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#### CHARACTERIZATION OF TRANSURANIC SOLID WASTES

#### FROM A PLUTONIUM PROCESSING FACILITY

#### by

### Ray Mulkin

#### ABSTRACT

Transuranic-contaminated wastes generated in the processing areas of the Plutonium Chemistry and Metallurgy Group at the Los Alamos Scientific Laboratory (LASL) were studied in detail to identify their chemical and physical composition. Nondestructive Assay (NDA) equipment was developed to measure transuranic activity at the 10-nCi/g level in low-density residues typically found in room-generated waste.

This information will supply the Waste Management Program with a more positive means of identifying concerns in waste storage and the challenge of optimizing the system of waste form, packaging, and environment of the storage area for 20-yr retrievable waste. A positive method of measuring transuranic activity in waste at the 10-nCi/g level will eliminate the need for administrative "control in a sensitive area, and will provide the economic advantage of minimizing the volume of waste stored as retrievable waste.

#### I. INTRODUCTION

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The radioactive waste resulting from the handling of uranium, plutonium, and other radionuclides has been recognized as a special problem since the beginning of the Manhattan Project. As the nuclear industry has developed and expanded, specific guidelines have been established for particular waste streams to control waste form and methods for storage in such a manner that the environment is adequately protected.

In 1970, the General Manager's Office of the U. S. Atomic Energy Commission (AEC) issued Immediate Action Directive No. 0511-21 specifying that solid waste contaminated with <sup>233</sup>U and its daughter products, plutonium, and transplutonium nuclides (except <sup>238</sup>Pu and <sup>241</sup>Pu) could continue to be stored in conventional AEC-approved burial grounds if their level of radioactivity did not exceed 10 nCi/g. Plutonium-238 and <sup>241</sup>Pu were to be handled as transuranics (TRU) when so indicated by <sup>239</sup>Pu impurities, or when required by local burial criteria.<sup>1</sup> Solid wastes contaminated to a level of greater than 10 nCi/g could no longer be buried, but were to be stored at AEC sites, segregated from other radioactively contaminated solid waste, with combustible and noncombustible TRU-contaminated waste packaged separately. The packaging and storage conditions were to be such that the packages could be readily retrieved in an intact, contamination-free condition for 20 yr.<sup>2</sup>

In order to meet the segregation, measurement, and packaging requirements<sup>3</sup> of retrievable storage it was recognized that the kinds of TRU-contaminated solid waste would

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have to be identified and categorized. In addition, the data obtained from a sorting study would be relevant to efforts aimed at optimizing the waste packaging, handling techniques, and storage facility designs required for retrievable storage. Waste treatment facilities can be more effectively designed if the characteristics of the influent stream are known. Finally, a knowledge of residue types, volumes, and radioactivity content as a function of origin is essential toward achieving a reduction in the amount of waste being generated.

The Plutonium Chemistry and Metallurgy Group operations in Technical Area(TA)-21 at LASL offered a unique study area which could be used for evaluating the generation of TRU waste. All unit operations involved in <sup>239</sup>Pu metal handling and a complete scrap recovery system are located in this area. Figures 1 and 2 describe a typical plutonium metal cycle and some of the major process residues handled by scrap recovery. Other operations in the study area include basic plutonium chemistry research, development work in Liquid Metal Fast Breeder Reactor (LMFBR) fuels, and development work using 80% <sup>238</sup>Pu as an energy source for Space Nuclear Systems and artificial hearts --providing an even broader spectrum of wastes.

### II. CLASSES OF RESIDUES

The radioactive waste examined in this study consisted of two major streams: residues generated by process operations in the glovebox or hood enclosures and residues generated in the operating room or area containing the transuranic process facilities. Packaged residues are normally sent to scrap recovery for measurement of the plutonium content or transuranic activity by Nondestructive Assay (NDA) techniques.

Residues are considered recoverable if the plutonium content is sufficient to warrant reclaiming and reusing, based on local criteria. Such packages of measured residues can then be defined as scrap or feed material for a scrap recovery process. Packaged residues not considered recoverable, but which are above 10 nCi/g in transuranic activity, are defined as retrievable waste and are logged into a "20-yr-retrievable waste drum." Disposable residues from an area containing transuranic processing facilities are those items containing less than 10 nCi/g of transuranic activity. Such lowlevel waste may be disposed of in a nonretrievable manner such as land burial, but in a controlled area.

<u>Process-generated residues</u> are usually classified as recoverable (scrap) or retrievable (waste), with a low probability of finding packages with transuranic activity less than 10 nCi/g. <u>Room-generated residues</u> are assumed to contain at least trace quantities of transuranic activity simply from having been in the process area, but are normally less than 10 nCi/g. A small portion could be more than 10 nCi/g, but recoverable levels would not be expected.

