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Plutonium and Environmental Metals in Man

Interlaboratory Meeting, May 9, 1973

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scientific laboratory
of the University of California
LOS ALAMOS, NEW MEXICO 87544

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Plutonium and Environmental Metals in Man

Interlaboratory Meeting, May 9, 1973

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Bio-Assay Chemical Analysis Section

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This work supported by the US Atomic Energy Commission's
Division of Biomedical and Environmental Research

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ABSTRACT

This paper presents the proceedings of an informal meeting on Plutonium and Environmental Metals in Man held in Albuquerque, NM on May 9, 1973.

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PLUTONIUM AND ENVIRONMENTAL METALS IN MAN

Interlaboratory Meeting May 9, 1973

by

Evan Campbell, Section Leader
Bio-Assay Chemical Analysis Section
James F. McInroy, Tissue Program

I. INTRODUCTION

An informal meeting was held in Albuquerque, New Mexico, on May 9, 1973, to discuss the various aspects of the analytical procedures and handling of human autopsy tissues for plutonium analysis. Three types, or cases, were defined with each having its own unique problems.

a. General population, no known occupational exposure to the actinide elements.

b. Former employees of AEC contractors or laboratories which had an exposure potential to actinide elements.

c. Transuranium Registry cases representing known exposures to plutonium and other actinides.

The purpose of the meeting was to coordinate the efforts of the three main laboratories [the Los Alamos Scientific Laboratory (LASL), Los Alamos, New Mexico; Battelle Northwest Laboratories (BNWL), Richland, Washington; Dow Chemical Company (Dow RF), Rocky Flats, Colorado], to carry out the tissue analysis program. By such an exchange of ideas, a degree of similarity between laboratories would enable the data from the laboratories to be evaluated on an equal basis.

A. Opening Remarks

Joseph Goldstein, M. D., "These sessions have been planned as purely technical, for without these technical capabilities this program will fail. The first priority within the AEC and in DBER are the plutonium programs...The Tissue Program's contribution to the overall use of plutonium will be the development of standards based on human data. This in no way reflects on the important work of plutonium effects studied in animals ...We must develop a solid program that is coherent and comparable. Unless the data coming out of the various laboratories are comparable, we are heading down a blind alley...I hope this meeting will consist of constructive criticism among yourselves. This interdisciplinary Tissue Program will continue with this kind of inter-laboratory communications."

B. General Discussion

The purpose of the tissue analysis program is to establish the level of Pu in the general population and detect any trend or changes in the Pu concentration. The importance of Pu in the nation's future and the need for more and complete human data were discussed. The 5 millirem per year exposure to the public has been proposed by the regulatory branch of the AEC. This is equal to approximately three 1 um particles of Pu deposited internally per person per year. The analytical chemist is confronted with what appears to be an almost insurmountable task in the future to detect this level of Pu in man. At the present, the analyst is working at the limits of today's technology.

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II. INTERLABORATORY CALIBRATION

Interlaboratory calibration is to insure an equal basis between laboratories. Approximately 1-1/2 years ago, the Los Alamos Scientific Laboratory prepared a set of plated standards of ^{239}Pu, ^{238}Pu, and ^{236}Pu on stainless steel. The plates were to be counted in the equipment of each participating laboratory. The BNWL has counted the plates for total alpha activity using the NTA counting method, but has not completed the analysis by alpha spectrometry. Upon completion of the alpha spectrometric analysis by BNWL, the plates will then be sent to Dow Chemical Company at Rocky Flats and finally returned to LASL for distribution within LASL. The plates will then be sent to any AEC contracting laboratory that would like to compare their counting techniques with the three participating laboratories. This set of plates has also been counted by the Colorado Department of Public Health and the agreement between LASL, BNWL, and Colorado was excellent. Because, at the time the plates were prepared, ^{236}Pu was being used as an internal tracer, ^{242}Pu was not included. The preparation of additional plates appeared unnecessary. The laboratories were asked to analyze the plates and return the raw count data to LASL for evaluation as soon as possible.

A. Quality Control

Beef bone (20 Kg), liver (25 Kg), and lung (25 Kg) were ashed to a gray white but not whitened. Samples were well mixed, weighed out, and sealed in plastic bags. Each bag was spiked by pipetting a known amount of ^{239}Pu into the powder. A sample of the spiking solution was included for evaluation. The pipetting error was minimal and has been evaluated. All counting data are to be sent to BNWL for evaluation. A set of 40 samples and a spiking solution have been sent to LASL, BNWL, DOW RF, and the Health and Safety Laboratory (HASL), New York.

