

CST ANALYTICAL CHEMISTRY PROCEDURE

Procedure Number: ANC103, R.0

Approval Date: 10/30/97

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TITLE: Spectrochemical Determination of Metallic Impurities in Plutonium Materials

Type of Procedure: <input type="checkbox"/> Safe Operating Procedure (SOP) <input type="checkbox"/> Quality Assurance (QA) <input type="checkbox"/> Quality Control (QC) <input checked="" type="checkbox"/> Analytical <input type="checkbox"/> Radiochemistry <input type="checkbox"/> QC Preparation <input checked="" type="checkbox"/> Inorganic <input type="checkbox"/> Instrument Operation <input type="checkbox"/> Organic <input type="checkbox"/> Miscellaneous <input type="checkbox"/> Sample Management <input type="checkbox"/> Computing	Status: <input checked="" type="checkbox"/> New <input type="checkbox"/> Major Revision <input type="checkbox"/> Minor Revision <input type="checkbox"/> Reviewed, No Change
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Type of Training Required: <input type="checkbox"/> Informal Qualification <input checked="" type="checkbox"/> Formal Qualification <input type="checkbox"/> Continuing Qualification	Form of Training: <input checked="" type="checkbox"/> Required Reading <input type="checkbox"/> Lesson Plan <input type="checkbox"/> Briefing <input type="checkbox"/> Other	Instruction: <input type="checkbox"/> Notify of Changes <input type="checkbox"/> Retrain on Changes <input type="checkbox"/> Retrain on Entire Revision <input type="checkbox"/> Training Not Required
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Signatures on File in CST-3 Document Control

SPECTROCHEMICAL DETERMINATION OF METALLIC IMPURITIES IN PLUTONIUM MATERIALS

Analyte: Metallic impurities	Method No.: ANC103
Matrix: Plutonium material	Accuracy Precision: 20–50%
Procedure:	Minimum Detection Limit: Analyte dependent, $\mu\text{g/g}$
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1. SCOPE

Impurities are determined in plutonium (Pu) materials (metal, carbide, nitride, oxide and Pu/U oxide) by emission spectrochemical analysis. It is performed by causing the constitutive atoms to emit light by heating the material in a electric arc. The light is dispersed as a function of wavelength using a grating spectrograph and the emission spectra recorded on photographic glass plates. The basis for semi-quantitative measurement is the dependence of the amount of energy emitted on the number of atoms present under standardized conditions. The Pu materials are first converted to oxide by heating in a electric furnace. Aliquots are mixed with either Ga_2O_3 or AgCl carrier which forms the basis for three carrier-distillation methods (see Source Material 14.3). Analyte concentrations in the range between the Limit Of Detection (LOD) and approximately $750 \mu\text{g/g}$ are determined. The procedure applies to the elements Ag, Al, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Fe, Ga, Hf, K, La, Li, Mg, Mn, Mo, Na, Nb, Ni, Pb, Rb, Re, Si, Sn, Sr, Ta, Ti, V, W, Y, Zn, Zr, or any other element that has measurable emission in the ultraviolet or visible region of the electromagnetic spectrum.

2. TERMINOLOGY

- 2.1. Analytical Batch—A suite of up to 13 samples having a similar matrix and processed as a unit using the same analytical method within a specific time period.
- 2.2. Calibration Standards—Matrix material containing analytes in known concentrations used to construct the calibration function for analytes quantification.
- 2.3. Internal Standard—An element not present in the sample that is added to samples and standards and used to monitor for long and short term fluctuation in signal or to correct for unspecified matrix effects.
- 2.4. Laboratory Control Sample (LCS)—A known matrix spiked with analyte(s) from an independent source to monitor the execution of the analytical method. If a matched matrix (e.g. characterized surrogate) is not available, commercially purchased standards are used to prepare the LCS. The LCS undergoes the same sample preparations and

cleanup methods as the samples. One LCS, if available should be analyzed with each analytical batch.