## III. PROCESS-GENERATED RESIDUES FROM A TYPICAL OPERATION

The first phase of this waste characterization study was the identification of process residues at the point of generation in the plutonium metal fabrication area. Processes generating residues include research and development work in casting, machining, welding, assembly, and disassembly, plus a variety of other experimental operations in fabrication, preparation of test specimens, and metal handling.

Process-generated residues from all operations are transferred through a conveyor system to one glovebox line for disposal. Material is removed from the glovebox by standard bag-out procedures and transferred to the scrap recovery area for assay, normally by use of a neutron coincidence counter. Packages with recoverable quantities of plutonium are transferred into scrap recovery and discardable items are logged into



Fig. 1. Plutonium metal cycle.



Fig. 2. Flow diagram of plutonium scrap recovery operations.

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a retrievable waste drum.

For this study, operation of the waste collection glovebox was manned by members of the research team. The first objective was to determine the amounts of various material types generated in such operations. Then each waste type was sorted into high and low contamination levels by both visual examination and by use of a gamma probe.

During the study period, 483 kg of residues were processed. Using a discard limit of 0.5 g of Pu/kg of waste (0.5 g/kg), 59 wt% of the material (containing less than 10% of the plutonium) was put into retrievable waste drums. Material types, composition, and plutonium content of each stream are shown in Table I. More detailed description of waste items observed are given in Appendix A.

The procedure of sorting waste before removal from the glovebox has economic practicality. Normally, the entire waste stream is transferred to scrap recovery and then sorted into waste types according to the recovery process used. However, when wastes were sorted before removal the costs were reduced since incurred recovery costs were only those associated with handling 41% of the waste stream.

IV. ROOM-GENERATED RESIDUES

Residues generated in an operation area or a room containing transuranic process facilities are usually of considerably lower contamination levels than the processgenerated residues. Since the radioactivity content is not typically expected to be at the recoverable level, these residues are more commonly referred to as room-generated waste or room trash. Much of the trash is not really contaminated at all, but some waste generated during maintenance work and cleaning work around process equipment such as transfer lines might contain some contamination.

These wastes are collected in polyethylene bags blaced inside 0.06-m<sup>3</sup> cardboard boxes which are inside 115-l drums covered with flame-retardant lids. The new Division of Waste Management and Transportation

PROCESS-GENERATED	RESIDUES FROM FABRICATION AREA
April	73 to September 73

TABLE I

				Sorted Waste Output			
Unsorted Waste Input		F	Retrievable Waste		Recoverable Scrap		
Material	Pu g	Net Wt. kg	Wt.% of Total <u>Waste</u>	(<0.5 Pu g	g Pu/kg waste) Net Wt. <u>kg</u>	(>0.5 g Pu 	Pu/kg waste) Net Wt. <u>kg</u>
Metal	127	166	34	29	123	98	43
Plastic	36	107	22	17	82	19	18
Rubber	23	39	8	3	17	19	23
Cellulosic	260	29	б			260	29
Glass & Ceramic	4	57	12	4	55	<1 99	2 82
Graphite	99	83	17			33	2
Floor Sweeping	33	2	1				· · · · · · · · · · · · · · · · · · ·
				53	284	529	199
Total	582	483	100	9	59	91	- 41
% of Total	100	100	100				

(DWNT) criteria of 10 nCi/g made it necessary to examine such materials to determine their actual level of contamination.

A hood-glovebox system built around a FIDLER (Field Instrument for the Detection of Low Energy Radiation) Counter was installed in the scrap recovery area. The thin sodium iodide crystal was positioned to interrogate thin, "pancake" shaped packages of low-density trash, and was referred to as the Pancake Counter. Development work, using analytically prepared standards in typical room trash, indicated that the Pancake was capable of detecting activity in the 1- to 10-nCi/g range using 16-keV L-x rays and 60-keV gamma rays.

After in-place calibration of the system, the 0.06-m<sup>3</sup> boxes of typical room trash were introduced into the hood. Waste was sorted by material type into 500- to 1000-g packages. A standard 10-s count of each package was compared to normal background radiation levels to determine the level of activity. Packages assaying higher than 10 nCi/g were transferred into the glovebox for disposal as retrievable waste. Other samples were repackaged for disposal by burial.

A mapping system was initiated to identify the point of origin of samples and the associated type of operation. Samples assaying at  $\geq 10$  nCi/g were traced to their source, and an attempt was made to determine the reason for the high level of activity. The higher levels of contamination were usually caused by residues from maintenance cleanup items, plutonium welding work materials, or plastic adapters used in transferring plutonium nitrate solutions.

During the waste characterization and composition studies, a routine logging system recorded origin of waste, weight of each room-trash box, and the major isotope being processed in the area from which the waste originated. Data obtained from this study will be used to determine means of reducing or eliminating waste streams from some process areas.

