト	h	10^{-21}	ゼプト	z
10^{1}	デカ	da	10^{-24}	ヨクト	y

表３．固有の名称とその独自の記号で表されるSI組立単位

組立量	SI 組立単位			
	名称	記号	他のSI単位による表し方	SI基本単位による表し方
平面角	ラジアン(a)	rad		$m \cdot m^{-1}=1$(b)
立体角	ステラジアン(a)	sr(c)		$m^2 \cdot m^{-2}=1$(b)
周波数	ヘルツ	Hz		s^{-1}
力	ニュートン	N		$m \cdot kg \cdot s^{-2}$
圧力，応力	パスカル	Pa	N/m^2	$m^{-1} \cdot kg \cdot s^{-2}$
エネルギー，仕事，熱量	ジュール	J	N・m	$m^2 \cdot kg \cdot s^{-2}$
工率，放射束	ワット	W	J/s	$m^2 \cdot kg \cdot s^{-3}$
電荷，電気量	クーロン	C		s・A
電位差（電圧），起電力	ボルト	V	W/A	$m^2 \cdot kg \cdot s^{-3} \cdot A^{-1}$
静電容量	ファラド	F	C/V	$m^{-2} \cdot kg^{-1} \cdot s^4 \cdot A^2$
電気抵抗	オーム	Ω	V/A	$m^2 \cdot kg \cdot s^{-3} \cdot A^{-2}$
コンダクタンス	ジーメンス	S	A/V	$m^{-2} \cdot kg^{-1} \cdot s^3 \cdot A^2$
磁束	ウエーバ	Wb	V・s	$m^2 \cdot kg \cdot s^{-2} \cdot A^{-1}$
磁束密度	テスラ	T	Wb/m^2	$kg \cdot s^{-2} \cdot A^{-1}$
インダクタンス	ヘンリー	H	Wb/A	$m^2 \cdot kg \cdot s^{-2} \cdot A^{-2}$
セルシウス温度	セルシウス度(d)	℃		K
光束	ルーメン	lm	cd・sr(c)	$m^2 \cdot m^{-2} \cdot cd=cd$
照度	ルクス	lx	lm/m^2	$m^2 \cdot m^{-4} \cdot cd=m^{-2} \cdot cd$
（放射性核種の）放射能	ベクレル	Bq		s^{-1}
吸収線量，質量エネルギー分与，カーマ	グレイ	Gy	J/kg	$m^2 \cdot s^{-2}$
線量当量，周辺線量当量，方向性線量当量，個人線量当量，組織線量当	シーベルト	Sv	J/kg	$m^2 \cdot s^{-2}$

(a) ラジアン及びステラジアンの使用は、同じ次元であっても異なった性質をもった量を区別するときの組立単位の表し方として利点がある。組立単位を形作るときのいくつかの用例は表４に示されている。
(b) 実際には、使用する時には記号rad及びsrが用いられるが、習慣として組立単位としての記号“1”は明示されない。
(c) 測光学では、ステラジアンの名称と記号srを単位の表し方の中にそのまま維持している。
(d) この単位は、例としてミリセルシウス度m℃のようにSI接頭語を伴って用いても良い。

表４．単位の中に固有の名称とその独自の記号を含むSI組立単位の例

組立量	SI 組立単位		
	名称	記号	SI 基本単位による表し方
粘度	パスカル秒	Pa・s	$m^{-1} \cdot kg \cdot s^{-1}$
力のモーメント	ニュートンメートル	N・m	$m^2 \cdot kg \cdot s^{-2}$
表面張力	ニュートン毎メートル	N/m	$kg \cdot s^{-2}$
角速度	ラジアン毎秒	rad/s	$m \cdot m^{-1} \cdot s^{-1}=s^{-1}$
角加速度	ラジアン毎平方秒	rad/s^2	$m \cdot m^{-1} \cdot s^{-2}=s^{-2}$
熱流密度，放射照度	ワット毎平方メートル	W/m^2	$kg \cdot s^{-3}$
熱容量，エントロピー	ジュール毎ケルビン	J/K	$m^2 \cdot kg \cdot s^{-2} \cdot K^{-1}$
質量熱容量（比熱容量），質量エントロピー	ジュール毎キログラム毎ケルビン	J/(kg・K)	$m^2 \cdot s^{-2} \cdot K^{-1}$
質量エネルギー（比エネルギー）	ジュール毎キログラム	J/kg	$m^2 \cdot s^{-2} \cdot K^{-1}$
熱伝導率	ワット毎メートル毎ケルビン	W/(m・K)	$m \cdot kg \cdot s^{-3} \cdot K^{-1}$
体積エネルギー	ジュール毎立方メートル	J/m^3	$m^{-1} \cdot kg \cdot s^{-2}$
電界の強さ	ボルト毎メートル	V/m	$m \cdot kg \cdot s^{-3} \cdot A^{-1}$
体積電荷	クーロン毎立方メートル	C/m^3	$m^{-3} \cdot s \cdot A$
電気変位	クーロン毎平方メートル	C/m^2	$m^{-2} \cdot s \cdot A$
誘電率	ファラド毎メートル	F/m	$m^{-3} \cdot kg^{-1} \cdot s^4 \cdot A^2$
透磁率	ヘンリー毎メートル	H/m	$m \cdot kg \cdot s^{-2} \cdot A^{-2}$
モルエネルギー	ジュール毎モル	J/mol	$m^2 \cdot kg \cdot s^{-2} \cdot mol^{-1}$
モルエントロピー，モル熱容量	ジュール毎モル毎ケルビン	J/(mol・K)	$m^2 \cdot kg \cdot s^{-2} \cdot K^{-1} \cdot mol^{-1}$
照射線量（Ｘ線及びγ線）	クーロン毎キログラム	C/kg	$kg^{-1} \cdot s \cdot A$
吸収線量率	グレイ毎秒	Gy/s	$m^2 \cdot s^{-3}$
放射強度	ワット毎ステラジアン	W/sr	$m^4 \cdot m^{-2} \cdot kg \cdot s^{-3}=m^2 \cdot kg \cdot s^{-3}$
放射輝度	ワット毎平方メートル毎ステラジアン	$W/(m^2 \cdot sr)$	$m^2 \cdot m^{-2} \cdot kg \cdot s^{-3}=kg \cdot s^{-3}$

