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7. Abstract This report describes the activities of the author on behalf of the Metallic Mercury Recycling Project. Data from tests are presented. The consistently higher level of activity of the filter residue versus the bulk mercury is discussed. Recommendations for the recycling procedure are made.		
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LIST OF TERMS

DOT
EPA
PUREX
SCA
TSD
WHC

U.S. Department of Transportation
U.S. Environmental Protection Agency
Plutonium uranium extraction
Surface contamination area
Treatment, storage, and disposal
Westinghouse Hanford Company

TSD

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1.0 INTRODUCTION

Metallic mercury is known to be a hazardous material and is regulated as such. The disposal of mercury, usually by landfill, is expensive and does not remove mercury from the environment. Recycling such a hazardous material is in keeping with the mission of the Hanford Site. Recycling also demonstrates a pro-active approach to hazardous substances. This test report outlines progress towards a process by which metallic mercury may be recycled and discusses the applicability of this method to the Hanford Site. The test plan for activities reported in this report is found in Appendix A.

1.1 GOALS, NEAR TERM AND LONG RANGE

The first and primary goal of this project is to demonstrate method(s) by which mercury may be recycled onsite. A long-range goal is to make the Hanford Site a closed system with respect to mercury, thereby eliminating the net importation of mercury onto the Hanford Site.

1.2 PROBLEM STATEMENT

The treatment of mercury to remove radionuclide content does not pose the major problem for this project. Mercury has no fission or decay product radioactive isotope with a half-life of greater than 45 days (Hg-206), and so would not be a contributor to the radionuclide content at the Hanford Site. Possible radionuclide contaminants include cesium-137, strontium-90, and other radioactive metals. These are not likely to be found inside the volume of mercury as amalgams or metals, since most radionuclides in Hanford Site processing facilities are in an oxidized form, precluding dissolution of these radionuclides in the mercury. Most, if not all, of the contamination of the mercury (including amalgams) is likely to be in the form of adhering powders and scum on top of the mercury (Kirk-Othmer Encyclopedia 1981).

In recycling mercury that is thought to be contaminated, a foreseen problem is getting acceptance of the decontamination process and the purity of the mercury. The following are some of the many requirements for denoting material as "nonradioactive."

- The U.S. Department of Transportation (DOT) limits for denoting material as nonradioactive are 0.002 $\mu\text{Ci/g}$ (2 nCi/g, 2,000 pCi/g) (49 CFR 173.403y 1989).
- Westinghouse Hanford Company (WHC) limits for denoting material as nonradioactive are 60 pCi/g alpha, 190 pCi/g beta, and 2 pCi/g gamma (Rathbun 1991); 60 pCi/g alpha and 200 pCi/g total activity or less than limits for specified nuclei (WHC-CM-4-10). Limits specified in WHC-CM-4-10 have been superseded by the U.S. Department of Energy moratorium on disposal of mixed (radioactive and chemically hazardous) waste. Guidelines for radiological release are currently in draft form.

- U.S. Environmental Protection Agency (EPA) drinking water limits are 15 pCi/L alpha and 50 pCi/L beta (40 CFR 141.25 1991). Mercury will not be used as drinking water; therefore, the drinking water limit does not apply.

Current practice for releasing samples to nonradioactive laboratories for analysis calls for limiting such releases to less than 50 pCi/g (by total activity analysis). However, samples sent to offsite laboratories are to be returned to the Hanford Site, limiting the "release" of these samples. The goal of the project was to decontaminate and prove decontamination of waste mercury to a level of 50 pCi/g or less. This level was chosen because it was equivalent to the current laboratory practice of sending samples to nonradioactive, offsite laboratories.

Draft documents and conversations with regulatory compliance personnel point to a drastic reduction in release limits. This will cause some difficulties in reaching the limit of detection these levels imply, but having some guidance about the levels will speed acceptance of the decontamination and recycling process. This is unlike the present situation, where once a material is in a surface contamination area (SCA), it is considered forever potentially contaminated, and no guidance is provided for setting release levels.

Regulatory requirements that govern recycling are generally much less stringent than equivalent treatment, storage, and disposal (TSD) processes (Appendix B). Even if the more stringent requirements are levied against this process, the 222-S Laboratories have fully permitted TSD units to which this process could be appended.

2.0 TESTS PERFORMED, RESULTS

Tests were done in both nonradioactive ("cold") and radioactive ("hot") laboratories. Cold laboratory tests involved the testing of the physical separations (pinhole filter and distillation). Hot tests consisted of analyzing samples of mercury and of filtration residue for total activity. This section describes those tests, and the results. The raw data on which the tests are based can be found in laboratory notebook WHC-N-120, Volume 6 (Appendix C).

2.1 COLD TESTS

Pinhole filtering is a method of purification largely limited to mercury, because mercury has a very high density and high surface tension compared to most substances. Pinhole filtering consists of pouring mercury through a filter paper funnel which has a pinhole in the bottom tip. Tests were conducted to determine whether or not the filter paper needed to be damp for best scum and particle adhesion. It was found that a damp filter produced the best adhesion. It was also noted that too large a hole in the bottom of the filter resulted in significant unwanted material coming through. This material was detected visually because pure mercury has a mirror surface

allowing impurities to easily be seen. Pinhole filtration fails if all the mercury comes through the hole because the scum concentrates on the "heel" of mercury that is unable to go through the pinhole (as a result of insufficient head pressure). The heel can be cycled back through the process.

Distillation tests have proceeded in cleaning, setup, and manual operation of the still. The still was modified to be more suitable for use in a SCA. The modifications included making barriers to mercury liquid leaving the confines of the still in the event of a failure of the still. Tube clamping and the installation of a vent system to a hood were among the modifications performed. The still was started up with pure mercury to test its operation. The still distilled mercury at a rate of several kilograms an hour, but the automatic operation feature (i.e., mercury level and output control) did not function. The cause of this malfunction was initially believed to be a defective electronic vacuum tube. A new vacuum tube was supplied by the vendor; however, the device did not operate correctly even with a new vacuum tube installed. Consultation and troubleshooting activities are ongoing, with an instrument technician aiding in this effort.

2.2 HOT TESTS

Both mercury and filter residues were analyzed for total activity to determine the activity of representative samples and the decontamination capabilities of the filtration method.

One of the objects of the fiscal year 1993 program was the optimization of a method for analysis to prove that the product of the process met or was less than the 50 pCi/g total activity goal.

All the mercury liquid samples showed very low activities (see Table 1 for data summary). One sample (MANO) was above the 50 pCi/g goal for the limit of detection according to the initial analysis. Upon reanalysis, MANO had an activity of less than 10 pCi/g, the discrepancy being caused by the difference in counting times and masses of sample. The counting time for the first analysis was only 5 minutes, which yielded a limit of quantitation that was barely adequate to meet the 50 pCi/g goal, so the scatter at that level would be high enough to explain the appearance of more activity than the 50 pCi/g goal.

The mercury was sampled before the filtering process; therefore, the sampling pipet had to dip through the material that later became the filter residue. Despite this, the level of activity in the metallic mercury was lower than the filter residue. The mercury was not analyzed after filtering because of low activity levels before the filtering treatment.

Table 1. Results of Analysis of Mercury Samples.

Sample number	Sample description	Mercury total activity (pCi/g)	Average filter residue total activity (pCi/g)	Standard deviation of filter residues (n-1) n=2	Filter as reported total activity (pCi/g)
AB	Acid blank (1.3 mL, 12M HNO3)	<16	0.86	1.1	<9.3
4H	222-S room 4H, used for hydroxide titrator	<43	116.90	2.4	119
UNK1	Unknown source in 222-S	<49	293.09	15.9	304
UNK15C	Aqueous scum from UNK1	N/A	545.27	11.8	554
MANO	Sealed bottle, for use in a manostat	62* -0.88** <10.4***	N/A too little residue	N/A	N/A
617	Cold mercury, found in 222-S, room 1D	<44	-13.45	2.1	<16.5
618	Cold mercury, found in 222-S, room 1D	<37	-4.35	1.3	<6.8
4S	Found in 222-S, room 4S	<55	118.60	0.5	118
BECK1	100 N Zone 1 manometer	<49	-59.07	0.4	<40
BECK2	100 N Zone 1 manometer	<44	278.07	42.9	<2230
202	PUREX manometer mercury, from the sampling gallery	-2.56** <9.9***	116.42	7.3	122
619	Cold used mercury 222-S, Room 1D	<46	-5.05	4.2	<8.4
HGCHGB	Mercury cold blank	<39	N/A	N/A	N/A

*First analysis attempt - 5 minute count time.

**Second analysis - 15 minute count time, as calculated from the counts per minute raw data.

***Second analysis, as reported by 222-S Laboratory.

There was only one measurement performed for each sample; therefore, no statistical data are reported for the mercury analyses.

The filter residues had widely varying limits of detection because of variations in residue mass. As a group, the residues had higher activities, some of which are quite significant. The values, presented here as pCi/g, may include a significant mass of mercury. The activity value of the residue is probably much higher because of the mercury heel (which is free from activity). The standard deviation reported is the "small sample" standard deviation.

The samples came from a variety of sources including: analysis hoods in 222-S Laboratory (UNK1, 4S, 4F, and MANO), plutonium uranium extraction (PUREX) (202), cold bench sources in 222-S (619, HG618, and HG617), and N Area (BECK1 and BECK2). The samples amounted to about 13 kg total. The samples were taken by the 222-S Hazardous Materials Group. The samples collected are probably representative of the types of mercury to be found onsite.

3.0 RECOMMENDATIONS FOR THE PROCESS

Based on the experience gained during the tests, the following suggestions will form the basis of any successful mercury reclamation process. This process should consist of an initial pinhole filtration step followed by additional pinhole filtrations if required, and an optional distillation step. The finished product mercury should then be analyzed by liquid scintillation.

Pinhole filtration should be conducted using filter paper that is damp and fairly rough. The pinhole should be about 1 mm in diameter. Residual water on the mercury (i.e., from the filtering step or water that was previously present) should be allowed to dry at ambient temperatures (do not use heat!) or should be removed with a transfer pipet. If a substantial amount of scum remains the process should be repeated. Water on the surface of the mercury needs to be removed because it interferes with the distillation process and carries with it any water-soluble impurities. Several hundred milliliters of mercury with a light scum may be treated with a single small (7-cm diameter) filter. Minimizing the amount of paper and other waste generated is essential to the efficiency of this method.