The Pancake Counter assay system proved accurate in measuring activity in low-density materials; however, activity contained in scrap metal objects and glassware (i.e., high-density items) could not be satisfactorily detected due to excessive attenuation of the low-energy gamma and x rays. Cross checks were thus begun between this system and a Multienergy Gamma Assay System (MEGAS) which not only measures low energies but is also capable of detecting higher energy gamma-ray emissions. Hardware was fabricated to facilitate assay of the standard 0.06-m<sup>3</sup> trash box, resulting in the designation of Box Counter. Boxes of typical low-density room trash were opened and their contents assayed by the Pancake Counter. Samples were then repackaged and scanned by the Box Counter for a total box count. (A detailed discussion of the development of this instrumentation is presented in LA-5904-MS)."

The Box Counter was installed in the Trash Monitoring Room at the scrap recovery area in 1974. It is now used as the routine monitoring system for room-generated waste associated with all plutonium operations in TA-21. Though originally calibrated for reporting activity from weaponsgrade plutonium, the MEGAS was programmed to identify the major transuranic isotope or fission product in a box and to calculate the appropriate activity of the box in In a 4-month period, 487 boxes were nCi/g. assayed using the Box Counter (see Table II for composition and Fig. 3 for activity distribution). Although 12% of the boxes assayed greater than 10 nCi/g, some contained <sup>238</sup>Pu\* and/or mixed fission products, so that only 8% were transferred to retrievable storage or returned to the sender. The

<sup>&</sup>lt;sup>\*</sup>The LASL retrievability limit was 100 nCi/g for <sup>238</sup>Pu materials.

COMPOSITION OF ROOM TRASH	
<u>v</u>	olume 🕯
Cellulosics	83
Chart paper, computer paper,	
surgeon's glove boxes, kraft	
paper, masking tape, cheesecloth	,
clothing - (coveralls, caps,	
booties, undershirts, shorts),	
paper towels	
Surgeon's gloves	4
50 % each rubber and plastic	
Plastic	4
Polyethylene bags, bags from fac	e
masks, reagent bottles and bags	
Styrofoam	3
Packing material and coffee cups	
from "In-Plant Coffee Room"	
Glass	1
Sample bottles, glass wool from	
room air prefilters	
Metal	5
Flashlight batteries, wire, con-	
duit, tin cans, aerosol cans,	
aluminum foil	

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Fig. 3. Activity distribution in room trash.

remaining 92% were sent to land burial as disposable waste.

Room-trash boxes are not compacted except for limited manual compression as they are being filled. Density data collected during a 4-month period showed that the boxes had an average density of 86 kg/m<sup>3</sup>.

A brief study was made in the plutonium processing facility while using the MEGAS instrumentation (see Fig. 4) to evaluate the economics of alternative methods for the disposition of room trash. Approximately 8% of the room-trash boxes contained sufficient TRU contamination to require retrievable storage. The economic analysis showed that the MEGAS operation is more economical than the alternative of administratively assigning all room trash to retrievable storage. (Table III shows the cost comparisons of the two alternatives.) This study considered only short-term costs, which included manpower, materials, on-site transportation, pit operation, trash volume, and alternative tasks for personnel. All factors indicate that long-term application of the MEGAS would show an even more favorable economic comparison.

### V. RETRIEVABLE WASTE

The retrievable waste stream from all the Plutonium Chemistry and Metallurgy Group operations, including scrap recovery, ccnsists of all process-generated residues that are below established recoverable limits combined with any room-generated residues that assay greater than 10 nCi/g.

The previously described evaluation of room trash demonstrated that certain operations, such as maintenance on process equipment--even though performed in open room areas under controlled conditions--did in fact result in trash contaminated to activity levels above 10 nCi/g. The next question to be answered was whether any of the process-generated wastes could be less than 10 nCi/g.



Fig. 4. MEGAS (Box Counter).

A glovebox glove, which had been cleaned by wiping thoroughly with wet cheesecloth, was assayed in the Pancake Counter. The measured activity in the package was over 1000 nCi/g. For a 750-g glove, this activity would be equivalent to 10 to 20 mg of 239Pu. In a second study, the contents of several 1-L ice crean cartons of dry chemicals were transferred to mixing jars and the empty cartons moved to the nearest bag-out station. Contact with plutonium-contaminated surfaces occurred only when the containers were placed on the glovebox floor and when they came in contact with three or four glovebox gloves during the transfer operation. Nevertheless, assay of the bag-out package showed a plutonium content of 700 nCi/g (equivalent to 5 mg in a 500-g package). Examination of many other items showed similar data which indicate that material from inside gloveboxes and process equipment will be above the 10-nCi/g level.