- 2.5. Method Detection Limit (MDL)—The minimum concentration of analyte that can be differentiated from the background in the sample matrix.
- 2.6. Spectroscopic Purity—A compound that is at least 99.99% pure.

3. SAFETY

3.1. All safety precautions involved in the manipulation and spectrochemical analysis of radioactive materials are described in the following procedures. These are available from Analytical Chemistry Document Control team of group CST-3 or online.

- SAF-1, “General Safety Procedure”
- QC-13, “Good Laboratory Practices for Analytical Chemistry”
- CMR-POL-001, “Radiation Protection Practices in the CMR Facility”
- SAF-4, “ALARA Program for CST Analytical Chemistry Groups in the CMR Building”

3.2. Waste handling and disposal is described in the following procedures.

- CMR-PLA-001, “CMR Waste Management Plan”
- CMR-SOP-006, “TRU Waste Bag Out for Certified WIPP Waste”
- ANC118, “Packaging Solutions or Solid Residue Bottles for Shipment to TA-55 Drum Handling Instructions for WIPP TRU Waste”
- SAF-5, “CLS-1 Spill Procedure and Waste Minimization Plan”

4. RESPONSIBILITIES AND TRAINING

4.1. The analyst is responsible for the following.

- 4.1.1. Preparing samples, standards and quality control samples.
- 4.1.2. Ensuring adequate supplies are on hand.
- 4.1.3. Ensuring that the instruments used are functioning properly.
- 4.1.4. Setting up and performing the analytical run, including samples, standards and Quality Control (QC) samples according to this procedure .
- 4.1.5. Reducing the data according to this procedure.
- 4.1.6. Performing initial QC review of the analytical data.
- 4.1.7. Scheduling re-analysis of samples.

- 4.1.8. Preparing data for Laboratory Information Management System (LIMS) entry.
- 4.1.9. Tracking Special Nuclear Material (SNM) inventory.
- 4.1.10. Preparing sample residues for shipment to TA-55.
- 4.2. The technical supervisor is responsible for the following.
 - 4.2.1. Scheduling instrument maintenance.
 - 4.2.2. Overseeing the performance of the analyst and ensuring QA/QC requirements are met or exceptions documented.
 - 4.2.3. Reviewing technical data and documentation according to procedure QA-9, "Maintaining Laboratory Notebooks and Logbooks."
- 4.3. The analytical operations leader is responsible for the following.
 - 4.3.1. Ensuring that samples are managed according to the procedure ANC917, "Sample Receiving and Inventory in Wing 7, CMR."
 - 4.3.2. Ensuring that analytical laboratory personnel have fulfilled their training requirements or are directly supervised by a certified analyst while performing spectrochemical analysis work.
 - 4.3.3. Ensuring that analytical laboratory personnel follow this procedure.
 - 4.3.4. If no technical supervisor is assigned, the operations leader assumes those duties.
- 4.4. The minimal job training requirements for a new analyst are the following.
 - 4.4.1. Reads and understands this procedure.
 - 4.4.2. Observes a certified analyst perform the complete analysis of a set of samples.
 - 4.4.3. The new analyst then analyzes a set of samples while being observed by a certified analyst.

5. INTERFERENCES

The major interference is due to the Pu emission spectra overlapping the analytical wavelengths of interest. The Pu spectra is minimized by using the carrier-distillation technique. Occasionally due to sample history or impurity content the technique is unable to suppress the Pu spectra. Grinding the sample in a mortar to a fine powder and ensuring the sample and carrier form a homogeneous mixture minimize or eliminate the interference (see Source Material 14.2).

6. SAMPLE STORAGE AND HANDLING

Samples and standards are stored in plastic vials within glove boxes according to analytical chemistry ALARA practices. The exception to this is $^{238}\text{PuO}_2$ which can not be stored in plastic due to the heat generated by spontaneous fission. This material type is segregated from ^{239}Pu and stored in tantalum (Ta) vials.