The quality control procedure in the Tissue Program at LASL was discussed and as soon as sufficient data are available a report will be prepared.

The existence of International Atomic Energy Agency (IAEA) tissue samples was noted and when more data are known on their availability, the information will be distributed to the participating laboratories. All laboratories are to give "top" priority to interlaboratory evaluation and quality control so that all data can be compared.

B. Sample Size

Because LASL is requesting both lungs, the whole liver, and both kidneys, the Laboratory has not been getting as many cases as might be available if smaller amounts of tissue were to be requested. Direct contact with the pathologist appears to be the only way in which the laboratories can obtain the desired sets of tissues.

LASL has been working with some 15-20 pathologists and most prefer not to take all of each organ. If 500 g of lungs, 500 g of liver, one kidney, and the standard sets of other organs were requested, the number of available cases could be increased. This would reduce the amount of tissue by only a factor of 2 or 3 and by proportional aliquoting give essentially the same statistical significance to the data. BNWL has been collecting these smaller weights routinely. An experiment to evaluate the use of smaller tissue weights in the LASL Program is in progress. If it can be established that smaller samples of tissue can be used without sacrificing detection levels, the program will benefit from increased number of samples. It was also mentioned that the ashing time of the large samples is also a problem and that lower recoveries have been observed as the tissue weight is increased. If less than both lungs are requested, a single lobe from the same anatomical location is necessary because of the lack of homogeniety in the distribution of Pu in the lung. It will be impossible to extrapolate analytical results to both lungs even with consistant selection of lung tissue. It appeared from the discussion that both lungs should be obtained when possible.

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It is possible that certain selected lymph nodes, such as the saddle lymph node (one of the large right tracheobronchial lymph nodes) may be an index to general population exposure to environmental Pu. The analysis of a large number of Transuranium Registry (TUR) cases may serve as a basis for this selection.

The legal problems concerning the granting of permission for autopsy and the size of the tissue samples removed were discussed and each laboratory was encouraged to consult their legal sections. Dow RF has not been confronted with this problem. They select only certain cases of interest and obtain a release directly from the family to permit the pathologist to remove the entire organ of choice for the purpose of analysis. There appears to be no problem when the laboratory personnel are actually in attendance at the autopsy. LASL and BNWL have experienced some problems when attempting to work with pathologists outside of the immediate laboratory area. It was suggested that a standard form releasing the tissue for "the advancement of medical knowledge" be sent to each pathologist. Some doubt was expressed about the acceptance of a second permit form by the pathologist since experience has indicated that this tends to discourage the next of kin from releasing the body for routine autopsy. It was also pointed out that the use of the local coroner's office pathologist or medical examiner is advantageous to the program. BNWL, however, has avoided coroner's cases to prevent possible legal involvement.

The possibility of pooling portions of organs from similar cases was discussed. With the detection levels that are presently accepted, the minimum tissue weight that could be analyzed to detect Pu in 95% of the general population is 300 g of lung, 100 g of liver, 300 g of kidney, and 1500 g of bone. If the median values of Pu concentration are accepted, a minimum of one kidney and 50 g bone are required. The smaller organs must be analyzed as a whole. Pooling of samples may more truly represent an average for the population, without regard to age, sex, disease, cause of death, etc., but would cause other errors in interpretation. Each laboratory should investigate this problem with the possibility of evaluating the general population in this manner.

The use of a smaller number of good samples with appropriate case histories, regardless of the detection limit of the program, is better than a large number of unidentifiable background samples. The statement was made that efforts should be directed toward the accumulation of excellent data on fewer samples rather than a large number of cases that would produce poor correlation with environmental levels.

C. Tissue Bank

A review of a meeting held to discuss the possibility of an Interagency Tissue Bank was presented. The purpose of the Tissue Bank would be to establish the pollutant levels in man with respect to age, geographical area, type of pollutant exposure and many other factors. Before a case or set of tissues would enter the bank, a complete work history, medical history, residential pattern, habit profile, and case review would be obtained. Each tissue would be analyzed for specific metals and organic compounds. Core samples would be taken from the larger organs. Portions of the minor organs including several bone site samples would be taken, also. The tissues would be homogenized and divided into two parts. One part would be frozen and held at -70° C and the other vacuum dried. It was recommended that the program include analysis for Pu, T, ^{131}I, and possibly some other of the more exotic nuclides, as well as the standard elemental analyses recommended. Since the specimen sizes would be relatively small, pooling of samples would be required for Pu analysis in the tissues provided from the bank.