表６．国際単位系と併用されるが国際単位系に属さない単位

名称	記号	SI 単位による値
分	min	1 min=60s
時	h	1h =60 min=3600 s
日	d	1 d=24 h=86400 s
度	°	1° =(π/180) rad
分	’	1’ =(1/60)° =(π/10800) rad
秒	”	1” =(1/60)’ =(π/648000) rad
リットル	l、L	1l=1 $dm^3=10^{-3}m^3$
トン	t	1t=10^3 kg
ネーパ	Np	1Np=1
ベル	B	1B=(1/2)ln10(Np)

表７．国際単位系と併用されこれに属さない単位で
SI単位で表される数値が実験的に得られるもの

名称	記号	SI 単位であらわされる数値
電子ボルト	eV	1eV=1.60217733(49)×10^{-19}J
統一原子質量単位	u	1u=1.6605402(10)×10^{-27}kg
天文単位	ua	1ua=1.49597870691(30)×10^{11}m

表８．国際単位系に属さないが国際単位系と
併用されるその他の単位

名称	記号	SI 単位であらわされる数値
海里		１海里=1852m
ノット		１ノット=１海里毎時=(1852/3600)m/s
アール	a	1 a=1 $dam^2=10^2m^2$
ヘクタール	ha	1 ha=1 $hm^2=10^4m^2$
バール	bar	1 bar=0.1MPa=100kPa=1000hPa=10^5Pa
オングストローム	Å	1 Å=0.1nm=10^{-10}m
バーン	b	1 b=100$fm^2=10^{-28}m^2$

表９．固有の名称を含むCGS組立単位

名称	記号	SI 単位であらわされる数値
エルグ	erg	1 erg=10^{-7} J
ダイン	dyn	1 dyn=10^{-5}N
ポアズ	P	1 P=1 dyn・s/cm^2=0.1Pa・s
ストークス	St	1 St =1$cm^2/s=10^{-4}m^2/s$
ガウス	G	1 G ^10^{-4}T
エルステッド	Oe	1 Oe ^(1000/4π)A/m
マクスウェル	Mx	1 Mx ^10^{-8}Wb
スチルブ	sb	1 sb =1$cd/cm^2=10^4cd/m^2$
ホト	ph	1 ph=10^4lx
ガル	Gal	1 Gal =1$cm/s^2=10^{-2}m/s^2$

表10．国際単位に属さないその他の単位の例

名称	記号	SI 単位であらわされる数値
キュリー	Ci	1 Ci=3.7×10^{10}Bq
レントゲン	R	1 R = 2.58×10^{-4}C/kg
ラド	rad	1 rad=1cGy=10^{-2}Gy
レム	rem	1 rem=1 cSv=10^{-2}Sv
X線単位		1X unit=1.002×10^{-4}nm
ガンマ	γ	1γ=1 nT=10^{-9}T
ジャンスキー	Jy	1 Jy=$10^{-26}W \cdot m^{-2} \cdot Hz^{-1}$
フェルミ		1 fermi=1 fm=10^{-15}m
メートル系カラット		1 metric carat = 200 mg = 2×10^{-4}kg
トル	Torr	1 Torr = (101 325/760) Pa
標準大気圧	atm	1 atm = 101 325 Pa
カロリー	cal	
ミクロン	µ	1 µ =1µm=10^{-6}m

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