#### TABLE III

AVERAGE MONTHLY COSTS BY CATEGORIES AND TOTALS FOR ALTERNATIVES

	ALL WASTE to Retrievable Storage	Use of MEGAS to Screen Waste
<u>Material</u>		\$ 90.00
Boxes, bags, tape/mo.	\$ 890.00	70.00
Drums, drum liners/mo.	\$ 890.00	160.00
Total/mo.		
Manpower	70.00	220.00
Counting/mo.	700.00	90.00
Documentation/mo.	770.00	310.00
Total/mo.		
Transportation	140.00	60.00
Total/mo.		
Disposal/Storage Area		20.00
( <u>&lt;</u> 10 nCi/g)	300.00	20.00
	300.00	40.00
Total/mo.		
	2100.00	570.00

GRAND TOTAL/MO.

The retrievable wastes resulting from all plutonium processing operations were processed through the hood-glovebox system equipped with the Pancake Counter during the same time period the room waste was being studied. As the packages of the process-generated waste were assayed by neutron coincidence counter or by the segmented gamma scan, those packages below recoverable limits were introduced into the glovebox and inspected for anything unusual in terms of chemical contaminants, liquids, evidence of oxidation or degradation, etc. The wastes were separated by material type and removed from the glovebox by standard bag-out methods. As drums of waste were filled with a single material type, the total content of each drum was reassayed to provide plutonium content as well as weight and volume of each material type.

The results obtained from the characterized retrievable waste are shown in Table IV, with a detailed discussion of each material type in Appendix B. The distribution of plutonium contamination on the various waste matrices is given in Figs. 5 and 6.

#### TABLE IV

## COMPOSITION OF RETRIEVABLE WASTE FROM PLUTONIUM CHEMISTRY AND METALLURGY GROUP November 1973 to May 1974

	We	ight	Vol	Lume			
Material	ka	% of Total	l	% of Total	Pu.a	g Pu/kg waste	Activity rCi/g
Cellulosic	205	5	1760	9	14	0.066	4700
Plastic	771	. 20	7400	39	121	0.157	11200
Process Solids	1225	32	3375	18	2781	2.271	161900
Metal	863	22	4388	23	165	0.191	13600
Glass	635	16	1407	8	85	0.134	9600
Rubber	189	5	520	3	39	0.208	14800
Total	3888		18850		3205		

## VI. CHEMICAL CONTAMINATION OF WASTE

An inventory of the chemicals used in the plutonium processing areas during one calendar year was compiled with more than 40 reagents identified. Preliminary estimates reveal that only minute quantities of these reagents enter the solid waste stream as contamination. None of the dry bulk chemicals are pyrophoric in nature. All organic liquids which are utilized have high vapor pressures. Consequently, the probability of these compounds entering the solid



Fig. 5. Activity distribution in retrievable waste.

waste stream during normal use and operation is remote. The inorganic bulk liquids (acids, hydroxides, caustics, etc.) are used in plutonium processing steps and are transferred to the liquid waste treatment facility. The absence of any free liquid in the solid wastes characterized and sorted during this study indicates that these chemicals enter the solid waste stream only as contaminants absorbed onto other items. A complete listing of chemicals used, and items which become contaminated with these chemicals, is given in Appendix C.

The large volume of nitric acid used (58 000 L/yr) and the nature of the processes imply that contamination of scrap recovery solid wastes with nitric acid is the rule rather than the exception. Obser-



Fig. 6. Activity distribution in process solid waste stream.

vations have indicated that oxidation-reduction reactions between nitric acid and other chemical contaminants which may be present have attained equilibrium before the contaminated waste material is packaged for retrievable storage. The observed diffusion of nitric acid through PVC bags and the ubiquitous presence of this compound in stored wastes indicate that internal corrosion of the presently used 17C or 17H drums may be accelerated by this chemical contaminant.

## VII. UNIT OPERATIONS RELATIONSHIP

The value of establishing predictable or reproducible relationships between unit operations and generated residues was studied. Possible guides considered were: waste generated per gram of plutonium processed, waste generated per man-hour, or materialtype distribution related to some standard or typical operation. Tables V, VI, and VII show the results of some of these studies from typical unit operations.

The validity or usefulness of this data can be questioned when one analyzes the factors contributing to types and amounts of residues generated. As an example, in Table VII the waste generated by ash leaching shows 23 wt% to be scrap metal. This study was made when the ash leaching operation was concentrating on offsite ash from the Central Scrap Management Office at Richland, and the scrap metal was primarily the inner shipping container. Had the same ash leaching equipment been used for processing locally generated ash from an incinerator in the same glovebox line, scrap metal would have been less than 5 wt%.

Thus, each process at each major ERDA operation will have its own unique set of circumstances influencing the amount and type of waste generated.