Comments were made to the effect that the existing AEC Tissue Program is also a

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Tissue Bank. The Program must understand what our present capabilities and resources are and what our future responsibilities may be. The evaluation of tissues from all communities is necessary to completely understand the possible contribution of AEC installations to the environment and, subsequently, to the general population.

D. Frozen and/or Formalin-Fixed Tissues

LASL receives mainly frozen samples prior to preparation for analysis. BNWL receives only formalin-fixed tissues while Dow RF collects fresh tissue only. There appears to be no major problems associated with the treatment or analysis of formalin-fixed tissues. Analysis of the excess formalin to determine the leaching possibility indicates that this is not a problem. However, there may be a problem with the fact that the formalin only fixes the outer few centimeters of the tissue and not the internal sections. This must be evaluated on formalin-fixed Transuranium Registry (TUR) samples by BNWL.

The problems with shipping were discussed. It was noted that formalin-fixed tissues must be shipped in liquid and that the dissection of lymph nodes from the lung is more difficult. The formalin-fixed tissues may present additional problems during ashing. All TUR cases are formalin-fixed. Sectioning to remove certain tissues prior to analysis is simplified by the character of the tissue. Embalmed tissue was discouraged particularly for TUR cases because the dissection of the tissues was difficult, if not impossible.

The removal of pulmonary lymph nodes by TUR was discussed. The handling of lungs from TUR cases has been carried out by filling the lungs with formalin and kneading the tissue to insure the filling of all the air spaces with formalin. The lung is then suspended in formalin for four days. The dissection of the tissue is simple and straight forward for freshly prepared tissue. There was no experience with the fixing of previously frozen tissues.

Only 30 - 40 identifiable lymph nodes with diameters of 1 mm or larger are normally found in any lung. There are problems of interpreting the amount of Pu in the lungs when the data from a dissected lung is compared to the data acquired from the analysis of a whole lung. The extrapolation of radiation dose to lung is not practical when you consider the nonhomogeniety of the Pu distribution between the lung and the pulmonary lymph nodes or lymphatic tissue.

Most laboratories expressed some difficulty in removing the lymph nodes even when the tissue is fixed with formalin. In most cases the tracheobronchial lymph nodes were removed but the deeper pulmonary lumph nodes were not. The concentration of Pu in the pulmonary lymph nodes is, therefore, associated with the results of the lung tissue analysis. It was decided that the next several TUR cases would be used to evaluate the possibility of selecting only the tracheobronchial lymph nodes, hilar lymph nodes or mediastinal lymph nodes as an index to exposure. If the saddle lymph node can be identified and removed individually, it would be run as a separate analysis. It will only be by using a variety of registry cases that such an indicator can be selected. Data from occupational inhalation exposures may be accumulated to indicate an organ or tissue that may be selected as an index of environmental exposure. This would not eliminate studies related to puncture wounds, nitrate inhalation, or other type exposures in our efforts to document the distribution of Pu in tissues.

The protocol prepared by the TUR for autopsy and tissue handling will be followed. The registry will remove the upper lymph nodes for this study.

III. SPLITTING OF SAMPLES

Multiple analyses of a tissue by splitting of a sample is impractical, particularly for interlaboratory verification. The liver appears to be the only tissue that may be split into parts and have some confidence

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in the homogeniety of Pu distribution. Special homogenization would be necessary for most tissues. The analysis of aliquots of the ashed tissue solution may serve as a sample for interlaboratory verification. However, replicate analyses by a single laboratory appears to be the more practical approach statistically. This, then, would place greater reliance on the interlaboratory calibration to validate each laboratory's capability and each laboratory would be performing its own analyses on its own population. Because of the complete quality control that is planned with interlaboratory calibration, it appears unnecessary to split samples.

A. Special Study of an Occupationally Exposed Case

LASL is presently involved in the analysis of tissue from a former employee who suffered a traumatic death. The case is assigned to the Transuranium Registry.

