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# FMPC RI/FS REPORT OPERABLE UNIT 4 LABORATORY SCREENING WORK PLAN

9-1-90

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COMMENTS

FMPC RI/FS PROJECT
OPERABLE UNIT 4 LABORATORY SCREENING WORK PLAN

GENERAL COMMENTS:

**COMMENT: 1. The last few appendices containing referenced analytical methods were not submitted with the work plan.**

RESPONSE: The appendices will be submitted with the laboratory screening work plan.

**COMMENT: 2. The treatability study work plan should state clearly what the study objectives are and how they will be met.**

RESPONSE: The objectives of the laboratory screening have been stated in the work plan.

**COMMENT: 3. Additional discussion should be provided regarding what the quality assurance/quality control (QA/QC) procedures are for the treatability study. In addition, the work plan should consider collection of additional samples to cover sample losses. Such as those caused by containers breaking, and analytical mistakes requiring re-analysis.**

RESPONSE: The bench-scale treatability plan will cover QA/QC procedures in depth.

**COMMENT: 4. The work plan should include a separate section clearly describing the roles and responsibilities of the U.S. Department of Energy and all its subcontractors involved in the treatability study.**

RESPONSE: This topic will be covered in the bench-scale treatability response.

**COMMENT: 5. The work plan should include a schedule with milestones so that the treatability study's progress can be tracked. Also, the plan should include examples of data collection sheets, to show what data will be recorded during each task, and a list of all standard test methods to be used during the study. If nonstandard methods are proposed, the reason for using the nonstandard method should be provided, along with the method.**

RESPONSE: Please see work plan #4.

**COMMENT: 6. The plan should discuss why it is not considering a mass balance of constituents and analyzing sample prior to and after leaching.**

RESPONSE: Please see the previous response.

SPECIFIC COMMENTS

**COMMENT: 7. Introduction, Page 1, Paragraph 1: "The samples are representative of the matrix of materials that are required to be treated in the full scale project." The work plan should clearly (1) identify the samples to which the plan refers and (2) state the purpose of the samples.**

RESPONSE: The samples to be used for this initial treatability study are those obtained by Westinghouse in Fall 1989. Silo 2 was sampled on 6/23/89 and Silo 1 was sampled on 7/6/89 and 7/25/89. These samples are listed in Table #1 under Task IA. The purpose of the study is to provide information about the removal of the target metals lead and uranium. Based on the solubilities of the radionuclides, it is anticipated that if the process is successful for uranium it will also remove the other radionuclides. Based on this information a subsequent work plan will be developed for treatability testing of the K-65 material including solidification, filtration, centrifugation, solids washing, solid/liquid separation, and leaching kinetics.

**COMMENT: 8. Introduction, Page 1, Paragraph 1: "The silo samples may not be quantitatively representative of the actual silo contents. However the samples are representative of the type of matrix found in the silos. As such, it is anticipated that the optimal treatment developed here will also be the optimal treatment for the representative material." The plan should define optimal treatment. Also, since the treatability study samples do not have the same relative proportions of the materials in the silos, it is unlikely that an optimal treatment can be developed.**

RESPONSE: This initial screening process is not designed to provide one optional treatment for the K-65 material. However, it is designed to provide information regarding the success of the acid extractions. The silo samples may not be quantitatively representative of the actual silo contents, but it is anticipated that if the lead and uranium concentrations can be reduced, this same

technology will be applicable to the new set of samples to be obtained by ASI/IT Corporation. Sentence will be reworded.

COMMENT: **9. Introduction, Page 1, Paragraph 1: "The second set of samples from this program will be subjected to the optimum treatability process, then analyzed for efficiency of separation." This sentence is unclear. The plan should clearly define what is meant by efficiency of separation.**

RESPONSE: "Efficiency of separation" simply refers to the success of extracting lead and uranium from the silo material.

COMMENT: **10. Introduction, Paragraph 4, Page 2: The second sentence states that lead and uranium will be tracked as target metals. The plan should state why these two parameters were selected.**

RESPONSE: Lead and uranium can be easily tracked throughout the extraction process. Uranium was chosen as a target metal because it is anticipated that if removal is successful for uranium, other radionuclides should be removed also.

COMMENT: **11. Introduction, Paragraph 5, Page 2: The last word in the last sentence should be "reduced" instead of "reduces."**

RESPONSE: The error has been corrected.