7. APPARATUS AND EQUIPMENT

- 7.1. Spectrograph: Jarrell-Ash or equivalent 3.4 m focal length with 15,000 and 30,000 groove/inch gratings giving reciprocal linear dispersions of 0.5 nm/mm and 0.25 nm/mm respectively. Must be capable of covering the ultraviolet and visible wavelength region from 240 nm to 900 nm.
- 7.2. Power Source: Spex Industries or equivalent delivering 0–17.5 Ampere direct current electric arc short circuited for a variable time span of 0–60 s.
- 7.3. Plate Developer: Wyng Lynch or equivalent capable of automatic and simultaneous development of three 4 × 10 inch photographic glass plates.
- 7.4. Plate Dryer: Forced air with electric heater to provide filtered air at a minimum of 50°C.
- 7.5. Microphotometer: Jarrell-Ash or equivalent capable of measuring line density or % transmittance, adjustable plate movement of 0.1–25 mm/min. and slit 0–25 μm wide and 0–2 mm in height.
- 7.6. Muffle Furnace: platinum (Pt) lined furnace with controlled atmosphere capable of reaching 1000°C
- 7.7. Analytical Balance: Mettler, or equivalent, with readout to 0.1 mg.
- 7.8. Mortars and Pestles made of tungsten carbide, boron carbide, and uranium metal.
- 7.9. Pt microspoon, Pt funnel, Pt weighing dish, Pt crucibles, Pt crucible holder, and Pt tipped tongs.
- 7.10. Sample arcing chambers as described in Los Alamos report LA-4622 (see Source Material 14.1).

8. REAGENTS

- 8.1. High purity ^{239}Pu compound converted to $^{239}\text{PuO}_2$ by heating in muffle furnace at 900°C to constant weight.

8.2. Graphite electrodes

8.2.1. 1.5 in. × 0.25 in. diameter tapered at one end to a point, ASTM Designation E130-66, type C-1.

8.2.2. 3 cm long × 8 mm diameter drilled at one end with a 4 mm diameter × 8 mm deep crater, ASTM Designation E130-66, type S-2.

8.2.3. 1.5 in × 0.25 in diameter with undercut cup, ASTM Designation E130-66, type S-4.

8.3. Co_3O_4 , Ga_2O_3 and AgCl of spectroscopic purity.

8.4. Master impurity mixes prepared by analytical chemistry personnel from spectroscopic purity oxide or other suitable compounds.

9. CALIBRATION AND STANDARDIZATION

NOTE: All compounds are ground individually in a mortar and sieved to pass a 325 mesh nylon screen prior to mixing.

9.1. Carrier - Internal Standard Mixture

Prepare the mixture by adding 0.0432 g of Co_3O_4 to 2.9568 g Ga_2O_3 in a mortar. Mix with a Pt microspoon and then grind with a pestle.

9.2. Master Impurity Mixture

Prepare a mixture using the compounds and weights listed in Attachment 3. Mix in a mortar with a Pt microspoon and then grind with a pestle.

9.3. Standards

NOTE: The following is an example of how to prepare a set of standards. The impurity elements and their concentrations are determined by the technical supervisor. Guidance can be found in Los Alamos report LA-6354 (see Source Material 14.5).

Prepare ~10 g of each of the 7 standards listed in Attachment 4.

9.3.1. Standard number 7 and 6 preparation

9.3.1.1. Weigh the amounts of PuO_2 , master impurity and carrier - internal standard mixture shown below assuming 17.1000 g as basis for calculation.