Based on the best estimate from urine analysis for Pu, the former employee had approximately 40 nanocuries Pu whole body. He was a 49-year old white male, and had had several incidents involving plutonium and americium during his 26 years of employment. Because of the importance of this particular case, these samples will not be ashed until all non-destructive analyses have been completed. The easily obtainable lymph nodes have been removed. The lung was inflated with dry nitrogen and suspended in a freezer. The upper left lobe was removed and sliced while frozen into sections approximately 5-7 mm thick. Each slice was kept frozen and counted on a Phoswich X-ray detector. Each section is currently being autoradiographed on X-ray film. Even six weeks of exposures have resulted in doubtful images that were too difficult to interpret. These slices will now be dissected to remove a 3mm layer of pleura and subpleural lung tissue. All identifiable lymph nodes will be removed. The upper left lobe will be analyzed in this manner to evaluate the techniques prior to continuing with the other four lung lobes. Microtome sections will be removed for nuclear tract alpha counting. Several lymph nodes will be treated to isolate the particulates and determine the particle size of the Pu-Am present. A single lymph node was removed initially for preliminary analysis, and it appears that both from alpha pulse height spectrometry and X-ray counting that 90% of the activity is $^{239-240}Pu$ with about 10% Am and only a trace of ^{238}Pu. Sections of bone including vertebrae, rib, sternum, and several pieces of soft tissue have been submitted to the University of Utah (Webster Jee) for special fission fragment counting.

B. Fission Fragment Counting for Occupational Exposure Cases

The autoradiogram using NTA is satisfactory for only extremely high levels of plutonium deposition in bone or soft tissue. A technique that appears promising is the use of fission fragment counting. Experience has shown that fission fragments can be seen on Lexan^R films when the bone is in contact with the film and exposed to a neutron source. The "old" NCR reactor used for activation of the specimens was very satisfactory because the bone image also became visible. A new reactor is being sought and the TUS-1 Ames reactor appears promising. The fission fragment tracks are clearly visible on the bone surface and inside of the bone structure. The punctate tracts (star) from the bone marrow region are identifiable. These punctate tracts indicate that the plutonium is associated with the microphage system. Soft tissue may be studied in the same manner. Tissues from LASL (case presented above) and several cases from Dow RF are being studied at this time and should be out of the reactor and evaluated in three to six months. There appears to be no problem in working with frozen tissue.

The bone was prepared by sawing a small section out of the bone area and fixing the section in acetone. The bone was then

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sliced on a special microtome and mounted on Lexan^R film for exposure. The following procedure was recommended by Dr. Jee to provide the best bone sections for this study: Remove transverse and lateral sections from the bone with a jeweler's hacksaw. A whole vertebra and sternebra is preferred. The sections should be sawed about 6 mm thick to avoid excess saw dust in the marrow region.

In a search for the proper neutron flux and low enough temperature the LASL-OWR reactor will be studied. Several ports are presently available and a new epithermal port may be available soon.

Soft tissue can be submitted to Dr. Jee as cases become available. These studies may indicate that plutonium is present in the microphage. However, the method to produce the outline of the bone image requires more study. Dr. Jee will distribute to each laboratory the sampling protocol. Films will be sent to Los Alamos for exposure in the OWR reactor to determine the temperature effect.

Existing tissues (only soft) presently in LASL Tissue Bank representing occupationally exposed cases can be submitted for fission counting to assist in the interpretation of the plutonium distribution.

Autoradiography and fission counting are comparable for high levels, but for low plutonium concentrations only fission counting can be used because of the increase in sensitivity

C. Internal Spiking (^{242}Pu vs ^{236}Pu)

Most laboratories have used ^{236}Pu (from HASL) as an internal spike to determine chemical yield of the analytical procedure. Plutonium 238 has become of considerable interest and the use of the ^{236}Pu spike is impractical because it is contaminated with ^{238}Pu. The high energy of ^{236}Pu also made the determination of ^{238}Pu difficult. Work at LASL has shown the use of ^{242}Pu to be much more desirable because:

1. The energy of ^{242}Pu is less than the ^{239}Pu and the ^{238}Pu. Therefore, the tailing effects and interferences observed with ^{236}Pu are eliminated.

2. The purity of ^{242}Pu that is available is significantly better than the ^{236}Pu originally used.

The spiking level used at Los Alamos is 1.66 dis/min. This does not interfere with the low levels of ^{239}Pu or ^{238}Pu normally found in the tissue of the general population. The interference of ^{239}Pu with the ^{242}Pu was questioned, but the energy separation appears to be far enough apart to fully discriminate by alpha pulse height analysis. High levels of ^{239}Pu on a poorly prepared plate would interfere with the recovery factor determined from the ^{242}Pu internal spike, and would have to be corrected. The alpha ratio of ^{239}Pu to ^{242}Pu in the spike solution is on the order of 10^{-5} and the ^{238}Pu to ^{242}Pu ratio is similar, based on the analysis submitted by the Lawrence Livermore Laboratories (LLL). LLL will be making this isotope available commercially. Each of the laboratories represented at this meeting is already on the shipping list and should receive their spike solution soon.