COMMENT: **12. Introduction, Paragraph 1, Page 3: The first sentence states that the optimum leaching medium and optimum conditions will be those that give the greatest lead and uranium removal. The plan should state how the optimum leaching medium and/or conditions will be determined if the medium and conditions corresponding to the greatest lead removal yield a relatively low uranium removal.**

**The remainder of the paragraph then apparently contradicts the first sentence. It states that the criterion for judging successful treatment will be determined by analyzing the leachate's lead content and multiplying that by the volume of the leachate. No mention is made of analyzing the leachate for uranium. This discrepancy should be resolved.**

RESPONSE: This study is only intended to be an initial screening analysis. The information generated from this experiment will be used to prepare a subsequent work plan to treat the samples obtained by IT Corporation. Uranium will be analyzed using the organic layer after lead has been analyzed using a HACH DRL-3 spectrophotometer. Appendix V describes the method used for uranium analysis.

COMMENT: **13. Introduction, Paragraph 2, Page 3: The second sentence states that passive radon detectors and/or an alpha-CAM detector will be used to measure radon emissions during testing. The plan should state when will the decision to use one detector over the other be made.**

RESPONSE: Passive radon detectors will be placed in the glove box and removed for analysis. An alpha-CAM detector will be used to measure radon emissions continuously during testing.

COMMENT: **14. The last sentence states that radon (misspelled as radion) emissions will be minimal in field operations. This statement should be substantiated or clarified since radon emission monitoring has not yet been conducted during treatability testing.**

RESPONSE: Based on the following assumptions;

- Radon and radium are in secular equilibrium in the contained sample.
- The Radium concentration is 192,600 pci/gm (OU-4 Remedial Investigation Report)
- Upon opening the sample container all of the enclosed radon will escape immediately into the surrounding atmosphere (assuming radon will bypass the carbon adsorbers).
- After the initial radon cloud is emitted, the contained radium will continue to decay into radon which will escape immediately into the surrounding atmosphere.
- The initial sample weighs five pounds

the worst-case calculations indicate that the instantaneous release of radon upon opening the container will be approximately 0.4 mCi and the radon release rate from a single opened sample container will be less than 3.6 uCi/hr. Samples will be handled inside a glove box. The glove box will use carbon adsorbers and HEPA filtration (in series) which is considered the best available technology to control emissions. Westinghouse

is to obtain all the necessary air permits and Federal NESHAP approval.

**COMMENT: 15. Introduction, Paragraph 3, Page 3: The first sentence states that some of the tasks in the overall treatability program are being performed by others. The work plan should describe the roles and responsibilities of each key individual/firm associated with the treatability testing.**

RESPONSE: Vitrification treatability is being addressed by WMCO in a separate program through Battelle's Northwest facility. The geotechnical feasibility testing program outlined in a document titled "K-65 Silo Residues and Subsoils for Feasibility Study Testing Plan" prepared by IT/ASI, dated August 27, 1989, has been completed. Because this work plan is intended only as an initial screening analysis, a more detailed description of each task and contractor will be included in the forthcoming treatability work plan.

**COMMENT: 16. Introduction, Paragraph 3, Page 3: The last sentence, which discusses the ability of the K-65 silo materials to be slurried, seems unnecessary and should be excluded from the treatability study work plan.**

RESPONSE: This section has been excluded from the work plan.

**COMMENT: 17. Introduction, Paragraphs 1 and 2, Page 4: The work plan states that the results of the program will influence selection of the most suitable remedial alternative, guide the methods to be used in the removal action for the silos, and satisfy the requirements outlined in Figures 5-3 through 5-6 of the initial screening of alternatives document for Operable Unit 4. The plan should clarify to which results it is referring - the results form the laboratory treatability study screening or subsequent treatability testing results.**

RESPONSE: The "results" refer to subsequent treatability testing results. The results from the laboratory screening will be used to develop the treatability work plan for the testing of samples to be obtained by IT Corporation. They will also serve as a rough guide to the reliability of Alternatives 8 and 9 of the FS report.

**COMMENT: 18. Task IA, Paragraph 1, Page 4: The table on the following page should be cited and discussed in this section. Also, the last sentence states that one of two**

**filter combinations will be used. The plan should state when the selection of the filter combination be made.**

RESPONSE: The glove box will be equipped with 2-HF resistant HEPA filters when working with hydrofluoric acid solutions (See Table #3, runs #19-24). At all other times one HEPA filter and one carbon filter will be used in series.

COMMENT: **19. Task IA, Untitled Table, Page 5: Many portions of this table should be explained. For example, the table should state what the three soil types presented for each of the two K-65 silos represent. The table should explain the multiple weights listed for the "brown" soil of Silo 1 and the "white" soil of Silo 2. Moreover, the table should explain why the total sample weight for silo 1 is almost twice the weight of the Silo 2 sample.**

RESPONSE: During geotechnical testing of the K-65 material, the samples were composited according to physical appearances. Results can be found in a certificate of Analysis, IT Geotechnical Services Project No. 482331, job number 303317 24.05.20, dated March 22, 1990. Material from Silo 1 was separated into 3 groups; brown, sandy brown, and light brown. The material from Silo 2 was also separated into 3 groups; wet muddy, white, and sandy brown. The weights listed are the actual weight amount of samples collected by Westinghouse during their sampling attempts. Based on these weight amounts and physical appearances, the samples will be composited as shown on a weight percent basis.