The distillation process will be automatic with the exception of introducing and removing mercury from the still. A note of caution is warranted because mercury, despite being a dense liquid, will bounce and roll like a solid but is very difficult to recapture. Care should be taken in any transfer operation.

Preparation for the analysis should start with about 50 μ l of mercury in a tared 22-mL glass scintillation vial. The mass of the mercury must be found by weighing because the pipetter does not deliver mercury accurately. Nitric acid is added to the vial in the amount of 1.3 mL of 12 M HNO_3 . This concentration was chosen to reduce the sample volume and yet keep dissolution steadier than 15 M (concentrated nitric). The nitric is followed by 3.7 mL of water to ensure that the concentrated mercuric nitrate and impurities do not precipitate either when the solution cools from the heat of dissolution or when the scintillation cocktail (17 mL) is added.

Likely mercury sources for future processing are: 222-S Laboratory, N Reactor manometers, various decommissioning activities, and dissolver thermowells in PUREX Tunnels (Black 1991). The dissolver thermowell mercury will not become available until the PUREX Tunnels are closed and remediated. The "word-of-mouth" method of announcement for the recycling service would seem to be the most efficient method. Electronic mail to the sources of the test program mercury would also be a means to announce the availability of this service.

4.0 CONCLUSIONS

This project has demonstrated that metallic mercury is a good candidate for reclamation and recycling. We have shown that most, if not all, of the potential contamination of mercury resides in the scum floating on the surface of the mercury. Pinhole filtration was demonstrated to be an inexpensive and easy way of removing residues from mercury. The analysis method is shown to be sufficient for present release practices, and should be sufficient for future release requirements. While the distillation method was not demonstrated fully, a mercury still has been prepared for surface contamination zone use. The self-contained operation (except electrical power) of the still was demonstrated. Regulatory restrictions (see Appendix B) on the recycling process are not excessive (compared to the radiological release restrictions) and should not burden the operators of the recycling process.

5.0 PLANS FOR FY-94 - EPILOGUE

Several thousand dollars have tentatively been allocated to procedure preparation and initial process runs in fiscal year 1994. The 222-S Pollution Prevention Council will provide this money and support. Procedure preparation and technologist training, to allow operation of the method by chemical technologists, will be completed. Determination of methods for the radiological release of mercury reclaimed by this recycling process will be an ongoing activity during fiscal year 1994, as it has been in fiscal year 1993.

6.0 ACKNOWLEDGMENTS

Stan Catlow and Roger Jones gave invaluable help in optimizing the analysis method and explaining liquid scintillation results.

7.0 REFERENCES

Black, D. 1991, *1991 Report on Hanford Site Land Disposal Restrictions for Mixed Wastes*, DOE/RL-91-43, U.S. Department of Energy, Richland Field Office, Richland, Washington.

Kirk-Othmer Encyclopedia, Third Edition, 1981, Volume 15, pg 152, Wiley-Interscience, New York, New York.

Rathbun L. A., 1991, *Releasable Concentrations for Radioactive Objects*, WHC-SP-0692, Westinghouse Hanford Company, Richland, Washington.

WHC-CM-4-10, *Radiological Protection*, Westinghouse Hanford Company, Richland, Washington.

49 CFR 173.403y, 1989, "General Requirements for Shipments and Packaging," *Code of Federal Regulations*, as amended.

40 CFR 141.25, 1991, "National Primary Drinking Water Regulation," *Code of Federal Regulations*, as amended.

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APPENDIX A

**Internal Memo, "METALLIC MERCURY RECYCLING
PROJECT REPORT AND INFORMAL TEST
PLAN-FISCAL YEAR 1992"**

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**Westinghouse
Hanford Company****Internal
Memo**

From: Process Chemistry Laboratory 12110-PCL92-076
Phone: 3-2182 T6-50
Date: September 29, 1992
Subject: METALLIC MERCURY RECYCLING PROJECT REPORT AND INFORMAL TEST
PLAN-FISCAL YEAR 1992

To: W. C. Allan (12) L5-53
cc: J. R. Jewett T6-50
K. E. Parker H1-60
MAB File/LB

This report details the activities of the Metallic Mercury Recycling Project for Fiscal Year (FY) 1992. Also included as an attachment is an informal test plan developed in FY92 for FY93.

PROJECT RATIONALE

Mercury is known to be a hazardous material and is regulated as such. Mercury is an easily recyclable material. Currently, contaminated mercury at the Hanford site is amalgamated with zinc and stored pending disposal. This project seeks to decontaminate mercury and make it available for reuse onsite or further refinement offsite.

PROGRESS REPORT

The metallic mercury recycling project was started late in the fiscal year, funding was approved in June by the Technology Development Steering Board.

The procurement of a metallic mercury distillation system was initiated in the last quarter of the Fiscal Year. The system is due to arrive the end of September, 1992. Due to the late arrival of the system, no installation or testing will be completed before the end of the Fiscal Year.

A search revealed that the following organizations are managing waste mercury: Hanford Restoration Operations ("Decontamination and Decommissioning"), Processing and Analytical Laboratories, and Waste Receiving And Packaging IIA (WRAP IIA).

Those interested in the process and its product include: Recycle Programs, and Excess, Surplus Sales and Shipping. An organization interested in this project as a potential customer and user of the process is WRAP IIA.

The WRAP IIA project is interested in this endeavor, as there is a plan for mercury waste handling at WRAP. The WRAP IIA mercury waste handling plan calls for retorting mercury waste, gathering the metallic mercury, amalgamating it, and disposing mercury as the zinc-mercury amalgam. The Metallic Mercury Recycling program fits in the WRAP IIA concept well, since the output of the retorting process (metallic mercury) could be recycled.

W. L. Allen
Page 2
September 29, 1992

12110-PCL92-076

Any waste (used pinhole filters, analysis waste) from the Metallic Mercury Recycling process could be retorted in the WRAP IIA mercury retort. Therefore, no mercury-containing waste need be generated by the recycling process.

A library search was made to determine the feasibility of recovering mercury from mercury-zinc amalgam currently stored as a waste. The idea appears reasonable, but may not be feasible with the distillation apparatus that is currently on order. However, the planned WRAP IIA mercury retort should be able to handle such material. Mercury-zinc amalgam could be a future source of mercury if the simple distillation of metallic (liquid) mercury provides sufficient decontamination.

A cursory set of inquiries was made to determine if similar technology had been used at other Department of Defense and Department of Energy sites. No similar projects are currently underway at the sites polled. The sites polled include, Savannah River Site, Oak Ridge Plant, and the Missouri River Office of the United States Army Corps of Engineers. Some of the contacts had vague recollections of mercury recycling projects in the past, but could give no specifics. These inquiries were made at the suggestion of W. C. Allan, with the Savings Through Sharing program in mind.

M. A. Beck, Scientist
Process Chemistry Laboratory

tth

Attachment (2)

METALLIC MERCURY RECYCLING INFORMAL TEST PLAN

1.0 INTRODUCTION

Metallic mercury is known to be a hazardous material and is regulated as such. The disposal of mercury, usually by landfill, is expensive and does not remove mercury from the environment. Recycling such a hazardous material is in keeping with the mission of the Hanford Site. Recycling also demonstrates a pro-active approach to hazardous substances. This informal test plan outlines a process by which metallic mercury may be recycled and states methods to prove the applicability of this method to the Hanford Site.

1.1 GOALS, NEAR-TERM AND LONG RANGE

The first and primary goal of this project is to demonstrate the possibility of recycling mercury onsite. A secondary goal is to make the Hanford Site a closed system with respect to mercury, thereby eliminating the net importation of mercury onto the Hanford Site.

1.2 PROBLEM STATEMENT

The treatment of mercury to remove radionuclide content should not pose the major problem for this project. Mercury itself has no fission or decay product radioactive isotope with a half-life of greater than 45 days (mercury-206), and so would not be a contributor to the radionuclide content in Hanford mercury. Possible radionuclide contaminants include cesium-137, strontium-90, and other radioactive metals. These are not likely to be found inside the volume of mercury as amalgams or metals, since most radionuclides in Hanford processing facilities are in an oxidized form, precluding dissolution of these radionuclides in the mercury. Most, if not all, of the contamination of the mercury, including amalgams (Reference 5), is likely to be in the form of adhering powders and scum of the mercury.

The major problem foreseen in recycling potentially contaminated mercury is getting acceptance of the decontamination process and the purity of the mercury. The following are requirements for denoting material as "non-radioactive".

- Department of Transportation: 0.002 $\mu\text{Ci/g}$ (2nCi/g, 2000pCi/g) (Reference 4).
- Westinghouse Hanford Company: 60pCi/g alpha, 190pCi/g beta, and 2pCi/g gamma (Reference 1), 60pCi/g alpha and 200 pCi/g total activity or less than limits for specified nuclei (Reference 2).
- EPA: drinking water limits are 15pCi/L alpha and 50pCi/L beta (Reference 3).

1.3 CURRENT VIEW OF THE METALLIC MERCURY RECYCLING PROCESS

As currently envisioned, the recycling process will involve the following steps:

- Collection

Collection of mercury from sources, such as current users of mercury, and organizations involved in decontamination and decommissioning facilities that contain mercury. Ensuring that possible source organizations are aware of the project is key to efficient use of the project resources. Waste disposal organizations also need to be aware of the project, so that they can make their customers aware of the mercury recycling project.

- Pinhole filtering

This process, in which mercury is allowed to drip through a pinhole made in a paper filter, serves to remove oxide scum and most mercury-insoluble impurities. Due to the high density of mercury, almost every compound or element will float on the surface, enabling pinhole filtering to be effective.

- Distillation

This purification step is based on well known technology. Apparatus is available commercially. However, few, if any, attempts have been made to apply this technique to the removal of radionuclides.

- Testing of product

Each batch of mercury will need to be tested after processing to certify it free from radioactive contamination. Proposed tests include Total Activity by Liquid Scintillation and Gamma Energy Analysis. Using pre-established decontamination factors to establish product purity is not possible because of the different sources of the process feed mercury. Decontamination factors would be impossible to calculate due to the difficulty of finding materials that would contaminate mercury reliably for decontamination studies. The most straight-forward method is to test each batch after processing.