Standard number 7

wt. of impurity elements	17.1000×0.003725 (Attach. 3) =	0.0637 g
wt. of master impurity mix	0.0637×1.890 (Attach. 3)=	0.1204
wt. of PuO ₂	$(17.1000-0.0637) \times 1.1338$ (Attach. 3)=	19.3158
wt. of PuO ₂ + master imp. mix		19.4362
wt. of carrier-int. std. mix	$19.4362 \times 0.04/0.96 =$	0.8097
constituents:	master impurity mix	0.1204 g
	carrier-internal std. mix	0.8097
	PuO ₂	<u>19.3158</u>
	Total	20.2459 g

Standard number 6

make 16.5000 g of the standard

wt. of impurity elements	$16.5000 \times 0.0018625 =$	0.03073
wt. of master impurity mix	$0.03073 \times 1.890 =$	0.0508
wt. of PuO ₂	$(16.5000 - 0.0307) \times 1.1338 =$	18.6729
wt. of PuO ₂ + master imp. mix		18.7236
wt. of carrier-int. std. mix	$18.7236 \times 0.04/0.96 =$	0.7802
wt. of no. 7 to furnish 0.0508 g of master impurity mixture	$20.2504 / 0.1204 \times 0.0508 =$	8.5442
wt. of carrier-internal std. mix in 8.5442 g of no. 7	$0.8142 / 20.2504 \times 8.5442 =$	0.3435
wt of PuO ₂ in 8.5442 g of no. 7 standard	$19.3158 / 20.2504 \times 8.5442 =$	8.1499
constituents:	no. 7 standard	8.5442
	carrier-internal std. mix (0.7802-0.3435) =	0.4367
	PuO ₂ (18.6729 - 8.1499) =	<u>10.5230</u>
	Total	23.4342 g

9.3.1.2. Place in mortar and mix thoroughly by stirring with Pt microspoon prior to grinding with pestle.

9.3.1.3. Remove the portion to be diluted to make the next standard.

9.3.1.4. Package the remaining material in a labeled plastic vial.

9.3.2. Prepare the other 5 standards by successive dilution with PuO₂ and carrier - internal standard mixture and grinding each in the mortar.

9.4. Standards evaluation

- 9.4.1. Reserve about 1 g of the PuO₂ matrix material for evaluation and confirmation of blank impurity concentrations.
- 9.4.2. Test each set of standard by comparing analytical curves with those obtained with the previous set of working standards.
- 9.4.3. The Laboratory Control Standard (LCS) is analyzed and results compared to the consensus value.

Continuing calibration is done by recording spectra of a set of the standards on each photographic plate along with the samples being analyzed.

10. QUALITY CONTROL

For this procedure to be in control the following QC requirements must be met.

- The standards graduate in analyte emission line intensity with increasing concentration.
- The average deviation of the standards from the least squares line of best fit is within 20%.
- The average deviation of the LCS is within 50% of the nominal concentration.

If these conditions are not met the source of the problem is determined, documented and the samples reanalyzed.

11. PROCEDURE

- 11.1. Receiving and opening ²³⁸PuO₂ sample container. This section describes the opening of the shipping container used specifically for ²³⁸PuO₂ samples.
 - 11.1.1. ²³⁸PuO₂ samples are received in a sealed Ta capsule sealed in a second Ta capsule and then wrapped in aluminum foil. This package is placed in a pipe nipple commonly referred to as a dog bone capped at both ends and then in a taped tin can. The tin can is filled with aluminum (Al) foil to prevent excess movement of the pipe nipple during shipment.
 - 11.1.2. While wearing a respirator and using an alpha counter the tin can is opened and each layer of packaging is monitored. The Ta capsules are entered into the glovebox train through the air lock.
 - 11.1.3. The two Ta capsules are opened using a pipe cutter. The ²³⁸PuO₂ sample is weighed, ground with a uranium mortar and pestle, and weighed. The first weight is a check on the shipping weight from TA-55 to Analytical Chemistry. The second weight subtracted from the first weight gives the amount of ²³⁸PuO₂ on the cheese cloth used for cleaning the uranium mortar

after grinding. The finely ground $^{238}\text{PuO}_2$ is placed in the opened smaller Ta capsule with the cut off end of the larger Ta capsule used as a cover.