COMMENT: **20. Task IB, Paragraph 1, Page 5: This section should discuss why the selected baseline analyses were chosen. Other than total organic carbon (TOC), no other organic parameters are being proposed for analysis. The plan should explain why.**

RESPONSE: The preliminary characterization task will consist of compositing the K-65 Samples and preparation for the acid extraction tests. When the data from this experiment can be analyzed, a more complete baseline study including organics will be incorporated in the subsequent bench-scale treatability work plan.

COMMENT: **21. Task IIA, Paragraph 1, Page 6: The second sentence cites room temperature. The plan should define room temperature and state whether this temperature will be recorded for each test. The third sentence references Appendix III: this appendix was not provided with the treatability study work plan. Also, the meaning of the third sentence is unclear.**

RESPONSE: The room temperature will be the actual temperature inside the glove box and that information will be recorded in a standard laboratory notebook. Appendix III will be included in the work plan to describe the HACH Micro COD Digester. The HACH digesters will use acid solutions to digest the samples at 100°C for a period of two hours. Al digestions will take place inside the glove box.

**COMMENT: 22. Task IIA, Table 1, Page 6: This table should define whether the weight basis does ratio is weight of sample to weight of acid or vice-versa.**

RESPONSE: In Table 2, the weight basis ratio is the weight of the acid to the weight of the sample.

**COMMENT: 23. Task IIA, Paragraph 1, Page 7: The discussion of sample digestion addresses extraction and analysis of lead but not uranium. The sixth sentence mentions disposal of sample solids but does not state how this will be done. The eight sentence cites an analytical procedure in Appendix IV. This appendix was not provided with the treatability study work plan. The ninth sentence discusses solvent substitution -- 1,1,1,-trichloromethane for carbon tetrachloride. The work plan should explain why the procedure was modified. In the sentence "Quantification of the lead will be by HACH DRL-3 should be clarified. The work plan should also state what conditions would require that the HACH DRL-3 instrument be placed inside the glove box.**

RESPONSE: Uranium analysis will be performed on the organic layer after the lead content has been determined by the HACH DRL-3. The HACH DRL-3 is a spectrophotometer used to measure the absorbance of the lead solution. Appendix IV has been added to describe this procedure. The uranium content will be determined as described in Appendix V (Spot Tests in Inorganic Analysis, F. Feigl). 1,1,1 Trichloromethane was substituted for carbon tetrachloride to satisfy WMCO's safety policy.

**COMMENT: 24. Task IIA, Paragraph 2, Page 7: The plan should state what the leaching procedures are for the second set of samples. The plan also should clarify whether the leachate from those samples be analyzed for lead and uranium or only lead.**

RESPONSE: The second set of samples will be extracted in the same manner as the first set except that specially designed teflon digestion bombs will be necessary. These will be

heated in a sand bath to 100°C inside the glove box. These samples will be analyzed for both lead and uranium.

**COMMENT: 25. Task IIB, Paragraph 2, Page 8: The work plan cites an analytical procedure for uranium in Appendix V; this appendix was not provided with the treatability study work plan.**

RESPONSE: Appendix V has been added to the work plan.

**COMMENT: 26. Task IIB, Paragraph 3, Page 8: This paragraph states that the amount of lead and uranium leached from the samples will be compared to the other leaching test results to determine success. This contradicts the earlier sections that discuss using only lead for comparison of test results. This discrepancy should be resolved.**

RESPONSE: Data will be collected for both the uranium and lead leached from the samples. The subsequent work plan will consider both target metals to develop an optimum process.

ADDITIONAL COMMENTS
General Comments

**COMMENT: 1. The processes described in this study do not coincide with the program goals, that is to screen technologies to determine the suitability for treating the K-65 residues to be able to dispose of them as Low Level Radioactive Waste or in a sanitary landfill. The study is geared only toward separation of lead and uranium from the residues, assuming if treatment removes these radionuclides, it will also remove the other radionuclides present. However, the major contaminants of concern in the K-65 residues are radium and thorium, since these are the radionuclides that have been measured at levels that justify the management and disposal standards at 40 CFR 191 as relevant and appropriate requirements. There are no radionuclide analyses planned in this study to ensure that radium and thorium will be removed. Thus, there is no evidence that treatment will be useful in meeting the goals of this study.**

RESPONSE: These samples yielding the best uranium and lead removal results will undergo a full spectrum of radiological analyses at IT Corporation's Radiological Sciences Laboratory.

