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SUBJECT: **Recommendations For Subsequent Sampling And Analysis Events, QA/QC Analytical Requirements**

Technical Review of Initial Sampling For TA-53 Surface Impoundments

The following are recommendations that would aid and expedite review of future sampling and analysis events. HRMB would like to suggest a data format checklist for subsequent QA/QC reviews so that results are both sequenced and standardized in such a manner that an efficient and timely review can be conducted. Also, comments on initial sampling of sludge and wastewater are itemized and a section pertaining to sampling and analysis strategy is included.

RECOMMENDATIONS:

Please refer to "Components of an Adequate Laboratory Quality Assurance/Quality Control Plan" (attachment 1) for a checklist of QA/QC minimum requirements.

Please refer to "Data Format Checklist" (attachment 2) for suggestions on data presentation.

Please provide a narrative description of all technical terms used in sampling and analysis, all calculations performed and all calculation variables defined. (ie. RPD stands for what, RPD is calculated by what formula, RPD refers to the spike or duplicate spike, the use of the term duplicate spike etc.)

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COMMENTS - RECOMMENDATIONS:

1. As stated in the Volatile Organic Components (**VOC'S**) QC Summary, request #11673, samples of both sludge and wastewater "were relatively radioactive". Also, the samples for Semi-Volatile Organic Components (**SVOC'S**), both sludge and wastewater were screened for radioactivity. Was the radioactive screening quantified and documented? In the future provide these results.

2. Ten sludge samples were analyzed for SVOC'S on 7/5-7/92, as stated in HSE-9 Semi-Volatile Organic Analysis Summary Of Analytical Results dated 7/8/91, by "capillary gas chromatography/mass spectrometry using methods consistent with EPA method SW-846". Which specific method was employed (ie. 8270)? State which specific EPA method is utilized in the summary report.

3. Semi-volatile organic analysis reported significant amounts of Tentatively Identified Compounds (**TIC'S**) in all samples of both sludge and wastewater. Subsequent accounts should report all constituents identified above Method Detection Limits (**MDL**) or indicate if TIC'S are below MDL. Please refer to attachment 1 for definition of MDL.

4. The volatile organic analysis report did not mention TIC'S. If TIC'S are found, report their presence and if they are above MDL, list them in all subsequent reports. Also, it was stated in the summary report that TIC'S were identified; however, at the end of the datasheets it states "TIC'S found - none". In the future please explain such discrepancies.

5. In reference to the SVOC analysis for sludge matrix it was stated that re-analysis of Benzidine confirmed the presence thereof. In the future provide a copy of the data sheet(s) for such re-analyses.

6. In reference to attachment 1, item 4(E): One check sample at or near the Practical Quantitation Limit (**PQL**) for a subset of the parameters should be performed and reported for every 20 samples. This information should be provided in following sampling and analysis events.

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7. Sludge samples analyzed for SVOC'S were reported as "extremely wet". On page 2 of Summary of Analytical Results For SVOC'S it was stated that "therefore a reduced amount of sample was used in order to use more drying agent". In the future provide reasoning for such an approach.

8. In the HSE-9 Semi-Volatile Organic Analysis Summary Of Analytical Results dated 8/8/91 for the four re-samples of wastewater it was stated that "surrogate recoveries for all the submitted samples were within EPA guidelines". The surrogate recoveries for all the samples were reported out of QC limits. In the current data set we note that surrogate percent recoveries are frequently "out of control". Please be informed that this will compromise or possibly invalidate future data.

9. Section 3.0 discusses the desire of LANL to insure that mixed waste is not introduced into any of the three surface impoundments. There it is stated that "Because the influent to the NW surface impoundment is the only possible influent source of mixed waste, this [northwest impoundment] sample will be sufficient to characterize each of the impoundments'. It is also suggested, however, that any of the impoundments could potentially receive sanitary waste, suggesting that the potential exists for mixed waste to be present in the south RAD impoundment. The HRMB, therefore, considers it necessary to take an annual mixed-waste sample from both the northwest and south impoundments in order to insure that no mixed waste is being introduced to the impoundments.