- 11.1.4. An inventory is taken and any needed materials such as electrodes, cheese cloth, and carrier-internal standard are entered into the glove box train along with the samples.
- 11.2. $^{238}\text{PuO}_2$ sample preparation. This portion of the procedure is performed in Room 7136.
 - 11.2.1. For the determination of routine and alkali elements described in Attachment 4, weigh 96 mg of the $^{238}\text{PuO}_2$ sample and 4 mg of the carrier internal standard ($\text{Co}_3\text{O}_4 + \text{Ga}_2\text{O}_3$) in a Pt dish. Mix well using a Pt microspoon and transfer into an 8 mm deep vented electrode ASTM type S-2 using a Pt funnel.
 - 11.2.2. Repeat this process 3 more times to prepare a total of four electrodes. Two of the electrodes will be used for the routine analysis and the other two for the alkali elements.
 - 11.2.3. For the refractory elements use 60 mg of the $^{238}\text{PuO}_2$ sample and 15 mg of the AgCl carrier. Transfer the mix to a ASTM type S-4 undercut electrode using a Pt funnel. Prepare two electrodes.
- 11.3. Removal of $^{238}\text{PuO}_2$ Samples from Room 7136
 - 11.3.1. The sample electrodes have been transferred to a wood block. The block is moved to the exit airlock. Under Special Air Test (SAT) conditions the inner door is opened and then the outer door.
 - 11.3.2. A damp piece of cheese cloth or similar material is placed on the airlock floor. A cardboard transfer box with taped corners is placed in the airlock.
 - 11.3.3. The wooden transfer block is gently placed in the transfer box. One person then places the transfer box cover on the box. The transfer box is removed and slid into a plastic bag held by the second person.
 - 11.3.4. The plastic is twisted shut and the bag and second person are monitored by health physics personnel. If the bag is contaminated, it is double bagged and rechecked. If it is not contaminated the second person carries the samples to Room 7134 and puts them in the open front box that leads to the arc box.
 - 11.3.5. During the SAT for the sample removal any trash generated is removed.

- 11.4. $^{239}\text{PuO}_2$ sample preparation This section is performed in the glove boxes in Room 7134.
- 11.4.1. For the routine and alkali elements weigh out 96 mg of the $^{239}\text{PuO}_2$ sample and 4 mg of the carrier internal standard ($\text{Co}_3\text{O}_4 + \text{Ga}_2\text{O}_3$) into a Pt weighing dish. Mix thoroughly with a Pt microspoon. Transfer to an 8 mm deep vented ASTM type S-2 electrode using a plastic transfer funnel. Prepare four electrodes.
- 11.4.2. For refractory elements weigh 60 mg of the $^{239}\text{PuO}_2$ sample and 15 mg of the AgCl carrier into a Pt weighing dish and mix thoroughly with a Pt microspoon. Transfer to a ASTM type S-4 electrode with a plastic transfer funnel. Prepare two electrodes for each sample.
- 11.4.3. Take the tray with samples to the arcing box through the dry box train.
- 11.4.4. Follow the procedures in Secs. 11.5 through 11.7 for arcing and development of photographic plates.
- 11.5. Spectrograph instrument operation
- 11.5.1. Select the proper instrument and excitation parameters for the carrier-distillation method being used from Table I

Table 1				
	Routine Plate	AgCl Plate	Alkali Plate	Cesium Plate
Grating (grooves per inch, mask)	30,000gpi, full	30,000gpi, full	15,000gpi, full	15,000gpi, full
Grating Rotation (degrees)	10.82	10.82	10.40	14.53
Split Filter	17 and 100% T	25 and 100% T	17 and 100% T	17 and 100% T
Slit Tube	22 mm	22 mm	20 mm	19 mm
Slit (width, height)	15 μm , 1.5mm	15 μm 1.5mm	20 μm 1.5mm with yellow filter #3389	20 μm 1.5mm with yellow filter #3389
Exposure	50 sec	5/45 sec	55 sec	55 sec
Amperage (closed circuit)	17.5 A	7.5 A	17.5 A	17.5 A
O ₂ , flow to arcing chamber	8 on scale, cylindrical chamber	8 on scale, round chamber	8 on scale, round chamber	8 on scale, round chamber

