Appendix T
Radiological Data Validation Procedures

Radiological Data Review and Validation Guidelines
Ra-226 by Radon Emanation

Stepan Company and Sears and Adjacent Properties RI/FS

1.0 Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results for Ra-226 by radon emanation analyses of environmental samples.

2.0 Purpose

The purpose of review and validation is to assure that the quality of each data point is known, and that each data point is flagged with a qualifier indicating the quality of that data point. In addition, data validation provides a review of laboratory quality control (QC) measures so that corrections to laboratory procedures can be implemented, if necessary. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages, and establishes criteria for applying appropriate data qualifiers to individual data points.

3.0 Criteria

This document provides criteria for evaluating Ra-226 by radon emanation data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab replicates, laboratory control samples (LCSs), cell constants, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers.
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.).
- Descriptions of each out-of-control situation, corrective actions taken, and resolution.
- Signature of the laboratory manager or designee.

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include the identification number of each scintillation detector calibrated, calibration date, identification, activity, certification, and expiration date of standard material. The midpoint voltage of the plateau curve for the photomultiplier tube in each detector, and raw count rate data for calibrations of each detector should also be included.

  Continuing calibration verification data should include identification of each detector system checked, date of the check, identification and activity level of standard material used for the checks, and raw count rate data from the detectors checked.

  Instrument background data should also be included in the calibrations data summary, including detector identification, count duration, background counts, and any statistical evaluation results for background counts.

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification (ID) numbers, ID of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector ID, aliquot size, date of analysis, and analyst's initials.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including ID of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample ID, activity results for sample

and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **LCS Data Summary.** The LCS summary should include date of LCS analyses, detector IDs, analyst's initials, LCS ID, activity, uncertainty and MDA for the LCS, with associated raw count data.

- **Cell Constants Data Summary.** This summary includes the identification of each cell/instrument combination, concentration in pCi/l of standards in solutions used to prepare "standard bubblers," time interval between initial and final deemanations, counts obtained for each cell/instrument combination, and efficiency or calibration factor for each cell/instrument combination. Background data including counts and count durations should also be shown for each cell.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 equivalent) showing the results for each sample (including blanks, duplicates/replicates, LCSs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain-of-custody (COC), sample ID, date of analysis (counting), detector ID, raw sample count data, background counts, count duration, aliquot used, detector efficiency, sample activity, error, and MDA results. The raw data should include any count data (background counts or spectra) necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

Calibration of alpha scintillation counters should be conducted at least quarterly using a standard traceable to the National Institute of Standards and Technology (NIST).

Flag results "estimated" (J) if the calibration was conducted using an expired NIST traceable standard. Flag results "rejected" (R) if the calibration was conducted using a standard that is not traceable to NIST.

Flag results "rejected" (R) if the plateau curve and the "midpoint" of the plateau were not determined during calibration, or have changed dramatically since the last calibration.

Calibration verification of alpha scintillation counters should be conducted at least weekly using a check source that approximates typical sample activity. The source check count rate should be within 3 standard deviations (3σ) of the source check count rate determined at the time of calibration.

Results from a counter that has a calibration verification result outside of the 3σ range should be flagged "estimated" (J).

Background should be measured at least weekly for each photomultiplier tube. Weekly background checks should be within 3σ of the background determined at the time of calibration. Flag results from counters with background count rates outside of the 3σ control limits as "estimated" (J).

3.4 Reagent Blanks

Reagent blanks are used to determine background counts due to environmental and reagent radiation sources and to monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5% of the samples. Flag results as "estimated" (J) if blanks were not run at a frequency of 5% or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered as positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities and MDAs to the Contract Required Detection Limit (CRDL). If blank results are consistently greater than the CRDL, it may be an indication of laboratory contamination. If blank MDAs consistently exceed the CRDL, it may indicate unacceptable counting times. Use professional judgment to determine if the samples should be flagged "estimated" (J) for these conditions.

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They are a measure of laboratory precision or degree of agreement of repeated measurements about acceptable ranges of concentrations. One replicate should be analyzed for each batch of 20 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S - R|}{(S + R)/2} \times 100$$

where:

S = Sample result
R = Replicate result

A control limit of $\pm$20% for water and $\pm$35% for soil samples for the RPD shall be used for sample results greater than 5 times the CRDL.

A control limit of $\pm$CRDL for water samples and $\pm$2x CRDL for soil samples shall be used for sample results less than 5 times the CRDL, including the case when only one of the sample/replicate pair results is less than 5 times the CRDL.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 LCSs

LCSs are defined as any quality assurance (QA)/QC internal laboratory standard, measurement control sample, or Environmental Protection Agency (EPA)-QC-crosscheck samples included in the daily analysis of regular samples. These samples have a known value, and a +/- value of uncertainty attached to them. Laboratory control samples should be run at a frequency of 1 per batch of 20 samples (5%). These samples provide an indication of laboratory accuracy.

A control limit of $\pm 3\sigma$ shall be used for LCSs with known values of less than 20 times the CRDL. The control limit will be based on the calculated counting error of the observed results for the LCS. Flag the results as "estimated" (J) if the known LCS value is outside of the range of $\pm 3\sigma$ of the observed LCS value.