**COMMENT: 2. This document should include estimates of the expected radionuclide emissions from this project, to ensure compliance with the radionuclide emissions standard at 40 CFR 61, Subpart H.**

RESPONSE: Please see the response to Comment #14.

**COMMENT: 3. This document has so many obvious flaws in it that it seems as if it was no even reviewed by U.S. DOE before submission to U.S. EPA.**

SPECIFIC COMMENTS

**COMMENT: 1. Page 2, paragraph 4 - The assumption that removal of lead and uranium will also remove other radionuclides in the residues should be justified for all radioisotopes in question, including thorium, radium, bismuth, and polonium.**

RESPONSE: The goal of this initial screening analysis is to track only the metals lead and uranium. Based on the solubilities of uranium and other radionuclides such as thorium, radium, bismuth, and polonium, if uranium is successfully leached from the material, we anticipate the other radionuclides to also be extracted. Full radionuclide analysis will be performed on representative samples.

**COMMENT: 2. Page 2, paragraph 5 - This paragraph is too general. What removal fraction of the radionuclides is required, and what residual levels of radionuclides are acceptable to allow the residues to be disposed of in a hazardous waste landfill?**

RESPONSE: Since this work plan is to be an initial screening effort only, the removal fraction required and residual levels will be addressed in a subsequent work plan.

**COMMENT: 3. Page 3, paragraph 1 - If the optimum leaching medium and conditions will be those that give that greatest lead and uranium removal, why is only the lead content to be determined in the leachate to judge the treatment's success? The uranium level and levels of other radionuclides should also be determined.**

RESPONSE: The uranium level will be determined from the organic layer after the lead to determine the uranium content is described in Appendix V (Spot Tests in Inorganic Analysis, F. Feigl).

**COMMENT: 4. Page 3, paragraph 2 - A continuous radon monitor should be used instead of a CAM to measure emissions from**

**the glove box. Also, quantify the statement that radon emissions will be minimal in field operations.**

RESPONSE: The glove box will use carbon absorbers and HEPA filtration (in series). Westinghouse is to obtain Federal NESHAP approval and to obtain all state and local permits that are required. An alpha-CAM will be used to continuously monitor all radon emissions inside the laboratory.

**COMMENT: 5. Page 4, paragraph 1 - How will the results of this treatability study be used to guide the methods used in the Removal Action for the silos? The EE/CA does not address treatment.**

RESPONSE: The EE/CA is a separate task not related to this study.

**COMMENT: 6. Page 4, Task IA - Describe how samples of the residues in Silos 1 and 2 will be composited by weighted average, according to physical properties.**

RESPONSE: Please see response to Comment 19 of first set.

**COMMENT: 7. Page 5, task IB - Preliminary baseline analyses should also include those for thorium bismuth and polonium.**

RESPONSE: When information from this study can be analyzed, a more complete baseline study will be performed on the new samples obtained by IT Corporation. Thorium, bismuth, polonium, and other radionuclides will be incorporated into this work plan. A few representative samples will have the additional radionuclides analyzed.

**COMMENT: 8. Page 7, paragraph 1 - The processes described on this page for Task IIA are not sufficient. Radium, thorium and uranium levels should be also determined by gamma scan or alpha spectroscopy in both the residue and the extract to justify that these radionuclides are not competing in this process with lead.**

RESPONSE: For the five most promising extraction techniques, radium, thorium and uranium levels will be analyzed in both the residue and the extract to justify there is no competition with lead in this process. This shall be part of the forthcoming work plan using samples obtained by IT Corporation.

**COMMENT: 9. Page 8, Task IIB - The analysis proposed for uranium is not appropriate. Alpa spectroscopy or fluorimetry**

**analysis should be performed instead. Also, checking for radon leaks around the glove box with a survey meter is inadequate. Radiation survey meters are incapable of detecting radon gas or radon decay products. If there are air leaks in the glove box, then it can be assumed that radon is leaking out of the glove box, too.**

RESPONSE: The analysis for uranium is appropriate because the goal is only to compare one leaching technique to another. Fluorimetry analysis will be conducted in the subsequent work plan. An alpha-CAM will be used continuously during testing to ensure there are no fugitive emissions from the glove box.

COMMENT: **10. Page 8, last sentence - The criteria for success is unacceptable. Radium and thorium are the contaminants of concern.**

RESPONSE: The goal of this work plan is to determine the magnitude of lead and uranium leached compared to other processes. The other radionuclides are expected to behave in a similar manner to uranium. Representative samples will be analyzed more extensively for additional radionuclides, at an off-site laboratory.