The present supply of ^{236}Pu from HASL is limited and the ^{236}Pu that is available from Oak Ridge is not sufficiently pure for spiking purposes. The ^{242}Pu from Oak Ridge has not been found to be satisfactorily pure for biological analysis of ^{239}Pu or ^{238}Pu. It was noted that during all of the discussions any reference to ^{239}Pu is in fact the combination of ^{239}Pu and ^{240}Pu. It was agreed between the laboratories that the use of ^{236}Pu as an internal spike eliminated the determination of low levels of ^{238}Pu and that all of the laboratories would adopt the use of ^{242}Pu as an internal spike as soon as possible.

D. Ashing of Samples

LASL is presently dry ashing all samples prior to a nitric acid-hydrogen peroxide treatment to whiten them.

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BNWL formally used wet digestion only, however, that laboratory is now converting to a dry ashing system followed by a nitric acid whitening procedure. Dow RF is presently wet ashing their samples. The Dow RF procedure is essentially the same as that used by BNWL. All laboratories are doing a final wet ash involving hydrofluoric acid. It was pointed out that LASL is using 30% hydrogen peroxide during the final nitric acid whitening procedure and that most of the wet ashing is carried out in Teflon beakers. The use of stabilized 90% or 50% hydrogen peroxide was discouraged because of the high concentration of metal ions used as an inhibitor.

The use of HF has caused some problems. However, all laboratories are using boric acid prior to the isolation of plutonium. The ashing procedure proposed during the discussion may be summarized as follows:

1. The wet tissue is dried by heating slowly at 100° - 200° C (less than 300° C) and finally ashed at 450° - 500° C.
2. The tissues are then whitened with nitric acid-hydrogen peroxide and treated with hydrofluoric acid.

A unique method of dry ashing was mentioned by BNWL: The specimen is placed in a preheated oven at approximately 400° C with a piece of tissue paper on top of the wet tissue. This procedure eliminates the caramelization and the foam that has been experienced by some of the laboratories. It was also pointed out that if the temperature is maintained at less than 300° C for greater than 24 hours the caramelization and foaming is less likely to occur. High frequency (microwave) or cold ashing techniques for large tissue samples were discouraged because of the problem of internal heating. The porcelain and Pyrex dishes that have been used for ashing tissues from occupationally exposed cases are normally discarded and not reused. Most of the laboratories have found that cleaning of the laboratories glassware with nitric acid using a standard procedure for washing was satisfactory for the low level general population type of samples but impractical for the higher levels of occupationally exposed cases. All Teflon ware is washed and rinsed with nitric acid prior to reuse.

E. Point of Spiking

Because of the impurities associated with the ^{236}Pu spike and the daughters from its decay, LASL decided early in the Tissue Program not to spike the wet tissue prior to ashing. The reason for this was the desire to re-analyze the sample as the chemical and instrumental techniques improved. Beef tissue samples spiked with ^{239}Pu prior to ashing were carried through the analytical procedure in order to estimate the overall recovery. It was pointed out by BNWL that dry ashing increased the yield over the wet ashing procedure and that in both cases Pu losses were minimal during the ashing phase. LASL adds their tracer at the time of aliquoting. BNWL has always used spiked control tissue because of their use of the NTA counting techniques. Dow RF run their samples immediately and analyze the entire sample spiked with a tracer at the time of wet ashing the tissue.

It was agreed that the new ^{242}Pu available for internal spikes should be added at the original wet or dry ashing stage. The technique of spiking was discussed.

F. Homogenization

Most tissues can be homogenized by many methods. The homogenization of the lung would presume that the particles would also become homogeneously dispersed. Homogenization prior to freeze drying would assist in the drying process if freeze drying of the tissue was required. This would be advantageous if solid aliquots were to be taken for other analyses or for storage. A similar procedure is used by the Physical and Chemical Branch of NIOSH in their Tissue Program.