TABLE V							
RETRIEVABLE	WASTE	GENERATED	PER	GRAM	OF	PLUTONIUM	PROCESSED

Unit Operation	Pu Processed, g	Bulk Waste, kg	kg Waste/g Pu
Ash Leaching	3 675	167	0.045
Ion Exchange	3 780	20	0.005
Alloy Processing	3 336	11	0.003

TABLE VI RETRIEVABLE WASTE GENERATED PER MAN-HOUR

Unit Operation	Man-Hours	Bulk Waste, kg	kg Waste/Man-Hour
Ash Leaching	320	167	0.522
Ion Exchange	160	20	0.125
Alloy Processing	240	11	0.046

TABLE VII

COMPOSITION OF RETRIEVABLE WASTE FROM UNIT OPERATIONS

Material Type	Composition, wt%						
	Ash Leaching	Ion Exchange	Alloy Processing				
Metal	23	0	22				
Plastic	14	26	17				
Rubber	7	12	0				
Cellulosics	11	2	9				
Glass	14	8	38				
Process Solids	31	52	14				

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## VIII. RELATIONSHIP OF WASTE GENERATION AND PLUTONIUM RECOVERY

Most of the information in this report has been limited to the room-generated and the process-generated residues immediately related to the glovebox operations in plutonium processing areas. Realizing that each part of the plant must assume an appropriate portion of waste such as scrubber solutions, seal liquid from house vacuum systems, and ion exchange effluents, an effort was made to "quantify" typical operations involved in the recovery of two common residue streams. The flowsheet in Fig. 7 describes the incineration of cheesecloth, through leach steps and ion exchange, to produce a product of pure plutonium nitrate. The flowsheet in Fig. 8 describes the production of plutonium metal from nitrate with the associated recovery of plutonium from the major residues--peroxide filtrate and the slaq and crucible. These studies show very clearly the need for improvements in handling liquid waste streams since the end products of liquid waste treatment account for over 95% of the total volume of waste generated.

CONCLUSIONS AND RECOMMENDATIONS IX. 1. The process residues and waste items associated with plutonium handling in gloveboxes were studied in order to more clearly define and evaluate the risks associated with placing these materials in interim 20-yr retrievable storage. The waste items in most cases were found to result from packaging, transfer, storage, and other handling of transuranic materials. A concerted effort should be made to eliminate as many items as possible, reduce the use rate of those items that cannot be eliminated, and look for substitutions that would result in smaller volumes or more easily treatable material. The recycling or reuse of packaging should also be fully evaluated.

2. A decision should be made concerning the amount of radioactivity permitted in transuranic wastes. Economic and ecological concerns are in conflict when considering the discard level of materials from scrap recovery operations going into retrievable waste. Improved process systems are needed in order to comply with the waste management policy<sup>3</sup> of reducing the amount of radioactivity in such waste and still having economical recovery. Recovery of plutonium from process residues, such as incinerator ash, to a lower level is of particular concern.

3. Process-generated residues should be sorted at the point of generation with guidance from trained scrap recovery personnel. Material type categories should, at a minimum, meet the criteria of separating combustibles and noncombustibles and could be coordinated with the recovery processes used.

More refined on-line measurement methods are needed to optimize quantitative methods which, when correlated with discard levels in recovery operations, can minimize the amount of material to be processed and the associated waste resulting from the additional handling.

4. NDA systems designed for on-line work, as described above, are needed to improve the handling of scrap and waste. In recovery operations it is not unusual to remove scrap from a glovebox after routine processing and find that NDA results show the package to be above the discard limit. An on-line system would eliminate the extra handling, additional PVC bags, and other supplies used in glovebox systems. 5. The level of chemical contamination in retrievable waste should be controlled to reasonable concentrations as described in "Guidelines for the Interim Storage of AEC-Generated Solid Transuranic Waste."<sup>5</sup>



Fig. 7. Incineration flowsheet.



Fig. 8. Plutonium metal production flowsheet.

The levels and types of chemicals observed in this study were relatively low and did not appear to present a serious hazard in normal retrievable storage.

One area of concern, however, is the potential hazard of certain cellulosic materials that have been exposed to concentrated nitric acid. Typical examples are cheesecloth or wipes used to clean around dissolvers in gloveboxes, and HEPA filters exposed to fumes from boiling nitric acid. Limited experimental data indicate some degree of nitration can occur leading to self-ignition and possible detonation at slightly elevated temperatures. Incineration of this type of waste should be standard practice until the hazard is more clearly defined.

6. Plant design has a decided influence on the amounts and types of residues to be treated. Facilities such as the new plutonium facility at Los Alamos and the new scrap recovery facility at Rocky Flats have incorporated many features in their design which will reduce the generation of waste and its transuranic content. The volume of room trash associated with processing areas will be greatly reduced by more careful planning of office areas and materials receiving areas. A waste characterization study in the new plutonium facility in 1980 would predictably be entirely different compared to the results in this report, even if the same number of people and the same processes were involved.