2.0 PLANNED TEST ACTIVITIES

The tests will be performed in two major phases -- cold (non-radioactive) tests and hot (radioactive materials and environment) tests. Before the testing starts, this test plan and a Job Safety Analysis must be approved and issued. Section 2 of this test plan will be pasted into notebook WHC-N-120, volume 6, prior to the start of testing.

2.1 COLD TESTS

Cold testing will consist of receiving the mercury still (Attachment 2), setting it up according to the manufacturer's instructions, adding those devices needed to run it in a Surface Contamination Area, and verifying the correct operation of the completed apparatus.

The devices needed for operation include a vacuum pump, mercury trap, and a funnel for mercury addition from inside a hood. The still itself will be positioned outside the hood.

Cold testing will also include demonstration of scum removal by pinhole filtration. Cold-element testing is impractical because of the immiscibility of mercury with solid salts aqueous solution, or amalgams formed by addition of solid metal analyte. Calculating the "true" value of the analyte spike is impossible under these conditions.

2.2 HOT TESTS

The first part of the "hot" tests is to determine the level of radioactive contamination present in mercury to be recycled. The tests can include, but are not limited to:

- dissolution in nitric acid followed by either 1) total activity determination by liquid scintillation or 2) mounting on a planchet for counting by proportional counting or,
- total gamma counting of the metallic mercury.

Some radioanalytical development work may be necessary to ensure the adequacy of the tests for radioactive contamination.

Pinhole filtering will be followed by tests to determine the efficiency of initial removal of scum. The mass gain of the filter will be measured to determine the amount of mercury and residue remaining on the filter. The mercury product and the filter will both be examined for radionuclide content.

After setting up the apparatus in a Surface Contamination Area and reverifying operation, mercury will be distilled and its purity tested by the tests described above.

3.0 PROGRAMMATIC ADDITIONS

The work described here is expected to be completed in FY93. The scope of work is greater than that originally planned for FY92.

A large part of the new scope of work is the effort to have the reclaimed mercury accepted as radiologically clean. In order to be accepted as radiologically clean, the activity levels must meet acceptance criteria yet to be established.

Part of the additional effort will go into determining the grade of the product mercury. The grade determination is necessary to help any onsite mercury users judge if the mercury meets their needs.

4.0 REFERENCES

- 1) Rathbun L. A., WHC-SP-0692, "Releasable Concentrations for Radioactive Objects," dated August 1991.
- 2) WHC-CM-4-10, "Radiological Protection" section 11.0, Rev. 0. paragraph 4.6, p. 14, dated June 28, 1991.
- 3) U. S. Environmental Protection Agency, 1991. National Primary Drinking Water Regulation, Title 40, Code of Federal Regulations, Part 141.25, U. S. Government Printing Office, Washington, D. C.
- 4) U. S. Department of Transportation, 1989. General Requirement for Shipments and Packagings, Title 49, Code of Federal Regulations, Part 173.403y, U. S. Government Printing Office, Washington, D. C.
- 5) Encyclopedia: Kirk-Othmer, 3rd edition, Volume 15, p. 152, Wiley-Interscience, New York, dated 1981.

EBERBACH MERCURY STILL

AUTOMATIC • INDUCTION HEAT • ECONOMICAL

A fast, economical producer of high purity distilled mercury, the Eberbach Mercury Still is truly automatic. Within four to six minutes after the start button is pressed, distilled mercury begins flowing into the receiving vessel at the bottom of the unit. The operation can be halted at any time by pushing the stop button. There are two pilot lights which show when: (1) power is on, and (2) solenoid valve is being automatically opened so raw mercury can flow into the still system.

Because this unit produces five to seven pounds of distilled mercury per hour with an electrical input of approximately 500 watts, the cost of distilling is but a small fraction of a cent per pound. Efficient 60-cycle induction heating generates heat only within the mercury to be distilled; at no time does the temperature of any component of the distilling system exceed 220 degrees C. The all-glass vacuum distillation chamber is fully insulated, and the condenser is cooled by forced air flowing through the baffled stack surrounding the condenser. The distillate is of high purity — mercury vapor enters the condenser through a restricted and elevated opening requiring a 180-degree change of direction from the surface of the boiling mercury. A large dissociation space in the distillation chamber minimizes contamination by splash or high energy mercury particles.

50-864
Mercury Still

Full electronic control maintains proper level of mercury in distillation chamber by automatically regulating flow from raw mercury reservoir into distilling system. Barometric pressure and line voltage are compensated for. Failures in any of the components, lack of adequate mercury supply or line interruption can cause no harm, for built-in safety devices function automatically. Provisions are also made to catch overflow or spillage of mercury. The Eberbach Mercury Still is designed for operation from sea level to an altitude of 6000 feet. The raw mercury reservoir holds 40 to 50 pounds of mercury. Before operation it is necessary to evacuate the system with a mechanical high vacuum pump; subsequent operations are automatic as described in the first paragraph until unit requires cleaning. The period of operation between cleanings depends solely upon how dirty the raw mercury is. Usually it is recommended that the raw mercury be run through a mechanical filter of the gold adhesion type before placing in the still. This preliminary cleaning is rapid and greatly increases the operating period between cleanings.

The still requires a floor space 22 inches wide by 17 1/4 inches deep and stands 82 1/2 inches high. The still operates direct from 115-volt, 50-60 cycle A.C. Complete with detailed instructions for installation, operation and maintenance. Catalog

Eberbach CORPORATION
ANN ARBOR, MICH.
ESTABLISHED 1943

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APPENDIX B

**Internal Memo, "MERCURY RECLAMATION AT
THE 222-S LABORATORY"**

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**Westinghouse
Hanford Company****Internal
Memo**

From: Facility Compliance
Phone: 6-7313 B2-19
Date: May 18, 1992
Subject: MERCURY RECLAMATION AT THE 222-S LABORATORY

81140-92-035

To: M. A. Beck T6-50
J. G. Francik T6-22

cc: S. L. Brey T6-12
L. P. Diediker T1-30
B. G. Erlandson B2-19
R. J. Landon B2-19
R. P. Marshall T6-14
M. L. Martin T6-20
E. H. Smith B2-19
B. L. Vedder B2-19
AGM File/LB

Reference: Internal Memo, A. G. Miskho to J. G. Francik, et al.,
"Reclamation of Methylene Chloride at the 222-S Laboratory,"
81140-92-007, dated February 6, 1992.

Per your request, Facility Compliance is offering guidance relating to the proposed mercury reclamation at the 222-S Laboratory. The referenced letter provided guidance regarding methylene chloride reclamation. Mercury reclamation can be conducted under the same requirements identified in the referenced letter regarding permitting requirements, treatability study requirements, and Annual Dangerous Waste reporting requirements. The requirements imposed upon the mercury reclamation are identified in the Washington Administrative Code 173-303-120(4).

The difference between the proposed methylene chloride and mercury reclamation activities is in the waste generation locations and use of reclaimed materials. The methylene chloride waste will be generated by the 222-S Laboratory and the reclaimed methylene chloride will be used again in 222-S Laboratory analytical procedures. Mercury waste will be accepted from other generators on-site into the 222-S Laboratory. The reclaimed mercury will be either sent off-site for further refining of the mercury (if radiological releases can be accomplished), or used on-site by facilities or organizations other than the 222-S Laboratory. The 222-S Laboratory will accept mercury waste from other generators since the generation rate of reclaimable mercury waste within the 222-S Laboratory will not result in any cost savings from mercury reclamation activities.

M. A. Beck, et al.
Page 2
May 18, 1992

81140-92-035

To accept mercury waste from other generators on-site, the 222-S Laboratory will need to develop a recordkeeping system to allow for accepting waste. Waste shipments of reclaimable mercury require the use of a Uniform Hazardous Waste Manifest (UHW) consistent with Hanford Site practices for on-site or off-site waste shipments. Waste shipments are considered off-site if waste is received from generators south of the Wye barricade. The 222-S Laboratory will need to receive manifested waste shipments and sign as the receiving facility. A copy of the signed UHW needs to be sent back to the generator of the mercury waste. Acceptance criteria should be established to prevent the receipt of mercury waste which is not amendable to the reclamation process.

The 222-S Laboratory will need to establish a waste storage area prior to reclamation as this mercury waste. The waste storage area needs to be operated as any other waste accumulation area. If waste at the generating site is accumulated in a satellite area, the 222-S Laboratory may accumulate waste for less than 90 days without a storage permit and start the 90-day clock upon receipt of the waste. If a generator has already started the 90-day clock, the 222-S Laboratory will either need to process the waste within that time frame (the clock cannot be reset), or store the waste in a greater than 90-day permitted storage area. The 222-S laboratory complex has a permitted storage area (connex boxes) for mercury waste, however, space within the connex boxes may preclude storage of this reclaimable waste. In addition, the connex boxes are discussed in the 222-S Laboratory Complex Dangerous Waste Permit Application. The permit application will require revision since all waste streams managed in the connex boxes requires discussion, although the mercury reclamation process itself will not be included in the permit application.

Finally, mercury reclamation activities as identified above should not proceed until a user of the reclaimed mercury is identified. If a user of the reclaimed mercury cannot be identified, the mercury will then require disposal and the reclamation operation will be declared as a hazardous waste treatment operation subject to all applicable permitting requirements.

If I can be of further assistance, please call me on 376-7313.

A. G. Miskho, Advanced Engineer
Facility Compliance

smb

APPENDIX C

**Taken from a
Controlled Laboratory Notebook
Westinghouse Hanford Company,
Richland, Washington.**

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Mercury RecyclingContacts

Bill Allan 6-9456 Technical Advisory Committee
of The Technology Development
Steering Board.

Possible Hg Sources

Glenn Van Sickle - 3-1184
Hanford Restoration operations (Duro)

Interested Programs

Glenn Van Sickle - 3-1184 MRO

Rob Silva - Waste minimization
2-0206

Excess, surplus sales and shipping

Murphy McCollum - Recycle Programs

WHC Analytical Laboratories - (PAL)

WRAP - Kent Parker 2-0452

Jim Peterson - mgr.

6/2/93 Jim Knight - 6-3289 - Performance
~ 40 lbs of mercury. Evaluation

Cynthia Smith - ?

Total Quality

? funding for FY94?

Vendor

Joe Novotny 313-665-8877

Eberbach

Buysen Rita Magnaghi - 6-5166

Survey of other sites

US Army Corps of Engineers

- Wendell Greenwald - Walla Walla
- Mearsha Dawkins - Missouri River Office
402-228-7324

- no recycling, had large Hg Superfund site.

Savannah River Labs

Chuck Coleman

803-725-1160

Gray Steid

803-725-8242

- amalgamated Hg

Joel Landen 803-725-6403

- called - will return call

Oak Ridge - K-25

K-25 Lab

Gene Clark 615-574-9580

9/25/92 Kent Parker WRAP IIA

- current plans - retort and amalgamate for RCRA burial
- interested in project, willing to be mentioned as a potential customer.

Include mention of additional scope in test plan and report. ^{FY 92}

9/25/92 Asked B. Allen for \$20,000 for FY 92 and FY 93 scope

9/29/92 Called Tony Michko

Rick Adams - ^{Fig 1} Encl Permitting } process
Jeff Luke } release

60 pCi/2000 g Mike Romas -
- 9402 ? has monitoring issue

WHC LM-4-10 Section 11 pg 14

60 pCi/g gross alpha
200 pCi/g total activity
or Appendix F limits are
not exceeded

Mike Romas -
Total activity, then survey outside]
≤ 50 pCi/g

? unknown source 17 bls/d/yr 0.2 bls/yr max 222-R emission.

Mercury Recycling contacts (cont.)

10/22/92 Marty Martin of 222-S Hazardous Waste Unit
 1) He is interested in taking over Hg recycling
 2) He is looking for non-222-S customers and would be willing to accept Hg from non-222-S customers.

11/20/92 Stan Catlow
 - Stored in organic cabinets (acid side)
 get should be either
 a) low liquid
 b) low salt
 or both
 and should be near neutral on transition
 acidic side.

4/5/93 ICPUREX Chemists
 IC ~~met~~ Mike Lindberg
 ICP - Mark Roberts
 (both on loan from Idaho (IDEL))

8/30/93 Bob Davis - ORNL
 - tel. number 372-3640 - for phone #

Bob W. Fyfe - HAS
 - Savannah River contact

9/1/93 Jay Warwick - 222-S - in 27045
 - regulatory release limit issue

12110-PCL92-076
ATTACHMENT 1
Page 2 of 4

- Collection

Collection of mercury from sources, such as current users of mercury, and organizations involved in decontamination and decommissioning facilities that contain mercury. Ensuring that possible source organizations are aware of the project is key to efficient use of the project resources. Waste disposal organizations also need to be aware of the project, so that they can make their customers aware of the mercury recycling project.

- • Pinhole filtering

This process, in which mercury is allowed to drip through a pinhole made in a paper filter, serves to remove oxide scum and most mercury-insoluble impurities. Due to the high density of mercury, almost every compound or element will float on the surface, enabling pinhole filtering to be effective.

- Distillation

This purification step is based on well known technology. Apparatus is available commercially. However, few, if any, attempts have been made to apply this technique to the removal of radionuclides.

- Testing of product

Each batch of mercury will need to be tested after processing to certify it free from radioactive contamination. Proposed tests include Total Activity by Liquid Scintillation and Gamma Energy Analysis. Using pre-established decontamination factors to establish product purity is not possible because of the different sources of the process feed mercury. Decontamination factors would be impossible to calculate due to the difficulty of finding materials that would contaminate mercury reliably for decontamination studies. The most straight-forward method is to test each batch after processing.

2.0 PLANNED TEST ACTIVITIES

The tests will be performed in two major phases — cold (non-radioactive) tests and hot (radioactive materials and environment) tests. Before the testing starts, this test plan and a Job Safety Analysis must be approved and issued. Section 2 of this test plan will be pasted into notebook WHC-N-120, volume 6, prior to the start of testing.

2.1 COLD TESTS

Cold testing will consist of receiving the mercury still (Attachment 2), setting it up according to the manufacturer's instructions, adding those devices needed to run it in a Surface Contamination Area, and verifying the correct operation of the completed apparatus.

Approved: *[Signature]*
11/23/92

12110-PCL92-076
ATTACHMENT 1
Page 3 of 4

The devices needed for operation include a vacuum pump, mercury trap, and a funnel for mercury addition from inside a hood. The still itself will be positioned outside the hood.

Cold testing will also include demonstration of scum removal by pinhole filtration. Cold-element testing is impractical because of the immiscibility of mercury with solid salts aqueous solution, or amalgams formed by addition of solid metal analyte. Calculating the "true" value of the analyte spike is impossible under these conditions.

2.2 HOT TESTS

The first part of the "hot" tests is to determine the level of radioactive contamination present in mercury to be recycled. The tests can include, but are not limited to:

- dissolution in nitric acid followed by either 1) total activity determination by liquid scintillation or 2) mounting on a planchet for counting by proportional counting or,
- total gamma counting of the metallic mercury.

Some radioanalytical development work may be necessary to ensure the adequacy of the tests for radioactive contamination.

Pinhole filtering will be followed by tests to determine the efficiency of initial removal of scum. The mass gain of the filter will be measured to determine the amount of mercury and residue remaining on the filter. The mercury product and the filter will both be examined for radionuclide content.

After setting up the apparatus in a Surface Contamination Area and reverifying operation, mercury will be distilled and its purity tested by the tests described above.

3.0 PROGRAMMATIC ADDITIONS

The work described here is expected to be completed in FY93. The scope of work is greater than that originally planned for FY92.

A large part of the new scope of work is the effort to have the reclaimed mercury accepted as radiologically clean. In order to be accepted as radiologically clean, the activity levels must meet acceptance criteria yet to be established.

Part of the additional effort will go into determining the grade of the product mercury. The grade determination is necessary to help any onsite mercury users judge if the mercury meets their needs.

Approved
[Signature]
11/23/92

11/23/92

On 11/20/92 replaced existing still "hose clamps" (linen rope) with plastic clamps. Added clamps in appropriate location. Linen "clamps" might work loose and may not be sufficient to hold the mass of mercury.

Cleaned off still, prepared hood. (on 11/22)

11/24/92 prepared catch bottle for glycerine procedure. (taped, got stopper and connectors).

12/4/92

12/4/92

On 12/3/92 cleaned p. still as per Flapjack instructions. All inside surfaces clean except bottom of furnace flask and a small bit of glycerine in between the intake and condenser tube. Used Duo-Seal pump for all ~~vacuum~~ vacuum. Learned from flt. intake house ~~vacuum~~ vacuum in 5A is preferable for mechanical wash. Used ~3L (vs 4L) of water for wash, and only 500m aliquoted water for rinse (vs 4L suggested by Eberbach).

Planning to melt, wash glycerine with hot water again today.

Got glass liquid scintillation vials, caps, for analysis study.

methods search

- mount prep for L.S. 4L 10cm above background
LA-548-11

operation ofushman LA 508-121

12/10/92

Pipette calibration studies

Pipette - Eppendorf 3 way adjustable 50-75-100, set at "100ul" using yellow tip. Pipette ID# 7782612386.

Vial used 20ml glass scintillation vial.

Tare: 16.80765
16.80765
16.80763

Kalamo WA45026 zeroed at 0g

Mark A. Bell
10/12/12/10/92

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12/10/42 Tare average 16.80764 g $\pm$ 0.00001 (100%)

1st addition "100ul" 17.952569g

1st addition mass 1.14505g

2nd addition "100ul" 19.10795g

2nd addition mass $\frac{1.15528g}{1.14426g}$

3rd addition "100ul" ~~20.2521~~
20.25221g

3rd addition mass 1.14426g
ave. 1.14819g $\pm$ 0.006g (100%)

same pipetter - with different (yellow type) tip to check for difference in tip mass due to a different tip.

Tare 16.80811g mass H₂ added

1st addition 17.95500g 1.14689g

2nd addition 19.10162g 1.14662g

3rd addition 20.25208g 1.15046g

Zero checked after use - 0.00007g
ave 1.14799 $\pm$ 0.002 (100%)

~1.4g H₂ $\frac{1 \text{ mole H}_2}{200.61g H_2}$ $\frac{1 \text{ mole H}_2 (H_2)_2}{1 \text{ mole H}_2}$ $\frac{2 \text{ mole HNO}_3}{1 \text{ mole H}_2 (H_2)_2}$ $\frac{L}{16 \text{ mole H}_2}$ = 9×10^{-4} minimum L conc HNO₃

if 4 mole HNO₃ = 1 mole H₂

minimum is 1.4ml H₂O₂ conc.

with 6 runs - 2 different tips 1.14809 $\pm$ 0.004g H₂ (100-1)

= average 1.14804 $\pm$ 0.015g at 99%

mark H. Bell 12/10/42 confidence limit

12/14/92 Pit Pinhole filter tests

- need to determine if the filter is best when wetted or dry.

filter - 47 mm TYPE H/E glass fiber
lot # 402608 (Gelman Science)

Pinhole made with a syringe - needle "N722" from Hamilton
0.028" diameter - max 12/14/92

Dirt from 2255A Bonestop. This dirt is a fine yellow loose type of material that can be expected to gather on equipment surfaces.

Dry Test

Hg appearance before

Hg had grains of the yellowish dust adhering to it, Hg even when Hg moved around

Hg appearance after test

Hg had grains of yellowish dust adhering to the surface. The dust seemed to be more uniformly distributed than prior to "filling".

Pinhole may be too small, as it took ~ 9 minutes, with tapping, to get almost all of the mercury through the pinhole.

not much dust adhered to the filter after the filter.

Wet Test

same as dry test

Hg, with some water in collection vial, was shiny. Only a small "scum" of material was attached to mercury. Scum was translucent. Overall appearance of Hg much better in this test than in the dry test. Hg flowed much better, and more adhered to filter as it did during the dry test. The filter picked up most, if not all the dust, judging by its appearance after the test. Note, not all the mercury would flow by gravity.

lightly capped wet test product, to see if the water inadvertently improved the appearance. 12/14/92 Mark Q. Beck

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12/14/92

conclusions from filtering tests
(pending appearance of wet test when dry)

- ① wetted filtering is preferred method.
- ② no attempt should be made to get last inch or so of Hg through pinhole.
- ③ larger filter paper, funnel should be used to increase Hg head (and therefore flow).
- ④ The pinhole size should be adequate for large head (pressure) values.
- ⑤ Textured side of filter should be used to help capture larger particles of dirt.

12/17/92

looked at Hg filter product after the water has evaporated off. Some ~~small~~ clumps of ~~scum~~ are on the surface of the Hg. most of the surface is bright and shiny per ACS specification (reagent chemicals).

Further filter filtering tests may be needed.
Filter may need to be washed with DI water prior to use, larger filters may need to be used to provide a greater surface area for particle adsorption.
mark A. Beck 12/17/92

2/17/93 Hg Dissolution test

Purpose: Determine if Hg dissolves cleanly (without spitting or sputtering, & what is the minimum of acid needed.

	Hg (g)	HNO ₃ conc. (M)	HNO ₃ volume	
"50ml"	.58g	15.9 7.95M	0.5ml	some minor fizzing
		added 0.5ml		
mark A. Beck			0.5ml of 15.9 M HNO ₃ - soln immediately	
12/17/93			got a yellow tinge to it. no other changes	

For ~100ul (1.14g Hg) $\text{Hg} + \text{HNO}_3 \rightarrow \text{Hg(NO}_3)_2 + \text{NO}_2 + \text{H}_2\text{O}$

$$1.14\text{g Hg} \frac{1\text{mole Hg}}{200\text{g Hg}} \frac{2\text{mole HNO}_3}{1\text{mole Hg}} \frac{L}{15\text{mole HNO}_3} = .00076\text{L} = 0.76\text{ml HNO}_3 (15\text{M})$$

~~used water bath to ensure a~~ ^{meq}
had water bath nearby to ensure a heat sink (per T5A).

no heat evolution noted (or at least none through a surgical ^{mask} surgeon's glove).

Experiment terminated 0550 after 720 minutes of acid-mercury contact.

Thoughts for next try, ① at least 12M HNO₃ with a slight excess of acid
2/17/93 mark G. Reed.

2/22/93

acid: 0.5ml water 1ml 15.9M HCl

$$15.9 \frac{\text{mole}}{\text{L}} \times 0.001\text{L} \frac{1}{0.0015\text{L final vol}} = 10.6\text{M}$$

$$0.50\text{ul Hg} \frac{1.14\text{g Hg}}{100\text{ul}} \frac{1\text{mole Hg}}{200\text{g Hg}} = 0.0029\text{mole Hg}$$

$$0.0029\text{mole Hg} \frac{2\text{mole HNO}_3}{1\text{mole Hg}} \frac{L}{10.6\text{mole HNO}_3} = 0.0005\text{L} = 0.5\text{ml } 10.6\text{M}$$

needed

50ul Hg, 0.5ml 10.6M HNO₃ added together at 1425.

- immediate rxn. brown NO_x fumes seen
small amount of heat generation @ 1440, noticing that rxn had slowed to negligible, added 0.75ml 15.9M HNO₃ - immediate formation of x-stals, yellowish tinge. additional water clarified and dissolved x-stals.
50ul Hg, 0.75ml 10.6M HNO₃ added together at 1425
in solution. rxn as above. some whitish accum cond. floc appearing, rxn slower than above (0.5ml HNO₃) at equivalent stages of Hg dissolution.

mark G. Reed 2/22/93

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2/22/93

0.5ul Hg 0.5ul ^{10.6M} HNO₃ (original)
 + 5.5ml 1453 noticed complete dissolution
 of Hg in this vial

50ul Hg 0.75ul ^{10.6M} HNO₃ - (originally)
 as 1453 no rxn noted. ~30ul Hg remain.

Excess HNO₃ (either ^{not} seems to cause a passivating film
 or xstate to appear. This may be due to the laboratory
 temp.

suggestion for next series of experiment

- 1) Use exact molar ratios to start.
- 2) add small amount of acid ^{HSCl} to finish
 metallic Hg dissolution.
- 3) add water to dissolve any Hg(NO₃)₂? xstate

2/26/93

50ul Hg with:
 (0.0029 mole Hg)

lab (hood) air temp: 19°C
 & water temp 21°C

conci:	15.9 M	12 M	10.6 M
volume	1 ml 15.9 M HNO ₃	1 ml 12.9 M HNO ₃ 0.3 ml Q water	1 ml 15.9 M HNO ₃ 0.5 ml Q water
common/s			
start at 9:55	* a film developed immediately rxn yellowish < 30 sec Dissolution began after a lag period Dissolution complete 1006 much NO ₂ fumes soln dark & brown red after dissolution.	reddish soln with gas, heat gas generation within 30 sec. Hg spinning around in beaker Dissolution complete 1002 NO ₂ only during active dissolution soln lighter than 15.9 M test beaker after dissolution	dark yellowish with lesser gas, heat generation than 12 M test. H ₂ also seen rolling around. bottom edge of beaker Dissolution complete 1002 soln much lighter than of others tests after dissolution.

Mark A. Beck 2/26/93

2/26/93

added sufficient water to bring each of previous tests or solns. up to 5 ml volume all solns now water white.

$$\frac{50 \mu\text{L H}_2 \cdot (1.4 \mu\text{L} / 1 \text{ mole H}_2)}{100 \mu\text{L} \cdot 200 \mu\text{L}} \cdot 0.005 \text{ L} = \frac{1}{2} \cdot 0.57 \text{ M H}_2$$

↑
see calibration
- approximate relationship

since $\therefore 0.57 \text{ M H}_2(\text{NO}_3)_2$

~~1 ml = 0.001 L $\times$ 15.9 mole / L = 0.005 L = 3.18 M HNO₃~~

~~3.18 mole HNO₃ / L~~

~~200 μ g 77 mm equation~~

~~2/26/93~~

Assuming

then remaining (excess acid)

$$\frac{15.9 \text{ mole HNO}_3}{\text{L}} \times 0.001 \text{ L} - 0.57 \text{ M H}_2(\text{NO}_3)_2 \cdot \frac{4 \text{ mole HNO}_3}{1 \text{ mole H}_2(\text{NO}_3)_2} = \frac{1 \text{ mole HNO}_3 \times 0.005}{1 \text{ mole H}_2}$$

$$0.0159 \text{ mole HNO}_3 - 0.0057 = 0.0102 \text{ mole HNO}_3$$

$$\Rightarrow \frac{0.0102 \text{ mole HNO}_3}{0.005 \text{ L}} = 2.04 \text{ M HNO}_3 \text{ remaining}$$

total ionic strength $\frac{1.47 \text{ M}}{2 \times 0.9 + 3 \times 0.57} = 3.42 \text{ M}$

Conclusions - Quasit H₂/HNO₃ ratio of $\frac{50 \mu\text{L H}_2}{1 \text{ mL } 15.9 \text{ M HNO}_3}$

- ② 12 M HNO₃ seems to work best (fastest, most controllably)
- ③ solns should be made up from ~20 °C or warmer HNO₃, water.

Mark A. Besh 2/26/93

40

2/26/43 Hg - Keegell

Liquid Scintillation Cocktail Method
Development

2ml Qwater in 2/0ml Instagel - XL
 shook, clear ~~soln~~ liquid resulted after
 air bubbles floated to surface

ml 0.57M $Hg(NO_3)_2$	ml Instagel	Results
2	6	Immediate turbidity. When shaken, near solid gel formation, cloudy (misty) with additional micro size air bubbles. - not suitable
1	6	immediate turbidity viscous (not nearly as thick as 2ml $Hg(NO_3)_2$ test) when shaken, significant cloudiness, heavier than - $Hg(NO_3)_2$ test.
0.5	6	immediate turbidity when $Hg(NO_3)_2$ soln added on shaking, turbidity disappeared.
0.7	6	immediate turbidity, cleared upon shaking
0.9	6	same as above.
* 1.0	6	added 0.1ml (100ul) to "0.9 ml" test vial. soln cleared after shaking.
0.9	6	because of discrepancy in results between * test and 1st 1.0 ml $Hg(NO_3)_2$ soln test, tested 0.9 and 0.7ml taken with new vials.
0.7	6	I had been using same vial for 0.9... to * 1.0 ml tests.
mark A. Keegell 2/25/43		0.9, 0.7ml (new vial) both turned clear.

Hg - Recycle

3/4/83

7/9/5 Liquid Scintillation setup seems to be satisfactory at this point. No further work on this should be necessary.

Now determining how to replace latex
~~room~~ rubber fittings with TYSON, to ensure
long tubing life.

3/18/43

3/18/63 Replaced all lagtex ^{to} tubing with tygon® tubing, added catch flask for overflow and waste mercury. Safety clipped slipped stopcock and all broken tubing.

3/22/93

3/22/93
Sending in RSA to get R#s assigned
customer sample #s Lab R#s (to be determined
and added later)

E#s assigned

~~10/20/2~~

~~10/20/2~~
Hg 1 Blank

E 8223

Hg 2 Blank

E 8221

Hg. 3 cold

E 8224

Hg 4 cold

E 8222 mark Rich
3/26/93

3/24/93

3/24/93
method for liquid scintillation
LA ~~548-111~~ 508-121
mount 548-111

put aluminum sheeting over direct paths for ~~mercury~~
mercury escape. Try trying to make the still a containment
in case of Hg spill or equipment (glass) breakage
M. J. Hill rev 3/26/93

3/25/93

Hg Recycle

need lucite catch canisters for front ports

 $\frac{1}{8}$ " thick x 19" wide x 4" high all holes $\frac{3}{16}$ "

3/29/93

make up of vials Used Eppendorf 4710 346206, set at 25.0 ul
had some difficulty ejecting an equivalent amount of Hg from tip.

Hg 1 March E8223

+ 17.08658

tare 16.77989

0.30591 g Hg

Hg 2 Blank E8221

17.05743

tare 16.72686 g

0.33057

Hg 3 Cold E8224

17.12402

tare 16.77952

0.34450

Hg 4 Cold E8222

17.14008

tare 16.81913 g

0.32095 g

Hg placed directly into scintillation vial

ejected residue from each attempt prior to sampling a new aliquot
may not be able to use volume as an accurate
measure of mass.added to each scintillation vial, 0.6 ml 12M HNO₃ = 0.0072 mole HNO₃.
$$0.3445 \text{ g Hg} \times \frac{1 \text{ mole Hg}}{200.59 \text{ g}} \times \frac{4 \text{ mole HNO}_3}{1 \text{ mole Hg}} \times \frac{1 \text{ mole HNO}_3}{1 \text{ mole Hg}} = 0.00681 \text{ mole needed} + 4\% \text{ excess added.}$$

by Hg 39 run eq.

considerable NO_x brown fume evolution.March 31, 1993
3/29/93

2/29/93

All gas evolution stopped, with total H_2 dissolution in ~15 minutes.

added 17.5 ml Instagel[®] to each vial initially cloudy, filimentous ~~or~~ in suspension because water white, and clear, (water with a few bubbles) upon shaking.

sample E8221, E8222, E8223, E8224
submitted 1205 2/29/93 to 5 Lab.

σ_m for mass 0.016239 $n=4$ $t_{95\%} = 3.182$ @ 95%
or average is 0.3254825 $\pm$ 0.0516 @ 95%
 $= 0.32 \pm 0.05g$ = 15% error @ 95%
— mass of H_2 must be weighed, not calculated from
from pipette volume

All E822X samples are identical, and so
do not necessarily reflect User (volume 20).
Mod. error in preparation

2/30/93

Raw data with notes by M Beck attached
as or following, pages.

Disc Discussion with R. J. Jones:

key points. General L.S.

Efficiency determined by quench (calibrated) ⁽¹⁰⁰⁾
Quench determined by external standard.
(S. source) H_{eff} - Quench - source
 $\frac{\text{net} - \text{bg} - \text{ground}}{\text{efficiency}} = 0 \text{ p.m.}$
empty stage
+ C_{S} 157
5.4 $\times 10^5$ imp/cm²

channel 1 is 5-1000 (α and β)
channel 2 is 600-1000 (α and β)

RCM% is random coincidence, caused by fluorescence
+ stray light.

Mark A. Beck,
2/31/93

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PAGE: 2

SAM	POS	CH	CFM	CSIG%	TIME	EL TIME	AVG HW	RCM%	ERR
1	3	1	101.93	3.62	30.00	63.07	242.0	20.24	
		2	38.80	5.86					
		ISO1 XEFF CH1:93.12						ISO1 DPM :29.31688	
2	4	1	81.93	4.03	30.00	94.69	242.0	11.06	
		2	39.47	5.91					
		ISO1 XEFF CH1:93.12						ISO1 DPM :7.839313	

EE8222
EE8222

*permanently
improvement of CS
shift*

cut line

*11" vs off day
shift*

*cut DR
F*

mark (1) B. d. 3/31/43

BEST AVAILABLE COPY

62549 3-19-93
H. Adelman

PAGE: 1

USER: 1 IS: TOTAL ACTIVITY PROBET TIME: 3.00
SAMPLE REPEAT: 1 CYCLE REPEAT: 1 GORIN POSITION
H#: 1 ADD: 1 GORIN ROM: 1 PHASE MONITOR: N
ROM-TIME: 0.10 INIT: 0.00
CHANNEL 1-UL: 1 UL: 1000 SIGMA: 0.10 SKE: 0.00 SKE: 0.00 SKE: 0.00 SKE: 0.00
CHANNEL 2-UL: 1000 UL: 1000 SIGMA: 0.10 SKE: 0.00 SKE: 0.00 SKE: 0.00 SKE: 0.00

SINGLE LABEL ORN SET UP ON 20 DEC 1991 13:12

UNKNOWN ID: TOTAL ACTIVITY UNKNOWN REPLICATES: 1
UNKNOWN NORM FACTOR 1301:2 1.00000
UNKNOWN UNITS 1301:OPM
UNKNOWN HALF LIFE CORRECTION: N
INDIVIDUAL UNKNOWN NORM FACTORS: N BACKGROUND QUENCH CURVES:
STANDARD ID: 55340 A-J QUENCH LIMITS LOW: 24.00 HIGH: 484.0
HALF LIFE (DAYS): 1301: N
STANDARD OPM 1301: 0.000000000

SAM	POS	CH	OPM	SIGMA	TIME	EL	TIME	AVG	#	ROM	ERR
91	44-	1	75.60	14.20	5.00	1.44	93.0			1.07	
		2	40.00	14.14							

BACKGROUND QUENCH CURVES: CONSTANT

CHANNEL 1
QUENCH CURVE COEFFICIENTS
A: 13.00000 210.00000000 0.000000000 0.000000000

BACKGROUND QUENCH CURVE CORRELATION TABLE

OPM	TIME	MEASURED OPM	CALCULATED OPM	PERCENT DIFF	FLAG
1	5.00	75.60	75.60	0.00	

CHANNEL 2
QUENCH CURVE COEFFICIENTS
A: 40.00000 210.00000000 0.000000000 0.000000000

BACKGROUND QUENCH CURVE CORRELATION TABLE

OPM	TIME	MEASURED OPM	CALCULATED OPM	PERCENT DIFF	FLAG
1	5.00	40.00	40.00	0.00	

BACKGROUND QUENCH LIMITS LOW: 24.00 HIGH: 1000.0
TOTAL QUENCH LIMITS LOW: 24.00 HIGH: 484.0

3/1/42

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BEST AVAILABLE COPY

PAGE: 1

SAM	POS	CH	SRM	ORIGIN	TIME	EL TIME	AVG SRM	SRM	SRM
1	**	7	1	51.00	11.45	5.00	11.01	237.0	5.12
			2	37.00	14.39				
			1301	NEFF	CH1: 90.59			1301 SRM	10.000000
2	**	4	1	74.40	10.77	5.00	15.57	233.0	10.41
			2	39.00	14.32				
			1301	NEFF	CH1: 71.57			1301 SRM	10.000000

ES23

ES24

Handwritten: 2. n. h. a. back 7/7/93

Hg Kezgle

3/21/93 Specific comments about ES 221-ES 224

The count was quite high for the 30 min counted sample ES 221, ES 222. These samples were counted ~~immediately~~ soon after receipt, estimated time of test of analysis was being 40 min for the last sample. The RCM's indicate that these samples did not have time to dark adapt sufficiently. Samples ES 223 and ES 224 had about 7 hours of dark adaptation time before (1344, probably to just after 20:23)

The similarity of the channel 2 results may imply a support the supposition that the events seen in the wide window (WH1) were low energy (possibly chemical) events.

The quench factor starts out fairly high, but does not change significantly over 7 hours or so.

The RCM as a measure of cooling, or dark adaptation time is supported by the very high RCM of the first (ES 221) sample and the much lower RCM of the second sample run. However, 1 point does not prove much.

Suggestions:

- ① 5 min count time should be adequate.
- ② These samples seem highly susceptible to a lack of dark adaptation. no rush analysis should be done.
- ③ The quench is quite high for these samples despite being water white and clear upon preparation. no samples past 24 hrs should be considered suspect.

$$29.31 \frac{\text{min}}{\text{count}} \times \frac{0.8221}{0.3705} \times \frac{1.0 \text{ Ci}}{2.26 \text{ gm}} = 40.2 \text{ } \mu\text{Ci/gm} - \text{below release limit}$$

can with a high RCM (fluorescence)

50

4/5/93 Hg Recycle

Point #3 on previous page is supported by the observation my observation that the cocktail turns yellow and slightly cloudy after ~ about 24 hours. The high HH (quench value) would imply that much additional quench might place the quench value outside of the efficiency vs. quench calibration, and thereby invalidate the data.

Try contacted Hg with Aluminized sheeting. no dissolution, or erosion of the sheeting was seen, even when the sheeting was scratched.

5/24/93

Samples available in the SCA

I.D.	amount	Comments (Source, etc.)	Location
4H	~50ml	off titrator	1 GA
UNK-1	25ml	some plastic waste, visually dirty	1 GA
Manu	~10ml	very clean looking, "for manometer"	1 GA
617	~25ml	"clean" - used	1 D
618	~40ml	" "	1 D
619	~50ml	" "	1 D
45	30ml	HMU stretcher underneath - unknown source	2 B
BECK-Hg-1	~100ml	R 2522 from Brad Schipperout	
BECK-Hg-2	~80ml	R 2522 100N noted a thin layer of carbonaceous liquid on surface may 6/2/93	

$$\sim 468 \text{ mL} \frac{2.72}{1.2} = 5.0 \text{ kg}$$

5/27/93

according to R Jones and S Caltor

$$\text{detection limit (cpm sample)} = \frac{2.72}{T_{\text{ac count}}} + 4.66 \sqrt{\frac{\text{background}}{T_{\text{ac count}}}}$$

4/2/a) Hg recycle
Hot tests

Preparing sample	sample R # Lab #	mass
HGAB-1	R 3931	(acid blank)
HG4H-1	R 3932	
HGUNK-1	R 3933	
HGMAVO-1	R 3934	
HG617-1	R R 3935	
HG618-1	R 3936	
HG619-1	R 3937	
HG45-1	R 3938	
HGBECK1-1	R 3939	
HGBECK2-1	R 3940	
HGCHGB-1	R 3941	

will ask for 10 minute count as a 10 min count

	40	80	150 cpm background
for	9.5 cpm	13.4 cpm	26.9 cpm net
will yield	4.8 pCi/g	6.8 pCi/g	9.7 pCi/g
and			

assuming 90% efficiency and $22.4 \mu\text{m} = 1 \mu\text{Ci}$
and using the formula on pg 50 for limit of detection

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14-0000-101 (9-89)

DON'T SAY IT ... Write It!

None on 6/2/93
DATETO Mark Beck

FROM

Used ~ 600ml 12M HNO₃
~ 14ml InstagelHG BECK 1-1and HG 45-1 both showed difficulty
in dissolution, after ~ 15 min of gentle shaking
both dissolved. All other samples dissolved
in < 5 min.packed in 6/3/93

+

TO MAKE LIFE LAST, PUT SAFETY FIRST

+

8/10/93

R 3931. BECK	HGAB-1
4271 TOT-ACT	< 1.65000E 01 pCi/G
R 3932. BECK	HG4H-1
4271 TOT-ACT	< 4.33000E 01 pCi/G
R 3933. BECK	HGUNK1-1
4271 TOT-ACT	< 4.93000E 01 pCi/G
R 3934. BECK	HGMAO-1
4271 TOT-ACT	6.21000E 01 pCi/G
R 3935. BECK	HG617-1
4271 TOT-ACT	< 4.44000E 01 pCi/G
R 3936. BECK	HG618-1
4271 TOT-ACT	< 3.71000E 01 pCi/G
R 3937. BECK	HG619-1
4271 TOT-ACT	< 4.64000E 01 pCi/G
R 3938. BECK	HG4S-1
4271 TOT-ACT	< 5.56000E 01 pCi/G
R 3939. BECK	HGBECK11
4271 TOT-ACT	< 4.94000E 01 pCi/G
R 3940. BECK	HGBECK21
4271 TOT-ACT	< 4.47000E 01 pCi/G
R 3941. BECK	HGCHGB-1
4271 TOT-ACT	< 3.95000E 01 pCi/G

posted in
8/10/93
Mark Beck

8/10/93

Note the result for Hg monosol, 62 $\mu\text{Ci/g}$ which is above the release limit. This result is most unexpected, as this mercury was in a bottle in a box, ~~thru~~ brought by ~~way~~ wrapped, and to all appearances, had never been used. Since this datum may be an artifact of the short counting time used (5 minutes), this poor sample will be reanalyzed.