10. Since many of the surrogate recoveries for both SVOC'S and VOC'S were "out of control", this initial waste characterization sampling and analysis event would be inadequate.

SAMPLING AND ANALYSIS STRATEGY

The RCRA Part B Permit Application section on Sampling and Analysis Strategy describes the plan for 1991 and subsequent sampling events as consisting of:

1) one initial water sample per impoundment per year (sampled during sludge-sampling event) + three additional water samples per impoundment per year
2) fifteen randomly-selected sludge samples per impoundment per year
3) water samples will be analyzed for Target Analyte List **(TAL)** metals, total volatile organic, and Base/Neutral/Acid **(BNA)** compounds
4) sludge samples will be analyzed for TCLP metals and total volatile organic and BNA compounds

The analytical data sheets for the 1991 sampling event, however, contains only the following information.

Northwest impoundment:

- water: 3 semi-volatile analyses
  3 VOC analyses + 1 trip blank
  3 EP toxicity analyses
- sludge: 3 semi-volatile analyses + 1 QC Blank
  3 VOC analyses + 1 trip blank
  3 EP toxicity analyses

Northeast impoundment:

- water: 3 semi-volatile analysis + 1 QC blank
  3 VOC analyses + 1 trip blank
  3 EP toxicity analyses
- sludge: 3 semi-volatile analyses
  3 VOC analyses
  no EP toxicity analysis

South impoundment:

- water: apparently 4 semi-volatile samples
  3 volatile analyses + 1 trip blank
  no EP toxicity analysis
- sludge: 3 semi-volatile analyses
  3 VOC analyses
  no EP toxicity analyses

The provided analytical results from the 1991 sampling fall short in several cases of the proposed sampling and analysis plan.

* The deficiencies in the 1991 analytical report are:
    1) no data from the "initial" water sample
    2) fifteen sludge samples were proposed, only three were provided
    3) no map was provided showing sample locations
    4) no clear explanation of "customer number" codes (e.g. AB-53NE-**TB**)
    5) no EP toxicity analysis for the South impoundment
    6) no PCB analyses for any of the impoundments

During future sampling events, HRMB will require complete TAL metals for the sludge samples as well as for the water samples. In addition, samples will need to include PCB analysis.

SLUDGE SAMPLING PROCEDURE

For future sludge-sampling events, the HRMB recommends the following methodology for the "Sampling and Analysis Stragegy".

- The surface impoundments should be subdivided into a grid of 441 (10'x10') uniquely-numbered blocks, in the case of the NW and NE impoundments, and 648 (10'x10') uniquely-numbered blocks in the case of the South impoundment.
- Fifteen sludge sample locations should be established in each impoundment, each one equidistant from impoundment walls and from each other.
- The locations of these fifteen sample sites should be given a block designation based on the master grid.
- In addition, one sludge sample should be taken within approximately 3 feet from the influent.
- Sampling should be done with a stainless-steel sludge sampler with core tip. The HRMB views this method as the only method proposed so far that is adequate for obtaining sludge samples.
- Should a situation arise where a sample cannot be obtained with a given block (e.g. inadequate sludge depth), an adjacent block should be sampled.
- If no sample can be obtained from any of the four adjacent blocks, then that sample location could be omitted.
- Future sampling reports provided to NMED should include the grid layout and map showing the location of each sample and designation as to whether the sample is sludge or water.

This sampling procedure assures a representative characterization of each impoundment. The spacing of sample locations will make it possible to define concentration gradients and identify potential "hot" areas.

WATER SAMPLING PROCEDURE

For future wastewater sampling events, the HRMB recommends the following procedure.