7. When evaluating process improvements to reduce residues generated, or when designing systems for stabilizing waste streams before storage, <u>all</u> resulting waste streams must be considered. Liquid waste must receive the same attention as solid waste when considering minimum releases to the environment.

#### ACKNOWLEDGMENTS

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# APPENDIX A RESIDUES GENERATED BY PLUTONIUM METAL FABRICATION

- 1. <u>Non-Pu Scrap Metal</u> Aluminum foil, tin cans, used scrap pipe (stainless steel, mild steel, and aluminum), small obsolete equipment, etc.
- <u>Plastic</u> Primarily polyvinyl chloride (PVC) from bag-in and bag-out operations, and some polyethylene and polypropylene.
- <u>Rubber</u> Mostly drybox gloves.
- 4. <u>Combustibles</u> Almost totally cheesecloth.

5. Glass

Broken laboratory-type equipment such as beakers, graduated cylinders, and Vycor castings sleeves.

- <u>Ceramics</u> Magnesium oxide liners and other refractories.
- 7. <u>Insulation</u> Transite board, asbestos pipe insulation.
- 8. Graphite

Primarily molds and crucibles from cast- . ing operations.

#### APPENDIX B

#### DESCRIPTION OF RETRIEVABLE WASTE BY MATERIAL TYPE

## 1. Cellulosics

All of the cheesecloth used in glovebox operations for cleanup work is reused as long as possible before it is transferred to recovery. In many cases, the cheesecloth has been exposed to nitric acid or oil. This material is guite high in plutonium concentration and is routinely counted and transferred to the incinerator. For this reason, none of the process-generated cellulosics were in the retrievable waste category. Cheesecloth used outside the gloveboxes during maintenance work and cleanup operations contribute a significant portion of the less than 0.05-g/kg stream. Wood filter frames of HEPA glovebox primary filters contribute heavily to the 0.4-q/kg stream.

## 2. Plastic

Retrievable plastic wastes consist primarily of PVC bags and bag stubs. Some sheet material used as temporary floor covering, and laboratory wares such as funnels, petri dishes, graduated cylinders, wash bottles, tubing, and gaskets also appear in this waste stream. If PVC bags are handled with any degree of care, contamination can be held to less than 0.1 g/kg.

#### 3. Process Solids

Incinerator ash, after being leached with nitric acid and calcium fluoride, is the main solid residue from scrap recovery operations. Any other solid material collected from glovebox cleaning is also leached in the same fashion. Other terms used on a local basis include sweepings, heels, ash heels, and leached residues. Present discard limit for this material based on the economics of recovery is 4 g/kg.

4. <u>Metal</u>

Retrievable metal wastes are represented by a diverse stream of nails, nuts, bolts, wiring, conduit, tin cans, stainless steel dressing jars, aluminum foil, lathe turnings, hacksaw blades, screw drivers, tweezers, hammers, hair dryers, hot plates, heating coils, vacuum cleaners, and furnaces. In this study most of the tin cans in which offsite ash had been received from the Central Scrap Management Office at Richland were less than 0.1 g/kg. The contamination present on tools and small equipment can vary greatly depending on usage, time in the glovebox, and cleaning effort.

#### 5. Glass and Ceramic

Retrievable glass and ceramic materials primarily include normal laboratory glassware such as beakers, cylinders, graduated cylinders, and 1-1 to 9-1 bottles. Heating mantles used in batch leaching operations are occasionally discarded, but are normally reduced to a small residue by volatilizing the silicon in a hydrofluorination treatment.

### 6. Rubber

In this study, almost all of the stream was glovebox gloves. When a box of room trash was found to be over 10 nCi/g, and the waste was merged with the process generated waste, some surgeon's gloves would contribute to this waste. Occasionally items such as rubber stoppers and tubing would appear.

## APPENDIX C

## STUDY OF CHEMICAL CONTAMINATION ON TRANSURANIC WASTES

## Process Chemicals Used in Plutonium Processing Area During CY 1973

		ANNUAL	i nam bangananan	
CHEMICAL	AREA	ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Aluminum Nitrate	Scrap Recovery	3182	Used to complex fluoride ions in nitrate solutions from reduction residue dissolvers, bulk fusion solutions, and in ion exchange feed adjustment.	Trace quantities may be found in exhaust filters, on dry box gloves, and on rubber window gaskets. If spilled, may be found in larger quantities on wet cheesecloth. Small amounts may also be found on cardboard transfer containers.
Ammonium Bifluoride	Metallography	0.5	May be used in combination with acids for metal etch- ing processes. Used in quantities of l g or less.	Found in very low quantities in cheesecloth when spilled. Disposal may cause contamination on PVC plas- tic bags or on polyethylene jar.
Calcium Fluoride	Scrap Recovery	93	Used with nitric acid for the leaching of incinerator ash.	Trace quantities may be found in exhaust filters, on dry box gloves, and rubber window gaskets. Larger quantities are found on heating mantles when process solutions boil over. Residual amounts are found in empty reagent bottles.
Carbon Tetrachloride	Fabrication	4 L	Used with dry ice in a dew point chamber. This product is no longer used.	Evaporated in glovebox exhaust system.