G. Dissolution of the Tissue Ash

LASL dissolves the white ash residue

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from each tissue in a fixed volume and analyzes only an aliquot. Since 1970, the following volumes and aliquots have been used for each tissue listed:

Tissue	Volume (cm^3)	Aliquot Analyzed (cm^3)
Liver	1000	500
Lung	1000	500
Bone	250	50
Kidney	100	50
Lymph Nodes	50	10
Gonads	50	10

Dow RF and BNWL have, to this date, analyzed the entire sample received. New facilities are being made available to both laboratories to enable them to store dissolved tissue samples. This will permit all laboratories to analyze aliquots of each solution and retain the balance for replicate or other analyses. The desirability of the three laboratories using uniform dissolution volumes and aliquot sizes for the analysis of similar tissues was agreed upon. The optimal volumes and aliquot sizes to be used will be decided upon at some future date.

IV. SUMMARY OF ANALYTICAL METHODS

A. BNWL

The ashed tissue is dissolved in 7.7 M HNO_3 and ion exchanged on AG 1-X2, 50-100 mesh column (5 mm x 70 mm). The column is prepared by washing the resin with 12 M HCl, 6 M HCl and finally with 7.7 M HNO_3. The sample is added to the column and the column washed with three column volumes of 7.7 M HNO_3. The column is then washed with six volumes of 10 M HCl and the washings analyzed for Am. The Pu is eluted from the column with 6 M HCl ~ 0.024% HI. Nitric acid is added to the eluate and evaporated to dryness. Concentrated H_2SO_4 (0.25 cm^3), water (1.5 cm^3), and two drops of thymol blue are added to the plutonium fraction. The pH is adjusted and electroplated on stainless steel (0.5 in. diam) at 240 ma for two hours. Ammonium hydroxide (5 cm^3) is added to the electrodeposition cell 30 sec before terminating the electrodeposition. The plates are washed with alcohol, dried, and counted.

B. Dow RF

The ashed tissue residue is dissolved in 9 M HCl and ion exchanged on an AG 1-X4, 100-200 mesh chloride form column. The column is washed with 7.2 M HNO_3 and two 12 M HCl washes to isolate Am. The plutonium is eluted with 50-60 cm^3 of 9 M HCl saturated with NH_4I. The eluate is treated with 0.5 cm^3 of concentrated H_2SO_4 and fumed to SO_3 vapors. The acid is neutralized with NH_3 gas (silica in NH_4OH interferes) and plated for two hours. The cell consists of a glass tube and 3/4 inch plate held in position with a finger cot.

Americium is determined on the 12 M HCl wash by evaporating the acid solution to dryness and dissolving the residue in 4 M HCl. Yttrium carrier is added and the pH adjusted to 2. The solution is extracted with DEHPA, back extracted with 8 M NH_4SCN and ion exchanged. The column is washed with 5 M NH_4SCN and finally eluted with 0.3 M HCl. The eluate is evaporated, ashed with aqua regia and electroplated in the same manner as Pu.

C. LASL

The residue is dissolved in 2 N HNO_3 and stored for analysis. The proper aliquot from each tissue solution is evaporated to dryness repeatedly with HF and then dissolved in 7.2 N HNO_3 and again evaporated to dryness. The residue is dissolved in 7.2 N HNO_3 (~500 cm^3 for lung and liver tissue), 0.2 gm Na NO_3 is added and allowed to stand overnight. The solution is centrifuged and the supernatant anion-exchanged on AG 1-X4; 50-100 mesh resin. The column is eluted with 50 cm^3 of 0.36 N-0.01 N hydrochloric-hydrofluoric acid and evaporated to dryness. Any visible residue is dissolved in 7.2 N HNO_3 and anion-exchanged on a 0.5 cm x 8 cm column. If the beaker is clean after evaporation, 2 cm^3 of 6 N HCl are added and evaporated to dryness to

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remove any nitric acid. The residue containing the plutonium is dissolved in the electrolyte (4% oxalic acid - 0.33 N HCl) and electroplated on 0.50 inch stainless steel plates at 200-250 ma for three hours. The solution is neutralized with concentrated NH_4OH and the plates are washed with distilled water, dried, and counted. Disposable cells are used and no mechanical stirring is employed.

The meeting was concluded with the possibility of meeting again in the near future when all interlaboratory calibrations have been completed.

The information reported above may have errors, in fact, because of interpretations of, or incomplete notes. The participants have not had an opportunity to review the statements.

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APPENDIX

ATTENDEES, TRANSURANIUM TISSUE ANALYSIS MEETING

Atomic Energy Commission (DBER)

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