A control limit of $\pm 2\sigma$ shall be used for LCSs with known values greater than 20 times the CRDL. Flag the results as "estimated" (J) if the known LCS value is outside of the range of $\pm 2\sigma$ of the observed LCS value. Flag the results as "rejected" (R) if the known LCS value is outside of the range of $\pm 3\sigma$ of the observed LCS value for known LCS results greater than 20 times the CRDL.

3.7 Cell Constants

The efficiency of each cell/instrument combination that is used in calculating activities, uncertainties, and MDAs must be reviewed by the data validator. In addition, background count rates for each cell should be verified.

The cell/instrument efficiency can be determined using the formula shown below:

$$E = \frac{C_N}{(A_S)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})}$$

where:

C_N = Net count rate of standard (CPM)
A_S = Activity of Ra-226 in standard (DPM)
λ = Decay constant of Rn-222
t_1 = Ingrowth time of Rn-222 (time interval between initial and final deemanation)
t_2 = Decay time of Rn-222 (time interval between deemanation and beginning of count)

The control limit for cell/instrument efficiencies is 3σ of the mean of the last 10 efficiencies. The efficiency should be close to 5.3 CPM/pCi. Flag results as "estimated" (J) if the efficiency falls outside of the 3σ control limit.

The background for each cell/instrument should be determined immediately after a sample has been counted to determine if the background count rate has changed. The control limit for background count rate is 3σ of the average of the last 10 background measurements. Background typically should be less than 0.2 CPM for environmental samples. Flag results as "estimated" (J) if the background count rate falls outside of the 3σ control limits.

3.8 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA:

3.8.1 Sample Result

$$A = \frac{CPM_G - Bkg}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})} \left[\frac{\lambda t_3}{(1 - e^{-\lambda t_3})} \right]$$

where:

A =	Ra-226 sample concentration (pCi/l or pCi/g)
CPM_G =	Gross sample count rate
Bkg =	Background count rate
2.22 =	Conversion factor (2.22 DPM = 1 pCi)
E =	Counting efficiency
Vol =	Sample mass or volume (grams for solids, liters for water)
λ =	Decay constant for Rn-222
t_1 =	Time interval between initial and final deemanation
t_2 =	Time interval between final deemanation and beginning of count
t_3 =	Sample count time

3.8.2 Counting Error

$$ER = \frac{1.96 \left(\frac{CPM_G}{t_3} + \frac{Bkg}{t_{Bkg}} \right)^{0.5} (\lambda t_3)}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})(1 - e^{-\lambda t_3})}$$

where:

ER =	2σ counting error (pCi/l or pCi/g)
CPM_G =	Gross sample count rate (CPM)
t_1 =	Time interval between initial and final deemanation
t_2 =	Time interval between final deemanation and beginning of count
t_3 =	Count time for sample (min)

Bkg =	Background count rate (CPM)
t_{Bkg} =	Count time for background (min)
2.22 =	Conversion factor (2.22 DPM = 1 pCi)
E =	Cell/instrument efficiency factor
Vol =	Sample mass or volume (grams for solids, liters for water)
λ =	Decay constant for Rn-222

3.8.3 MDA

$$MDA = \frac{4.66\ (Bkg/t_3)^{0.5}\ (\lambda t_3)}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})(1 - e^{-\lambda t_3})}$$

where:

MDA =	Minimum detectable activity
Bkg =	Background count rate (CPM)
t_1 =	Time interval between initial and final deemanation
t_2 =	Time interval between final deemanation and beginning of count
t_3 =	Count time (min)
2.22 =	Conversion factor (2.22 DPM = 1 pCi)
λ =	Decay constant for Rn-222
E =	Cell/instrument efficiency factor

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.9 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory and have the appropriate data submitted.

After the data package review is completed, detailed review of sample results should begin. The summary report form shown in Attachment 2 should be completed for each batch of samples. A general assessment of data quality for sample batches should be provided on this form. The data quality flags for each sample should be listed on the Radiochemical Analysis Analytical Results form shown in Attachment 3. One form is required for each sample. The forms for each sample in a batch should be attached to the Summary Report Form for that batch.

The data quality flags used for radiological sample results are shown below:

J = Indicates the analyte is present, but the reported value may not be accurate or precise because the associated QA/QC was unacceptable. The result is considered "estimated."

R = Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."

U = Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

-D = Qualified because laboratory duplicate control limits were exceeded.

-S = Qualified because matrix spike recovery control limits were exceeded.

-C = Qualified due to instrument calibration problems.

-B = Qualified due to blank contamination problems.

-Q = Qualified due to reasons not stated above–refer to the text of the report.

4.0 References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA–Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Radiochemical Data Validation Guidelines–Ra-226 Analysis of Soil and Water by Radon Emanation. Rocky Flats Plant, Golden, Colorado. Version 2.1. Revised September 1990.