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## APPENDIX C (cont)

CHEMICAL	AREA	ANNUAL ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Cerous Nitrate	Metallography	0.5	Used in quantities of 1 q or less in combination with several types of acids for metal etching.	Not usually found in waste but could be found in cheesecloth if spilled and in polyethylene jar used for liquid disposal.
Chlorothene	Metallography	416 £	Used as a lubricant for sample polishing of fuel pellets in metallography processes.	Evaporated in glovebox exhaust system.
Copper Granules	Fabrication	0.5	Used for compacting and compression testing.	Small amounts may have been found in glovebox floor sweepings if spilled.
Copper Shot	Fabrication	0.9	Used in compression and compacting testing.	Not normally found as waste con- taminant, but could have been spilled and consequently be pre- sent in floor sweepings.
Diethylene triamine	Metallography	1.1	Standard epoxy catalyst used for setting up fuel pellets for metallographic processes.	Usually found in floor sweepings from grinding operation and may be found in glovebox exhaust (HEPA) filters.
Epoxy Cement	Metallography	6 L	Epoxy cement used to mount samples for etching.	Found in floor sweepings and on grit paper as well as exhaust filters and in cheesecloth.

CHEMICAL	AREA	ANNUAL ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Ethanol	Metal Production	416 &	Used to dehydrate plutonium peroxide cake prior to hydrofluorination step.	Filtered into a 50-ℓ glass jar contained inside a 114-ℓ metal drum and transported to Bldg. 257 for liquid waste processing.
Ferric Nitrate	Scrap Recovery	9.1	Used in hydroxide precipi- tations in combination with aluminum nitrate to act as a carrier precipitator.	Residual amounts found in cardboard transfer containers and if spilled could be found in cheesecloth and floor sweepings. Particles may be found adhering to exhaust filters and dry box gloves.
Ferrous Ammonium Sulfate	Scrap Recovery	14	Used in combination with urea and hydroxylamine nitrate in the reduction of plutonium to the tri- valent state prior to the cation exchange process.	Resulting solution from ion exchange is transferred in a trailer tank to Bldg. 257 for liquid waste treatment. In a case of a leaky pipe or valve it could be found in cheesecloth in retrievable waste.
Iodine Crystals	Metal Production	11	Used with calcium metal as a booster for reduction of the plutonium fluoride to plutonium metal during the bomb reduction procedure.	Found in the reduction slag which is sent to recovery in stainless steel cans. May also be found on PVC plastic bags resulting from transfer. Can be found in small amounts in exhaust filters.
Lithium Fluoride	Metallography	0.5	Used in etching fuel pel- lets in metallography.	Found in exhaust filters, dry box gloves, and PVC plastics.

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# APPENDIX C (cont)

CUEMICAL		ANNUAL	UCE DECONTONI	
	AREA	550E (Kg)	USE DESCRIPTION	WASTE TIEMS CONTAMINATED
Magnesium Oxide	Metal Production	2455	Used as a crucible mater- ial and as a packing sand during bomb reduction of plutonium fluoride.	May be found on stainless steel cans and PVC bags used in transfer to recovery section.
Nitric Acid (Bulk) Technical Grade	Scrap Recovery 52	,300 l	Used in all leaching pro- cesses: ash, graphite, surface contaminated materials and in bulk fusion and dissolution of sand slag and crucible.	Found in residual amounts on all leached and pickled materials such as dry box gloves, plastic, glass and ceramics, metals and graphite. Also found in all process residues from bulk fusion and ash leaching.
Nitric Acid (Analytical Reagent)	Scrap Recovery 5	290	Used in ion exchange column regeneration, washing, and if needed, during column elution.	Found in spent ion exchange resin. (Nitric Acid is so commonly used it may be found on most items; filter-aid, line filters, dry box gloves and gaskets, and on cheese- cloth used in cleaning.)
Nitric Acid (Analytical Reagent)	Metal Production	399	Used to adjust nitrate con- centration prior to peroxide precipitation and to treat filtrate after precipitation.	Found in exhaust filters, dry box gloves, and PVC plastics.
Nitric Acid (Technical Grade)	Metallography	258	Used in etching fuel pellets in metallography.	Disposed of in a self-contained acid drain in dry box line and therefore may be a contaminant on polyethylene bottle and PVC bag. It may also be found on exhaust filters and dry box gloves.