Cl (mas) discovered that instead of the 8 lbs of mercury I had thought were required for a cold (non radioactive) test, it will require 13 lbs. Also for date PCL has in 222-54 4.190 kg of metallic mercury.

I called around at Jim Jewett's suggestion I called Susan Jones of 2-plant and Sam Wurbut of 2-plant. Mr. Jones offered 4 kg cold metallic mercury. Mr. Wurbut offered ~500 ml of metallic mercury.

On 8/10, there now should be sufficient mercury to do the cold tests.

Need to get Sam's Hg released, and both bottles shipped to 222-54.

8/18/93

I now have enough mercury to do cold tests (distillation setup).

for see note { added 2.2 kg Hg - noticed that there is an air bubble that the mercury bubbles through in order to drain the reservoir. This air bubble is in the upper reservoir drain tube, near the lower part of the valve.
2.8 kg added
* The pump is connected, but the tubing is open to the atmosphere through a valve.
Mark A. Beck 8/18/93

54

8/18/93

The rise in the level of the mercury in the cistern is fairly slow.

The level in the cistern is much higher than it should be ($\approx 1/2"$ from overflow) and higher than the level in the supply tube i.e. levels "T" and "O" are not equal.

Sealed the cistern connection to the atmosphere by taking a pipet bulb and varying the levels in the cistern and also the still head.

The air bubble trapped in the supply tube must be causing the head difference.

Also the "barometer" between cistern level and the top of the mercury measures $23 1/2"$ - a bit low.

The tubing I used to go from the trap bottle and the outlet (product side arm tube) has collapsed, this may be why the pump cannot achieve a good vacuum ~~and~~ above the mercury. This tubing must be replaced.

~~How~~

Recovery plan: Follow cleanout instructions for mercury removal.

Suggestion: Instead of letting the mercury level hit the barometer bottom with all the bumping, bumping and surging. Try leaking air into the vacuum side.

(Difficult to do with Hg in the product side arm test tube)

The As the mercury is added via the receiver, the level in the 1st (outer) barometer is about $1 1/2"$ higher than the level in the cistern (Why?)

applied vacuum, barometer at $26 3/4"$, cistern to top of Hg.

Hg at $7/8"$ level is distilling flask.

Mark. Beck 9/18/93

8/18/97

Level "B" $7\frac{1}{8}$ " from bottom of flask was reached - power ON - 1323

Fan is operating, power light on.
Mercury distilled over quite quickly
at 1335 ~ 1335 (very approximately) was first
Hg in side arm tube.

Shut still off at 1345.

- Victor disconnected pump, power and.
- Did not see if the valve was operational.
- The valve operation is the next cold test.

The rigorous control of mercury addition is critical for clean operation.

Mark A. Beck
8/18/97

8/19/97

0955 - power on

- mercury level in flask is
about $\frac{1}{2}$ of $7\frac{1}{8}$ " = $\frac{3}{16}$ " above the bottom
(measured from $7\frac{1}{8}$ " mark made on the
inside of the flask). A flashlight is needed
to see inner part of flask.

0957 - some Hg seen condensing on side of flask.
There is no apparent apparent way to
determine if Hg is being distilled, except for
a rise in the ~~mercury~~ level in the side arm tube,
or product flask level increase.

1014 1st product in flask.

1042 - shut down - no valve actuation, and a loud
hum (which may be an undocumented alarm) is heard.

The air coming from the fan (condensation portion) just now
cooler than previous and the beach had stopped fogging
in the system

Mark A. Beck 8/19/97

56

8/19/43 added more mercury Hg to system by manually opening the reserve valve. Still then started up without the load pump.

Hg level very low (2 5/8 from block that the vacuum system sits on.

No problems with turning on still.

Either a) the automatic shutdown mechanism is not working.

b) The manual mercury level is inadequate and valve (reserve) action is not needed

shut off tube, still ~1830.

but before doing so, noted that the tube envelope was cold (see pg 7 59 d. of instruction.

Removed the tube - noted that the tip said "2050 A"

called Joe Novotny - a) will check if tube correct
b) will send new tube

8/23/43

8/24/43 Lat test (Filter) notes
 pasted in on 8/24/43.

94-2000-101 (9-50)

DON'T SAY IT --- Write It!

DATE _____

TO _____ FROM _____

8/23/43

MBECK-1 was very shiny prior to filtering
 minimal residuals on filter. pinhole seemed much smaller
 than HG202 filter pinhole (as was gauged by fluoride).

MBECK-2 had very little caught in the filtrate.

"45" had dark grey solid, heavy deposits

44 had some residue on the filter but not as much as the
 "45" sample had.

94-2000-101 (9-50)

DON'T SAY IT --- Write It!

DATE _____

TO _____ FROM _____

8/23/43 clearing hood 1 16C

HG-202 - from Jim Hilliard: Purax - had some clear
 liquid on top - ? organic organic
 liquid adhered onto Kim Wipes easily, general feel was
 similar to that of water, not greasy - , no activity by p/o and
 considerable solid (dark, grey grey and black) were left
 on the filter

+

TO MAKE LIFE LAST, PUT SAFETY FIRST

+

50

94-0000-101 (0-00)

DON'T SAY IT ... Write It!

DATE _____

TO _____ FROM _____

8/27/93 "618" - very heavily loaded with residue
 "617" - much residue, not nearly as heavy as 600, 619

UNK-1

UNK 50M-1 - drum sample from UNK-1, 100 ml

The goal of this test series is to determine
 how the amount of activity in the fission
 residue from pinhole filtration.

The filtration was done with wet filter paper
 (Whatman #40, 9.0 cm). I cut the paper
 with a razor blade and used the smallest
 orifice, of 2 that appear with folded
 filter paper.

needed ~ 8-10 ml 10M HNO₃ and ~ 20 ml
 Fastagel
 note lower HNO₃ conc.

Mark H. Bell
 8/26/93

8/24/97

Decided not to submit

HG-MANUF-1 - as the amount of sample would be too small

10M HNO₃ = 2 parts 15M in 1 part water

6ml water 12ml 15M HNO₃

- J1044 - added 500ul 10M HNO₃

needed ~8ul 10M HNO₃
~20ul in 4200

added 500ul 10M HNO₃ } To all samples
added ~300ul 10M HNO₃ } except HG-AB-2
added 100ul 10M HNO₃ } (and blank)

HG-45F-1 had a large ball of mercury that wouldn't dissolve and some some flakes (flocculent type material)

UNK17F-1 had light brown of brown floc and ppt. & no mercury seen

UNK15CM-1 had a light brown ppt, grey in color

HG-4MF had a light colored light floc.

HG-617R-1 had a very small bit of mercury undissolved.

HG-619F-1 had a 25ul Hg in it

~~HG-202F~~ HG-202-1 had a few flakes

HG-202F-1 had very small light ppt; dark flakes, Am made two large clumps, but still transparent in color. Marked as such after 8/29/97

80

8/24/93

HG 8/8F - very large undissolved
mercury, otherwise OK

HG MAAC-2 small mercury ball
left - otherwise OK

8/1/93

2050A tube is correct replacement
for CL502A.

Joe Horvath is sending the new tube
as soon as it arrives in stock at Eberbach.

Jay Workwith of 222-S (274-27045 office)
is exploring the regulatory release
limit issue (moratorium waste designation
avoidance).

MP: 11/1/93
2/12/93

Em Borela
8-26-93

PAGE: 1

26 AUG 1993 02:37

ID:LMCS TOTAL ACT.

USER: 8

COMMENT:

PRESET TIME : 15.00

DATA CALC : AUTO DPM H# : YES SAMPLE REPEATS: 1 PRINTER : STD
COUNT BLANK : YES IC# : NO REPLICATES : 1 RS232 : EDIT
TWO PHASE : NO AGC : NO CYCLE REPEATS : 1
SCINTILLATOR: LIQUID LUMEX: NO LOW SAMPLE REJ: 0
LOW LEVEL : NO HALF LIFE CORRECTION DATE: none

WIDE OPEN WINDOW ZERROR: 0.00 FACTOR: 1.000000 BKG. SUB: 0

SAM NO	POS	TIME MIN	H#	WIDE CPM	WIDE ZERROR	WIDE DPM	WIDE EFF-1	LUMEX %	ELAPSED TIME
B1	**1	15.00	85.9	65.33	6.39	78.40	83.33	0.05	15.62
Blank Average				DPM for	WIDE :	78.40	COEF. OF VAR:	0.000	

<i>MC666K</i>	HG AB-2	1	**3	15.00	146.7	62.47	6.53	0.15	79.53	0.15	31.83
	HG 49F-1	2	**4	15.00	272.4	167.33	3.99	113.24	87.32	0.02	48.04
	HG 49F-1	3	**5	15.00	258.8	148.60	4.24	91.75	87.33	0.01	64.19
	HG 49F-1	4	**6	15.00	255.6	59.73	6.68	-0.72	76.90	0.01	80.38
	HG 49F-1	5	**7	15.00	268.9	55.13	6.95	-9.08	79.54	0.02	96.54
	HG 619F-1	6	**8	15.00	279.0	56.53	6.87	-6.45	78.57	0.08	112.68
	HG 49F-1	7	**9	15.00	253.0	241.53	3.32	194.80	88.41	0.00	128.87
	HG BECK 1F-1	8	**10	15.00	307.3	49.47	7.34	-17.99	81.88	0.04	145.03
	HG BECK 2F-1	9	**11	15.00	193.4	62.07	6.55	1.43	77.74	0.16	161.23
	HG 202 F-1	10	**12	15.00	248.6	60.60	6.63	-5.52	83.15	0.01	177.44
	HG 202 F-1	11	**1	15.00	252.7	147.07	4.26	89.50	87.59	0.01	193.73
	HG 49K13CA-1	12	**2	15.00	184.4	655.07	2.02	624.56	93.19	0.03	209.96
	HG 619F-1	13	**3	15.00	315.3	51.33	7.21	-12.24	77.60	0.03	226.21

These values used to generate the p/q results found on the analytical card.

C-37

WMC-SD-WM-DTR-030 Rev. C

the Harada
8-25-93
PAGE: 1

ID:LMCS TOTAL ACT.

25 AUG 1993 18:13

USER: 8 COMMENT:

PRESET TIME : 15.00
 DATA CALC : AUTO DPM H# : YES SAMPLE REPEATS: 1 PRINTER : STD
 COUNT BLANK : YES IC# : NO REPLICATES : 1 RS232 : EDIT
 TWO PHASE : NO AGC : NO CYCLE REPEATS : 1
 SCINTILLATOR: LIQUID LUMEX: NO LOW SAMPLE REJ: 0
 LOW LEVEL : NO HALF LIFE CORRECTION DATE: none

WIDE OPEN WINDOW XERROR: 0.00 FACTOR: 1.000000 BKG. SUB: 0

SAM NO	POS	TIME MIN	H#	WIDE		WIDE DPM	WIDE EFF-1	LUMEX %	ELAPSED TIME
				CPM	XERROR				
B1	##-1	15.00	86.2	66.80	6.32	77.85	85.81	0.08	15.64
			Blank Average	DPM for	WIDE :	77.85	COEF. OF VAR:	0.000	
1	##-3	15.00	146.4	65.20	6.40	2.18	81.48	0.24	31.85
2	##-4	15.00	267.5	160.40	4.08	110.12	85.33	0.03	48.07
3	##-5	15.00	255.8	144.20	4.30	84.97	88.57	0.01	64.22
4	##-6	15.00	252.3	59.53	6.69	-1.47	77.94	0.02	80.38
5	##-7	15.00	265.0	55.67	6.92	-11.39	83.77	0.03	96.52
6	##-8	15.00	274.9	53.40	7.07	-9.85	78.54	0.17	112.70
7	##-9	15.00	253.2	246.33	3.29	196.02	89.95	0.00	128.86
8	##-10	15.00	302.5	48.67	7.40	-18.18	81.57	0.09	145.05
9	##-11	15.00	192.8	63.67	6.47	1.78	79.95	0.35	161.27
10	##-12	15.00	247.3	60.67	6.63	-0.70	78.64	0.01	177.49
11	##-1	15.00	248.9	144.27	4.30	81.91	90.30	0.01	193.80
12	##-2	15.00	185.4	643.00	2.04	605.80	94.05	0.05	210.09
13	##-3	15.00	307.4	55.20	6.95	-3.19	73.93	0.05	226.38

copy of analytical cards

C-39

HG017E-1		count 15 minutes	
16.9842	16.6414g Tare	16.5714	
.3428			
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4
Paul Kell	Sam Burch		
Time Completed			
Date			

37

2

Sample No	J 1051-4271	BECK	Date	8-9-93	Time Recd	12:58	Priority	28
Designation	TOT-ACT	LA-548-111	Result Units	PC1/G	Charge Code	MDR21	Customer ID	0
Sample Size	?					HGANK1F1		
Remarks Calculations Results 16.7616 -16.6268g Tare .1358g $\frac{91.35 (V)}{2.22 (1.1850)} = 3.0 \text{ two } \mu\text{g/g}$ count 15 minutes								
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5				
Paul Kell	Sam Burch							
Time Completed								
Date								

4

Sample No	J 1052-4271	BECK	Date	8-9-93	Time Recd	12:59	Priority	28
Designation	TOT-ACT	LA-548-111	Result Units	PC1/G	Charge Code	MDR21	Customer ID	0
Sample Size	?					HGANK-2		
Remarks Calculations Results 17.2826 -16.7216g Tare .5610g $210.4 \mu\text{g/g}$ count 15 minutes								
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5				
Paul Kell	Sam Burch							
Time Completed								
Date								

1

Sample No	J 1049-4271	BECK	Date	8-9-93	Time Recd	12:57	Priority	28
Designation	TOT-ACT	LA-548-111	Result Units	PC1/G	Charge Code	MDR21	Customer ID	0
Sample Size	?					HGAB-2		
Remarks Calculations Results 17.2606 -16.6508g Tare .6098g $\frac{4.20}{2.22 (1.1850)} = 2.4 \mu\text{g/g}$ count 15 minutes $19.3 \mu\text{g/g}$								
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5				
Paul Kell	Sam Burch							
Time Completed								
Date								

2

Sample No	J 1050-4271	BECK	Date	8-9-93	Time Recd	12:58	Priority	28
Designation	TOT-ACT	LA-548-111	Result Units	PC1/G	Charge Code	MDR21	Customer ID	0
Sample Size	?					HGANK-2		
Remarks Calculations Results 17.1480 -16.7179g Tare .4301g $\frac{4.504}{2.22 (1.1850)} = 1.77 \mu\text{g/g}$ count 15 minutes								
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5				
Paul Kell	Sam Burch							
Time Completed								
Date								

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Copy of analytical cards

7

Sample No.	Sample Name	Date	Time issued	Priority
J 1055-4271	BECK	8-9-83	13: 2	2A
Concentration	Method Standard	Result Units	Charge Code	Remarks
TOT-ACT	LA-548-111	PC1/G	MDR21	0
Sample Size	Customer ID			
?	HG4SF-1			
Remarks, Calculations, Notes				
$\begin{array}{r} 17.4071 \\ 16.649 \text{ tare} \\ \hline 742g \\ 1948 \\ 2.22 (742) = 1.18 \times 10^{-6} \text{ g/g} \end{array}$				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mark	Tom			
Yes	Yes	Yes	Yes	Yes
Date	Time Completed	Lab Unit No.		
		PC1/G	8/30	

6

Sample No.	Sample Name	Date	Time issued	Priority
J 1055-4271	BECK	8-9-83	13: 2	2A
Concentration	Method Standard	Result Units	Charge Code	Remarks
TOT-ACT	LA-548-111	PC1/G	MDR21	0
Sample Size	Customer ID			
?	HG4SF-1			
Remarks, Calculations, Notes				
$\begin{array}{r} 17.6345 \\ 16.7910 \text{ tare} \\ \hline 8435g \\ 46.8 \mu\text{g/g} \end{array}$				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mark	Tom			
Yes	Yes	Yes	Yes	Yes
Date	Time Completed	Lab Unit No.		
		PC1/G	8/31	

8

Sample No.	Sample Name	Date	Time issued	Priority
J 1055-4271	BECK	8-9-83	13: 2	2A
Concentration	Method Standard	Result Units	Charge Code	Remarks
TOT-ACT	LA-548-111	PC1/G	MDR21	0
Sample Size	Customer ID			
?	HG4SF-1			
Remarks, Calculations, Notes				
$\begin{array}{r} 16.9953 \\ 16.6874 \text{ tare} \\ \hline 1379g \\ 2.10 \mu\text{g/g} \end{array}$				
Analyst - 1	Analyst - 2	Analyst - 3	Analyst - 4	Analyst - 5
Mark	Tom			
Yes	Yes	Yes	Yes	Yes
Date	Time Completed	Lab Unit No.		
		PC1/G	8/31	

7/22/83

9

Remarks Calculations Results				
$\begin{array}{r} 16.6546 \\ -16.65209742 \\ \hline .0026 \end{array}$ $2.23 \times 10^{-3} \text{ g/g}$				
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5
Mark O. Heck	Em. Davis			
Date	Time Completed	Lab Unit Mgr		

Count 15 minutes

analytical card

11

Remarks Calculations Results				
$\begin{array}{r} 17.2413 \\ -16.80574242 \\ \hline .4356 \end{array}$ $2.82 \text{ (500)} = 1.22 \times 10^{-3} \text{ g/g}$				
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5
Mark O. Heck				
Date	Time Completed	Lab Unit Mgr		

Count 15 minutes

analytical card

12

Remarks Calculations Results				
$\begin{array}{r} 17.1669 \\ -16.68794242 \\ \hline .4789 \end{array}$ $2.82 \text{ (500)} = 5.54 \times 10^{-3} \text{ g/g}$				
Analyst: 1	Analyst: 2	Analyst: 3	Analyst: 4	Analyst: 5
Mark O. Heck				
Date	Time Completed	Lab Unit Mgr		

Count 15 minutes

analytical card

66

9/22/93 call w Jim Hilliard
202 sample galling manometer
sample^T - 1 manometer - ?

figures ~ 6 lbs/manometer

see pg 50 for descriptions of other
"hot" samples. with cci

6 FCCI Prod. Echilperoot

HEC2

100A

Zone 1 - 1. Heater
manometer

7 steam generators, primary
and secondary, cooling containment

DOT 200 $\mu\text{Ci/g}$
limit

49 CFR 173.403 subpart
I(y)

suggested Excess: procurement
of rectifier

5 lb/inner container

Robert Nielsen - gave me (m.p.) a so light
copy of WHC "Magadous waste radiation release
protocol"

- very low LLD's (2 $\mu\text{Ci/g}$ Pb)

notes from trouble shooting the still on 7/20/93
- the valve seems to work fine to shut down the still
it is sufficient to, a shut down occurs. It is
said to be set correctly (above) to to at power.

- The valve doesn't seem to operate, even after
replacing the tube (as indicated in the notes of the still
tube was observed compared to the new one).

- The red line on the piston indicates the lowest
point to which the Hg should be allowed.

Robert - G. Hill 9/22/93

- 9/24/43 - notes on 9/20/ test continued.
- call for valve solenoid or (OVR)
(not found, broken)
 - need to reprime still, ~~and pump~~
since air got in to still head, which
should be under vacuum

9/24/43 paper - backage, - will call Monday
(president of corp.)
Result of call - to Eberbach - w. Tol. Novotny

not used

marked
10/6/43

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DATE
FILMED

7/28/94

END