Attachment 1

Radiochemical Data Completeness Checklist for Ra-226 Analysis by Radon Emanation of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
   - ___ ID of each detector
   - ___ Dates of last efficiency factor check certificates and DPMs of check sources; counts obtained; count durations
   - ___ Midpoint voltage of plateau curve for photomultiplier tube in each detector
   - ___ Background counts obtained for each alpha scintillation detector with count times

   B. ___ Reagent Blanks Data Summary
   - ___ ID of each cell/instrument combination used
   - ___ Analyst initials
   - ___ Date reagent blanks were analyzed
   - ___ ID of samples analyzed with the reagent blanks
   - ___ Type of method blank used, minimum detectable activity (MDA) of method
   - ___ Volume of aliquot for reagent blanks

   C. ___ Replicate Sample Data Summary
   - ___ ID of each cell/instrument combination used
   - ___ Analyst initials
   - ___ Date sample and replicates were analyzed
   - ___ Sample IDs, values obtained for sample and replicates
   - ___ Count durations of sample and replicates
   - ___ Volume of aliquot for sample and replicates
   - ___ Calculated uncertainties and MDAs

   D. ___ Lab Control Samples (LCSs) Data Summary
   - ___ ID of each detector used
   - ___ Analyst initials
   - ___ Date LCSs were analyzed
   - ___ ID of LCSs
   - ___ Values obtained for LCSs with uncertainty and MDA
   - ___ True value of LCSs with uncertainty
   - ___ ID of samples analyzed with the LCSs

E. ___ Cell Constants Data Package

___ ID of each cell/instrument combination

___ Concentration in pCi/l of solutions used in "standard bubblers"

___ Time interval between initial and final deemanations

___ Count obtained and count durations for each cell/instrument combination

___ Background count rate and count duration for each cell

3. ___ Sample Results Package

A. ___ Sample Summary Data

___ Printed report of results and counting errors for samples and reruns

___ MDA calculated for each isotopic analysis for samples with activity less than MDA

B. ___ Sample/MDA Raw Data

___ Date of analysis

___ Background CPM

___ ID of each cell/instrument combination used

___ Calculated MDA

___ Calculation sheets including, sample ID, cell/instrument identification sample counts, background counts, count durations, sample aliquots used, cell constant values, time interval between initial and final deemanations, time interval between final deemanation and counting, calculated sample activity uncertainty, and MDA

Date: _____

Attachment 2

Stepan Company and Sears and Adjacent Properties
Radiological Data Assessment
Summary Report Form

Batch No.: _____ Site: _____

Laboratory: _____ No. of Samples/Matrix: _____

_____ Reviewer: _____

Sample Numbers: _____

Ra-226 Analysis by Radon Emanation Data Assessment Summary		
	Ra-226	**Comments**
1. Holding Times		
2. Calibrations/Calibration Verification		
3. Blanks		
4. Lab Replicates		
5. Lab Control Samples		
6. Recovery Factors		
7. Sample Calculations		
8. Overall Assessment		

V = Data had no problems.
J = Data acceptable, but qualified as estimated.
R = Data rejected.
X = Problems, but do not affect data. See comments.

Data Quality: _____

_____

_____

Action Items: _____

Comments: _____

Note: Data summary tables are attached.

_____ _____

Reviewer Signature Date

Radiological Data Review and Validation Guidelines
Isotopic Analyses by Gamma Spectroscopy

Stepan Company and Sears and Adjacent Properties RI/FS

1.0 Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results from gamma spectroscopy analyses of environmental samples.

2.0 Purpose

The purpose of review and validation is to assure that the quality of each data point is known, and that each data point is flagged with a qualifier indicating the quality of that data point. In addition, data validation provides a review of laboratory quality control (QC) measures so that corrections to laboratory procedures can be implemented, if necessary. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages, and establishes criteria for applying appropriate data qualifiers to individual data points.

3.0 Criteria

This document provides criteria for evaluating gamma spectroscopy data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab replicates, laboratory control samples (LCSs), chemical recovery, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers.
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.).
- Descriptions of each out-of-control situation, corrective actions taken, and resolution.
- Signature of the laboratory manager or designee.

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include energy vs channel, efficiency vs energy, resolution [Full-Width Half Maximum (FWHM)] vs energy, background data used in the calibration, and calibration verification process.

  The energy calibration documentation should include the detector and system identification numbers, date of calibration, calibration source geometry, energy range of the system (in KeV), memory (in channels) of the detector system, calibration results (equation for energy vs channel or system gain and offset), standard reference isotopes, and certificates and activity values for calibration standard.

  The efficiency calibration documentation should include detector and system identification numbers, date of calibrations, calibration geometries, efficiency results (plot of efficiency vs energy or equation for curve) standard reference isotopes and efficiencies, and certificates and activity values for standard calibration and reference sources.

  The resolution calibration documentation should include a listing of names and activities of the isotopes used to determine system resolution and results of resolution calculations for each system. FWHM for the Co-60 peak at 1,332 KeV should be specifically listed.

  Background documentation should include results of background checks for each system, including detector identification, date of background collection, count duration, and counts in each region of interest (ROI).