Table 1 (cont.)				
Plate emulsion (Kodak or equivalent)	3(SA-1), full cassette	2(SA-1), loaded in left and middle sections of cassette	3(1-N), full cassette	1(1-N), loaded in middle section of cassette
Electrodes (ASTM)	Type C-1 and S-2	type C-1 and S-4	type C-1 and S-2	type C-1 and S-2

- 11.5.2. Load photographic plates into the film cassette in the darkroom and place the cassette into the cassette holder in the spectrograph.
- 11.5.3. Turn on the master switch for the spectrograph and rack the photographic plates to the starting position to record spectra
- 11.5.4. Clean the arc stand and atmosphere chamber with water and cheese cloth.
- 11.5.5. Move sample electrodes into arcing box and vent electrodes with proper venting tool as described in Los Alamos report LA-4622 (see Source Material 14.1).
- 11.5.6. Turn on coolant water for the electrode clamps by turning the light switch on for the arc box.
- 11.5.7. Load first sample electrode into clamps and press ignitor button. Maintain the proper electrode gap during the burning of the sample. Remove the used electrodes and save the PuO₂ in a recovery container. Continue the process until all electrodes have been shot.
- 11.5.8. Rack down photographic plate cassette to approximate position 62, then stop. Put rack on 1 mm increment, then rack to position 57.
- 11.5.9. The photographic camera light shield is now in position and it is safe to open the cassette door, pull the cassette out and take the cassette to the darkroom and develop the plates.

11.6. Operation of Photographic Plate Developing Unit

11.6.1. To start the automatic developer open the following.

- Nitrogen tank valve and set the regulator to 65 psi.
- Hot and cold water valves and set the pressure regulators to 30 psi.
- Hot and cold water filter valves.

- 11.6.2. Set solution level controller to appropriate position for photographic plates.
- 11.6.3. Set the timers to the appropriate position for the developer solution and fixer.
- 11.6.4. Turn water temperature controller to on position.
- 11.6.5. Check the level of the chemicals in the supply tanks. For proper operation they should be above the lowest black indicator lines marked on the side of the tanks. Refer to Attachment 1 for preparation of photographic chemicals.

NOTE Do not begin developing plates until the temperature of the chemicals reaches 68°C and the pressure gauge indicator light for the solution tanks is green.

11.7. Development of photographic plates

- 11.7.1. Start up developing unit as described in Sec. 11.6.
- 11.7.2. Set the trough drum spacer to proper position as measured by plate holder.
- 11.7.3. Turn off room light and insert plates into plate holder.
- 11.7.4. Place plate holder in trough and place cover over trough.
- 11.7.5. Turn on developer. At this point the red light is on. When the red light goes out push the start button. After 20 min. a high pitched buzzer goes off to indicate that the process is complete.
- 11.7.6. Remove plates, rinse with distilled water and dry with paper towels instead of cloth to prevent leaving lint on the plates.
- 11.7.7. Place plates in a plate holder and leave them in the dryer for 20 min.
- 11.7.8. After surveying the plates for possible contamination take them to Room 7170 for microphotometry and storage.

12. CALCULATIONS AND DATA REPORTING

12.1. Visual comparison

- 12.1.1. Align the plate with a master plate which identifies the analytical lines.
- 12.1.2. Visually compare the blackening of the lines produced by the sample with the blackening shown by the corresponding lines of the standards.
- 12.1.3. Enter the concentration value obtained on the log sheet.