# APPENDIX C (cont)

		ANNUAL		
CHEMICAL	AREA	ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Oil, Heat Treating	Fabrication	19 é	Use in hot bath for heat treating metal parts	Found in cheesecloth when spilled and in exhaust filters from vapor- ization and on dry box gloves and window gaskets. Also found on PVC plastic bags.
Oil, Hydraulic	Fabrication	853 l	Hydraulic fluid used to operate NC (numerically controlled) machine.	May be found on cheesecloth when leakage in system occurs. Trans- ferred to Bldg. 257 in metal cans for liquid waste disposal.
Oil, Lubricating	Metallography	2	Machining oil and a grinding and polishing lubricant.	Transferred to recovery in glass jars. May be found in cheesecloth when spilled, and on PVC plastic when bagged out of line.
Oil, Machining	<sup>238</sup> Pu Research	7	Machining oil	Transferred to recovery in glass jars.
Oil, Vacuum Pump	Fabrication	19 L	Operational fluid for diffusion pump.	Drained into tin can when changed and transferred to Bldg. 257 for liquid waste treatment.
Oil, Vacuum Pump	Fuels Research	38 L	Operational fluid for vacuum pump.	Transferred to recovery in glass jars or sent to Bldg. 257 for liquid waste disposal.

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## APPENDIX C (cont)

CHEMICAL	AREA	ANNUAL ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Oxalic Acid	Scrap Recovery	421	Used to precipitate the eluate from the anion- exchange system.	Residual amounts found in poly- ethylene transfer bags and may be found on cheesecloth if spilled. Filtrate is recycled to ion ex- change feed adjustment.
Paint, Krylon Clear	Fabrication	1.5 %	Used in changing dry box windows.	Found on rubber window gaskets and occasionally in exhaust filters.
Potassium Chloride	Metal Production	32 l	Used in combination with sodium chloride for elec- trorefining plutonium.	Packaged in tin can and bagged out of line in PVC plastic. Trace amounts may be found in exhaust fil- ters.
Potassium Hydroxide	Scrap Recovery	9318	Used to manufacture scrub solution for off-gases from the hydrofluorination system and those from the SCC dis- solvers. It is used for treating electrorefining salt residues.	Scrub solutions are transferred to Bldg. 257 for liquid waste treat- ment.
Potassium Pyrosulfate	Scrap Recovery	982	Used in bulk fusion in combination with sodium fluoride to treat insoluble plutonium-bearing solids.	Residual amounts found in cardboard transfer containers and in exhaust filters.

# APPENDIX C (cont)

CHEMICAL	AREA	ANNUAL ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
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Silicon Metal	Fabrication	0.028	Used in experimental cast- ing work.	Not found in waste stream.
Sodium Chloride	Electrorefining	41	Mixed with potassium chloride and used in the electrorefining process of plutonium.	Packaged in tin cans and bagged out of line in PVC plastic. Trace amounts may be found in exhaust filters.
Sodium Fluoride	Scrap Recovery	184	Used in bulk fusion in combination with potassium pyrosulfate to treat insol- uble plutonium-bearing solids.	Residual amounts found in transfer containers and trace amounts may be found in exhaust filters.
Sodium Hydroxide	Scrap Recovery	1888	Used to prepare scrub solution for incinerator off-gases. Also used to clean oxalate storage tank.	Solutions transferred to Bldg. 257 for liquid waste treatment.
Sodium Nitrite	Scrap Recovery	318	Used for the oxidation of plutonium from the triva- lent state to the tetra- valent state.	Small quantities may be found in cardboard transfer containers, in exhaust filters, and in cheesecloth if spilled.
Sulfuric Acid	Metallography	8	Used in metal sample etching.	Disposal in self-contained acid drain may result in contamination of PVC plastic and polyethylene plastic jar.

		ANNUAL		
CHEMICAL	AREA	ISSUE (kg)	USE DESCRIPTION	WASTE ITEMS CONTAMINATED
Sulfuric Acid	Metal Production	25	Used in adjustment of ni- trate feed prior to perox- ide precipitation.	Found in resulting solution fil- trate which is transferred to recovery. May be found as a con- taminant on some glassware, plastics, and dry box gloves.
Trichloroethylene	Scrap Recovery	10	Used to reduce viscosity of oils during filtration step in recovery process.	Residue may be found in filter- aid.
Trichloroethylene	Fabrication Metal Production	632 30	Used in ultrasonic baths for cleaning and with cheesecloth for cleaning metal parts.	Evaporated in line.
Triethylene Tetra- mine	Metallography	0.5	Epoxy catalyst used in metallography.	Usually found in floor sweepings and may be found in exhaust filters.
Urea	Scrap Recovery	9	Used in ion exchange feed solutions to remove nitrite ions.	Not usually found in solid waste.