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification (ID) numbers, ID of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector ID, aliquot size, date of analysis, and analyst's initials.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including ID of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample ID, activity results for sample and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **LCS Data Summary.** The LCS summary should include date of LCS analyses, detector ID, analyst's initials, LCS ID, activity of each nuclide in the LCS, and raw count data (counts in ROIs).

- **Chemical Recovery Data Summary.** This summary includes the activity of each tracer used, net counts for each isotope of interest, efficiency for each isotope of interest, count duration, and calculated chemical recovery for each sample analyzed.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 equivalent) showing the results for each sample (including blanks, duplicates/replicates, LCSs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain-of-custody (COC), sample ID, date of analysis (counting), detector ID, raw counts for each isotope of interest, background counts, tracer counts and tracer activity, chemical recovery, count duration, aliquot used, detector efficiency, sample activity, error, and MDA results. The raw data should include any count data (background counts or spectra) necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures

should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

3.3.1 Calibration

For gamma spectroscopy measurements, the detectors must be calibrated to obtain the counting efficiency for each of the radionuclides with a standard traceable to the National Institute of Standards and Technology (NIST). Each detector should have been calibrated with an gamma standard that covers the energy range of the nuclides of interest, and efficiencies should be determined using standards in the same geometries as the sample counting geometries. The calibration should have been conducted within one year of the analysis date. The annual calibration should include energy vs channel calibration and efficiency vs energy calibration. A calibration of FWHM vs energy calibration may also be necessary if required by the peak search and analysis software used for the system.

Verify that the standards used to prepare efficiency and calibration verification standards are unexpired and traceable to NIST. Flag the results "rejected" (R) if the standards are not traceable to NIST. Flag the results "estimated" (J) if they were obtained with expired NIST traceable standards.

Verify that calibrations have been performed at least annually. Flag results as "rejected" (R) if annual primary calibrations have not been performed. Verify that efficiency calibrations have been performed for each sample geometry. Flag results as "rejected" (R) if they were obtained using an incorrectly calibrated detector.

3.3.2 Calibration Verification

A calibration verification should have been performed weekly with an independently prepared verification standard. The calibration verification should verify the energy vs channel, efficiency vs energy, and FWHM vs energy (if appropriate) calibrations. As part of the calibration verification, a background spectrum should also be collected weekly. If the calibration verification was not performed, all results should be flagged "estimated" (J).

The energy vs channel system verification check should show that the system gain is 1.00 Kev/channel $\pm$ 0.03. Flag results as "estimated" (J) if the system gain falls outside of this range. Verify that the Co-60 1,332-Kev peak has not shifted more

than 2 channels from its position at the time of calibration. Flag results as "estimated" (J) if the peak has shifted more than 2 channels.

Verify from the efficiency vs energy plot for each detector that the latest efficiency value for a given energy is within 3σ of the efficiency at the same energy from the latest efficiency vs energy calibration. Flag the results "estimated" (J) if the efficiency has changed more than 3σ.

Verify that the latest background count for each peak of interest is within 3σ of the background at the time of calibration. Flag results "estimated" (J) if the current background is outside of the range of $\pm 3\sigma$ of the background established at the time of calibration.

3.4 Reagent Blanks

Reagent blanks are used to determine background counts due to environmental and reagent radiation sources and to monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5% of the samples. Flag results as "estimated" (J) if blanks were not run at a frequency of 5% or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered as positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities to the Contract Required Detection Limit (CRDL) for each nuclide. If blank samples consistently show concentrations greater than the CRDL for nuclides of interest, it may be an indication of laboratory contamination. If blank MDAs consistently exceed the CRDL, it may indicate unacceptable counting times. Use professional judgment to determine if the samples associated with such blanks should be flagged as "estimated" (J) for these conditions.

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They are a measure of laboratory precision or degree of agreement of repeated measurements about acceptable ranges of concentrations. One replicate should be analyzed for each batch of 20 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S \quad R|}{(S \quad R)/2} \quad 100$$

where:

S = Sample result
R = Replicate result

A control limit of ±20% for water and ±35% for soil samples for the RPD shall be used for sample results greater than 5 times the CRDL.

A control limit of ±CRDL for water samples and ±2x CRDL for soil samples shall be used for sample results less than 5 times the CRDL, including the case when only one of the sample/replicate pair results is less than 5 times the CRDL.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 LCSs

LCSs are defined as any quality assurance (QA)/QC internal laboratory standard, measurement control sample, or Environmental Protection Agency (EPA)-QC-crosscheck samples included in the daily analysis of regular samples. These samples have a known value, and a +/- value of uncertainty attached to them. Laboratory control samples should be run at a frequency of 1 per batch of 20 samples (5%). These samples provide an indication of laboratory accuracy.

A control limit of ±3 shall be used for LCSs with known values of less than 20 times the CRDL. The 3 control limit will be based on the calculated counting error of the observed results for the LCS. Flag the results as "estimated" (J) if the known LCS value is outside of the range of ±3 of the observed LCS value.

A control limit of ±2 shall be used for LCSs with known values greater than 20 times the CRDL. Flag the results as "estimated" (J) if the known LCS value is outside of the range of ±2 of the observed LCS value. Flag the results as "rejected" (R) if the known LCS value is outside of the range of ±3 of the observed LCS value for known LCS results greater than 20 times the CRDL.

3.7 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA:

3.7.1 Sample Result

$$A = \frac{CPM_N}{(2.22)(E)(Vol)(AB)}$$

where:

CPM_N =	Net CPM in peak. Area of peak (counts) corrected for background and divided by count duration in minutes.
2.22 =	Conversion factor (2.22 DPM = 1 pCi).
E =	Counting efficiency.
Vol =	Volume or mass of sample (liters for water, grams for solids).
ABH =	Abundance (number of photons emitted per 100 atom decays of the radionuclide).

3.7.2 Counting Error

$$ER = \frac{1.96\left(\frac{CPM_G}{T_S} + \frac{Bkg}{T_{Bkg}}\right)^{0.5}}{(2.22)(E)(Vol)(AB)}$$

where:

ER =	2σ counting error
CPM_G =	Gross sample count rate (CPM)
T_S =	Count time for sample (min)
Bkg =	Background count rate (CPM)
T_{Bkg} =	Count time for background (min)
2.22 =	Conversion factor (2.22 DPM = 1 pCi)
E =	Counting efficiency

Vol = Volume or mass of sample (liters for water, grams for solids)
AB = Abundance (number of photons emitted per 100 atom decays of the radionuclide)

3.7.3 MDA

$$MDA = \frac{4.66\ (Bkg/T)^{0.5}}{(2.22)(E)(Vol)(AB)}$$

where:

MDA = Minimum detectable activity
Bkg = Background count rate (CPM)
E = Counting efficiency
Vol = Sample volume or mass (liters for water, grams for solids)
AB = Abundance (number of photons emitted per 100 atom decays of the radionuclide)

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.8 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory and have the appropriate data submitted.

After the data package review is completed, detailed review of sample results should begin. The summary report form shown in Attachment 2 should be completed for each batch of samples. A general assessment of data quality for sample batches should be provided on this form. The data quality flags for each sample should be listed on the Radiochemical Analysis Analytical Results form shown in Attachment 3. One form is required for each sample. The forms for each sample in a batch should be attached to the Summary Report Form for that batch.

The data quality flags used for radiological sample results are shown below:

J = Indicates the analyte is present, but the reported value may not be accurate or precise because the associated QA/QC was unacceptable. The result is considered "estimated."

R = Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."

U = Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

-D = Qualified because laboratory duplicate control limits were exceeded.

-S = Qualified because matrix spike recovery control limits were exceeded.

-C = Qualified due to instrument calibration problems.

-B = Qualified due to blank contamination problems.

-Q = Qualified due to reasons not stated above–refer to the text of the report.

4.0 References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA–Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Radiochemical Data Validation Guidelines–Analyses by High Resolution Gamma Spectrometry. Rocky Flats Plant, Golden, Colorado. Version 1.1. Revised January 1991.

Attachment 1

Radiochemical Data Completeness Checklist for Gamma Spectrometric Analyses of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
   - ___ ID of each detector
   - ___ Dates of the calibration check; channel by channel printout, certificates and DPS values of check sources; count durations; calibrated energy (in KeV) for each peak of interest; calibrated centroid channel number for each peak of interest; observed channel number for each peak of interest; offset value; and calculated slope from the lease squares fit of the calibration data
   - ___ Full-Width Half Maximum (FWHM) of the peaks
   - ___ Energy range of the gamma detection system in (KeV); channels of memory
   - ___ Geometry, matrix, weight that efficiency curve is constructed for; line intensity of each nuclide of interest; counts per second observed for each peak of interest; DPS value of each nuclide; observed efficiency, observed energy, observed channel number of each nuclide; and plot of energy vs efficiency
   - ___ Integrated area of the peak regions of interest (ROIs); count duration
   - ___ Dates of last background spectra including spectra and/or channel by channel printout; count durations; counts durations; counts obtained for the peak ROIs; and statistical evaluation of the latest background data compared to a long-term background spectra

   B. ___ Reagent Blanks Data Summary
   - ___ ID of each detector used
   - ___ ID of the blank (i.e., if Ottawa Sand for Soil, etc.)
   - ___ Integrated area of peak ROIs
   - ___ Count duration
   - ___ Date reagent blanks were analyzed
   - ___ ID of samples analyzed with the reagent blanks
   - ___ Type of method blank used, minimum detectable activity (MDA) of method
   - ___ Volume of aliquot, weight, matrix, and geometry for reagent blanks

C. ___ Replicate Sample Data Summary
- ___ ID of each detector used
- ___ Date sample and replicates were analyzed
- ___ Analyst initials
- ___ Sample IDs, values obtained for sample and replicates
- ___ Count durations of sample and replicates
- ___ Calculated uncertainties and MDAs

D. ___ Lab Control Samples (LCSs) Data Summary
- ___ ID of each detector used
- ___ Analyst initials
- ___ Date LCSs were analyzed
- ___ IDs, aliquot size, weight, and geometry of LCS
- ___ Integrated areas of LCS peaks
- ___ Background counts
- ___ Count duration
- ___ Values obtained for LCSs with uncertainty and MDA
- ___ Known value of LCSs with uncertainty
- ___ ID of samples analyzed with the LCSs
- ___ Results of statistical evaluation for accuracy

3. ___ Sample Results Package

A. ___ Sample Summary Data
- ___ Printed report of results for samples and reruns
- ___ MDA calculated for each nuclide of interest for samples with activity less than MDA

B. ___ Sample/MDA Raw Data
- ___ Background spectra for each detector showing background counts accumulated for each nuclide of interest
- ___ Count duration for background
- ___ Date of analysis
- ___ Computer calculations sheet including detector identification number; date of analysis; sample number; names of nuclides detected; count duration; energy and channel number for each analysis; integrated area for each peak ROI; FWHM of each peak of interest; peak width for each ROI; calculated counts per second for each nuclide of interest; weight, matrix, and geometry of the samples; and calculated activity and uncertainty of the samples

Date: _____

Attachment 2

Stepan Company and Sears and Adjacent Properties
Radiological Data Assessment
Summary Report Form

Batch No.: _____ Site: _____

Laboratory: _____ No. of Samples/Matrix: _____

_____ Reviewer: _____

Sample Numbers: _____

Gamma Spectrometric Analyses Data Assessment Summary	
	Comments
1. Holding Times	
2. Calibrations/Calibration Verification	
3. Blanks	
4. Lab Replicates	
5. Lab Control Samples	
6. Recovery Factors	
7. Sample Calculations	
8. Overall Assessment	

V = Data had no problems.
J = Data acceptable, but qualified as estimated.
R = Data rejected.
X = Problems, but do not affect data. See comments.

Data Quality: _____

_____

_____

Action Items: _____

Comments: _____

Note: Data summary tables are attached.

_____ _____

Reviewer Signature Date

Radiological Data Review and Validation Guidelines
Isotopic Analyses by Alpha Spectroscopy

Stepan Company and Sears and Adjacent Properties RI/FS

1.0 Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results from alpha spectroscopy analyses of environmental samples.

2.0 Purpose

The purpose of review and validation is to assure that the quality of each data point is known, and that each data point is flagged with a qualifier indicating the quality of that data point. In addition, data validation provides a review of laboratory quality control (QC) measures so that corrections to laboratory procedures can be implemented, if necessary. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages, and establishes criteria for applying appropriate data qualifiers to individual data points.

3.0 Criteria

This document provides criteria for evaluating alpha spectroscopy data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab replicates, laboratory control samples (LCSs), resolution, chemical recovery, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers.
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.).
- Descriptions of each out-of-control situation, corrective actions taken, and resolution.
- Signature of the laboratory manager or designee.

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include efficiency, resolution and background data used in the calibration, and calibration verification process.

  The efficiency documentation should include detector and system identification numbers, date of calibration, standard reference isotopes and efficiencies, and certificates and activity values for standard calibration and reference sources.

  The resolution documentation should include a listing of names and activities of the isotopes used to determine system resolution, range in alpha energy of the detector system(s), memory (in channels) of the detector system(s), and results of resolution calculations for each system.

  Background documentation should include results of background checks for each system, including counts in each region of interest (ROI) and count duration.

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification (ID) numbers, ID of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector ID, aliquot size, date of analysis, and analyst's initials.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including ID of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample ID, activity results for sample and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **LCS Data Summary.** The LCS summary should include date of LCS analyses, detector ID, analyst's initials, LCS ID, activity of each nuclide in the LCS, and raw count data (counts in ROIs).

- **Chemical Recovery Data Summary.** This summary includes the activity of each tracer used, net counts for each isotope of interest, efficiency for each isotope of interest, count duration, and calculated chemical recovery for each sample analyzed.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 equivalent) showing the results for each sample (including blanks, duplicates/replicates, LCSs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain-of-custody (COC), sample ID, date of analysis (counting), detector ID, raw counts for each isotope of interest, background counts, tracer counts and tracer activity, chemical recovery, count duration, aliquot used, detector efficiency, sample activity, error, and MDA results. The raw data should include any count data (background counts or spectra) necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

For alpha particle measurements, the detectors must be calibrated to obtain the counting efficiency for each of the radionuclides with a standard traceable to the National Institute of Standards and Technology (NIST). Each detector should have been calibrated with an alpha standard representative of the target radionuclides within one year of the analysis date. The standard should have been prepared in the geometry and weight ranges expected to be encountered.

Verify that the standards used to prepare efficiency and calibration verification standards are unexpired and traceable to NIST. Flag the results "rejected" (R) if the standards are not traceable to NIST. Flag the results "estimated" (J) if they were obtained with expired NIST-traceable standards.

A calibration verification should have been performed weekly with an independently prepared verification standard. The measured efficiency value should not be more than three standard deviations (3σ) from the value determined at the time of calibration. Results for samples analyzed after a verification beyond control limits and before the next adjacent acceptable verification should be flagged "estimated" (J). If the calibration verification was not performed, all results should be flagged "estimated" (J).

Alpha spectrometers require a weekly energy vs channel calibration verification, with a source having at least two alpha emitters. The results form any detector where the energy calibration is more than three channels out of calibration should be flagged "estimated" (J).

Background spectra should be collected on each detector at least monthly for alpha spectrometer systems. Flag results "estimated" (J) if the current background is outside of the range of $\pm 3\sigma$ of the background established at the time of calibration.

3.4 Reagent Blanks

Reagent blanks are used to determine background counts due to environmental and reagent radiation sources and to monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5% of the samples. Flag results as "estimated" (J) if blanks were not run at a frequency of 5% or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered as positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities to the Contract Required Detection Limit (CRDL). If blank results are consistently greater than the CRDL, it may be an indication of laboratory contamination. If blank MDAs consistently exceed the CRDL, it may indicate unacceptable counting times. Use professional judgment to determine if the samples should be flagged "estimated" (J) for these conditions.

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They are a measure of laboratory precision or degree of agreement of repeated measurements about acceptable ranges of concentrations. One replicate should be analyzed for each batch of 20 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S - R|}{(S + R)/2} \times 100$$

where:

S = Sample result
R = Replicate result

A control limit of $\pm$20% for water and $\pm$35% for soil samples for the RPD shall be used for sample results greater than 5 times the CRDL.

A control limit of $\pm$CRDL for water samples and $\pm$2x CRDL for soil samples shall be used for sample results less than 5 times the CRDL, including the case when only one of the sample/replicate pair results is less than 5 times the CRDL.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 LCSs

LCSs are defined as any quality assurance (QA)/QC internal laboratory standard, measurement control sample, or Environmental Protection Agency (EPA)-QC-crosscheck samples included in the daily analysis of regular samples. These samples have a known value, and a +/- value of uncertainty attached to them. Laboratory control samples should be run at a frequency of 1 per batch of 20 samples (5%). These samples provide an indication of laboratory accuracy.

A control limit of $\pm 3\sigma$ shall be used for LCSs with known values of less than 20 times the CRDL. The control limit will be based on the calculated counting error of the observed results for the LCS. Flag the results as "estimated" (J) if the known LCS value is outside of the range of $\pm 3\sigma$ of the observed LCS value.

A control limit of $\pm 2\sigma$ shall be used for LCSs with known values greater than 20 times the CRDL. Flag the results as "estimated" (J) if the known LCS value is outside of the range of $\pm 2\sigma$ of the observed LCS value. Flag the results as "rejected" (R) if the known LCS value is outside of the range of $\pm 3\sigma$ of the observed LCS value for known LCS results greater than 20 times the CRDL.

3.7 Recovery Factors

An isotopic tracer solution is used to spike each sample prior to analysis by alpha spectroscopy. The tracer used should have chemical behavior similar to the target radionuclides. For most procedures, the recovery is determined using an isotope of the analyte of interest. This isotope is one that is not expected to occur in the samples to be analyzed.

The chemical recovery is calculated based on the net count rate (CPM) obtained from the tracer, the actual activity or disintegration rate (DPM) contained in the spike of the tracer solution, and the instrument efficiency. The above terms are related in the following formula:

$$\text{CPM tracer found/DPM} = \text{eff} \times \text{chem recovery}$$

Chemical recoveries for plutonium and americium analyses should be greater than 20%, but less than 105%. Chemical recoveries for isotopic uranium analyses should be greater than 30%, but less than 105%.

Flag results "rejected" (R) if these criteria are not met and the sample activity is below the MDA.

For plutonium and americium analyses, flag results "estimated" (J) if the chemical recovery is greater than 10%, but less than 20%, and the sample activity is greater

than the MDA. Flag these results "rejected" (R) if the chemical recovery is less than 10% and the sample activity is greater than the MDA.

For uranium analyses, flag results "estimated" (J) if the chemical recovery is greater than 20%, but less than 30%, and the sample activity is greater than the MDA. Flag uranium results "rejected" if the chemical recovery is less than 20% and the sample activity is greater than the MDA.

3.8 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA:

3.8.1 Sample Result

$$A = \frac{CPM_G - Bkg}{(2.22)(E)(Vol)(R)}$$

where:

A = Sample concentration (pCi/l or pCi/g)
CPM_G = Gross sample count rate
Bkg = Background count rate
2.22 = Conversion factor (2.22 DPM = 1 pCi)
E = Counting efficiency
Vol = Sample mass or volume (grams for solids, liters for water)
R = Chemical recovery

3.8.2 Counting Error

$$ER = \frac{1.96\left(\frac{CPM_G}{T_S} + \frac{Bkg}{T_{Bkg}}\right)^{0.5}}{(2.22)(E)(Vol)(R)}$$

where:

ER = 2σ counting error (pCi/l or pCi/g)
CPM_G = Gross sample count rate (CPM)
T_S = Count time for sample (min)
Bkg = Background count rate (CPM)
T_{Bkg} - Count time for background (min)
2.22 = Conversion factor (2.22 DPM = 1 pCi)
E = Counting efficiency
Vol = Sample mass or volume (grams for solids, liters for water)
R = Chemical recovery

3.8.3 MDA

$$MDA = \frac{4.66\ (Bkg/T)^{0.5}}{(2.22)(E)(Vol)(R)}$$

where:

MDA = Minimum detectable activity
Bkg = Background count rate (CPM)
T = Count time (min)
2.22 = Conversion factor (2.22 DPM = 1 pCi)
E = Counting efficiency
R = Chemical recovery

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.9 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory and have the appropriate data submitted.

After the data package review is completed, detailed review of sample results should begin. The summary report form shown in Attachment 2 should be completed for each batch of samples. A general assessment of data quality for sample batches should be provided on this form. The data quality flags for each sample should be listed on the Radiochemical Analysis Analytical Results form shown in Attachment 3.

One form is required for each sample. The forms for each sample in a batch should be attached to the Summary Report Form for that batch.

The data quality flags used for radiological sample results are shown below:

J = Indicates the analyte is present, but the reported value may not be accurate or precise because the associated QA/QC was unacceptable. The result is considered "estimated."

R = Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."

U = Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

-D = Qualified because laboratory duplicate control limits were exceeded.

-S = Qualified because matrix spike recovery control limits were exceeded.

-C = Qualified due to instrument calibration problems.

-B = Qualified due to blank contamination problems.

-Q = Qualified due to reasons not stated above–refer to the text of the report.

4.0 References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA–Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Radiochemical Data Validation Guidelines–Isotopic Analysis by Alpha Spectrometry. Rocky Flats Plant, Golden, Colorado. Version 2.1. Revised September 1990.

Attachment 1

Radiochemical Data Completeness Checklist for Alpha Spectrometric Analyses of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
   - ___ ID of each detector
   - ___ Dates of last efficiency check including spectra and/or channel by channel printout, certificates, and DPMs of check sources; counts obtained; count durations; and channels selected for regions of interest (ROIs)
   - ___ Proper channel numbers of isotopes of interest based on calibration of data of Pu, Am, and U standards
   - ___ Total memory (channels per detector)
   - ___ Energy range of the alpha detection system (KeV)
   - ___ Gain (KeV/channel) of the alpha detection system
   - ___ Dates of last background spectra including spectra and/or channel by channel printout; count durations; counts obtained; and channels selected for ROIs

   B. ___ Reagent Blanks Data Summary
   - ___ ID of each detector used
   - ___ Analyst initials
   - ___ Date reagent blanks were analyzed
   - ___ ID of samples analyzed with the reagent blanks
   - ___ Type of method blank used, minimum detectable activity (MDA) of method
   - ___ Volume of aliquot for reagent blanks

   C. ___ Replicate Sample Data Summary
   - ___ ID of each detector used
   - ___ Analyst initials
   - ___ Date sample and replicates were analyzed
   - ___ Sample IDs, values obtained for sample and replicates
   - ___ Count durations of sample and replicates
   - ___ Volume of aliquot for sample and replicates
   - ___ Calculated uncertainties and MDAs

D. ___ Lab Control Samples (LCSs) Data Summary

___ ID of each detector used
___ Analyst initials
___ Date LCSs were analyzed
___ ID of LCSs
___ Values obtained for LCSs with uncertainty and MDA
___ True value of LCSs with uncertainty
___ ID of samples analyzed with the LCSs

E. ___ Chemical Recovery Data Summary

___ Efficiency factor provided for each detector used
___ ID of each detector used
___ Net counts obtained for each isotopic tracer used
___ Count duration
___ DPM value of each isotopic tracer
___ Calculated chemical recovery

3. ___ Sample Results Package

A. ___ Sample Summary Data

___ Printed report of results and counting errors for samples and reruns
___ MDA calculated for each isotopic analysis for samples with activity less than MDA

B. ___ Sample/MDA Raw Data

___ Background measurements including counts and count durations of samples and backgrounds taken during the same weekly time period
___ Date of analysis
___ Background CPM
___ Computer calculations sheet including sample IDs, detector IDs, isotopes of interest, counts obtained for samples, background counts obtained, isotopic tracer counts obtained, count durations, DPMs of tracer used, aliquots of sample and tracer, detector efficiency, chemical recovery, activities obtained for samples, uncertainties, and MDAs

Date: _____

Attachment 2

Stepan Company and Sears and Adjacent Properties
Radiological Data Assessment
Summary Report Form

Batch No.: _____ Site: _____

Laboratory: _____ No. of Samples/Matrix: _____

_____ Reviewer: _____

Sample Numbers: _____

Alpha Spectrometric Analyses Data Assessment Summary				
	Isotopic Uranium	**Isotopic Plutonium**	**Am-241**	**Comments**
1. Holding Times				
2. Calibrations/Calibration Verification				
3. Blanks				
4. Lab Replicates				
5. Lab Control Samples				
6. Recovery Factors				
7. Sample Calculations				
8. Overall Assessment				

V = Data had no problems.
J = Data acceptable, but qualified as estimated.
R = Data rejected.
X = Problems, but do not affect data. See comments.

Data Quality: _____

_____

_____