12.2. Microphotometry

- 12.2.1. Follow standard methods for calibrating the photographic emulsion (see Source Material 14.6).
- 12.2.2. Measure the percent transmittance, or optical density, of the analytical and internal standard lines listed in Attachment 2 in the spectrograms of the standards and samples.
- 12.2.3. Measure the percent transmittance, or optical density, of the background associated with the analytical lines.
- 12.2.4. Using the BASIC language program ALLCAL described in LA-6010 (see Source Material 14.4) generate analytical working curves of intensity versus concentration.
- 12.2.5. Determine the concentration of the analyte in the sample by entering the percent transmittance into the computer program.
- 12.2.6. Enter the concentration value obtained on the log sheet.

12.3. Data entry into spreadsheet prior to LIMS entry

Report results on sample as received basis as $\mu\text{g/g}$.

13. PRECISION AND ACCURACY

The relative standard deviation is 20% or better when an internal standard is used and the line intensities are measured with a microphotometer. The estimated relative standard deviation for a visual comparison is 50%. The use of microphotometry without an internal standard gives intermediate precision. Accuracy has not been determined since there is no Pu material that has been certified as to its impurity content.

14. SOURCE MATERIALS

- 14.1. J. E. Rein, G. M. Matlack, G. R. Waterbury, R. T. Phelps, C. F. Metz, "Methods of Chemical Analysis for FBR Uranium-Plutonium Oxide Fuel and Source Materials," Los Alamos National Laboratory report LA-4622 (March 1971).
- 14.2. C. J. Martell, "The Effect of Particle Size on the Carrier-Distillation Analysis of PuO_2 ," Los Alamos National Laboratory report LA-5454 (February 1974).
- 14.3. B. F. Scribner, H. R. Mullins, "Carrier-Distillation Method for Spectrographic Analysis and its Application to the Analysis of Uranium-Base Materials," J. Res. Nat. Bur. Stand. A37, 379-389 (1946).
- 14.4. C. T. Apel, "ALLCAL: An Interactive BASIC Program for Spectrochemical Calculations," Los Alamos National Laboratory report LA-6010 (September 1975).

- 14.5. C. J. Martell, W. M. Myers, "The Evaluation of Spectrographic Standards for the Carrier-Distillation Analysis of PuO₂," Los Alamos National Laboratory report LA-6354 (May 1976).
- 14.6. ASTM Methods for Emission Spectrochemical Analysis, "Recommended Practices for Photographic Photometry in Spectrochemical Analysis," American Society for Testing Materials (1968).

15. ATTACHMENTS

Attachment 1. Preparation of Darkroom Solutions (1 page).

Attachment 2. Analytical Lines and Detection Limits (1 page).

Attachment 3. Master Impurity Mixture (1 page).

Attachment 4. Composition of Routine Standards (1 page).

ATTACHMENT 1.

PREPARATION OF DARKROOM SOLUTIONS

1. Developer-Kodak D-19 Solution or equivalent
 - 1.1. Place disc shaped Teflon stirrer in 4-L glass beaker.
 - 1.2. Fill beaker to 3800 mL mark with tap deionized water.
 - 1.3. Place beaker on hot plate, turn on stirrer and heat water to 36°C.
 - 1.4. Turn off heat and slowly add contents of one envelope of Kodak D-19 powder.
 - 1.5. When solution clears and there is no undissolved powder in the beaker turn off the stirrer.
 - 1.6. Transfer D-19 solution into brown glass bottle for storage.

2. Acid Solution or Short Stop
 - 2.1. Fill 4-L acid bottle to ~1 L with deionized water.
 - 2.2. Add 16 mL glacial acetic acid to the water.
 - 2.3. Fill to 3-L line with deionized water.

3. Fixer - Kodak Rapid Fixer or equivalent
 - 3.1. Place a 2 in. magnetic stirring bar into 4-L glass beaker and place on electric stirrer.
 - 3.2. Pour 1.9 L of deionized water into bottle and turn on stirrer.
 - 3.3. Add 946 mL of Solution A to the water.
 - 3.4. When the water and Solution A are thoroughly mixed add 104 mL of Solution B.

CAUTION: *Do not mix solutions A and B without water.*
 - 3.5. Dilute to 3.8 L mark with deionized water and stir until solution is well mixed.
 - 3.6. Store between 4–27°C, shelf-life is 4 weeks.

NOTE: Solution A and B are designations used by Kodak. Part numbers for these solutions are GXKK197-3247 and GXKK173-3013 respectively.

4. Changing Darkroom Solutions. For this procedure refer to the Wing-Lynch Model 4E service manual (#10-1001014). This is kept on the window ledge in Room 7174.

ATTACHMENT 2.

ANALYTICAL LINES AND DETECTION LIMITS

Element	Wavelengths, nm		Detection Limit, ug/g	
			<u>Ga₂O₃ Carrier</u>	<u>AgCl Carrier</u>
Ag		328.06	1	
Al	394.40	396.15	5	
B		249.67	5	
Ba	455.40	493.40	2	
Be	313.04	313.10	1	
Bi		306.7	1	
Ca	393.36	396.84	3	
Cd	326.10	346.62	10	
Ce	418.66	395.25		100
Co	340.51	304.40		2
Co	252.13	253.59		3
Cr	283.56	302.15	5	
Cs		852.11	50	
Cu	327.39	324.75	1	
Fe	248.32	302.21	5	
Ga	403.29	294.36		100
Hf	339.98	286.63		25
K	766.49	769.89	5	
La	394.91	408.67		25
Li		670.78	1	
Mg	280.27	285.21	1	
Mn	257.61	280.10	1	
Mo	313.25	319.39	20	2
Na	588.99	589.59	2	
Nb	405.89	407.97		10
Ni	300.24	300.36	5	
Pb	280.19	405.78	5	
Rb		780.02	10	
Re	346.04	346.47		25
Si	288.16	251.61	5	
Sn	326.23	317.50	5	
Sr	407.77	421.55	5	
Ta		301.25		100
Ti	323.45	334.94		5
V	318.34	318.54		3
W	400.87	407.43		10
Y	371.03	360.07	324.22	25
Zn		334.50	5	
Zr	339.19	343.82		100

ATTACHMENT 3

MASTER IMPURITY MIXTURE

Element	Compound	Wt. of Element	Wt. of Compound	Conc. of Std. 7
Fe	Fe ₂ O ₃	200 mg	286.0 mg	500 µg/g
Si	SiO ₂	200	427.9	500
Ni	NiO	200	254.5	500
Al	Al ₂ O ₃	200	378.0	500
Na	Na ₂ C ₂ O ₄	100	291.4	250
Mg	MgCO ₃	100	346.8	250
Ca	CaCO ₃	100	249.7	250
Cr	Cr ₂ O ₃	100	146.1	250
Zn	ZnO	40	49.8	100
Pb	PbO	40	43.1	100
Cd	CdO	40	45.7	100
Sn	SnO ₂	40	50.8	100
Mo	MoO ₃	40	60.0	100
Cu	CuO	20	25.1	50
Mn	MnO ₂	20	31.7	50
Sr	SrCO ₃	20	33.7	50
B	H ₃ BO ₃	10	57.2	25
Be	BeO	10	27.8	25
Bi	Bi ₂ O ₃	<u>10</u>	<u>11.2</u>	<u>25</u>
	Total	1490	2816.5	3725 or 0.3725%

gravimetric factor = wt. of compounds / wt. of elements = 2816.5 / 1490 = 1.890

gravimetric factor = PuO₂ / Pu = 1.1338

ATTACHMENT 4.

COMPOSITION OF ROUTINE STANDARDS

Standard No.

	1	2	3	4	5	6	7
Element							
Fe, Si, Ni, Al	5 µg/g	10 µg/g	25 µg/g	50 µg/g	100 µg/g	250 µg/g	500 µg/g
Na, Mg, Ca, Cr	2.5	5	12.5	25	50	125	250
Pb, Zn, Cd, Sn, Mo	1	2	5	10	20	50	100
B, Be, Bi	0.25	0.5	1.25	2.5	5	12.5	25
Co	500	500	500	500	500	500	500