- 4 wastewater samples taken annually for each impoundment.
- the sample locations should represent the approximate center point within each of four quadrants in an impoundment
- the samples should be taken at mid-depth using a discreet interval (zone) sampler

Section 3.2.6, "Sample Preservation and Handling" states that 'If [potentially radioactive samples are] analyzed off-site, placards bearing the word "RADIOACTIVE" shall be placed inside the cooler... so as to be obvious when opening the cooler'. The HRMB recommends that all potentially radioactive containers shipped off-site have placards both inside **and** outside of the cooler.

If you have any questions regarding these matters, please contact Ms. Teri Davis or Mr. Danny Katzman of my staff at 827-4300.

Sincerely,

Ed Horst, RCRA Program Manager
Hazardous and Radioactive Materials Bureau

EH/td/dk

cc: TD
DK
BS

ATTACHMENT 1

Components of an Adequate Laboratory Quality Assurance/Quality Control Plan

New Mexico Hazardous and Radioactive Materials Bureau
Technical Support Group
(505) 827-4300

1. All constituents identified above the MDL must be reported.

   The Method Detection Limit is defined as the estimated concentration at which the signal generated by a known constituent is three standard deviations above the signal generated by a blank, and represents the 99% confidence level that the constituent does exist in the sample.

2. The "tune" of the GC/MS for volatile organic constituents must be checked and adjusted (if necessary) each twelve (12) hour shift by purging 50 ng of a of a 4-bromofluorobenzene (BFB) standard. The resultant mass spectra must meet the criteria given in Table 1 before sample analysis proceeds.

3. The "tune" of the GC/MS for semi-volatile organic constituents must be checked and adjusted (if necessary) each twelve (12) hour shift by injecting 50 ng of a Decafluorotriphenylphosphine (DFTPP) standard. The resultant mass spectra must meet the criteria given in Table 2 before analysis proceeds.

4. For every 20 samples perform and report:

   A. Duplicate spike for organics.

   B. Duplicate sample analysis for inorganics.

   C. Reagent blank, results provided for organic work.

   D. Surrogate and spike recoveries. See item 10.

   E. One check sample at or near the Practical Quantitation Limit for a subset of the parameters.

5. Analytical results must not be "blank corrected."

6. Any deviation from EPA-approved methodology must have a Written Standard Operating Procedure and NMED approval.

7. Detection limits must be generally in line with those listed in Appendix IX to §264.

8. The laboratory must document:

   A. That all samples were extracted, distilled, digested, or prepared (if appropriate) and analyzed within specified holding times.

   B. That if a sample for volatile analysis is received with headspace, this is reported.

   C. The date of sample receipt, extraction and analysis for each sample.

   D. Any problems or anomalies with the analysis should be documented.

   E. That all solids were analyzed dry or that the reported results are corrected to reflect a dry weight basis.

9. The name and signature of the lab manager must appear on each report.

10. The laboratory's historical surrogate and spike recoveries should fall within plus or minus 20% of the true value. The reported surrogate and spike recoveries must fall within: 1. the historical (statistically based) acceptance limits, generated at the laboratory or 2. the limits tabulated by the appropriate method from the current edition of SW-846, whichever limit is narrower. The actual historical recoveries must be submitted to HRMB with the analysis.

TABLE 1

BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of the base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

TABLE 2

BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

ATTACHMENT 2.

DATA FORMAT CHECKLIST

Include on Lab Data Sheets;

Location of Sample:

Type of Sample (Matrix):

Lab Sample ID:

Client Sample ID:
(include table which relates lab to client sample ID)

Date Sampled:
Date Received:
Date Extracted, If Appropriate:
Date Analyzed:

Name And Telephone # Of Analyst:

Signature Of Lab Manager:
(typed name and telephone number)

Receiving Sample Weight/Volume:

Result, w/concentration units:

Detection Limit:

List All TIC'S At Or Above MDL:

QA/QC Which Pertains To Sample Should Be Obvious:

Include in a Narrative Summary;

QA/QC Summary Listing All Technical Problems Encountered and Listing All Samples Effected:

Summary Of Results By Sample For All Parameters Identified In The Project:

Report Presence Of TIC'S: