

CHARACTERIZATION OF NATURALLY OCCURRING RADIOACTIVE  
MATERIAL (NORM) IN OIL AND GAS INDUSTRY EQUIPMENT AND  
WASTES

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Oil and Gas Industry Equipment and Wastes

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## Executive Summary

In February of 1991, a cooperative agreement between the U.S. Department of Energy (DOE) and the American Petroleum Institute (API) was established for the purpose of developing and implementing a program to characterize naturally occurring radioactive material (NORM) in oil and gas industry equipment and wastes. Under this agreement, personnel from the Idaho National Engineering and Environmental Laboratory (INEEL) were to take the lead in the project in collaboration with personnel from Grand Junction Projects Office (GJPO) of the DOE, who were to provide analytical services. Funding sources included API, the Gas Research Institute (GRI, through API) and DOE Fossil Energy (DOE-FE) office in Metairie, Louisiana. The API NORM study as it was referred to, had four main tasks:

1. Review available literature and proposed regulations related to NORM;
2. Prepare and field-test a Sampling and Analysis Plan (S&A Plan) for the collection and analysis of samples;
3. Collect and analyze samples from oil and gas production equipment, wastes, and facilities for NORM; and
4. Summarize the results of these analyses in a final report.

Task 1 was completed in June of 1992 (White 1992). Completion of Task 2 required several meetings between API, INEEL, and GJPO personnel to reach consensus on the nature and scope of sampling to be performed. Originally, a comprehensive sampling program was envisioned that would cover the contiguous United States and Alaska. However, funding levels were not adequate to develop and implement such a program. Instead, a limited sampling program was agreed upon whereby sampling would focus on areas of known contamination. These areas of known contamination were identified from a prior nationwide gamma exposure rate survey (Otto 1989), and were tabulated by state. Total sample numbers were apportioned to each state depending on the frequency of occurrence of elevated exposure levels. Because the number and type of samples collected were driven primarily by budgetary constraints rather than statistical considerations, valid spatial or temporal extrapolation of the results are limited.



The S&A Plan called for the collection of approximately 600 soil, pipe scale, and waste material samples (e.g. tank bottoms, sludges, etc.) and the analysis of these samples for concentration measurements of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ . Twenty measurements of  $^{222}\text{Rn}$  release from pipes containing barite scale were to be made along with  $^{222}\text{Rn}$  emanation measurements from the removed scale. Five samples were to be investigated for Ra solubility and 10 gas transmission lines were to be analyzed for  $^{210}\text{Pb}$ . At the suggestion of API, sampling was to be performed by API member companies to ensure that proprietary information about sites would not be released. INEEL prepared a brief field sampling manual to serve as an instruction manual for sampling. This manual was included as an appendix to the S&A Plan. Each participating member company was to designate a field representative that would be responsible for coordinating the sampling activities for that company.

The S&A Plan was completed in 1993, and was then field tested by INEEL at a site in West Texas. Measurements of  $^{222}\text{Rn}$  release from pipes were also conducted at that site. Revisions to the S&A Plan were subsequently made based on the experience gained from the field testing exercise. The S&A Plan was finalized in early 1994 (White, 1994) at which time API began contacting member companies and arranging for sample received.

From January 1994 through March 1997, API was successful in collecting 78 samples through its member companies. An additional 29 samples were collected by INEEL personnel during the field testing phase of the project, bringing the total sample count to 107. Forty-two measurements of Rn emanation and Rn release from used injection well tubing containing scale were also made at the GJPO laboratory. No  $^{210}\text{Pb}$  samples from gas delivery pipe were collected.

Because of poor success in getting samples collected and shipped to the analytical laboratory, all participants agreed in 1997 to redirect remaining funds available for this project to another NORM study. The purpose of this report is to summarize the data from the samples collected by API and INEEL as part of the original API NORM study. In these samples, Ra isotope concentrations ranged from 0.42 pCi/g to 7,400 pCi./g for  $^{226}\text{Ra}$  and from 0.24 pCi/g to 4,296 pCi./g for  $^{228}\text{Ra}$ . Radon (Rn) emanation fractions ranged from 0.02 to 0.06 and Rn release rates from pipes ranged from 0.47 to 2.9 pCi/s.

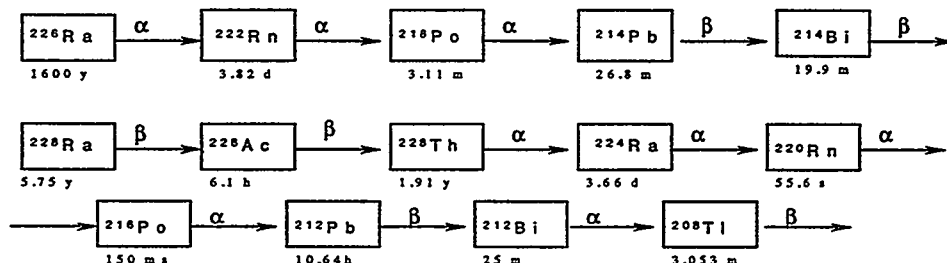
# 1. Introduction

The presence of naturally occurring radioactive materials (NORM) in oil and gas production and processing facilities has been known since the 1930s. Although widely dispersed in the earth's crust, elevated levels of NORM in oil and gas production and delivery equipment and wastes appears to be restricted to certain geographic areas, notably those where barite scales ( $\text{BaSO}_4$ ) are formed. Radionuclide contamination in oil and gas production equipment and wastes appears to be limited to the following materials (Otto, 1989; Baird et al., 1990):

1.  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  co-precipitated in some barite scales on the interior of production tubing and other equipment;
2. Sludges and sands from petroleum production equipment, containing isotopes of Ra, Th, and U;
3. Radon gas (primarily  $^{222}\text{Rn}$ ) emanating from Ra-contaminated materials;
4. Deposits containing  $^{210}\text{Pb}$  on the interior surfaces of pipes and other equipment used in the production and transmission of natural gas; and
5. Water produced during the extraction of oil and gas (produced waters).

The primary hazard associated with NORM is the inhalation of  $^{222}\text{Rn}$  decay products generated by the decay of naturally occurring  $^{226}\text{Ra}$  (Figure 1). Unlike Ra,  $^{222}\text{Rn}$  is a noble gas that is free to diffuse from the solid matrix in which it is formed. Furthermore,  $^{222}\text{Rn}$  decays with a half-life of 3.82 days through a series of short-lived alpha- ( $^{218}\text{Po}$ ,  $^{214}\text{Po}$ ) and beta-emitting ( $^{214}\text{Pb}$ ,  $^{214}\text{Bi}$ ) daughter products. Because of its much shorter half life (56 seconds),  $^{220}\text{Rn}$  produced from the  $^{228}\text{Ra}$  decay series is not considered a significant problem (Figure 1). Radon daughters are solids that can combine electrostatically to dust particles. In this solid form they can be deposited in the lung following inhalation, resulting in increased radiation dose to the lung tissue. Exposure to high concentrations of Rn daughters has been documented to cause

premature lung cancer in uranium miners. Exposure from gamma photons emitted during alpha and beta decay of Rn and Rn daughter radionuclides may also occur, but present much less of a health hazard than the inhalation of Rn daughters.



**Figure 1. Simplified decay series for  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  showing half lives and decay products important to analysis by gamma spectroscopy.**

## 1.1 Background

To date, federal regulation of radioactivity in wastes has been limited to those wastes specifically covered by the Atomic Energy Act of 1953 (AEA) and its amendments. However, NORM radionuclides are categorically excluded from regulation under the AEA, and the Nuclear Regulatory Commission (NRC) has decided not to seek legislative authority over NORM. The handling and disposal of NORM is therefore not currently regulated by either the Environmental Protection Agency (EPA) or the NRC. However, EPA is continuing to evaluate the need for regulation of NORM wastes, based on regulatory authority provided under Section 6 of the Toxic Substance Control Act (TSCA). This provision authorizes the Administrator of the EPA to regulate the disposal of any class of substances for which unregulated disposal would present an unreasonable risk of injury to human health or the environment.

To date, several states including Texas, Louisiana, and Mississippi have implemented NORM regulations that focus on the oil and gas industry (Cameron, 1996), and many other states are considering implementation of such regulations. Smith et al.

(1998) identified five general categories of NORM regulation by states: (1) development of NORM exemption standards or action levels; (2) license requirements for parties possessing handling, or disposing of NORM; (3) release of NORM-contaminated equipment and land; (4) worker protection; and (5) disposal of NORM wastes. Action levels for managing petroleum industry wastes as NORM wastes vary between states, ranging from 5 to 30 pCi g<sup>-1</sup> of total Ra (<sup>226</sup>Ra plus <sup>228</sup>Ra). Several states have established dual action levels distinguished by the Rn emanation rate of the NORM-contaminated waste. In these states, the action level is typically 5 pCi g<sup>-1</sup> total Ra if the Rn emanation rate is in excess of 20 pCi m<sup>-2</sup> s<sup>-1</sup> and 30 pCi g<sup>-1</sup> total Ra if the Rn emanation rate is below 20 pCi m<sup>-2</sup> s<sup>-1</sup>.

Although <sup>222</sup>Rn is the primary concern from a public health standpoint, regulations to date have generally been based on activity concentrations of Ra in the contaminated material. However, the relationship between <sup>222</sup>Rn concentration and radiation dose to humans is highly variable, depending in part on the <sup>222</sup>Rn emanation fraction. Because of the complex way that the physical properties of the scale materials may interact, direct measurement of the emanation fraction are needed to better estimate the exposure potential of oil field NORM.

In general, state regulations for the release of NORM-contaminated lands are derived from standards developed pursuant to the Uranium Mill Tailings Radiation Control Act of 1978 (Title 42, United States Code 7901, et seq.). These generally allow for the release of lands for unrestricted use provided the total Ra concentration in the upper 15 cm of soil is # 5 pCi/g, as averaged over 100 m<sup>2</sup>. Several states have established two release standards based on the Rn emanation rate of the NORM remaining in the soil: if the Rn emanation rate is above 20 pCi m<sup>2</sup> s<sup>-1</sup>, the release standard is 5 pCi g<sup>-1</sup> of total Ra, and if the Rn emanation rate is below that level, the release standard is 30 pCi g<sup>-1</sup> (Smith et al., 1998).

## 1.2 Information Needs

In order to ensure that NORM regulations applied to the oil and gas industry are both reasonable and practicable, information is needed on the range of activity concentrations (activity per unit mass of material) of the various radionuclides present

within oil and gas production and delivery equipment and wastes. This information must be available to accurately assess the potential risks associated with NORM contamination before decisions can be made regarding how materials containing NORM should be handled, stored, and disposed. Radon emanation rates from the different types of contaminated materials are also important in the determination of the potential impact to health and the environment from Ra-bearing materials.

The purpose of this API NORM study was to initiate a nationwide characterization of important NORM radionuclides in oil and gas production and delivery systems and wastes, focusing on known or suspected "problem areas". These geographic areas of concern were tentatively identified during a previous national survey based on external gamma radiation measurements (Otto, 1989). The goal of the present program was therefore to provide a cursory characterization of the NORM contaminated waste produced by oil and gas industry production facilities within the United States. Attempts were made to quantify reasonable upper bounds for NORM concentrations in various types of materials relating to the petroleum industry by sampling in areas the Otto study identified as being unlikely to have elevated NORM levels. The resulting data were intended to help supply the information necessary to determine risk to workers, the general public, and the environment. Due to the nationwide distribution of the types of facilities examined during this program, budget constraints limited the thoroughness with which the characterization was performed, which somewhat restricts the ability to extrapolate information gained through this program to all oil and gas producing areas in the country.

The data collected during the API NORM study were designed to help answer questions relating to the three general areas of interest summarized below:

**1. Radionuclide Concentrations:**

- What are the typical ranges of radium (Ra) concentrations that can accumulate in materials such as pipe scale, sludges, sands, and soil.

**2. Radon Emission and Emanation Fraction:**

- What are the typical ranges of Rn exhalation from pipes in storage yards that contain Ra bearing scale?
- What is a reasonable range of values for the Rn emanation fraction from NORM waste materials, pipe scales and production facility sludges?

**3. Environmental and Human Health:**

- At what types of facilities and from what types of processes might workers be at risk from NORM radionuclides?
- From what types of facilities and from what types of processes might members of the general public be at risk from NORM radionuclides?
- From what types of facilities and from what types of processes might the environment be at risk from NORM radionuclides?



## **2. Sampling Strategy and Statistical Basis**

The general strategy behind the sampling design applied to the API NORM study was described in detail in the S&A Plan (White, 1994). This included descriptions of:

1. The types of samples that were to be collected and measurements that were to be performed during the API NORM program;
2. The statistical basis applied to the determination of the sample number and location;
3. The rationale for collecting each type of sample or measurement, including descriptions of how the data resulting from each type of sample or measurement would be used;
4. How the general locations to be used for these samples and measurements were to be determined;
5. How the specific locations for sampling were to be selected in the field; and
6. The number of samples or measurements to be collected at each facility, and the quantity of each sample collected.

A primary factor in the development of the sampling strategy applied to the API NORM program was the level of funding available for sample analysis. Based on a scenario discussed earlier between INEEL and API, a limit to the total number of samples of each type was instituted. The target number for each type of sample that were to be analyzed is provided in Table 1 below. A total of 10% field QA/QC samples were included throughout the sampling program to verify the quality of the results observed.



**Table 1.** Breakdown by sample type of the target number of samples to be collected and analyzed during the API NORM Program

<b>Target No. of Samples<sup>1</sup></b>	<b>Sample Type</b>
599	Radium concentration samples
5	Radionuclide solubility measurements
20	Whole pipe Rn release measurements
30	Radon emanation fraction measurements
10	Gas pipes samples for <sup>210</sup> Pb
10	Alpha track measurements on gas pipe interiors

<sup>1</sup> including 10% QA/QC Samples

No samples were analyzed for uranium or thorium concentration. This decision was based on the assumption that with the likely exception of produced sands, these parent radionuclides would remain essentially immobile in comparison to their Ra daughter products. The parent U and Th are therefore expected to remain within the geologic formation when the more-soluble Ra is removed.

As a means of optimizing the information that generated from this program, sample collection relied heavily on the performance of gamma surveys at each field location sampled. Although somewhat time consuming to conduct in the field, the extensive reliance on gamma surveys provide several important benefits to the program, including the following:

1. Gamma surveys help to optimize the sampling program by identifying sample locations where elevated radioactivity levels are present. In this type of sampling program, a purely random selection of sampling locations would likely result in the analysis of a large number of samples showing zero or minimal levels of contamination, thereby failing to provide adequate answers to many of the questions that this program was designed to help answer.

2. The costs of conducting gamma surveys are much less than the costs of analyzing samples. Any additional information that can be extrapolated from gamma survey in conjunction with the analytical data therefore result in a more cost-effective program.
3. By recording the results of gamma surveys, it will be possible to revisit areas to collect additional or follow-up samples at some later date, provided that either additional funds are made available or additional information becomes necessary or desirable.

Despite the constraints placed on the number of samples and measurements made, a valid statistical design was applied to the API NORM Program wherever possible. Where this was not practical, the most reasonable approach was used. Several types of samples collected as part of this program do not lend themselves to the use of a statistical design. For example, the design for several types of samples is based on the earlier survey by Otto, which did not employ a valid statistical design. In other cases, notably the collection and analysis of  $^{210}\text{Pb}$  samples from gas delivery systems, the number of samples was restricted to a very limited number. This reflected the specific purpose of these samples, which was to determine the feasibility of the sampling procedure or to observe what may be considered a bounding case, rather than to conduct a nationwide characterization of this parameter. General descriptions of the strategies applied to the collection of the various types of samples are provided below.

## 2.1 Radium Concentration Samples

The purpose of collecting and analyzing samples for Ra concentration was to evaluate the extent and distribution of NORM contamination within petroleum industry equipment and waste. This information is needed before an assessment of the risk associated with these materials may be performed. Samples of pipe scale, production equipment wastes, and soil were analyzed for concentrations of both  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ . Analytical results from these Ra concentration samples provided the core of the data collected.

A total of approximately 600 samples (including field QA/QC samples) was allocated for this portion of the program. As a national program, this placed a severe restriction in terms of the statistical approach to be employed. Furthermore, the sampling strategy was largely dependant on the information provided by the Otto survey, which did

not use strict statistical procedures. Despite these deficiencies, this program was designed to help answer many of the basic questions that remain regarding the presence of NORM contamination in the petroleum industry. An approximate breakdown of the numbers and percentage of the different types of Ra concentration samples is provided in Table 2 below.

**Table 2.** Target number of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  samples to be taken for each sample type of Ra concentration sample.

Number Samples	QA/QC <sup>a</sup> Samples	TOTAL	Percent of total	SAMPLE TYPE
250	25	275	46.0	Pipe scale samples
10	2	12	2.0	Pipe yard soil samples
133	14	147	24.5	Waste samples
16	2	18	3.0	Michigan Samples <sup>c</sup>
133 <sup>b</sup>	14	147	24.5	Soil samples
		599	= Total $^{226}\text{Ra}$ and $^{228}\text{Ra}$ concentration samples	

<sup>a</sup> Represents field replicates of 10 percent of the samples collected

<sup>b</sup> Represents one composite surface and one composite subsurface sample for each of 50% of the production facilities at which waste is sampled.

<sup>c</sup> Michigan samples represent 5% of the production facility waste and soil samples.

All pipe scale, waste, and soil samples were analyzed for concentrations of Ra isotopes and  $^{40}\text{K}$  using gamma spectroscopy by the Radon Laboratory at the Grand Junction Project Office of the U.S. Department of Energy. Samples were not analyzed for uranium or thorium concentration. Potassium-40 is of interest because it contributes to the natural background exposure rate. Typical soils contain 5 to 20 pCi/g of  $^{40}\text{K}$ . A high-purity germanium (HPGe) solid-state detector was used for these analyses.

Prior to analysis, samples were dried and ground to a consistent matrix, and were then placed in a sample container for counting. Sample counting was based on the EPA procedure for analysis of gamma emitting radionuclides in drinking water (EPA 1980; "Procedure 901.1 in "Gamma Emitting Radionuclides in Drinking Water"). Differences between the soil and water media are accounted for in the calibration standard. Samples were sealed in their counting containers for a minimum of 20 days

before counting to allow  $^{222}\text{Rn}$  and progeny to grow into secular equilibrium with the parent  $^{226}\text{Ra}$ . The gamma emitting  $^{222}\text{Rn}$  daughters,  $^{214}\text{Bi}$  and  $^{214}\text{Pb}$ , which are also in secular equilibrium with  $^{222}\text{Rn}$ , were then counted (see Figure 1). Additional details are available in the S&A Plan (Appendix A).

## 2.2 Pipe scale samples

Information on the concentration of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  in pipe scale is necessary to determine the degree of risk to workers and to the public from these materials, and to evaluate the need for special disposal requirements. The purpose of collecting pipe scale samples was therefore to determine the concentration of Ra isotopes that accumulate within these materials.

Although pipe cleaning and storage yards were not surveyed as part of the earlier national survey conducted by Otto, information from the Otto survey was still be applied to the selection of pipe cleaning and storage yards for the collection of pipe scale samples. This was done by applying certain assumptions regarding the geographic areas from which each facility receives pipe. Primary among these is that NORM concentrations in the pipe scale from a given pipe yard are correlated with the frequency with which production facilities located within the same general geographic area as the pipe yard indicated elevated gamma readings during the earlier study by Otto. It was therefore assumed that used pipe was shipped to the pipe cleaning yards that are relatively nearby.

Pipe scale samples were to be collected from five different pipe yards identified by API. At each of the five pipe cleaning and storage yards selected, scale from a total of 50 pipes was to be sampled. Field replicates were also to be collected from five of the 50 pipes sampled at each pipe yard, making the total of 55 pipe scale samples to be collected at each pipe yard. Based on the results of a gamma survey of the site, the 10 pipes with the highest gamma readings were to be sampled. Results from these 10 hottest pipes provided the upper limit for Ra concentrations in the pipe scale from these yards. The remaining 40 samples were to be chosen randomly from the population of pipes surveyed that showed gamma readings in excess of  $50 \mu\text{R hr}^{-1}$  to provide a reasonable range of Ra concentration values for the yard. The total number of pipes surveyed that exhibited gamma exposure rates of at least  $50 \mu\text{R hr}^{-1}$  were noted.

## 2.3 Production Facility Waste Samples

Wastes from petroleum production activities were sampled to determine the Ra concentrations found in waste materials other than pipe scale that are generated from oil production facilities. A database listing the locations by state and county at which Otto conducted measurements was provided by API. General locations for collecting samples were determined using these data and by calculating a weighted sample distribution. This represented the percent of elevated gamma measurements found during the Otto survey and distributed by county, giving equal weight to the different types of equipment in which elevated readings were observed (e.g. sumps, separators etc.), even though a different number of measurements were made on each type of equipment.

The criterion for the selection of the locations from which these samples were to be collected was based on the  $50 \mu\text{R hr}^{-1}$  (including background) surface gamma survey criterion imposed by the state of Louisiana as a general limit for exposure. The sampling population of interest was therefore defined as all locations in which the Otto study found equipment that exhibited external gamma readings of greater than  $50 \mu\text{R hr}^{-1}$  in any of the following equipment types: flow lines, heater treaters, separators, sumps, and water lines. From Otto's gamma survey data, a total of 1573 measurements exceeded the  $50 \mu\text{R hr}^{-1}$  criterion. Weighting samples by the relative number of elevated readings in the different types of equipment using this criterion was designed to allow for the evaluation of a large number of sites while concentrating the sampling at sites from which elevated readings have been observed by Otto.

Much of the state of Michigan was not included in the Otto study, and concerns have been expressed recently regarding the potential for NORM contamination in that state. To correct this omission, approximately five percent of the production facility samples (soil and production waste) were to be collected in Michigan.

Table 3 provides a breakdown of samples to be taken in each county based on the Otto survey data and 133 production facility samples. Sixteen samples plus two field replicates samples were allocated to the State of Michigan. Ideally, one waste sample was to be taken at each facility, resulting in a total of 133 facilities sampled. A complete summary of all the measurements made by Otto on the five equipment types considered here is provided in the S&A Plan (Appendix A).

**Table 3.** Summary of samples within counties with NORM contaminated equipment (> 50  $\mu\text{R hr}^{-1}$  as measured in Otto report). Based on equal weighting of 5 equipment types: flow lines, heater-treaters, separators, sumps, and water lines.

State/County	Suggested Sample Number	State/County	Suggested Sample Number	State/County	Suggested Sample Number
<b>ALASKA</b>		<b>MISSISSIPPI</b>		<b>OFFSHORE</b>	
	1	JASPAR	1	GULF	16
OFFSHORE	2	JONES	3	<b>OS SUBTOTAL</b>	<b>16</b>
UNREPORTED	3	MARION	3		
<b>AK SUBTOTAL</b>		SMITH	1	<b>TEXAS</b>	
		WALTHALL	3	BROOKS	4
<b>ALABAMA</b>	3	WAYNE	1	CHAMBERS	1
ESCAMBIA	3	<b>MS SUBTOTAL</b>	<b>12</b>	FRANKLIN	1
<b>AL SUBTOTAL</b>				GRAY	2
		<b>MONTANA</b>		GRAYSON	1
<b>CALIFORNIA</b>	1	SHERIDAN	3	HIDALGO	3
KERN	1	<b>MT SUBTOTAL</b>	<b>3</b>	JIM WELLS	1
<b>CA SUBTOTAL</b>				KENEDY	4
		<b>NORTH</b>		KLEBERG	3
<b>ILLINOIS</b>	2	DAKOTA		MITCHELL	4
FAYETTE	5	DIVIDE	1	NUECES	3
UNREPORTED	7	<b>ND SUBTOTAL</b>	<b>1</b>	PECOS	1
<b>IL SUBTOTAL</b>				ROBERTS	1
		<b>NEW MEXICO</b>		THROCKMORT	
<b>KANSAS</b>	1	LEA	4	-TON	1
STAFFORD	1	SAN JUAN	3	UPTON	1
<b>KS SUBTOTAL</b>		UNREPORTED	4	WALLER	1
		<b>NM SUBTOTAL</b>	<b>11</b>	WARD	2
<b>LOUISIANA</b>	3			WILLACY	1
ACADIA	1	<b>OKLAHOMA</b>		WOOD	2
ASSUMPTION	3	CANADIAN	1	YOUNG	1
IBERIA	3	CARTER	3	<b>TX SUBTOTAL</b>	
LAFOURCHE	1	CLEVELAND	1		<b>38</b>
PLAQUEMINES	2	CREEK	4	<b>WYOMING</b>	
ST. MARTIN	1	GARVIN	1	UNREPORTED	
TERREBONNE	3	KAY	1	<b>WY SUBTOTAL</b>	<b>1</b>
VERMILLION	17	KINGFISHER	3		
<b>LA SUBTOTAL</b>		SEMINOLE	2	<b>TOTAL</b>	<b>1</b>
		STEPHENS	3		
		<b>OK SUBTOTAL</b>	<b>19</b>		<b>133</b>

## 2.4 Soil samples

Soil samples were to be collected at each pipe cleaning yard used to collect scale samples and at many of the production facilities used to collect waste samples. The production facilities to be sampled for soil were chosen randomly by API from among the facilities sampled for waste. The soil sampling design followed methods used elsewhere for characterizing soils contaminated with uranium mill tailings (e.g. Williams et al., 1989). At each facility sampled, collection personnel first conducted a gamma survey with a properly calibrated exposure rate meter, and mapped out hot spots where gamma readings of greater than  $50 \mu\text{R hr}^{-1}$  are found. The  $50 \mu\text{R hr}^{-1}$  criterion was chosen because this represents the calculated exposure rate from a surface soil containing a concentration of  $30 \text{ pCi g}^{-1}$  of  $^{226}\text{Ra}$ . These calculations were performed with the Microshield code (Grove Engineering, 1992).

Based on the results of the gamma survey, the sample collection personnel were to select the single "hottest" area for collection of soil samples, even if no readings in excess of the  $50 \mu\text{R hr}^{-1}$  criterion were found. A rectangular area of at least  $100 \text{ m}^2$  ( $1100 \text{ ft}^2$ ), with a minimum dimension of 5 m (16 ft) was surveyed. From each such grid area, one surface and one subsurface composite sample of 9 soil cores was collected. The 9 core locations will be spaced equidistant over the  $100 \text{ m}^2$  ( $1100 \text{ ft}^2$ ), so as to include the entire plot. Surface soil composite samples were collected from the top 15 cm (6 in) and the subsurface samples from the 15-30 cm (6-12 in) layer. This sampling approach essentially follows that proscribed by the Louisiana regulations. Implementation of this technique was designed to allow for the maximization of the number of sampling sites, so as to achieve the most extensive national representation possible, given the available funding levels. Although analytical costs are limiting, substantial additional data will be provided from the results of the initial gamma survey, the information from which may be compared with the results of the soil sample analysis.

## 2.5 Radium Solubility Measurements

A subsample of five pipe scale samples found to have elevated Ra concentrations was to be used to determine the solubility of the Ra-containing material. The purpose of determining the solubility of pipe scale was to evaluate the potential mobility of the Ra from contaminated scale material in water solutions. The samples chosen for this analysis

were to include those with relatively high Ra concentrations. No statistical design was applied to this portion of the program.

## 2.6 Whole Pipe Radon Release Measurements

The purpose of collecting Rn samples from NORM contaminated pipe samples was to determine the rate at which Rn is released to the environment from pipes containing Ra-bearing scales. Twenty sections of NORM-contaminated pipe from a single site in West Texas were selected for sampling. A statistically-based geographic distribution was not applied to this portion of the study.

Radon release rates from pipes were measured by collecting and subsequently measuring the amount of Rn released from the pipe over a timed interval. This was accomplished by fitting a column containing activated charcoal to one end of the pipe section and a supply of Rn-free air to the other end. A continuous flow of Ra-free air was used to sweep out any Rn that emanates from the Ra-contaminated scale. The Rn thus purged from the interior of the pipe was subsequently collected on the charcoal column. A quantitative analysis of the Rn collected on the charcoal was provided by gamma analysis. Additionally the interior connected volume of the pipe section was determined by evacuating the pipe and allowing it to return to atmospheric pressure through a dry gas meter. Additional information on the procedures used to measure whole pipe Rn release is provided in Rood et al. (1998).

## 2.7 Radon Emanation Fraction Measurement

Some of the  $^{222}\text{Rn}$  atoms created by the decay of  $^{226}\text{Ra}$  present in the pipe scale (or other Ra-bearing material) will be retained within the crystal lattice of the host mineral for a time sufficient to allow further decay into  $^{218}\text{Po}$ . Other atoms, especially those formed near the surface of the mineral grain, escape the crystal lattice as free gaseous Rn atoms. The fraction of the total Rn atoms produced by the Ra parent that are released as free gaseous Rn atoms is termed the *radon emanation fraction*. Measurements of the Rn emanation fraction for NORM contaminated pipe scales and sludges are useful in determining the potential exposures to workers and the general public from Rn released from sites containing these NORM contaminated materials.



Radon emanation fraction measurements were to be performed on 30 samples comprised of approximately 15 pipe scale samples and 15 process facility waste (sludge) samples provided by API member companies. In contrast to the sludge samples provided to the GJPO Analytical Laboratory for Ra analysis the sludge samples provided for Rn emanation fraction were not burned to remove the organic fraction. These scale samples were to be obtained from the pipe by the processes that is routinely used to clean scale from pipe.

The emanation measurements were made by evacuating the sample with dry nitrogen gas, sealing it in a container, then counting it immediately afterward using a NaI detector. The 352 keV  $^{214}\text{Pb}$  and 609 keV  $^{214}\text{Bi}$  were observed because of high count yields and high detector counting efficiency. A second count was made several weeks afterward. Counting at two different times allows for calculations of the Rn emanating fraction based on the theoretical ingrowth curve.

Figure 2 shows the theoretical  $^{222}\text{Rn}$  ingrowth curve. The sample activities at times  $t_1$  and  $t_2$  ( $I_1$  and  $I_2$ ) are described by Equations (1) and (2) below.

$$I_1 = I_o + N(1 - e^{-\lambda t_1}) \quad (1)$$

$$I_2 = I_o + N(1 - e^{-\lambda t_2}) \quad (2)$$

Where:  $I_1 = \text{Activity at time } t_1$   
 $I_2 = \text{Activity at time } t_2$   
 $I_o = \text{Bound Rn Activity at time } t_o$   
 $N = \text{Free, or Emanating Rn Activity at Radioactive Equilibrium}$   
 $\lambda = \text{Rn Decay Constant}$

These two equations can be solved simultaneously for the unknown quantities  $I_o$  and  $N$ . Substituting  $A$  for  $(1 - e^{-\lambda t_1})$  and  $B$  for  $(1 - e^{-\lambda t_2})$  simplifies the equations. The solutions are given in Equation (3). The emanation fraction  $F$  is computed from Equation (4).

$$N = \frac{(I_1 - I_2)}{A - B}, \quad I_o = \frac{I_2 A - I_1 B}{A - B} \quad (3)$$

$$F = \frac{N}{N + I_o} \quad (4)$$

It should be noted that it is not necessary to determine the actual sample activities ( $I_1$  and  $I_2$ ) at times  $t_1$  and  $t_2$ . The net count rates ( $C_1$  and  $C_2$ ) observed at  $t_1$  and  $t_2$  are proportional to the activities ( $I_1$  and  $I_2$ ) through a calibration constant, which cancels out when the final ratio is taken in Equation (4). Thus  $C_1$  and  $C_2$  may be substituted for  $I_1$  and  $I_2$ .

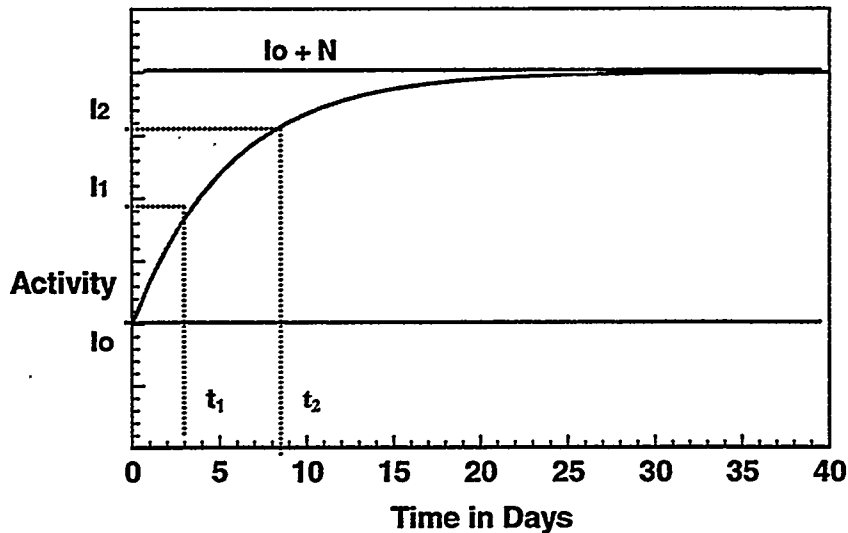


Figure 2. Theoretical radon ingrowth curve defining  $I_0$ ,  $I_1$ , and  $I_2$ .

## 2.8 Gas pipe $^{210}\text{Pb}$ samples

Segments of gas transmission lines were to be analyzed for  $^{210}\text{Pb}$  contamination for the purpose of determining the extent to which this Rn daughter radionuclide accumulates on the interior of gas pipes and equipment. Selection of sampling sites were to be based on several factors including known Rn concentrations within the natural gas during the lifetime of the pipe, the length of service of the pipe, and whether access to the pipe was readily available. This portion of the study was therefore intended to serve only as a pilot study rather than as general characterization of  $^{210}\text{Pb}$  accumulation in gas transmission lines. A total of 10 samples of gas transmission lines were to be collected by GRI and sent to GJPO for analysis of  $^{210}\text{Pb}$  concentration.

## 2.9 Alpha Track Measurements on Gas Pipe Interiors

The first decay product of  $^{210}\text{Pb}$  is  $^{210}\text{Po}$ , which itself decays by alpha emission. The half-life of  $^{210}\text{Po}$  is 138 days, so that after a few years, the activity of  $^{210}\text{Po}$  approaches equilibrium with the activity of  $^{210}\text{Pb}$ . The number of "alpha tracks" recorded on a special detection film placed on the interior of the pipe will be proportional to the  $^{210}\text{Po}$  activity. This allows for an estimation of the amount of  $^{210}\text{Pb}$  found on the interior of the gas transmission line.

The same ten samples of gas delivery pipe or equipment provided by GRI for analysis at GJPO for  $^{210}\text{Pb}$  contamination were to be used to determine the extent of residual  $^{210}\text{Po}$  contamination. Alpha track procedures were to be used to determine the presence of residual activity in the pipe before surface contamination is removed.

### 3. Results and Discussion

This section provides summaries of the analytical results from samples collected as part of the API NORM program. Raw data is also provided in tabular form in Appendix B.

Problems in the proposed sampling strategy became apparent shortly after the field test was completed and sampling was to have commenced. Most of the samples requested from member companies by API were not collected. Furthermore, it was often clear that samples received were not collected in accordance with procedures outlined in the S&A Plan, and sample documentation was often incomplete. In some cases, no sample identification was provided and improper sample containers were used resulting in leakage of a sample from its shipping container prior to analysis. Collectively, these inadequacies prevented our reaching established sampling target numbers which, in turn, make interpretation of the data generated difficult.

Between January 1994 and March 1997, a total of 78 samples were collected by API member companies. An additional 71 samples were collected by INEEL personnel during the field testing of the S&A Plan, making a total of 149 samples available during the project. A summary of the number and type of samples collected during the API NORM program is provided in Table 4.

A summary of sample results by medium is provided in Table 5. Additional information on each sample type is provided in subsequent sections.

**Table 4.** Sample type and number of samples collected by participants in the API NORM study

Sample Type (Collected By...)	Number
Solid Matrix Samples (API Member Companies)	78
Solid Matrix Samples (INEEL)	29
Total Solid Matrix Samples Collected	107
Radon Emanation Measurements (INEEL)	22
Radon Release from Pipes (INEEL)	20
Total	149

**Table 5.** Numbers, types, and average Ra concentrations and  $^{222}\text{Rn}$  emanation fractions for the NORM samples collected.

Sample Type	Sample Number	Mean Values (Standard Deviation)			
		$^{222}\text{Rn}$ Emanation Fraction	$^{222}\text{Rn}$ Pipe Flux ( $\text{pCi s}^{-1}$ )	$^{226}\text{Ra}$ ( $\text{pCi g}^{-1}$ )	$^{228}\text{Ra}$ ( $\text{pCi g}^{-1}$ )
Whole pipe Rn release flux	20		1.2 (7.4)		
Soil	42			82.3 (160)	8.93 (13)
Production waste (sludge, tank bottoms, etc.)	28			238 (351)	66.0 (95)
Scale <sup>a</sup>	35	0.036 (0.021)		1403 (1434)	1852 (1658)
Other miscellaneous samples	2			274 (220)	193 (143)

a.  $^{222}\text{Rn}$  emanation measurements were performed using aliquots from 22 of the 35 scale samples

### 3.1 Radium Concentration Samples

Mean  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  concentrations for pipe scale, soil, and sludge samples collected during the NORM characterization program are provided in Table 6. Radium-226 concentrations in these samples ranged from below detection limits to 2762 pCi g<sup>-1</sup>, while  $^{228}\text{Ra}$  concentrations ranged from below detection limits to 4296 pCi g<sup>-1</sup>. Although concentrations of both isotopes were significantly higher in the pipe scale samples in comparison with other media, concentrations were highly variable within each medium, as indicated by the large standard deviations shown in Table 6. Pipe scale Ra concentrations were generally in the same range as reported for uranium mill tailings (Rogers et al., 1984).

Radium concentration data are provided in Table B-1 (pipe scale), Table B-2 (soil), and Table B-3 (waste) of Appendix B for each sample individual sample collected.

	$^{226}\text{Ra}$ concentration (pCi g <sup>-1</sup> )	$^{228}\text{Ra}$ concentration (pCi g <sup>-1</sup> )	Ratio $^{226}\text{Ra} / ^{228}\text{Ra}$
<b>Pipe Scale</b>	1403 (1435)	1852 (1658)	2.00 (2.00)
<b>Soil</b>	82.3 (160)	8.9 (13.3)	6.7 (8.9)
<b>Waste</b>	238 (352)	65.9 (95.4)	8.54 (20.6)

### 3.2 Radium Solubility Measurements

No Ra solubility measurements were performed as part of the API NORM Characterization Program.

### 3.3 Whole Pipe Radon Release Measurements

Radon-222 release measurements were conducted on twenty pipe segments containing barite scale from a single site in Texas (Table 7). Based on the Ra concentrations measured for these 20 samples, the pipe scale would be regulated in those states that have enacted NORM regulations.

The  $^{222}\text{Rn}$  flux rates from the ends of these pipe segments ranged from 0.46 to 2.7  $\text{pCi s}^{-1}$ , with a mean of 1.1  $\text{pCi s}^{-1}$  and a standard deviation of 0.62  $\text{pCi s}^{-1}$ . By way of comparison, federal regulations limit Rn flux from uranium mill tailings to 20  $\text{pCi m}^2 \text{s}^{-1}$ . This suggests that health risks associated with the above ground storage of NORM-contaminated pipe are comparable to those of a properly remediated uranium mill tailings pile. Wilkening, et al. (1972) give a worldwide average of 0.43  $\text{pCi m}^{-2} \text{s}^{-1}$  for radon-flux density from natural soils, with a range from 0.006 to 140  $\text{pCi m}^{-1} \text{s}^{-1}$ . Thus, the Rn production potential of a single section of NORM-contaminated pipe from this site is comparable to approximately 2.5  $\text{m}^2$  of average ground surface. Additional information on these measurements is provided in Rood et al., 1998.

**Table 7. Radon flux and emanation fraction measurements for 20 pipe segments collected at a site in northern Texas.**

Pipe No.	Scale Volume (liters)	Average Scale Thickness (cm)	Radon Flux (pCi s <sup>-1</sup> )	Radon Emanation Fraction	Weight Gain Ratio	<sup>226</sup> Ra (pCi g <sup>-1</sup> )	Loss on Drying (%)
1	3.44	0.25	0.81	0.06	1.04	895	6.7
2	6.72	0.49	1.65	0.04	1.03	2150	10.7
3	3.24	0.21	0.46	0.03	1.03	1978	5.5
4	3.12	0.23	0.81	0.03	1.03	1941	5.3
5	12.22	1.08	2.81	0.03	1.00	2156	20.7
6	7.63	0.54	1.03	0.05	1.03	398	9.0
7	6.84	0.53	0.89	0.05	1.04	425	9.4
8	2.96	0.20	1.08	0.03	1.03	2748	4.9
9	3.85	0.26	0.51	0.03	1.03	1042	6.2
10	3.65	0.24	1.05	0.02	1.03	2322	5.0
11	4.00	0.28	2.40	0.04	1.04	2224	5.8
12	3.27	0.23	0.92	0.03	1.03	2629	4.1
13	4.23	0.28	0.65	0.04	1.04	717	7.3
14	4.21	0.28	0.92	0.03	1.04	2338	5.5
15	4.85	0.33	1.97	0.04	1.04	2762	5.2
16	4.91	0.33	0.68	0.035	1.04	707	8.8
17	3.56	0.25	1.03	0.030	1.03	2437	4.2
18	4.77	0.32	0.54	0.034	1.04	658	6.5
19	3.98	0.26	0.89	0.033	1.03	2256	4.6
20	3.64	0.25	0.65	0.057	1.04	592	6.5



### **3.4 Radon Emanation Fraction Measurements**

Radon emanation fraction measurements were conducted for the same 20 pipe segments for which whole pipe Rn release measurements were determined (Table 7). Radon emanation fractions for scale from these 20 pipe segments ranged from 0.020 to 0.063 with a mean of 0.04 and standard deviation of 0.01. These values are substantially lower than emanation fractions measured for uranium mill tailings of comparable  $^{226}\text{Ra}$  content, which typically range from 0.1 to 0.3 (Rogers et al., 1984), implying that health impact assessments for disposal of uranium mill tailings should not be simply extrapolated to NORM scale materials since the emanation fraction of the two materials differs substantially. This difference is largely due to different physical characteristics of the two media. Additional information on Rn emanation fraction measurements is available in Rood et al., 1998.

### **3.5 Gas Pipe $^{210}\text{Pb}$ Samples**

No gas pipe segments were received from GRI, so  $^{210}\text{Pb}$  measurements were not conducted during this study.

### **3.6 Alpha Track Measurements on Gas Pipe Interiors**

No gas pipe segments were provided by GRI, so alpha track measurements on pipe interiors were not conducted.

## 4. CONCLUSIONS

Although the information gained will be useful in terms of characterizing NORM activities in oil and gas equipment and wastes, valid extrapolation of results either spatially or temporally is tenuous at best. As the number of each type of sample to be collected or measurement to be conducted during this program was driven primarily by budgetary constraints rather than by statistical considerations, conclusions resulting from the data are difficult to interpret. Furthermore, the lack of success at receiving viable samples from participating organizations resulted in the termination of entire portions of the program.

Despite these problems, some general conclusions may be made. First, it is readily apparent from the data collected during this and other subsequent programs that Rn emanation from barite pipe scale is considerably lower than emanation from uranium mill tailings. Furthermore, Rn flux from undisturbed NORM-contaminated barite scale remaining in production tubing is minimal (Rood et al., 1998). From a health standpoint, this suggests that wherever possible, NORM-contaminated pipe scale should not be removed from the pipe. It also suggests that regulations developed for pipe scale that are based on Rn emanation rates from uranium mill tailings may be overly conservative.

It is also evident that Ra concentrations in pipe scale, soils, and wastes are highly variable. Additional data is needed on Ra concentration levels and on the relationship between concentration and Rn emanation not only for pipe, but also for soil and waste materials.



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**Appendix A**

**SAMPLING AND ANALYSIS PLAN**



**Sampling and Analysis Plan:**  
**Characterization of Naturally Occurring**  
**Radioactive Material (NORM) Contamination**  
**of Oil and Gas Industry Equipment**  
**and Wastes**

**Center for Environmental Monitoring and Assessment**  
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## ACRONYMS

AEA	Atomic Energy Act
ALARA	<u>A</u> s <u>L</u> ow <u>A</u> s <u>R</u> easonably <u>A</u> chievable
API	American Petroleum Institute
ASTM	American Society of Testing and Materials
cpm	Counts per minute
CRCPD	Conference of Radiation Control Program Directors
DMS	Data Management System
DPM	Disintegrations per Minute
DQOs	Data Quality Objectives
EPA	Environmental Protection Agency
GJPO	Grand Junction Project Office
GRI	Gas Research Institute
HpG	High purity Germanium
ID	Identification
ID	Inside Diameter
INEL	Idaho National Engineering Laboratory
LDEQ	Louisiana Department of Environmental Quality
LDR	Landfill Disposal Restrictions
MDC	Minimum Detectable Concentration
NIST	National Institute of Standards and Testing
NORM	Naturally Occurring Radioactive Material
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
RPD	Relative Percent Difference
S&A	Sampling and Analysis
TRCR	Texas Regulations for Control of Radiation
TSCA	Toxic Substances Control Act
UMTRA	Uranium Mill Tailings Remedial Action



# 1. Introduction

The presence of naturally occurring radioactive materials (NORM) in oil and gas production and processing facilities has been known since the 1930s. Although widely dispersed in the earth's crust, elevated levels of NORM in oil and gas production and delivery equipment and wastes appears to be restricted to certain geographic areas, notably those where barite scales ( $\text{BaSO}_4$ ) are formed. Radionuclide contamination in oil and gas production equipment and wastes appears to be limited to the following (Otto, 1989; Baird et al., 1990):

1.  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  co-precipitated in some mineral scales on the interior of production tubing and other equipment;
2. Sludges and sands from petroleum production equipment, containing isotopes of radium, thorium and uranium;
3. Radon gas (primarily  $^{222}\text{Rn}$ ) emanating from radium-contaminated materials;
4. Deposits containing  $^{210}\text{Pb}$  on the interior surfaces of pipes and other equipment used in the production and transmission of natural gas; and
5. Water produced during the extraction of oil and gas (produced waters).

## 1.1 Background

On a national basis, the handling and disposal of NORM is not currently regulated by either the Environmental Protection Agency (EPA) or the Nuclear Regulatory Commission (NRC). To date, federal regulation of radioactivity in wastes has been limited to those wastes specifically covered by the Atomic Energy Act of 1953 (AEA) and its amendments. However, NORM radionuclides are categorically excluded from regulation under the AEA, and the NRC has decided not to seek legislative authority over NORM. EPA is continuing to evaluate the need for regulation of NORM wastes. The regulatory authority exists for EPA to do so under Section 6 of the Toxic Substance Control Act (TSCA), which authorizes the Administrator of the EPA to regulate the disposal of any class of substances for which unregulated disposal would present an unreasonable risk of injury to human health or the environment.

The Conference of Radiation Control Program Directors (CRCPD), a national organization consisting of the directors of regulatory agencies responsible for radiation control in the various states, has drafted model state regulations for the control of NORM. These model regulations are currently in their seventh draft. The intent of the CRCPD in developing this model is to provide states with guidelines for developing regulations and licensing



requirements pertinent to their specific NORM problems. Qualifications and procedures for issuance of both general and specific radioactive material licenses and conditions for regulatory exemption are provided. Standards for worker protection and release limits for radioactive material effluent are also described.

Recently, both Louisiana and Texas have applied the CRCPD guidelines to provide a basis for the development of proposed NORM regulations. Both states are continuing to work in conjunction with the oil industry through the American Petroleum Institute (API) and their respective state oil and gas organizations to develop workable and practical regulations based on the groundwork established in the CRCPD guidelines. A number of oil-producing states are currently considering issuing regulations to govern NORM, many of which will likely follow the guidelines established by the CRCPD.

In Louisiana, the Department of Environmental Quality (LDEQ) initially implemented a rule for NORM in April 1991, a revision to which was issued in June 1992. The original focus of the Louisiana rule was on the release of equipment and land which had been involved in oil and gas operations, and the release of land which had been used for the cleaning of pipe and equipment. The original rule provides a general exposure rate limit of  $25 \mu\text{R/hr}$  above background, and a soil contamination limit of 30 pCi/g including background averaged over any area of 100  $\text{m}^2$ . Background soil radium concentrations are typically around 1 pCi/g, and background exposure rates are approximately 10-20  $\mu\text{R/hr}$ . Originally, LDEQ proposed an exposure limit of 50  $\mu\text{R/hr}$  including background, but this was revised to 25  $\mu\text{R/hr}$  excluding background prior to issuance of the rule in 1991. The most recent version of the Louisiana rule provides exemptions from the NORM regulation where the following conditions are met:

1. Concentrations of  $^{226}\text{Ra}$  or  $^{228}\text{Ra}$  within the contaminated material are less than 5 pCi/g above background;
2. Concentrations of "technologically enhanced"  $^{226}\text{Ra}$  or  $^{228}\text{Ra}$  do not exceed 30 pCi/g averaged over any 100  $\text{m}^2$  surface (provided that radon emanation from the surface does not exceed 20 pCi/ $\text{m}^2\text{s}$ ); and
3. Concentrations of any other NORM radionuclides do not exceed 150 pCi/g at any time.

Equipment contaminated with NORM is also now exempt from the Louisiana requirements if the maximum radiation exposure level of the equipment does not exceed 25  $\mu\text{rem/hr}$  above background at any accessible point.

The Louisiana rule also calls for strict adherence to the "ALARA" ("as low as reasonably achievable") concept of limiting exposures to ionizing radiation wherever practicable. Specific requirements of the rule call for pipe cleaning sites to be surveyed by the Louisiana Nuclear Energy Division before being authorized for release to the

general public. Similarly, oil and gas fields must be surveyed for radioactivity if they contained a disposal pit.

In Texas, the third draft of the Texas Regulations for Control of Radiation (TRCR Part 46), "Licensing of Naturally Occurring Radioactive Materials (NORM)" was issued by the Texas Department of Health in August 1991. The proposed Texas regulations also draw heavily from the CRCPD guidelines in that, if adopted, they will establish radiation standards for the "possession, use, transfer, transport, storage, and/or disposal of NORM and the recycling of NORM-contaminated materials". Licenses issued in Texas will be of two types, general and specific, as proposed by the CRCPD guidelines. General licensees in Texas must establish and adhere to worker protection standards similar to those found in Nuclear Regulatory Commission standards for radiation protection (10 CFR Parts 19 and 20), which include exposure limits and notification provisions.

According to the draft Texas regulations, equipment and soils must be within the specified contamination limits before being released for unrestricted use. The proposed Texas requirements recognize that the 5 pCi/g concentration limit established by the federal government for uranium mill tailings may be appropriate for soils contaminated with NORM, but is not necessarily applicable to NORM in solid scales because of the differing physical properties. The rationale is related to the expected lower radon emanation rates from the solid scale material as compared with mill tailings. Compared with the Louisiana rule, the Texas draft regulations provide similar (although somewhat less stringent) concentration limits for the release of areas for unrestricted use. These include a radon emission limit of  $20 \text{ pCi m}^{-2} \text{ s}^{-1}$  and  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  concentration limits of 30 pCi/g averaged over a maximum depth of 15 cm of soil below the surface.

Specific licenses under the Texas regulations would be required by manufacturers and distributors of products containing NORM, and for persons decontaminating equipment and facilities contaminated with NORM. Such activities would require a special license because they would involve greater potential for radiation exposure to workers and members of the general public. It is anticipated that pipe cleaning facilities could be required to apply for a specific license.

A noteworthy exemption is recognized in the draft Texas regulations regarding oil and gas production activities. This exemption states that "produced waters from crude oil and natural gas production are exempt from the requirements of these rules if the produced waters are re-injected in a well approved by the Railroad Commission of Texas as a Class II Injection and Disposal Well". More than 90 percent of the produced water in Texas is currently injected at permitted facilities, of which there are an estimated 5,800 within the state of Texas (Bohlinger, 1990).

On the national level, EPA is in the process of developing effluent limitation guidelines for the oil and gas point source categories pursuant to the Clean Water Act (CWA). As the result of a settlement agreement from a suit filed against EPA by the Natural Resources Defense Council, EPA must promulgate such rules. Effluent limitations for the offshore subcategory were to be promulgated by January, 1993, and those for the coastal subcategory are to be proposed by January 1995. Limitations for the onshore subcategory were promulgated in 1979 and require zero discharge of effluent in waste streams.

## **1.2 Information Needs**

In order to develop regulations that are both workable and practicable, information is needed on the range of activity concentrations (activity per unit mass of material) of the various radionuclides present within oil and gas production and delivery equipment and wastes. This information must be provided to assess the potential risks associated with NORM contamination before decisions can be made regarding how materials containing NORM should be handled, stored, and disposed. Radon emanation fraction from the different types of contaminated materials is also an important factor in the determination of the potential impact to health and the environment from radium-bearing materials.

The purpose of this study is to initiate a nationwide characterization of important NORM radionuclides in oil and gas production and delivery systems and wastes, focusing on known or suspected "problem areas". These geographic areas of concern were tentatively identified during a previous national survey based on external gamma radiation measurements (Otto, 1989). The goal of the present program is therefore to provide a reasonable characterization of the NORM contaminated waste produced by oil and gas industry production facilities within the United States. Sampling will be focused on areas where elevated activities are known (or suspected) to exist, based primarily on the earlier study by Otto. Where possible, however, attempts will be made to quantify reasonable upper bounds for NORM concentrations in various types of materials relating to the petroleum industry by sampling in areas the Otto study identified as being unlikely to have elevated NORM levels. These data will help to supply the information necessary to determine risk to workers, the general public, and the environment. Due to the nationwide distribution of the types of facilities examined during this program, budget constraints will limit thoroughness with which the characterization is performed. This may somewhat restrict the ability to extrapolate information gained through this program to all oil and gas producing areas in the country.

In general, data is needed on concentrations of radioactive materials in oil and gas industry equipment and wastes, and the potential level of hazard resulting from these materials to workers as well as to the general public. Data collected should therefore provide input needed by regulatory bodies to make reasonable decisions regarding

acceptable radionuclide concentrations in NORM material. Ideally, the data collected should provide the information necessary to answer questions relating to three areas of interest, as summarized below:

**1. Radionuclide Concentrations:**

- ! What are the typical ranges of radium concentrations that can accumulate in materials such as pipe scale, sludges, sands, and soil.

**2. Radon Emission and Emanation Fraction:**

- ! What are the typical ranges of radon exhalation from pipes in storage yards that contain radium bearing scale?
- ! What is a reasonable range of values for the radon emanation fraction from NORM waste materials, pipe scales and production facility sludges?

**3. Environmental and Human Health:**

- ! At what types of facilities and from what types of processes might workers be at risk from NORM radionuclides?
- ! From what types of facilities and from what types of processes might members of the general public be at risk from NORM radionuclides?
- ! From what types of facilities and from what types of processes might the environment be at risk from NORM radionuclides?

It is intended that information gained during this NORM characterization program will help answer these and other questions relating to NORM in the oil and gas industry. Data collected during the program will be provided to state and national regulatory bodies for their use in the development of reasonable and workable regulations on the distribution of NORM radionuclides in equipment and wastes from the oil and gas industry.

### **1.3 Purpose of the Sampling and Analysis Plan**

This Sampling and Analysis (S&A) Plan has been developed for the NORM Characterization Program, and describes the information to be gained through the program, how the required information is to be collected, and the anticipated form and content of the final data. The S&A Plan provides detailed procedures describing the work to be performed, how and why the work will be performed, and who will be responsible for conducting the various aspects of the work. The S&A Plan has been prepared with input from all parties involved with the program. Where appropriate, portions of the procedures described in the S&A Plan will be field tested by personnel of the Idaho National Engineering Laboratory (INEL) and the Grand Junction Project Office (GJPO), as well as representatives of the cosponsor organizations prior to their use in the field.

The importance of a Sampling and Analysis Plan is described in an EPA document "Test Methods for Evaluating Solid Waste: Volume II: Field Manual Physical/Chemical Methods" (USEPA, 1986). The excerpt below is taken from this document:

"The initial -- and perhaps most critical -- element in a program designed to evaluate the physical and chemical properties of a solid waste is the plan for sampling the waste. It is understandable that analytical studies, with their sophisticated instrumentation and high cost, are often perceived as the dominant element in a waste characterization program. Yet, despite that sophistication and high cost, analytical data generated by a scientifically defective sampling plan have limited utility, particularly in the case of regulatory proceedings.

The sampling and analysis plan is usually a written document that describes the objectives and details the individual tasks of a sampling effort and how they will be performed. The more detailed the sampling plan, the less the opportunity for oversight or misunderstanding during sampling, analysis, and data treatment.

To ensure that the sampling plan is designed properly, it is wise to have all aspects of the effort represented. Those designing the sampling plan should include the following personnel:

1. An end-user of the data, who will be using the data to attain program objectives and thus would be best prepared to ensure that the data objectives are understood and incorporated into the sampling plan.
2. An experienced member of the field team who will actually collect the samples, who can offer hands-on insight into potential problems and solutions, and who, having acquired a comprehensive understanding of the entire sampling effort during the design phase, will be better prepared to implement the sampling plan.
3. An analytical (scientist), because the analytical requirements for sampling, preservation, and holding times will be factors around which the sampling plan will be written. A sampling effort cannot succeed if an improperly collected or preserved sample or an inadequate volume is submitted to the laboratory for...testing. The appropriate analytical (scientist) should be consulted on these matters.
4. An engineer should be involved if a complex manufacturing process is being sampled. Representation of the appropriate engineering discipline will allow for the optimization of sampling locations and safety during sampling and should ensure that all waste-stream variations are accounted for.

5. A statistician, who will review the sampling approach and verify that the resulting data will be suitable for any required statistical calculations or decisions.
6. A quality assurance representative, who will review the applicability of standard operation procedures and determine the number of blanks, duplicates, spike samples, and other steps required to document the accuracy and precision of the resulting data base."

Input into the generation of this S&A Plan has been provided by the program sponsors (U.S. Department of Energy, American Petroleum Institute, and Gas Research Institute), the field personnel responsible for collecting the samples, quality assurance/quality control experts, data management experts, affected parties (e.g. landlords, neighbors), and regulatory bodies.

## **1.4 Data Quality Objectives**

In order to provide the information needed to develop reasonable regulations, specific Data Quality Objectives (DQOs) must be developed. DQOs are quantitative and qualitative statements developed by the users of the data that specify the *quality* of data required during a specific data collection and analysis activity. DQOs impact statistical sampling design, sampling techniques analytical procedures, documentation procedures, etc. The establishment of technical monitoring objectives will lead to a better definition of DQOs of the monitoring plan. These are typically specified in terms of six characteristics: precision, accuracy, representativeness, completeness, comparability, and detection limit. These characteristics are discussed below. For further information on DQOs see: "Data Quality Objectives for Remedial Response Activities, Development Process" (USEPA, 1987).

### **1.4.1 Precision**

Precision measures the reproducibility of measurements under a given set of conditions, and may be applied to activities in the field as well as to analytical procedures. Specifically, precision represents a quantitative measure of the variability of a group of measurements in comparison to their mean value. The overall precision of measurement data is a mixture of sampling and analytical factors. Analytical precision is much easier to control and quantify than sampling precision. There are more historical data related to individual method performance and the "universe" is limited to the samples received in the laboratory. In contrast, sampling precision is unique to each site. Precision is usually stated in terms of standard deviation, but other measurements such as the coefficient of variation (relative standard deviation), range (maximum value minus minimum value) and relative range are common.

**1.4.1.1 Sampling Precision** -- Sampling precision may be determined by collecting and analyzing co-located or field replicate samples and then creating and analyzing laboratory replicates from one or more of the field samples. The analytical results from the co-located or field replicate samples provide data on overall measurement precision; analysis results from the laboratory replicates provide data on analytical precision. Subtracting the analytical precision from the measurement precision defines the sampling precision.

In terms of the NORM Characterization Program, precision objectives for the analysis of samples in the laboratory may be established by Chem-Nuclear Geotech at the Grand Junction Project Office (GJPO) who will conduct the analyses. Precision objectives for the field activities cannot be defined, however, because much of the basis of the Characterization program is provided by the results of the earlier Otto study. Definition of precision objectives would therefore require a statistical breakdown of the data collected during the Otto study to determine the proper number of samples. Such a detailed statistical breakdown of the Otto study is not possible.

In an effort to minimize the impact our inability to establish sampling precision objectives to the extent practicable, the NORM Characterization Program will apply good sampling procedures that apply a composite sampling approach wherever practicable. This will allow us to draw the best sample possible under the constraints described above. Field replicates will be used where practicable.

**1.4.1.2 Analytical Precision** -- Analytical precision is defined as the degree of agreement between individual measurements made in an identical manner using the same test procedure. Precision is determined in the analytical chemistry laboratory by analysis of laboratory duplicates. Table 1.1 summarizes the target precision values expected for the analyses of samples for this project. These values are expressed as the relative percent difference (RPD), which is calculated as follows:

$$\%RPD = \frac{\text{difference between results}}{\text{average of results}} \times 100$$

## **1.4.2 Accuracy**

Accuracy provides a measure of the bias inherent to the measurement system, which is often difficult to measure for the entire data collection activity. Sources of error include the sampling process, field contamination, sample handling, the sample matrix, sample preparation and analytical techniques. Sampling accuracy may be

determined by evaluating the results of field/trip blanks, while analytical accuracy may be estimated through the use of known and unknown QA/QC samples and matrix spikes.

The largest source of bias in the NORM Characterization Program will be that associated with sampling design, as error from the analytical procedures applied to this program is expected to be relatively small. This is again due largely to the dependence on information gathered in the Otto study for the determination of sampling locations. For this reason, the accuracy objective for sample collection will necessarily be relatively nonrestrictive.

Analytical accuracy is the degree of agreement between a measurement and the accepted or true value. Accuracy is determined in the analytical chemistry laboratory by analysis of reference standards and spiked samples. Table 1.1 summarizes the target accuracy values expected for the analyses of samples for this project. These values are expressed in % Recovery which is calculated as follows for spiked samples and for reference standards:

$$\%Recovery = \frac{(Spiked\ sample\ results) - (Unspiked\ sample\ result)}{Spikeamount} \times 100$$

Accuracy calculation for reference standard is:

$$\%Recovery = \frac{(Expectedvalue) - (Measuredvalue)}{Acceptedvalue} \times 100$$

**Table 1.1 Analytical chemistry laboratory data quality objectives.**

Parameter	Analytical Method	Precision %RPD	Accuracy (% Recovery)
Radium-226	Gamma Spectroscopy	< 20%	< 10 %
Radium-228	Gamma Spectroscopy	< 20%	< 10 %
Radium-226	Alpha Spectroscopy	< 20%	< 10 %
Lead-210	Liquid Scintillation	< 20%	< 10 %

The precision and accuracy numbers reflected in Table 1.1 are the acceptance criteria values that the Chem-Nuclear Geotech Analytical Chemistry Laboratory will use to analyze samples for this project (see Analytical Laboratory Administrative Plan, procedure QP-7 in Appendix A). These values represent the overall uncertainty in making the measurement, and include the counting errors, errors in standards, sample homogeneity, analytical errors,



etc. associated with the measurement.

**1.4.2.1 Accuracy and Precision of Pipe Volume Measurements** – Measurement of the pipe volume involves evacuating the pipe with a vacuum pump, and then returning it to atmospheric pressure with ambient air. The measured volume of the pipe is the amount of air required to fill the pipe to atmospheric pressure. This volume will be determined with a dry gas meter.

In the absence of high vapor pressure compounds contaminating the interior of the pipe, the precision of these measurements should be within 2%, with an estimated overall uncertainty of 4%. In the event that the pipes are contaminated with high vapor pressure compounds or gas sorbing compounds the accuracy of the measurements may suffer significantly. Moreover the extent to which the procedure fails will be dependant upon the characteristics of the particular compound(s) involved. A value of 25% will be used as an upper limit of the overall uncertainty that may be obtainable from a worst case situation.

**1.4.2.2 Accuracy and Precision of Radon Exhalation from Pipe** – The Whole Pipe Radon Release Measurements are a form of radon flux measurement, and involves collection of the radon exhaling from the end of the pipe on activated charcoal and subsequently assaying the charcoal for radon by a spectral gamma analysis.

The analytical precision of the flux measurement is driven by the analysis of the charcoal sample for gamma activity. The precision obtainable for a given counting time is dependant upon the delay between sample collection and gamma analysis, and the amount of radon collected on the charcoal. The estimated analytical precision for a sample with a measured value of  $0.01 \text{ pCi}_A\text{sec}^{-1}$  is approximately 7% at the 99% confidence level. This is based on a counting time of 40 minutes for the sample and no more than a seven hour delay between sample collection and gamma analysis. For these counting conditions the estimated detection limit is  $6 \times 10^4 \text{ pCi}_A\text{sec}^{-1}$ . Because the analytical precision is driven primarily by counting statistics, the precision will improve substantially as the activity of the sample increases. The estimated precision for a sample measuring  $0.5 \text{ pCi}_A\text{sec}^{-1}$  is approximately 1%.

The overall uncertainty for the gamma analysis of an individual sample is determined by the precision with which a sample can be counted and the certainty of the calibration of the counting system with the selected counting geometry. The counting geometry used for this measurement will be a 4 inch diameter by 2 inch high hermetically sealed can containing approximately 180 grams of activated charcoal.

The gamma counting system calibration is based on repeated analysis of two different radium-on-activated charcoal standards. These standards provide the same counting geometry as the samples and were prepared from

$^{226}\text{Ra}$  solutions with certified activities traceable to the National Institute of Standards and Technology (NIST). Gamma analysis of the two standards shows a disagreement between the two standards of approximately 2.5%, which is well within the certified uncertainties of the two standards (3.7% and 1.3% at the 99% confidence level). Combining the uncertainty associated with calibration of the gamma system with the remaining relevant uncertainties results in an estimate of the total analytical uncertainty, at the 99% confidence level, which ranges from approximately 11% at a measured value of 0.01 pCi sec<sup>-1</sup> to approximately 5% at a value of 0.5 pCi sec<sup>-1</sup>.

**1.4.2.3 Radon Emanation Coefficient Measurements** -- The radon emanation fraction is that fraction of the  $^{222}\text{Rn}$  atoms produced by the decay of  $^{226}\text{Ra}$  that recoil out of the mineral grain or other material which contains the parent radium atoms and behave as free gaseous radon atoms. The technique used to measure the emanation fraction is based on the difference of a gamma analysis of the sample with all of the free gaseous radon removed and an analysis in which the radon is in secular equilibrium with the radium parent. The analytical precision to which the emanation fraction of a given sample may be determined is dependent upon the emanation fraction. In the case where the emanation fraction is large, the uncertainty of the difference between the two measurements is small relative to the value of the difference. On the other hand, as the emanation fraction approaches zero the uncertainty of the difference between the two measurement results approaches infinity.

It is anticipated that the emanation fraction of pipe scale samples may be extremely small and therefore result in very large uncertainties associated with the measured values of the emanation fraction. The analytical precision is largely influenced by counting statistics. Wherever reasonable the counting conditions will be controlled to produce a minimum of 1 million net counts for a given gamma analysis, for which the uncertainty due to counting statistics is approximately 0.1%. If the emanation fraction of the sample is 1% then the uncertainty of the measured emanation fraction is approximately 40% at the 99% confidence level. If the emanation fraction is 5% then the uncertainty is reduced to approximately 8% at the 99% confidence level.

An estimate of the analytical uncertainty will be provided for each of the analytical results that is based on the actual counting statistics of the analysis. In addition duplicate analyses will be performed on 10% of the samples. The results of these duplicate analyses will provide an estimate of the overall analytical precision, which includes the random uncertainties attributed to counting statistics as well as any other relevant sources of random variation. Because the emanation fraction measurement is not dependent upon calibration of counting efficiency relative to a reference standard the systematic uncertainties are likely to be very small.

#### **1.4.2.4 Accuracy and Precision of Alpha-Track Measurements on Gas Pipe Interiors**

-- The alpha-track measurements will consist of placing a standard size piece of alpha-track registration film (Kodak

LR-115, Type II) onto the surface to be measured. The film will be packaged in a holder which provides a cover of sufficient thickness to degrade the 5.3 MeV alpha from  $^{210}\text{Po}$  to an energy below the 4 MeV detection threshold energy of the LR-115. The alpha particle ejected from the measurement surface which intersects the plane of the LR-115 film will produce a latent track. These latent tracks will be made visible by processing the film in a caustic developing bath. A specified area of the film will be examined under a microscope and the number of tracks counted. The resulting track density is compared to a standard exposure to determine an estimate of the alpha activity.

The standard exposure consists of exposing the LR-115, in the same film holder which will be used for the pipe samples, to the alpha field from an electroplated  $^{241}\text{Am}$  source of known activity for a fixed length of time. Because the alpha particle emitted from the  $^{241}\text{Am}$  is more energetic (5.48 MeV) than the 5.30 MeV alpha from  $^{210}\text{Po}$ , the nature of track formation in the LR-115 does not allow the standard exposure to be used as a true calibration. Alpha particles of different energy will produce a slightly different number of visible tracks per disintegration in the alpha-track film due to geometric considerations. Moreover the  $^{210}\text{Po}$  atoms which remain attached to the pipe interior following the removal of surface contamination are likely to be distributed at various depths within the metal. This distribution of depth will serve to broaden the energy distribution of alpha particles which intersect the LR-115 film plane, resulting in some alpha particles not producing visible tracks.

The analytical precision obtainable from a source which has a uniform distribution of alpha activity is driven primarily by poisson counting statistics. Thus the greater number of tracks counted, the better the precision. The analytical precision available by manual track counting from a uniformly distributed alpha source is typically within 5%. However as the alpha source deviates from a uniform distribution the precision obtainable from a randomly located sample becomes poorer. It is anticipated that the alpha source on the interior surfaces of the gas pipe samples will be reasonably uniform, and a value of 15% will be used as the estimated analytical precision available from this technique. As mentioned above there will be no true calibration of the technique, only a comparison to a standard source. Thus the best estimate of the accuracy obtainable from this technique is 50%.

### **1.4.3 Representativeness**

Representativeness describes the degree to which sample data accurately and precisely represent some characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are selected properly and a sufficient number of samples are collected. The sample strategy and statistical basis for each sample

type is described in Section 3 of this S&A Plan.

Representativeness is addressed by describing sampling techniques and the rationale used to select sampling locations. Sampling locations can be biased (based on existing data, instrument surveys, observation, etc.) or unbiased (completely random or stratified random approaches). Either way, the rationale used to determine sampling locations must be explicitly explained. If a sampling grid is being utilized, it should be shown on a map of the site. The type of sample, such as a grab or composite sample, as well as the relevant SOP for sample collection should be specified. SOPs for sample collection, analysis, identification and tracking, data management and waste disposal are provided in Section 4. Additional SOPs are contained in Appendix A to the S&A Plan.

Representativeness can be assessed by the use of co-located samples. By definition, co-located samples are collected so that they are equally representative of a given point in space and time. In this way, they provide information relative to both precision and representativeness.

Because the sampling locations for the NORM Characterization Program will be based primarily on data provided by the earlier study by Otto, error due to representativeness of the sampling is expected to be relatively large. Additional problems involve the accessibility of pipe cleaning and storage yards, the accessibility for sampling of pipes within the pipe cleaning and storage yards, and the budgetary constraints of the program. Some of these problems can be minimized through the use of composite sampling procedures wherever possible, and by focusing on the bounding case where composite samples are not practical.

For these reasons, the accuracy objective for sample collection must necessarily be both arbitrary and nonrestrictive. Replicate samples will be used where possible (e.g. soil samples), but will not be used for the sampling of waste materials in drums, etc.

#### **1.4.4 Completeness**

Completeness is defined as the percentage of measurements made which are judged to be valid measurements. The completeness goal is essentially the same for all data uses: that a sufficient amount of valid data be generated. It is important that critical samples are identified and plans made to achieve valid data from them. A nominal value of 90% will be applied to the data collected for this program.

#### **1.4.5 Comparability**

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through the use of standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units. Only when precision and accuracy are known can data sets be compared with confidence.

Procedures described in the Louisiana regulations will be followed for conducting gamma surveys, and the resulting data will therefore be comparable to data collected in compliance with the Louisiana regulations. The approach used in the Otto study will also be applied where practicable during the gamma surveys. Data generated during this program should therefore yield data comparable to that generated during the Otto study. Sludge samples from drummed wastes collected using a Coliwasa sampler will use standard procedures for this type of sampling. Data collected using the Coliwasa sampler will therefore be comparable to data generated in earlier sampling programs. No standard procedures exist for the collection of pipe scale samples, nor for the collection of sludge samples for the purposes of determining the radon emanation fraction. Therefore, no comparability standards are applicable to these portions of the NORM Characterization Program.

#### **1.4.6 Detection Limit**

The DQO for detection limits is dependant on the analytical technique applied. The laboratory technique applied to radium samples will be gamma spectroscopy using a High Purity Germanium (HpG) detector. Minimum detectable concentration (MDC) limits for  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  are 1.0 pCi/g (Section GS-7.1 of the Operational Procedures for Gamma-Ray Spectroscopy, Appendix A).

The detection limit for whole pipe radon release measurements is estimated to be  $6 \times 10^4$  pCi  $\text{sec}^{-1}$ . This estimate is based on a delay time of 7 hours between the end of sample collection and gamma analysis and a counting time of 40 minutes.

## 2. Regulatory Requirements

All pertinent state and federal regulations for the shipping and disposal of the samples collected for analysis during the NORM Characterization Program will be complied with. Shipping containers will be surveyed with a calibrated gamma exposure rate meter prior to shipping to ensure compliance with U.S. Department of Transportation regulations. Once analyzed, the samples originating from the same site will be packaged and returned to the collection site. If necessary, the sample shipment will be diluted to ensure that the total activity concentration of the shipments is less than the 2 nCi/g concentration limit imposed by the U.S. Department of Transportation (Code of Federal Regulations 49 CFR 173.403). Because all samples will be returned to the site at which they were collected following analysis, quantities of waste generated during this program will be minimal.

The Landfill Disposal Restrictions (LDR) now in place should not impact the analysis of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  since sample excess will be returned to the site where it originated, and the analytical technique employed does not generate mixed waste. A small amount of mixed waste is generated during the  $^{210}\text{Pb}$  analysis. The procedure generates a corrosive/radioactive liquid scintillation cocktail. At present, Chem-Nuclear Geotech is working toward obtaining a conditionally exempt small quantity generator status. The laboratory is also exploring other analytical techniques for the determination of  $^{210}\text{Pb}$  that will not create a mixed waste.



### 3. Sampling Strategy and Statistical Basis

The purpose of this section of the Sampling and Analysis Plan is to describe the general strategy behind the sampling design applied to the NORM Characterization Program. Included are descriptions of:

1. The types of samples to be collected and measurements to be performed during this program;
2. The statistical basis, where appropriate, applied to the determination of the sample number and location.
3. The rationale for collecting each type of sample or measurement, including descriptions of how the data resulting from each type of sample or measurement will help provide the information necessary to answer the questions posed in Section 1.2 above;
4. How the general locations to be used for these samples and measurements were determined;
5. How the specific locations for sampling will be chosen in the field; and
6. The number of samples or measurements taken at each facility, and the quantity of each sample collected.

A primary factor in the development of the sampling strategy to be applied to the NORM Characterization Program is the level of funding available for sample analysis. Based on a scenario discussed earlier between INEL and API, a limit to the total number of samples of each type has been instituted. Based on this, the approximate number of each type of sample to be analyzed is provided in Table 3-1 below. A total of 10% field QA/QC samples has been assumed throughout the sampling program to verify the quality of the results observed. These samples have been included in the sample numbers cited in Table 3.1. The specific breakdown of the various types and quantities of QA/QC samples, as well as other pertinent aspects of the QA/QC program are discussed in the accompanying QA/QC Plan (Appendix B).

It should be pointed out that because the number of each type of sample and measurement conducted during this program is driven primarily by budgetary constraints rather than by statistical considerations, the conclusions that may be made from the results of the program will clearly be limited. Although the information gained will be useful in terms of characterizing NORM activities in oil and gas equipment and wastes, valid extrapolation of results either spatially or temporally will be somewhat limited.



**Table 3-1. Breakdown by sample type of the samples to be collected and analyzed during the NORM Characterization Program**

No. of Samples <sup>1</sup>	Sample Type
599	Radium concentration samples
5	Radionuclide solubility measurements
20	Whole pipe radon release measurements
30	Radon emanation fraction measurements
10	Gas pipes samples for <sup>210</sup> Pb
10	Alpha track measurements on gas pipe interiors

<sup>1</sup> including QA/QC Samples

Two additional points should be made concerning the basic strategy behind the NORM Characterization Program. First, as agreed earlier, no samples will be analyzed for uranium or thorium concentration. This decision was based on the assumption that with the likely exception of produced sands, these parent radionuclides should remain essentially immobile in comparison to their radium daughter products. Uranium and thorium are therefore expected to remain within the geologic formation when the more-soluble radium is removed. Second, as a means of optimizing the information that will be generated from this program, we will be relying heavily on the performance of gamma surveys at each field location sampled. Although somewhat time consuming to conduct in the field, the extensive reliance on gamma surveys will provide several important benefits to the program, including the following:

1. Gamma surveys will help to optimize the sampling program by identifying sample locations where elevated radioactivity levels are present. In this type of sampling program, a purely random selection of sampling locations would likely result in the analysis of a large number of samples showing zero or minimal levels of contamination. Such sampling would not provide adequate answers to many of the questions that this program is designed to help answer. By skewing the sampling population toward relatively "hot" locations identified during an initial survey of the facility, not only will these "hot" locations be identified, but the additional information generated during the survey will facilitate extrapolation of the results to areas that were surveyed but from which samples were not collected.
2. The costs of conducting gamma surveys are much less than the costs of analyzing samples. Any additional information that can be extrapolated from gamma survey in conjunction with the analytical data will therefore result in a more cost-effective program.
3. By conducting a survey and recording the results, it will be possible to revisit areas to collect additional or

follow-up samples at some later date, provided that either additional funds are made available or additional information becomes necessary or desirable.

Despite the constraints placed on the number of samples and measurements made, a valid statistical design has been applied to the NORM Characterization Program wherever possible. Where this is not practical, the most reasonable approach has been used, and the rationale applied is provided. Several types of samples to be collected as part of this program do not lend themselves to the use of a statistical design. For example, the design for several types of samples is based on the earlier survey by Otto, which did not employ a valid statistical design. In other cases, notably the collection and analysis of  $^{210}\text{Pb}$  samples from gas delivery systems, the number of samples is restricted to a very few. This reflects the specific purpose of these samples, which is to determine the feasibility of the sampling procedure or to observe what may be considered a bounding case, rather than to conduct a nationwide characterization of this parameter.

For the various portions of this program where a valid statistical design can be applied, a variety of potential statistical sampling designs exist. These include stratified, systematic, and random sampling designs, using either grab or composite sampling to meet sampling objectives. To be considered a valid statistical design, the various sources of environmental variability including space, time, sample collection, sample handling, sample processing including subsampling, and measurement must all be taken into consideration. The parameters of interest in any monitoring program will include several of these sources of error. The development of data quality objectives requires an analysis of these sources of error, an estimate of their magnitude and a review of methods to reduce the overall variability in a cost effective manner. For further information see "Statistical Methods for Environmental Pollution Monitoring" (Gilbert, 1987).

General descriptions of the strategies applied to the collection of the various types of samples are provided below.

### **3.1 Radium Concentration Samples**

The purpose of collecting and analyzing samples for radium concentration is to evaluate the extent and distribution of NORM contamination within petroleum industry equipment and waste. This information is needed before an assessment of the risk associated with these materials may be performed. This portion of the Program will involve the analysis of several different types of samples for concentrations of both  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ , and will include samples of scale from pipe, production equipment wastes, and soil. Analytical results from these radium concentration samples will provide the core of the data to be collected in the program.

A total of approximately 600 samples (including field QA/QC samples) has been allocated for this portion of the program. As this is a national program, this places a severe restriction in terms of the statistical approach to be employed. Furthermore, the sampling strategy will be largely dependant on the information provided by the Otto survey, which did not use strict statistical procedures. Despite these deficiencies, this program has been designed to help answer many of the basic questions that remain regarding the presence of NORM contamination in the petroleum industry.

An approximate breakdown of the numbers and percentage of the different types of radium concentration samples is provided in Table 3-2 below. The total number of samples collected will depend on budget constraints and sample accessibility. Ten percent field replicate QA/QC samples has been assumed, as discussed in the accompanied QA/QC Plan (Appendix B). Brief descriptions of how each different type of sample will be selected, and the rationale behind choosing these sample numbers is provided below. Detailed procedures for sample collection, analysis, and data management are provided in Section 4 (Standard Operating Procedures).

**Table 3-2.** Approximate number of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  samples to be taken for each sample type of radium concentration sample.

SAMPLES	QA/QC <sup>a</sup>	TOTAL	% OF TOTAL	SAMPLE TYPE
250	25	275	46.0	Pipe scale samples
10	2	12	2.0	Pipe yard soil samples
133	14	147	24.5	Production facility waste samples
16	2	18	3.0	Michigan Samples <sup>c</sup>
133 <sup>b</sup>	14	147	24.5	Production facility soil samples
599 = Total $^{226}\text{Ra}$ and $^{228}\text{Ra}$ concentration samples				

<sup>a</sup> Represents field replicates of 10 percent of the samples collected

<sup>b</sup> Represents one composite surface and one composite subsurface sample for each of 50% of the production facilities at which waste is sampled.

<sup>c</sup> Michigan samples represent 5% of the production facility waste and soil samples.

### 3.1.1 Pipe scale samples

Information on the concentration of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  in pipe scale is necessary to determine the degree of risk

to workers and to the public from these materials, and to evaluate the need for special disposal requirements. The purpose of collecting pipe scale samples is therefore to determine the concentration of radium isotopes that accumulate within these materials.

Although pipe cleaning and storage yards were not surveyed as part of the earlier national survey conducted by Otto, information from the Otto survey may still be applied to the selection of pipe cleaning and storage yards for the collection of pipe scale samples. This requires the application of certain assumptions regarding the geographic areas from which each facility receives pipe. Primary among these is that NORM concentrations in the pipe scale from a given pipe yard are correlated with the frequency with which production facilities located within the same general geographic area as the pipe yard indicated elevated gamma readings during the earlier study by Otto. It has therefore been assumed that used pipe is typically shipped to the pipe cleaning yards that are relatively nearby.

Pipe scale samples will be collected at five different pipe yards identified by API. Each of the yards selected must employ "rattling" or some other process that routinely removes scale from the pipe, and this process will be used to collect sample material. The scale sample material will be therefore be removed from the pipe at the pipe yard, and shipped to GJPO for preparation and analysis.

At a minimum, one of the pipe yards sampled will be representative of the Gulf Coast region, and will be selected based on its anticipated receipt of pipe from counties in elevated NORM contamination levels were observed frequently during the Otto survey. Ideally, this yard will represent the site with the greatest potential for elevated contamination to which API has access. Consideration will also be given to the length of operation of the facility, which will be of importance in the selection of soil sampling locations to be conducted at the same locations. This initial site will therefore represent the upper limit in terms of anticipated NORM contamination levels. Pipe from this first yard selected will be shipped to an oil production facility near Grand Junction for whole pipe radon release measurements prior to sampling the pipe scale.

Four additional pipe yards will be selected by API, and will be located within the geographic area covered by the Otto survey. This sampling scheme will provide a range of pipe cleaning and storage facility locations from which to compare NORM contamination. It is recognized that access may not be made available to all pipe cleaning and storage yards that will be considered for this program. In choosing the yards to be sampled, therefore, API will generate and prioritize a list of potential pipe scale sampling locations. If the preferred facility is not made available, the next facility on the list will be substituted, and so forth, until all sites to be sampled are identified.

At each of the five pipe cleaning and storage yards selected, scale from a total of 50 pipes will be sampled.

Field replicates will also be collected from five of the 50 pipes sampled at each pipe yard, making the total of 55 pipe scale samples to be collected at each pipe yard. A gamma survey will first be conducted of all readily accessible pipe present in the yard prior to the collection of the samples, with the results for each pipe recorded. From the results of the survey, the 10 pipes with the highest gamma readings will be sampled. This will provide an upper limit for radium concentrations in the pipe scale from these yards. The remaining 40 samples will be chosen randomly from the population of pipes surveyed that showed gamma readings in excess of 50  $\mu\text{R/hr}$  will provide a reasonable range of radium concentration values for the yard. The total number of pipes surveyed that exhibited gamma exposure rates of at least 50  $\mu\text{R/hr}$  will be noted.

### **3.1.2 Production Facility Waste Samples**

The purpose of sampling wastes from production activities is to determine the radium concentrations found in waste materials other than pipe scale that are generated from oil production facilities. This will allow for the evaluation of the extent to which elevated radium concentrations occur within these wastes. This information is necessary to determine the risk to workers from these waste materials, and to evaluate the need for special disposal requirements.

A database listing the locations by state and county at which Otto conducted measurements has been provided by API. General locations for collecting samples have been determined using these data and by calculating a weighted sample distribution. This represents the percent of elevated gamma measurements found during the Otto survey and distributed by county, giving equal weight to the different types of equipment in which elevated readings were observed (e.g. sumps, separators etc.), even though a different number of measurements were made on each type of equipment.

The criterion for the selection of the locations from which these samples will be collected has been based on the 50  $\mu\text{R/hr}$  (including background) surface gamma survey criterion imposed by the state of Louisiana as a general limit for exposure at the time the Otto survey was conducted. The sampling population of interest is therefore defined as all locations in which the Otto study found equipment that exhibited external gamma readings of greater than 50  $\mu\text{R/hr}$  in any of the following equipment types: flow lines, heater treaters, separators, sumps, and water lines. From Otto's gamma survey data, a total of 1573 measurements exceeded the 50  $\mu\text{R/hr}$  criterion. Weighting samples by the relative number of elevated readings in the different types of equipment using this criterion will allow for the evaluation of a large number of sites, while concentrating the sampling at sites from which elevated readings have been observed by Otto.

Much of the state of Michigan was not included in the Otto study, and concerns have been expressed recently regarding the potential for NORM contamination in that state. To correct this omission, approximately five percent of the production facility samples (soil and production waste) will be chosen in Michigan.

Table 3-3 provides a breakdown of samples to be taken in each county based on the Otto survey data and 133 production facility samples. Sixteen samples, plus two field replicates samples, have been allocated to the State of Michigan. Therefore eight Michigan facilities will be sampled and two soil samples (surface and subsurface) will be taken at each of four of these sites. Ideally, one sample will be taken per facility, resulting in a total of 133 facilities sampled. Access to 133 facilities is not assured however, so in certain circumstances, a central facility that receiving waste from several surrounding facilities may be visited and multiple samples will be taken.

A complete summary of all the measurements made by Otto on the five equipment types considered here is provided in Appendix D. It will be the responsibility of API to identify facilities and equipment for sampling in the counties listed in Appendix D that exceed the 50 pCi/hr limit.



**Table 3-3.** Summary of samples within counties with NORM contaminated equipment (> 50 pCiR/hr as measured in Otto report). Based on equal weighting of 5 equipment types: flow lines, heater-treaters, separators, sumps, and water lines.

State/County	Suggested Sample Number	State/County	Suggested Sample Number	State/County	Suggested Sample Number
<b>ALASKA</b>		<b>MISSISSIPPI</b>		<b>OFFSHORE</b>	
	1	JASPAR	1	GULF	16
OFFSHORE	2	JONES	3	<b>OS SUBTOTAL</b>	<b>16</b>
UNREPORTED	3	MARION	3		
<b>AK SUBTOTAL</b>		SMITH	1	<b>TEXAS</b>	
		WALTHALL	3	BROOKS	4
<b>ALABAMA</b>	3	WAYNE	1	CHAMBERS	1
ESCAMBIA	3	<b>MS SUBTOTAL</b>	<b>12</b>	FRANKLIN	1
<b>AL SUBTOTAL</b>				GRAY	2
		<b>MONTANA</b>		GRAYSON	1
<b>CALIFORNIA</b>	1	SHERIDAN	3	HIDALGO	3
KERN	1	<b>MT SUBTOTAL</b>	<b>3</b>	JIM WELLS	1
<b>CA SUBTOTAL</b>				KENEDY	4
		<b>NORTH</b>		KLEBERG	3
<b>ILLINOIS</b>	2	<b>DAKOTA</b>		MITCHELL	4
FAYETTE	5	DIVIDE	1	NUECES	3
UNREPORTED	7	<b>ND SUBTOTAL</b>	<b>1</b>	PECOS	1
<b>IL SUBTOTAL</b>				ROBERTS	1
		<b>NEW MEXICO</b>		THROCKMORTON	1
<b>KANSAS</b>	1	LEA	4	UPTON	1
STAFFORD	1	SAN JUAN	3	WALLER	1
<b>KS SUBTOTAL</b>		UNREPORTED	4	WARD	2
		<b>NM SUBTOTAL</b>	<b>11</b>	WILLACY	1
<b>LOUISIANA</b>	3			WOOD	2
ACADIA	1	<b>OKLAHOMA</b>		YOUNG	1
ASSUMPTION	3	CANADIAN	1	<b>TX SUBTOTAL</b>	<b>38</b>
IBERIA	3	CARTER	3		
LAFOURCHE	1	CLEVELAND	1	<b>WYOMING</b>	
PLAQUEMINES	2	CREEK	4	UNREPORTED	
ST. MARTIN	1	GARVIN	1	WY	1
TERREBONNE	3	KAY	1	<b>SUBTOTAL</b>	<b>1</b>
VERMILLION	17	KINGFISHER	3		
<b>LA SUBTOTAL</b>		SEMINOLE	2	<b>TOTAL</b>	<b>133</b>
		STEPHENS	3		
		<b>OK SUBTOTAL</b>	<b>19</b>		



Ideally, this portion of the study should involve the in situ collection of samples directly from the equipment that is in operation. Aside from the difficulty of sampling that would result from the need to dismantle production equipment to gain access to the contents, such sampling would require a disruption of normal operating activities. Such a disruption to site operations is not justified for this program. Where access may be obtained, in situ samples will be collected, otherwise, the remaining samples will be obtained from production facility waste stored in drums located on the facility. In addition, at facilities that store production wastes generated from production facilities in the surrounding area, multiple drums may be sampled. An initial gamma survey will be conducted within the facility to determine the exact location of sampling. This survey will concentrate on waste handling and disposal areas, but will also include equipment areas.

It is assumed sludges, oil and water emulsions, sediments, sands, and other materials that have been produced as wastes will be stored in drums on site, and samples will be collected from these drums. These wastes are generally solid or highly viscous since most of the liquid portion of the waste is drained off before storage in the drum. Some sites may contain drums from several facilities while others only have drums derived locally.

A modified Coliwasa sampler will be used to collect a composite sample of a cross section of the material within the drum. The collection tube of the Coliwasa sampler will be inserted into the top of the drum and driven to the bottom of the drum with minimal disturbance to the contents, such that the entire depth of the drum is sampled. This way, a representative sample of the contents of the drum will be provided. The "oily" mass of the sample will be measured and removed by an API member company before shipment to GJPO.

If all facilities selected for sampling are accessible to sample collection personnel, then 133 sites will be sampled. However, it is anticipated that not all sites selected will be accessible. In such cases, a centrally located waste storage facility will be selected which receives production facility waste from the general area surrounding the inaccessible facility. Such a central facility may provide more than one sample if more than one production facility in the region proves to be inaccessible.

At all accessible production facilities, all waste drums will be surveyed and the surface gamma exposure readings recorded. All drums showing surface gamma exposure readings in excess of 50  $\mu$ R/hr will then be identified and a waste sample will be collected from the waste drum having a gamma exposure reading closest to the mean reading for this population of drums. This will provide a representative value for waste drums at each site.

A second waste sample will be collected at each of a predetermined set of 10% of the production facilities sampled. This second sample will be collected from the waste barrel which yielded the highest surface gamma

reading. These additional analyses will provide data on the bounding case for the facility, indicating the highest radium concentration waste present.

Where centrally-located common waste collection areas are substituted for production facilities, a minimum of two samples will be collected. These samples will be collected from the barrels showing the mean and the high surface gamma exposure reading. If the common waste collection area is being substituted for more than two production facilities from the original list of sample sites, additional samples will be collected such that the number of drums sampled will equal the number of sampling facilities represented. In other words, if a regional waste collection facility replaces five different inaccessible production facilities, samples from a total of five different barrels should be collected. All barrels sampled will be among those identified in the initial gamma survey as having surface gamma readings in excess of 50  $\mu\text{R/hr}$ , and will include the barrel with the average gamma reading, the barrel with the highest reading, and three additional barrels randomly selected from among the remaining barrels above 50  $\mu\text{R/hr}$ .

The advantages of this sampling approach are that the operations of the site will not have to be disrupted to collect samples, and that the sample collected will represent a composite of the waste material accumulated from the facility sampled. For some drums, it may be too difficult to drive the Coliwasa sampler through the waste material. In this case, a sample will be taken from the top of the drum and it will be assumed that this sample is representative of the entire drum contents.

Because this approach is based on the Otto study, it does not represent a statistically valid sample design. The advantage to this approach is that it will allow for a quantification of the exposure measurements made during the Otto study, thereby allowing some extrapolation of the results to areas covered by the Otto survey but not sampled during this program. By including some samples from the "hottest" drums, information may be provided regarding the "worst case" situation. This represents a reasonable approach, given the budget limitations on number of samples to be analyzed. It is not possible to determine a required number of samples without more specific objectives, and a statistically based exploratory survey.

### **3.1.3 Soil samples**

Soil samples will be collected at each pipe cleaning yard used to collect scale samples and at many of the production facilities used to collect waste samples. Soil samples will be collected at the following production facilities:

1. All facilities designated among the 10% of the sites at which a second sample representing the "hottest" drum is collected;
2. All common waste storage facilities that are substituted for inaccessible production facilities; and
3. Half of the remaining production facilities sampled, selected randomly by API prior to initiating the sampling.

The production facilities sampled for soil will be chosen randomly by API from among the facilities sampled for waste. The soil sampling design will follow methods used elsewhere for characterizing soils contaminated with uranium mill tailings (e.g. Williams et al., 1989). At each facility sampled, collection personnel will first conduct a gamma survey with a properly calibrated exposure rate meter, and map out hot spots where gamma readings of greater than 50  $\mu$ R/hr are found. The 50  $\mu$ R/hr criterion was chosen because this represents the calculated exposure rate from a surface soil containing a concentration of 30 pCi/g of  $^{226}\text{Ra}$ . These calculations were performed with the Microshield code (Grove Engineering, 1992).

The initial survey should cover the grounds in general, but should be more heavily focused in the vicinity of heater-treaters, sumps, waste accumulation areas, waste water impoundments, and other locations identified as potential accumulation points for NORM. The purpose of the general survey is to identify "hot" spots that will provide the locations for the soil sampling, while determining the areal extent of NORM contamination throughout the facility.

Based on the results of the gamma survey, the sample collection personnel will select the single "hottest" area for collection of soil samples, even if no readings in excess of the 50  $\mu$ R/hr criterion are found. This must be a rectangular area of at least 100 m<sup>2</sup> (1100 ft<sup>2</sup>), with a minimum dimension of 5 m (16 ft). If additional hot spots are found, they will be identified on the map generated during the initial gamma survey of the site. No additional hot spots will be sampled. From each grid area, one surface and one subsurface composite sample of 9 soil cores will be collected. The 9 core locations will be spaced equidistant over the 100 m<sup>2</sup> (1100 ft<sup>2</sup>), so that the entire plot area is included. A 1 inch (2.5 cm) diameter or larger soil coring tool will be used to extract soil. The total soil volume collected in nine soil cores of this size be adequate to provide a field duplicate sample for QA/QC purposes, where required. The locations of each individual core will not be selected based on gamma screening measurements. The surface soil composite sample will be collected from the top 15 cm (6 in) and the subsurface samples from the 15-30 cm (6-12 in) layer. This sampling approach essentially follows that proscribed by the Louisiana regulations.

Implementation of this technique will allow for the maximization of the number of sampling sites, such that the most extensive national representation possible is achieved given the available funds. Although analytical costs are limiting, substantial additional data will be provided from the results of the initial gamma survey, the information from which may be compared with the results of the soil sample analysis.

### **3.2 Radium Solubility Measurements**

A subsample of five pipe scale samples found to have elevated radium concentrations will be used to determine the solubility of the radium-containing material. The purpose of determining the solubility of pipe scale is to evaluate the potential mobility of the radium from contaminated scale material in water solutions. Selection of these samples will be done by GJPO personnel in the laboratory. The samples chosen for this analysis will include those with relatively high radium concentrations. No statistical design is necessary for this portion of the program.

### **3.3 Whole Pipe Radon Release Measurements**

The purpose of collecting radon samples from NORM contaminated pipe samples is to determine the rate at which radon may be released to the environment from pipes containing radiumbearing scales. This will provide information necessary to estimate risks from radon and its short lived progeny at these facilities.

Twenty sections of NORM-contaminated pipe representing 2 sections from each of 10 different wells will be selected for sampling by API or API member company personnel. The 20 pipe sections will be randomly selected from the population of NORM contaminated pipe at those pipe yards selected by API. API or the API member company will determine the appropriate identification number (see section 4.3.1) for each pipe section, and this number will be permanently marked on the pipe. Because the focus of this portion of the study is on the relationship between the radon emission rate from pipes and the thickness and radium concentration of the scale, a statistically-based geographic distribution is not strictly necessary. Rather, personnel from API or the appropriate API member company will arrange to ship the pipe sections selected for this portion of the study to an oil production facility, preferably near Grand Junction, Colorado. GJPO Radon Laboratory personnel will then travel to that facility and perform the radon release measurements. Following analysis of the radon release rates, custody of the pipe sections will be returned to API or the appropriate API member company.

These pipes will be shipped to a commercial pipe cleaning facility where the entire scale contents of the pipe sections will be collected. The scale that is recovered from each pipe section will be homogenized and a 2 lb (0.9 kg) aliquot will be extracted using the cone and quarter or other suitable method. At the time that the scale is removed from the pipe section the total mass of the scale removed from the pipe will be determined. The pipe scale samples will then be forwarded to the GJPO Analytical Laboratory for radium analysis.

Radon release rates from pipes will be measured by collecting and subsequently measuring the amount of radon released from the pipe over a timed interval. This will be accomplished by fitting a column containing activated charcoal to one end of the pipe section and a supply of radonfree air to the other end. A continuous flow of radon-free air will be used to sweep out any radon that emanates from the radium contaminated scale. The radon thus purged from the interior of the pipe will be collected on the charcoal column. A quantitative analysis of the radon collected on the charcoal will be provided by gamma analysis. Additionally the interior connected volume of the pipe section will be determined by evacuating the pipe and allowing it to return to atmospheric pressure through a dry gas meter.

### **3.4 Radon Emanation Fraction Measurement**

Radon and its short lived daughter products pose a significantly greater potential health risk if the radon is released from the source material and is available to migrate as an integral part of the near ground level atmosphere where radon and its' daughter products may be inhaled. Conversely if the radon atoms are retained within the NORM contaminated materials the inhalation hazard is minimized and the radiation hazard is dominated by the occurrence of a localized gamma field associated with the contaminated materials.

Some of the  $^{222}\text{Rn}$  atoms that are created by the decay of  $^{226}\text{Ra}$  present in the pipe scale (or other radium bearing material) will be retained within the crystal lattice of the host mineral for a time sufficient to allow further decay into  $^{218}\text{Po}$ . Other atoms, especially those formed near the surface of the mineral grain, will be more likely to escape crystal lattice as free gaseous radon atoms. The fraction of the total radon atoms produced by the radium parent that are released as free gaseous radon atoms is termed the radon emanation fraction. Measurements of the radon emanation fraction for NORM contaminated pipe scales and sludges are useful in determining the potential exposures to workers and the general public from radon released from sites containing these NORM contaminated materials.

Radon emanation fraction measurements will be performed on 30 samples comprised of approximately 15 pipe scale samples and 15 process facility waste (sludge) samples. These samples will be provided by API or API

member companies. In contrast to the sludge samples provided to the GJPO Analytical Laboratory for radium analysis the sludge samples provided for radon emanation fraction will not be burned to remove the organic fraction. It is critically important for the utility of these measurement results that these samples be provided in the same physical form as they occur in the field. For this reason these scale samples should be obtained from the pipe by the processes that is routinely used to clean scale from pipe.

### 3.5 Gas pipe $^{210}\text{Pb}$ samples

Segments of gas transmission lines will be analyzed for  $^{210}\text{Pb}$  contamination for the purpose of determining the extent to which this radon daughter radionuclide accumulates on the interior of gas pipes and equipment. This portion of the NORM Characterization Program will rely on a limited number of samples collected from locations where the opportunity for accumulation of  $^{210}\text{Pb}$  is anticipated to be the highest. Selection will therefore be based on several factors including known radon concentrations within the natural gas during the lifetime of the pipe, the length of service of the pipe, and whether access to the pipe is readily available (See Appendix F). This portion of the study is therefore intended to serve only as a pilot study rather than as general characterization of  $^{210}\text{Pb}$  accumulation in gas transmission lines. A statistically designed sampling scheme is therefore not required for  $^{210}\text{Pb}$  sampling. A total of 10 samples of gas transmission lines will be collected by GRI and sent to GJPO for analysis of  $^{210}\text{Pb}$  concentration.

There are several complications involved with measuring  $^{210}\text{Pb}$  concentrations in these materials:

1. Because only low energy gamma radiation is given off by  $^{210}\text{Pb}$ , this radionuclide cannot be detected by surveying the exterior of the pipe with hand held scintillometers.
2. Deposits containing the  $^{210}\text{Pb}$  are anticipated to be of extremely low mass in comparison to the scale materials sampled from oil production piping. This may create a problem in obtaining an adequate sample size.
3. It has not yet been determined whether the material containing the  $^{210}\text{Pb}$  is removable from the interior of the pipe.

Analytical techniques involving the use of an organic solvent has been proposed as a first step. If this technique fails to produce adequate results, further work on sample analysis will be halted and GRI will be consulted regarding what additional steps, if any, should be taken. A limited amount of funding (approximately \$5,000) will be

available for this phase of the project.

### **3.6 Alpha Track Measurements on Gas Pipe Interiors**

The first decay product of  $^{210}\text{Pb}$  is  $^{210}\text{Po}$ , which itself decays by alpha emission. The half-life of  $^{210}\text{Po}$  is 138 days, so that after a few years, the activity of  $^{210}\text{Po}$  approaches equilibrium with the activity of  $^{210}\text{Pb}$ . The number of "alpha tracks" recorded on a special detection film placed on the interior of the pipe will be proportional to the  $^{210}\text{Po}$  activity. This will allow for an estimation of the amount of  $^{210}\text{Pb}$  found on the interior of the gas transmission line.

The same ten samples of gas delivery pipe or equipment provided by GRI for analysis at GJPO for  $^{210}\text{Pb}$  contamination will be used to determine the extent of residual  $^{210}\text{Po}$  contamination. Alpha track procedures will be used to determine the presence of residual activity in the pipe before surface contamination is removed.

## 4. Standard Operating Procedures (SOPs)

The samples to be collected during the NORM Characterization Program represent a variety of different types. In some cases, the form taken by the samples will be similar to those collected for the UMTRA Program or other previous large-scale radionuclide assessment programs. Where NORM Program samples are similar to those collected for other programs, sample collection and analysis procedures have been adopted with the appropriate modifications. In some cases, however, samples collected during the NORM Program are of a type not previously associated with radioactive monitoring and assessment programs. New procedures have been designed for the collection and analysis of these sample types.

Samples collected for analysis during the NORM Characterization Program include:

- ☐ pipe scale samples;
- ☐ production facility waste samples;
- ☐ soil samples;
- ☐ radium solubility measurement samples;
- ☐ whole pipe radon release measurements;
- ☐ radon emanation fraction; and
- ☐ gas pipe samples.

The purpose of this section is to provide detailed procedures to be applied to the different aspects of the NORM Characterization Program in the form of Standard Operating Procedures, or SOPs. Included are SOPs for Sample Collection (Section 4.1), Sample Analysis (Section 4.2), Sample Identification and Tracking (Section 4.3), Data Management (Section 4.4), and Waste Disposal (Section 4.5). Some of the analytical procedures applied to this program have been used previously by the analytical laboratory in Grand Junction during other monitoring and assessment programs. Rather than rewriting existing SOPs, such SOPs are incorporated into this S&A Plan as written. Copies of these previously existing SOPs are provided in Appendix A to this document.

Each SOP contained in this S&A Plan includes detailed descriptions of the rationale for using the specific procedures applied to the Program. To avoid burdening sample collection personnel with information not useful in the field, instructions for the collection of samples in the field are repeated in Appendix E. Appendix E is therefore designed to provide the field sampling personnel with a more detailed and useful "cook book" description of how to select, collect and ship each type of sample. As such, Appendix E represents the field manual for sample collection, and should accompany each team member while collecting samples.



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## 4.1 Sample Collection SOPs

Specific procedures for the collection of each type of sample are provided below. These procedures are to be used by the field by personnel charged with collecting NORM Characterization Program samples whenever possible. Any deviation from the sample collection SOPs described herein must be documented and explained by the field collection personnel in the sample collection documentation that accompanies the sample to the analytical laboratory. The QA/QC procedures applied during the sample collection process will be limited to the collection of replicate samples of 10% of the samples. Selection of the specific samples to be replicated will be predetermined randomly by API. These and other QA/QC procedures are summarized in the QA/QC Plan (Appendix B of this document).

Most of the samples collected during the NORM Characterization Program will be collected by on-site personnel affiliated with either API or GRI member companies. This implies a certain degree of inconsistency in the sampling process, and highlights the need for clear and concise sample collection SOPs. All personnel participating in the collection of samples shall be familiar with the appropriate sample collection SOPs prior to working in the field. These procedures describe:

- ⊘ How field personnel determine the specific location at which a sample will be collected (including how to survey the site at which the sample will be collected);
- ⊘ How the sample will be collected by field personnel;
- ⊘ The quantity (in terms of either volume or mass) of the sample to be collected;
- ⊘ The sample containers to be used in the field for the collection and shipment of the samples;
- ⊘ Any applicable QA/QC procedures that must be followed during sample collection; and
- ⊘ Any ancillary information that must be recorded by the field personnel.

### 4.1.1 Pipe scale samples collection SOPs

The pipe cleaning and storage yards at which pipe scale samples will be collected will be determined by API

personnel prior to the initiation of sample collection, as described in Section 3 (Sampling Strategy) of this document. The procedure for selecting the pipe yards to be sampled is not repeated here. Selection of the specific pipes from which scale samples will be collected will be conducted in the field, by on-site personnel from API member companies. This selection process is therefore included here along with the procedures to be applied to the collection of the samples.

Fifty pipe sections will be selected for sampling at each pipe cleaning and storage yard included in the survey. Production pipe is typically stacked horizontally in "pipe racks", sorted by size and quality, with each layer of pipe within the rack separated by wooden timbers. Because of this arrangement, access to pipes other than those on the top (and possibly the sides) of each stack will be difficult. Only those pipe sections that are readily accessible will be included in the sampling. Accessibility of individual pipe sections will be determined in the field by the sample collection personnel. If a basic configuration of pipe in the yard deviates significantly from the anticipated configuration, sample collection personnel will devise an alternative method for determining accessibility, and will document the procedure used in the sample collection documents that will accompany the samples to the analytical laboratory.

In addition to the accessibility of the pipe, selection of pipes to be sampled will be based on an initial gamma survey of the pipe storage area. The SOP for conducting facility gamma surveys is provided in Section 4.1.9 and Appendix E. The ends of all accessible pipe sections in all pipe racks present within the yard will be surveyed, with the resulting gamma readings recorded for each pipe. Using the results of this survey, pipe scale will be collected from the 10 accessible sections of pipe that exhibit the highest gamma readings. This will provide an upper limit for radium concentrations within the accessible pipe scale from these yards. The remaining 40 samples will be chosen randomly from among the sections of accessible pipe from which gamma readings in excess of 50  $\mu\text{R/hr}$  are observed. This approach will provide a reasonable range of radium concentration values for the yard in addition to the bounding case.

A list of materials needed to collect pipe scale samples is provided in Table 4-1. The sample collection process for pipe scale samples is outlined below:

1. **Map the pipe yard:** A hand-drawn map of the pipe yard shall be produced before any measurements are taken or samples collected. Each pipe stack should be assigned a number, and the total number of pipes within each stack should be estimated and recorded. This map will also be used during the soil sampling procedure.

**Table 4.1** Sampling kit contents necessary to collect pipe scale and soil samples from a single pipe yard (50 scale samples and 5 field replicates; 1 composite surface and 1 composite subsurface soil sample).

Quantity	Description
1	Gamma survey meter, calibrated and with check source
1	Tape measure
1	Sample collection form
1	Roll of engineers flagging
56	16 oz (1 pint) wide mouth plastic jar
56	Sample labels
1	Soil core sampler
1	Chalk or duct tape for numbering and identifying pipe
2	Disposable aluminum pans (approximately 8 x 12 inches)
1	Large spoon or other utensil for mixing soil

2. **Identify the accessible pipes:** Once the map of the pipe yard is complete, the sample population will be defined. The sample population will consist of all uncleaned, readily-accessible pipe with scale present. This activity will be coordinated with the operators of the pipe yard. Pipes are considered "accessible" if they can be removed from the stack without the need for removing any other pipe first. Only those pipes located on the top layer and possibly those at the end of rows within the stack are therefore expected to be "accessible", although additional pipes may be available depending on the specific configuration employed at the yard. The assumption has therefore been made that the accessible pipes are representative of the rest of the pipe within the rack.

Pipe with outside diameters of greater than four inches will be excluded as these larger diameter pipes generally contain less scale and therefore represent a lower probability of radioactive contamination. Each accessible pipe will be assigned and marked with an identification number using chalk or duct tape. The sample number for each pipe will be based on its position in the stack and on the number of the stack (e.g. number 14-23 would represent the 23rd pipe in the 14th stack). This procedure will allow for easy identification of the pipes from which actual samples will be collected.

3. **Survey accessible pipes:** All accessible, uncleaned pipes identified will be surveyed using hand held gamma exposure rate meters provided by API member companies. The gamma readings will be taken at one end of the pipe, with the results recorded. The survey meters used will be checked for accuracy using a standard source, and must be within 15% of the true count.

4. **Identify 10 "hottest" pipes:** From the results of the survey of accessible, uncleaned pipes, the 10 "hottest" pipes will be identified. These pipes will provide the first 10 samples collected from each pipe yard. The pipes selected will be labeled to identify them as sample pipes.
  
5. **Select additional pipes for sampling:** An additional 40 will be randomly selected for sampling from the remaining population of uncleaned, accessible pipes that have gamma readings in excess of 50  $\mu$ R/hr. Selection will be performed using a random number table, with the first appropriate number designating the stack, and the second number the pipe within the stack. These additional sample pipes will also be labeled for identification.
  
6. **Collect the samples:** Pipes selected for sampling will be "rattled" (or cleaned with whatever alternate process is typically used at the site) to remove scale accumulated within the pipe. A representative sample of the scale removed will be collected by the sample collection personnel, making sure that the material collected is from the pipe in question. Sufficient scale material will be collected from each pipe to fill one 16-oz., wide-mouth plastic jar, representing a total of approximately 1.0 kg (2.2lb) per sample. A second scale sample will be collected for five of the 50 pipes, including at least one of the "hottest" ten pipes sampled. These will constitute the 10% field replicate samples required for QA purposes, as described in the QA/QC Plan (Appendix B).

All samples will be properly labeled and shipped to the analytical laboratory in Grand Junction, CO (see Sample Identification and Tracking SOPs, Section 4.3 below), where they will be analyzed for  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  (see Sample Analysis SOPs, Section 4.2 below). Information that must be recorded for each pipe sample collected include:

- ⊘ pipe and stack number
- ⊘ gamma survey reading on end of pipe
- ⊘ outside diameter of the pipe
- ⊘ estimated average thickness of the scale
- ⊘ pipe wall thickness (or inside diameter)

#### **4.1.2 Production facility waste sample collection SOPs**

The selection of petroleum industry production facilities at which to collect waste samples will be

determined by API prior to the initiation of sample collection. The procedure used to determine which facilities to sample is described in Section 3 (Sampling Strategy), and is not repeated here. It is not likely that all production facilities selected by API for sampling will be made accessible by the facility owners. As described in Section 3, samples from facilities to which access cannot be obtained will be replaced with samples collected from common waste storage sites serving the region that includes the initially selected production facility. Selection of the specific locations within each facility at which to collect waste samples is considered here, as are the procedures for collecting the samples.

Collection of production facility waste samples will be performed by on-site personnel from API member companies. It has been assumed that sludges, oil and water emulsions, sediments, sands, and other materials that have been produced as wastes will be stored in drums on site. Samples will be collected from these drums, with liquid samples being avoided. Mixed wastes of this type will consist of different types of materials separated out into different phases within the drum.

At each production facility selected for sampling, selection of the drum or drums to be sampled will be determined from the results of an initial gamma survey of the drums at the facility. A population of drums that have exposure readings greater than 50  $\mu\text{R/h}$  above background will first be defined during a gamma survey of the facility. From that population of drums, individual drums will be identified and sampled (see Appendix E for details). At a predetermined set of 10% of the production facilities sampled, a second drum will also be sampled. This second drum will be the drum identified with the highest gamma reading. The SOP for conducting gamma surveys is provided in Section 4.1.9. and in Appendix E.

The sample collection process for production facility waste samples is outlined below:

1. **Map the production facility:** A hand-drawn map of the production facility shall be produced before any measurements are taken or samples collected. In addition to any buildings, roads, etc. identified on the map, all flow lines, heater-treaters, separators, sumps, water lines, and waste storage or disposal areas should be located, to the extent possible. This map will also be used during the soil sampling procedure.
2. **Survey waste accumulation areas:** All waste accumulation areas within the facility will be surveyed, using hand held gamma exposure rate meters provided by API member companies. The exposure rate meters will be checked for accuracy using a standard source, and must be within 15% of the true count. A table will be created in which exposure measurements of all waste drums with average gamma readings in excess of 50  $\mu\text{R/h}$  will be recorded, referenced to assigned barrel identification numbers. Where possible, measurements

shall be taken on the top and at least two sides of each waste drum, with the average of these three readings recorded for the drum.

**3. Identify the maximum drum and determine the mean exposure rate for the drums over 50  $\mu$ R/hr:**

Using the table of recorded exposure readings, identify the drum with the highest exposure reading and determine the mean for all drums with readings in excess of 50  $\mu$ R/h. This is done for the purpose of determining the "average" barrel from within this population.

**4. Collect the samples:** Waste samples will be collected using a modified Coliwasa sampler, consisting of a piece of electrical conduit approximately 3.5 ft long and with a diameter no less than 1 inch. A list of the equipment needed to collect these samples is provided in Table 4.2.

Drive the sample tube from the top to the bottom of the drum, taking care to minimize disturbance to the contents of the drum. Because the entire depth of the drum is sampled, the resulting composite sample is representative of all phases of waste material present in the drum. Fifty-five gallon drums are approximately 3.0 ft high, so approximately 6 inches of the electrical conduit should protrude above the drum after the conduit has been driven through the contents of the drum. Care must be taken to avoid puncturing the drum liner.

Remove the sample material from the electrical conduit using the wooden dowel. Each sample collected in this manner will yield a sample of about 1 pint.

If the barrel selected for sampling contains substantial quantities of liquid material, an alternate barrel will be substituted. For a sample to be collected from the barrel representing the average gamma reading for the facility, the replacement should be the barrel with the next closest reading to the mean value. For barrels representing the highest gamma readings, the barrel with the next highest reading should be substituted.

If the drum contents are hard, making it difficult to drive the Coliwasa sampler through the material in the drum, a sample may be "scooped" from the contents of the top 6 inches (15 cm) of the drum. This must be noted on the field sample collection form, and it will therefore be assumed that the waste material is homogeneous throughout the drum. If the material is oily, then a 1 quart sample (about 4.0 lb, 1.8 kg) will be collected, otherwise, a 1 pint (2 lb, 0.9 kg) sample will be collected.

If the drum being sampled has been identified by API as among the ten percent field replicates, the sampling

procedure must be repeated, with a second similar waste sample collected from the same drum as provided the original sample. Field personnel should take every precaution to ensure that the replicate sample is collected in the identical manner as the original sample.

**Table 4.2** Sampling kit contents necessary for collection of each soil and production waste drum sample collected at a production facility.

Quantity	Description
<b>Production Waste Samples:</b>	
1	Gamma survey meter, calibrated and with check source
2	Sample Collection Forms (one each for waste and soil sample to be collected at the site)
1	Pocket calculator
1	Electrical conduit (3.5 ft long, 1.0 inch diameter)
1	Wooden dowel (about 1/2 inch diameter, 3/2 ft long)
1	16 oz (1 pint) wide mouth plastic jar <sup>2</sup>
1	24 oz (1 quart) wide mouth plastic jar
<b>Soil Samples:</b>	
1	soil core sampler
2	16 oz (1 pint) wide mouth plastic jar <sup>2</sup>
2	disposable aluminum pans, 8 x 12 inches (approx.)
1	large spoon or other utensil for mixing soil
1	rinse bottle and paper towels for cleaning sampler between cores
<sup>1</sup>	Soil samples will only be collected at 50% of the production facilities at which waste samples are collected
<sup>2</sup>	Duplicate sample containers will be needed for any QA/QC (field replicates) samples collected at the facility for both waste and the soil samples.

All samples will be properly labeled and shipped to the Chem-Nuclear Geotech analytical laboratory in Grand Junction, CO (see Sample Identification SOPs, Section 4.3 below), where they will be analyzed for <sup>226</sup>Ra and <sup>228</sup>Ra (see Sample Analysis SOPs, section 4.2 below). If the sample contains oily material, then it is the responsibility of the sampler to indicate so and send the sample to a laboratory identified by API for removal of the oily material. It is the responsibility of the laboratory performing the removal of oily material to record, on the sample collection form, the sample mass before and after removal, and forward the sample to Chem-Nuclear Geotech for analysis. Information that must be recorded for each waste sample collected include:

- ☐ location within the facility
- ☐ gamma survey readings for the barrel
- ☐ state and county in which the facility is located
- ☐ type of facility



- 3 any information available regarding the source of the waste

### 4.1.3 Soil sample collection SOPs

Soil samples will be collected at each pipe yard used to collect pipe scale samples, and at a subset of the production facilities used to collect waste samples. The determination as to which of the production facilities will be used to collect soil samples will be made by API. The equipment needed for the collection of these samples are included in the equipment lists for the collection of associated pipe scale samples (Table 4-1) and production facility waste samples (Table 4-2). Selection of the specific locations within each facility at which to collect soil samples is considered here, as are the procedures for collecting the samples.

Collection of soil samples from pipe yards and production facilities will be performed by on-site personnel from API member companies. The soil sampling design generally follow methods used elsewhere for characterizing radium contaminated soils (Williams et al., 1989) as well as procedures described in the Louisiana regulations. Selection of the specific location within the facility at which to conduct the soil sampling will be based on the initial facility gamma survey.

The sample collection process for soil samples is outlined below:

1. **Survey the facility grounds:** Using the maps of the pipe yard or production facility generated during the pipe scale and production facility waste sampling procedure, a general gamma survey of the facility grounds will be performed. The survey meters will be provided by API member companies and will be checked for accuracy using a standard source. Survey meters must be within 15% of the true count. Although the entire area of the facility will be surveyed, the survey will focus on areas where soil contamination is most likely to occur. For pipe yards, this would include any areas where pipe cleaning operations were conducted. For production facilities, this would include areas around flow lines, heater-treaters, separators, sumps, water lines, and waste storage or disposal areas. Representative gamma readings will be recorded on the facility map, and all "hot spots" where gamma readings in excess of 50  $\mu\text{R/h}$  are found will be identified and located.
2. **Identify "hottest" 100m<sup>2</sup> area:** From the results of the survey of facility grounds, the single "hottest" 100m<sup>2</sup> area of surface contamination will be identified for sampling, and the location of the soil sample collection area within the facility will be noted on the map. This must represent a rectangular area of at least 100 m<sup>2</sup> (1100 ft<sup>2</sup>), with a minimum dimension of 5 m (16 ft).

3. **Collect the samples:** From the identified sampling location, one surface and one subsurface composite sample of 9 soil cores will be collected. The 9 core locations will be spaced equidistant over the 100 m<sup>2</sup> area selected, so that the entire plot area is included. The surface soil composite sample will be collected from the top 6 inches (15 cm) and the subsurface samples from the 6-12 inch (15-30 cm) layer. The locations of each individual core will NOT be selected based on gamma screening measurements. The individual surface or subsurface cores will be placed into disposable aluminum pans, one for surface soil compositing and one for subsurface soil compositing. Once all 9 surface and subsurface soil cores are collected in the aluminum pans, the composited samples will be blended using a large spoon or other utensil. Once thoroughly mixed, a representative sample of each (surface and subsurface) will be placed into a labeled 16-oz wide-mouth plastic jar. The jar should be filled completely with soil sample. Sampling equipment must be washed or rinsed to remove most traces of the soil between each of the 9 sample collection sites. If the facility being sampled has been identified by API as one of the ten percent at which field replicates will be collected, a second pair of soil samples must be collected. These field replicates should be collected from the same surface and subsurface sample material composited within the aluminum pans for the original soil samples.

All soil samples will be properly labeled and shipped to the Chem-Nuclear Geotech analytical laboratory in Grand Junction, CO (see Sample Identification and Custody SOPs, section 4.3 below), where they will be analyzed for <sup>226</sup>Ra and <sup>228</sup>Ra (see Sample Analysis SOPs, section 4.2 below). Information that must be recorded for each waste sample collected include:

- ☐ location of the sample within the facility;
- ☐ gamma survey readings for the sample area;
- ☐ state and county in which the facility is located;
- ☐ type of facility sampled; and
- ☐ any information available regarding the source of the contamination; (e.g., Is there a waste accumulation, sump, heater-treater, etc. nearby? Does the facility have any record of an incident that may explain the elevated readings?).

#### **4.1.4 Radium solubility sample collection SOPs**

A subsample of five pipe scale samples will be used to determine the solubility of the radium-containing material within the pipe. Selection of these samples will be performed by GJPO personnel in the laboratory from

among those samples collected for analysis of radium concentration, so no additional sample collection procedures are necessary. Only those samples with relatively high radium concentrations will be selected.

#### **4.1.5 Whole Pipe Radon Release Samples**

As discussed in Section 3.3, twenty sections of NORM contaminated pipe will be shipped to an oil production facility near Grand Junction, Colorado where personnel from the GJPO Radon Laboratory will determine the radon release rate from the pipe. Following the radon release measurements these pipe sections will be returned to a commercial pipe cleaning facility for recovery and sampling of the scale. The scale from each section of pipe will be removed and collected by an under-reaming process. Following this process the total quantity of scale recovered will be homogenized and a representative aliquot (2 lb) will be obtained and shipped to the GJPO Analytical Laboratory for radium analysis. The radon release rate can then be correlated with the average radium content and volume of scale.

The approach used to determine radon release rate from production pipe is to collect all of the radon that emanates from the radium-bearing scale within the pipe section on activated charcoal. The amount of radon activity on the charcoal will be quantitatively determined using spectral gamma analysis. The average radon release rate (in  $\text{pCi sec}^{-1}$ ) will be calculated for each pipe section using sampling period information and making the appropriate corrections for radioactive decay.

The Radon Laboratory Technical procedure RN-FLUX-U, Radon Flux Measurements Using the Large Area Activated Charcoal Collector Method will be used as the basis for these measurements, with appropriate modifications in terms of the cross sectional area of sample collection. The Large Area Activated Charcoal Collector (LAACC) described in Technical Procedure RN-FLUX-U will not be used. This will be replaced by a 2 ft. length of 1-inch PVC pipe that will hold the same volume of charcoal as is routinely used in the LAACC.

In addition to the radon release measurements, the interior connected volume of each of the sections of tubing will be measured. This measurement will be accomplished by evacuating the tubing with a portable vacuum pump. The volume of air required to return the tubing to atmospheric pressure will be metered with a dry gas meter. As the pipe sections are selected by a representative of the appropriate API member company, the pipe will be permanently marked with a unique identification number. This ID number will contain characters 1 through 5, and 8 through 10 of the sample ID from the scheme described in section 4.3.1. Place holders, such as "#" should be used for the sample type designator. An example of the ID number applied to one of the pipe sections is PI057##010. This identifies the pipe section as coming from a pipe storage yard at a facility which has been assigned the number

57 by API, and is the tenth pipe selected for sampling at that facility. This pipe identification number will then be used to develop the sample ID's for both the whole pipe radon release charcoal samples as well as the pipe scale samples to be analyzed for radium content, and will ensure that the two sets of results may be correlated.

Personnel from the API member company will prepare the individual pipe sections for shipment by capping both ends of the pipe with thread protectors or a similar cover type. These covers will then be secured to the pipe with duct tape in order to trap any loose scale within the pipe. The API member company representative will also be responsible for ensuring that the pipe shipment meets all appropriate Department of Transportation (DOT) regulations. This includes a requirement that surface contamination on the exterior of the shipment not exceed 2.2 dpm per 100 cm<sup>2</sup> (49 CFR 173.443). Additionally, the contact surface exposure rate on the shipment must not exceed 200 mR/h (49 CFR 173.441). Upon arrival at the oil production facility, the pipe and the vehicle hauling the pipe shall be cleaned thoroughly to remove any surface contamination.

Two adapters will be needed for each section of tubing to be sampled. One adapter will be comprised of a 2-inch by 1-inch PVC reducer bushing with a 2-foot length of 1-inch ID PVC pipe to hold approximately 180 grams of charcoal. The pipe will be fitted with a wire screen and nylon mesh at the bushing end to retain the charcoal. The other end will be capped with a one-hole rubber stopper. This end will also be fitted with a nylon mesh to retain the charcoal. The second adaptor will be used to connect the other end of the tubing to a supply of dry nitrogen. This adapter will be comprised of a 2-inch PVC reducer bushing stepped down to 1/8-inch pipe thread.

The PVC reducer bushings will be held against the end of the pipe to be sampled with a short length of 2 1/2-inch rubber hose. Hose clamps will be used to clamp the rubber hose onto the end of the pipe to be sampled and the PVC reducer bushing. A light coating of high vacuum grease may be used to ensure a good seal. The PVC bushings and hose sizes described above are used for 2 3/8" production tubing. Other sized fittings will be required for other pipe sizes.

Standard compressed gas bottles of nitrogen will be used as the gas source. There are two advantages to using compressed nitrogen for this purpose: (1) compressed nitrogen is essentially free of radon, and therefore will require no background radon measurement; and (2) compressed nitrogen is very dry and will therefore not load the charcoal with water, which competes with radon for sorption sites.

A standard two stage regulator will be used to drop the tank pressure down to approximately 5 PSIG. The outlet of the regulator will be connected to a needle valve, which is in turn connected to a 0 to 50LPM rotameter (flow rate meter). The exhaust from this rotameter will be connected to a manifold which distributes the nitrogen to

10 separate outgoing lines. Each line will be connected to a valve, followed by a 0 to 5LPM rotameter. This last rotameter will be plumbed directly into the PVC reducer bushing. Thick-walled, 1/2-inch rubber tubing will be used to fabricate the manifold and all connections.

The nitrogen tanks will be chained to the side of the pipe rack on which the tubing samples are held. The plumbing connections will then be made to the ends of the sections of production tubing. The sections of tubing will be purged with at least 5 volumes of nitrogen to remove any residual radon prior to attaching the charcoal-filled samplers. Assuming that the tubing sections are 30 ft. in length and have a 2 3/8 inch ID, this will require approximately 130 L of nitrogen for each section of tubing. This purging will be accomplished by adjusting the total flow from each nitrogen tank to 50 LPM with the needle valve. Then the valves leading to the individual tubing sections will then be adjusted to balance the flow resulting, in a flow rate of 5 LPM into each of the 10 tubing sections. This flow rate will be maintained for at least 26 minutes.

**4.1.5.1 Whole Pipe Radon Sample Collection** -- When the sample sections of pipe have been purged for at least 26 minutes, the total flow rate to the manifold will be reduced to 1 LPM. A 0 to 0.5LPM rotameter will then be inserted in the nitrogen supply line immediately upstream of the existing 0 to 5LPM rotameter. Again the valves leading to the individual sections of tubing will be adjusted to balance the flow, such that a flow rate of 0.1 LPM into each of the 10 sections of tubing will be achieved.

A short length of 2 1/2-inch ID rubber tubing will be connected to the exhaust end of the section of tubing to be sampled. To begin the sample collection a sample adapter will be inserted into the rubber tubing until the PVC reducer bushing mates with the end of the pipe. The hose clamp will be tightened to hold the sample adapter in place. The date and time of the start of the exposure will be recorded in the laboratory notebook, along with the sample ID.

Following an exposure period of 72 ± 4 hours, the sample adaptor will be removed from the tubing. The rubber stopper will then be removed and the charcoal emptied onto a mixing pad. Appropriate care will be exercised to ensure complete recovery of the charcoal. The charcoal sample will then quickly be mixed on the mixing pad and transferred to a gamma counting can. An identification label will be affixed to the can containing the charcoal sample. The label will contain the following information:

- ! Sample ID number;
- ! Date and time for start of exposure;
- ! Date and time for end of exposure; and
- ! Initials of the person collecting the sample.

**4.1.5.2 Volume Measurements** – The following procedures will be used to measure the volume of the connected interior portion of each section of tubing selected for whole pipe radon release measurements will also be determined. The apparatus required for the volume measurement includes the following items:

1. Welch Duo-Seal Vacuum Pump, Model 1400 (or equivalent);
2. Vacuum gauge 0 to 30 inches of Hg, with 0.1-inch graduations;
3. 500 mL Erlenmeyer Vacuum Flask, with rubber stopper and hose barb;
4. 5 teflon two-way stopcocks (valves);
5. 2 $\frac{1}{2}$ -inch by 1-inch PVC reducer bushing (glue-pipe thread);
6. 2-inch by 1-inch PVC reducer bushing (glue-glue);
7. 1-inch by 2-inch PVC reducer bushings (glue-pipe thread);
8. 2-inch by 1/8-inch galvanized reducer bushings (pipe thread);
9. 1/8-inch pipe thread to 2-inch tubing "T";
10. 1-inch pipe thread plug;
11. 2 $\frac{1}{2}$ -inch ID, rubber radiator hose, 4 inches in length;
12. 2-inch ID, rubber radiator hose, 4 inches in length;
13. Eight 3-inch hose clamps;
14. 1/4-inch ID by 5/8-inch OD rubber tubing;
15. Singer DTM-115 dry gas meter (or equivalent);
16. Dwyer differential pressure gauge,  $\surd$  2 inches water, full scale;
17. Hamilton model S-1000, 1 L syringe;
18. High vacuum grease;
19. Four 1/4-inch tubing "T" connectors;
20. 0-10-LPM rotameter;
21. Digital watch or stopwatch;
22. Materials to construct a portable sunshade; and
23. Notebook.

Figure 1 shows a sketch of the apparatus that will be used in the volume measurements. The procedure is as follows:

1. Install the 1-inch pipe thread plug into the 2 $\frac{1}{2}$ -inch by 1-inch PVC reducer bushing to complete the reducer bushing cap.

2. Assemble the remaining reducer bushings and the 1/8-inch pipe thread by tubing "T" fitting.
3. Attach the valves V-1 and V-3 with 1/4-inch rubber tubing to the tubing "T" to form the reducer bushing assembly.
4. Determine the volume of the PVC reducer bushing assembly, from the valves to the plane of intersection with the production tubing. This may be accomplished by weighing the amount of deionized water required to fill the requisite volume, and converting this into a volume using the density of the water. Measure the volume of the reducer bushing cap in the same manner.

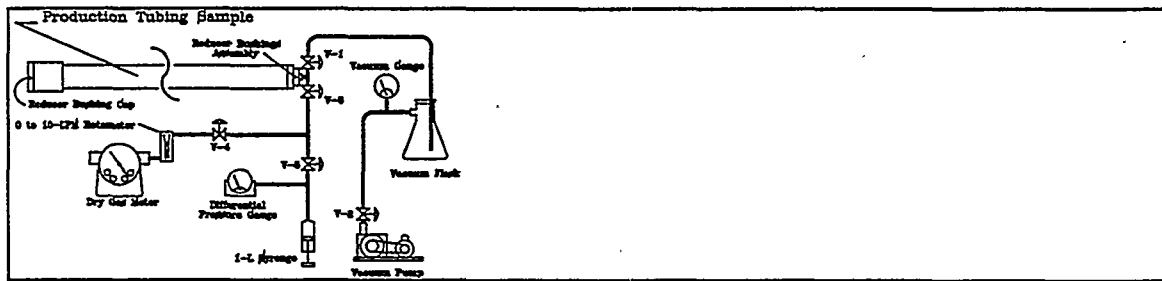


Figure 1. Apparatus Used to Measure Volume of Production Tubing Sample.

5. Erect a portable sunshade to shield the sample production tubing from direct sunlight, if the measurements must be conducted outside. Approximately 30 minutes should be allowed for the tubing to reestablish an equilibrium temperature once the sunshade has been erected.
6. Connect the vacuum pump to V-2 with rubber tubing, then the open side of V-2 to a tubing "T" connector with 1/4-inch rubber tubing.

7. Connect one of the open ends of the "T" connector to the vacuum gauge, and the other to the side hose barb of the Vacuum flask with  $\frac{1}{8}$ -inch rubber tubing.
8. Connect the hose barb in the rubber stopper of the flask to V-1 with  $\frac{1}{8}$ -inch rubber tubing.
9. Connect the open side of V-3 to a tubing "T" connector.
10. Connect one end of the "T" connector to V-4 with  $\frac{1}{8}$ -inch rubber tubing.
11. Connect the other end of the "T" connector to V-5.
12. Connect the open end of V-5 to another "T" connector.
13. Connect the differential pressure gauge to one side of the second "T" connector, and the 1-L syringe to the other side. Be sure that the syringe piston is positioned at the 1-L mark.
14. Connect the open end of V-4 to the exhaust side of the 1 to 10-LPM rotameter.
15. Connect the inlet side of the rotameter to the exhaust side of the dry gas meter.
16. Lightly coat the outside surface of the male threaded end of the production tubing with high vacuum grease.
17. Slide the length of  $\frac{1}{2}$ -inch ID rubber hose on the end of the tubing so that slightly less than 2 inches of the hose extends beyond the end of the tubing.
18. Secure the rubber hose to the tubing with 2 hose clamps.
19. Slide the 2-inch PVC reducer bushing assembly into the exposed end of the rubber hose until it butts against the end of the tubing and secure the bushing with 2 hose clamps.
20. Install the  $\frac{1}{2}$ -inch PVC reducer bushing cap on the other end of the tubing section in the same manner.
21. Close all valves, then turn on the vacuum pump and open V-1, V-2, V-3, and V-4. The reading on the dry



gas meter should begin to increase.

22. Allow several liters to flow through the dry gas meter, then close V-3.
23. Check the system for vacuum leaks between the vacuum pump and V-3 by evacuating the system until no noticeable increase in the gauge vacuum is observed. The gauge vacuum should correspond to the local barometric pressure uncorrected for altitude. Seal any leaks. **CAUTION!** The presence of a high vapor pressure contaminant compound in the tubing may act like a vacuum leak.

Once the apparatus is erected and checked out, pipe volume may be determined. This is done in the following sequence of steps:

1. Ensure that V-1 is closed and V-2 is open, then evacuate the apparatus to V-1 until no noticeable increase in the vacuum is seen on the gauge. Note the gauge reading and record it in the notebook.
2. Open V-1 and evacuate the production tubing sample until there is no noticeable increase in the gauge reading. This may take several minutes to accomplish. Note the gauge reading and record it in the notebook. A gauge reading of 0.5 inches of mercury less than the reading obtained with V-1 closed may result from contamination of the tubing with a high vapor pressure compound, and should be noted in the notebook.
3. Record the beginning reading on the dry gas meter. With V-4 fully open, and V-5 closed, close V-1 and open V-3 just enough to produce a flow rate of approximately 5 LPM. Note and record the time (to the nearest 10 seconds) that V-3 was opened.
4. Allow the tubing sample to fill with air through the dry gas meter until all noticeable movement of the meter dial has stopped. Then close V-4 and record the ending meter reading in the notebook.
5. Open V-5 and adjust the syringe until the differential pressure gauge reads  $0 \pm 0.05$  inches of water. Note and record the ending time to the nearest 10 seconds.

The total volume measured by the dry gas meter is the difference between the ending and beginning readings corrected by the calibration factor for the dry gas meter. The total measured volume of the tubing is the corrected dry gas meter volume added to the displaced volume (in liters) measured by the syringe. Compute the elapsed time

required to complete the measurement as the difference between the ending and starting times. This will be called the elapsed measurement time.

If the presence of a high vapor pressure compound is suspected an approximate correction may be made as follows:

1. Close V-3 and open V-1. Ensure that V-2 is open. Evacuate the sample section of production tubing until no noticeable increase in the gauge vacuum occurs. Note the gauge reading and record it in the notebook as the starting vacuum.
2. Close V-2 and note the time.
3. Wait for as long as was required to make the preceding volume measurement, e.g. the elapsed measurement time. Then close V-1, isolating the vacuum gauge from the tubing. Note the gauge reading and record this in the notebook as the ending vacuum.

The partial pressure (in inches of Hg) due to the presence of a high vapor pressure contaminant in the tubing ( $P_C$ ) is the difference between the starting and ending vacuum. The partial pressure of the contaminant is subtracted from atmospheric pressure ( $P_A$ ) to yield the partial pressure of air ( $P_a$ ) in the tubing. The best estimate of atmospheric pressure is obtained from the maximum vacuum gauge reading obtained with V-1 closed and V-2 open. A measurement correction factor ( $C_F$ ) is computed by dividing the estimate of atmospheric pressure ( $P_A$ ) by the partial pressure of air in the tubing ( $P_a$ ). The measured volume is then multiplied by this correction factor to provide a corrected estimate of the volume.

#### **4.1.6 Radon Emanation Fraction Measurements**

Thirty samples are to be collected for measurement of the emanation fraction of radon. Fifteen of these samples will be pipe scale samples and 15 will be production facility waste samples (sludge samples). The radon emanation fraction is strongly dependent upon the distribution of the parent radium atoms within the host mineral grain. If the radium is located at or near the grain boundaries, as it often is uranium ores, the emanation fraction can be reasonably large. If, on the other hand, the radium atoms are distributed fairly uniformly throughout the mineral grain, then the radon emanation fraction is likely to be very small because most of the radon atoms that are produced cannot escape the crystal lattice of the host mineral grain before they decay into non-gaseous  $^{218}\text{Po}$ . Therefore, any

process that serves to break down the grain size of the sample material will almost certainly increase the emanation fraction.

It is therefore critically important that the pipe scale and sludge samples be sampled in such a manner as to preserve the grain size and form of the materials of concern. For example, it is of interest to know the typical radon emanation fraction of scale that has been cleaned out of used production tubing. For this measurement to be useful the sample of pipe scale must be obtained in the same manner as that typically used to clean scale from pipe. If several different techniques are employed, and each produces waste material of different grain size, then scale samples should be provided that represent each technique.

The sample selection criteria and sampling methodology detailed in section 4.1.1, with the modifications described above, will be used to obtain the pipe scale samples. The sample selection criteria and sampling methodology detailed in section 4.1.2 will be used to obtain the production facility waste samples, except that no ashing or burning of the samples to remove organic matter will be performed.

#### **4.1.7 Gas pipe $^{210}\text{Pb}$ samples**

Ten samples of gas delivery pipe will be collected from locations where the opportunity for accumulation of  $^{210}\text{Pb}$  is anticipated to be the highest. A simple calculation was performed in order to estimate potential  $^{210}\text{Pb}$  concentrations in gas transmission lines (see Appendix F). This calculation was based on an assumed 24 inch gas transmission line delivering approximately  $3 \times 10^6$  scfd of natural gas and having a borehole radon concentration of 1000 pCi/L. Maximum  $^{210}\text{Pb}$  concentrations were estimated to be about  $1.4 \times 10^6$  pCi per square inch ( $4.8 \times 10^6$  dpm per  $100 \text{ cm}^2$ ) and would occur approximately 3 mi from the borehole. These concentrations represent "worst case" estimates based on a constant radon input rate and equilibrium conditions. It is believed that most of the  $^{210}\text{Pb}$  contamination will be in the "oily" residue deposited on the inside of the gas delivery pipe.

The pipe samples will be collected by GRI member companies for analysis at the Chem-Nuclear Geotech Laboratory in Grand Junction. The samples to be analyzed for  $^{210}\text{Pb}$  will be the same samples on which alpha-track measurements are to be made by the Radon Laboratory (see section 4.1.8). It will be assumed that lead-210 and polonium-210 are in, or close to, secular equilibrium.

#### **4.1.8 Alpha track measurement samples**

The ten gas delivery pipe samples provided by GRI for  $^{210}\text{Pb}$  analysis will also be used for alpha track

analysis to determine the extent of residual  $^{210}\text{Po}$  contamination on the interior surfaces of the pipe. Samples will be selected on the basis of expected elevated NORM levels and on availability of the sample material, and are therefore not based on a valid statistical design. Subsequent radiochemical analysis of the pipe interior where the measurement was made will indicate the level of equilibrium between  $^{210}\text{Pb}$  and  $^{210}\text{Po}$ .

The number of "alpha tracks" recorded on detection film placed on the interior of the pipe will be proportional to the surficial  $^{210}\text{Po}$  activity and therefore, an estimate of the amount of  $^{210}\text{Pb}$  found on the interior of the gas transmission line can be made.

**4.2.8.1 Exposing the Alpha-Track Film** -- The alpha-track detector consists of a strip of Kodak LR-115, Type II, alpha-track registration film. The detector strip is 16 mm wide, with the length varying from 20 to 100 mm depending upon the size and shape of the sample material. The detector strips will be packaged in a holder providing a 20- $\mu\text{m}$  thick layer of polyester to act as a degrader. The degrader is necessary to slow the 5.3-MeV  $\alpha$ -particle from the  $^{210}\text{Po}$  so that it can produce the ionization damage necessary to form a latent track. The detector package will be applied to the sample with the sensitive side of the LR-115, covered by the degrader, against the pipe surface to be sampled. The detector package will be held in place with duct tape.

The initial exposure period will be 24 hrs. This exposure length has been chosen to provide acceptable counting statistics at the track density corresponding to an activity level of 300dpm per 100  $\text{cm}^2$ , which is commonly used as a release limit for items contaminated with transuranic materials. In the event that a 24 hr. exposure period produces a track density that is too high to accurately count ( $> 100$  tracks/ $\text{mm}^2$ ), the exposure will be repeated using a proportionately shorter exposure period.

#### 4.1.9 Gamma surveys

As noted in the above sections, several sample procedures will require that an initial gamma survey of the site be conducted prior to the collection of samples. Gamma surveys fall into three basic categories;

- X Soil
- X Production facility waste drums
- X Pipes in pipe yards.

Soil gamma surveys are to be performed at each pipe yard visited and at 50% of the production facilities visited. It is the responsibility of API to identify which of the production facility sites will be sampled for soil. The purpose of the soil gamma survey is to identify a 100  $\text{m}^2$  area with the highest gamma exposure reading. The

purpose of production facility gamma survey is to define the piece of equipment or waste drum with the highest gamma exposure reading. The purpose of the pipe yard gamma survey is to identify the population of accessible pipes that have the highest gamma exposure reading. Details of the gamma survey procedure are presented in Appendix E and are not repeated in here.

## 4.2 Sample Analysis SOPs

Detailed descriptions of sample preparation and analytical procedures, including counting times and detection limits, are provided for each general type of sample collected for the NORM Characterization Program. Organizations and individuals responsible for sample preparation and analysis are also identified. Where possible, existing analytical SOPs already in place at GJPO will be used and incorporated into the S&A Plan by reference. These existing SOPs are provided in Appendix A.

### 4.2.1 Pipe scale samples

In preparation of analysis, pipe scale samples (including QA/QC samples) will be dried at 110°C, crushed to approximately 3 inch grain size, and ground to 80 mesh. This sieve size that is consistent with the calibration standards used at the analytical laboratory at GJPO. The quantity of ground material to be used for each sample is approximately 1 kg (2.2 lb). They will then be sealed in a can for a period of at least 21 days to allow for ingrowth of radon.

Analysis of pipe scale samples for concentrations of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  will be performed at the laboratory at GJPO using gamma spectroscopy. The GJPO gamma spectral laboratory consists of six intrinsic germanium detectors coupled with a multichannel analyzer and associated electronics. An automatic sample changing system has been installed to enable the system to operate on continuous basis. For a 50 minute count time, the minimum detectable concentration of  $^{226}\text{Ra}$  is 1 pCi/g. For samples with higher activities, the count time will be much shorter. The 609 keV gamma photon from  $^{214}\text{Bi}$  (a  $^{222}\text{Rn}$  daughter product) will be used to determine  $^{226}\text{Ra}$  concentrations. Gamma photons originating from  $^{228}\text{Ac}$  are used to measure  $^{228}\text{Ra}$ . Procedures are in place at GJPO for correcting for density of the material. These procedures will be performed in accordance with established quality assurance procedures currently in place at GJPO.

A detailed SOP for gamma counting at the GJPO is provided in Appendix A (Section A.3, procedure GS-7.4). The current version at time of analysis will be used. If the sample contains enough oil that grinding and blending by the above method cannot be accomplished, the sample will be canned as received with no sample preparation. If sufficient sample volume exists, a duplicate sample will be canned and analyzed to determine the homogeneity of those samples that cannot be prepared.

#### **4.2.2 Production facility samples**

The physical state of samples of production facility wastes will vary, and may include liquids, sludges, emulsions, or solids. If the sample visually appears "oily", then the organic fraction of the samples (including QA/QC samples) will first be removed by API member company laboratory. The original sample mass and the mass of the organic fraction will be recorded on the sample collection form. The sample will then be sent to Chem Nuclear Geotech for analysis of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  concentrations using gamma spectroscopy. Removal of the organic fraction prior to analysis is not expected to affect sample results since radium is not anticipated to be associated with the organic fraction.

#### **4.2.3 Soil samples**

Samples of soil collected at pipe cleaning and storage yards and production facilities will be analyzed using established analytical procedures described in Section 4.2.1 for the pipe scale samples.

#### **4.2.4 Radium solubility samples**

Radium-226 solubility will be determined on a subsample of five of the pipe scale samples found to have elevated radium concentrations. A portion of the sample will be leached with deionized water at a ratio of 10 parts water to one part sample and the resulting water will be analyzed by Chem-Nuclear Geotech procedure C-5. This SOP is contained in Appendix A. The results will be reported in activity of radium-226 per unit weight of sample.

The ASTM procedure will not be used for this measurement because the procedure uses sulfuric acid ( $\text{H}_2\text{SO}_4$ ) to leach the contaminant. The sulfate ion ( $\text{SO}_4$ ) will react with the radium ion to form  $\text{RaSO}_4$  which is extremely insoluble. Therefore little of the radium will be leached from the material.

#### **4.2.5 Whole pipe radon release measurements**

The radon-on-activated charcoal samples generated from the procedures detailed in Section 4.1.5 will be allowed to ingrow for a minimum of 4 hours prior to gamma analysis. The gamma analysis will be performed following the procedures detailed in the Radon Laboratory Technical procedure RNFLUX-U, "Radon Flux Measurements Using the Large Area Activated Charcoal Collector Method". This procedure is found in Appendix A of this document.

Calibration of the gamma counting system which will be used to analyze the radon-on-charcoal samples is provided in the Radon Laboratory Technical Procedure RNFLUX-C, Calibration of a Computer Automated Gamma Counting System for Radon Flux Measurements. These Technical Procedures are included in Appendix D of this sample and analysis plan.

#### **4.2.6 Radon emanation fraction measurements**

The pipe scale and production facility waste samples will be analyzed to determine the radon emanation fraction using the procedures detailed in the Radon Laboratory Technical Procedure RNREF-U, "Measurement of Radon Emanation Fractions". This procedure is found in Appendix A of this document.

#### **4.2.7 Gas pipe $^{210}\text{Pb}$ samples**

Measurement of  $^{210}\text{Pb}$  presents several problems that were discussed in Section 3.5. Because this measurement is not normally performed, several different procedures will have to be experimented with to remove  $^{210}\text{Pb}$  from the interior of the gas transmission line. The approach to be attempted first is to use an organic solvent to remove the "oily" residue from the interior of the transmission line. Once the material has been removed,  $^{210}\text{Pb}$  analysis will be by liquid scintillation counting. The procedure for liquid scintillation counting is described in Appendix A (Method C-6). A limited number of samples are expected to be processed since limited resources have been allocated to this phase of the project and success of the procedure is uncertain. If the initial trials of the procedure are successful, then more samples will be processed following approval by API and GRI to continue.

#### **4.2.8 Alpha track measurement samples**

Following exposure, the detector strips will be processed to reveal the latent tracks produced by the  $\alpha$ -particles. This process is comprised of etching the detector strips in hot sodium hydroxide solution for a specified length of time. This treatment results in a transformation of the latent tracks into holes ranging up to  $15\mu\text{m}$  in diameter. The etching procedure is described in detail in the Radon Laboratory Technical Procedure RN-ATE-U, "Standard Practice for Etching Kodak LR-115, Type II Alpha Track Registration Film", which may be found in Appendix A of this S&A Plan.

To determine a measure of the alpha activity of the sample surface, the net track density of the sample detector strips will be measured. The gross track density (exposed track density plus the background track density)



will be measured using a standard optical microscope with a total magnification of 100x. An ocular reticle that describes an area of 1.00 mm<sup>2</sup> on the specimen will be used as a counting aid.

The total area counted will be 40 mm<sup>2</sup>. This will be accomplished by locating 40 sequential 1-mm<sup>2</sup> fields of view along a traverse which bisects the 16-mm wide detector strip. By examining the film along a line down the center of the film strip, edge effects are avoided, and nonuniform track densities due to hot particles in the sample are averaged over larger distances.

The background track density (tracks/mm<sup>2</sup>) will be determined by counting the number of tracks in a 200-mm<sup>2</sup> area from an unexposed film strip. This background film strip will be taken from the same batch as the regular detector strips. The net track density is determined by subtracting the background track density from the gross track density. This net track density is divided by the detector efficiency to provide an estimate of the alpha decay density from the sample surface.

The area of measure of the ocular reticle will be calibrated by comparison with a stage micrometer. An approximate calibration of the detector efficiency will be obtained by performing a standard exposure of the detector package with a certified <sup>241</sup>Am button source. The certified activity of the <sup>241</sup>Am source is NIST traceable.

The standard exposures will be performed by placing the active side of the button source against the sensitive side of the detector package for a fixed length of time. The detector area will be larger than the active area of the button source so that all of the  $\alpha$ -particles emitted from the front side of the button source will intersect the plane of the detector. The detector strips from these standard exposures will be processed in the same manner as the sample detector strips.

The detector strips from these standard exposures will be analyzed by counting an area large enough to encompass all of the possible tracks produced by the button source. The area will be measured and the total number of observed tracks will be corrected by subtracting an appropriate number of tracks attributable to background.

The total number of decay events from the button source is determined by multiplying the certified activity of the source in becquerels (1 Bq = 1 disintegration per second) by the exposure length in seconds. The detector efficiency is then computed by dividing the total net track count by the total number of decay events from the source during the exposure period. Ten such standard exposures will be conducted, and the average of the individually computed detector efficiencies will be used as the best estimate of the average detector efficiency.

The accuracy with which the detector efficiency can be known is determined by the counting statistics associated with various track measurements and the accuracy with which the exposure length can be controlled. However, this efficiency is strictly valid only for the same geometry and  $\alpha$ -particle energy distribution as that exhibited by the button source. These conditions are not met for the samples for two reasons. First the energy of the  $\alpha$ -particles from the  $^{241}\text{Am}$  source is 5.48 MeV, which is slightly different in energy from the 5.30 MeV  $\alpha$ -particles from  $^{210}\text{Po}$ . Secondly, and more important, the energy distribution of the  $\alpha$ -particles emitted from the button source is relatively narrow by design. In fact, great care is taken to minimize energy loss from the emitted  $\alpha$ -particles by electroplating the parent atoms on the surface of the source. On the other hand, the  $^{210}\text{Po}$  atoms in the sample material are likely to be distributed over a depth range of several microns. This depth distribution is due to the recoil energy imparted to the nucleus from previous decay events. This distribution in depth of the  $^{210}\text{Po}$  atoms will serve to broaden the energy distribution of the  $\alpha$ -particles emitted from the sample surface, which in turn will affect the number of tracks produced for a given exposure.

Since it is beyond the scope of this project to develop an exact efficiency calibration, the above described methodology will be employed with the understanding that the uncertainty of the measurement is not well known, and may be as high as 50%.

### **4.3 Sample Identification and Tracking SOPs**

The purpose of this section of the NORM Characterization Program Sampling and Analysis Plan is to establish procedures for identifying and handling all samples from the time the sample is collected until the analysis is complete and the material has been returned to its point of origin, and for recording the information necessary to accompany each sample. Included are SOPs for Sample Labeling at the time of collection (Section 4.3.1), Sample Documentation and Recordkeeping (Section 4.3.2), and Sample Management (Section 4.3.3). These SOPs are designed to allow for the tracking of samples and to convey information regarding the samples. No formal chain-of-custody procedures are to be applied to this program because the sampling and analysis described in this document is not being conducted in response to any regulatory requirements.

#### **4.3.1 Sample labeling**

The purpose of a sample label is to establish and maintain the identity of the sample. A sample label will be completed in the field by the sample collection personnel for each sample collected, and shall remain with the sample until it has been returned to the facility from which it was collected.

The following information shall be recorded in waterproof ink on the sample label:

1. **Sample ID number:** Sample identification (ID) numbers will be assigned to each physical sample collected. The basic requirement of the sample ID number is that it must be unique and can therefore be used to distinguish any individual sample from other similar samples. This permits the tracing of any sample throughout the process from collection to disposal.

The sample ID number shall be displayed prominently on the sample label, and shall be in the form of a ten- or eleven-digit alphanumeric code. The following form will be used:

XX123YY456R

where:

- ⊘ The first two digits (XX) represent the type of facility from which the sample was collected. These should fit into one of three categories, the designations for which are provided below:

PI = pipe cleaning and storage yard

PF = oil production facility

GA = gas production and delivery system

- ⊘ The next three digits (123) represent sequential numbers assigned by API to the different facilities from which samples are collected. Separate sequences shall be maintained for each facility type, starting with 001. API will maintain records of which facilities are sampled for future reference.

- ⊘ The next two digits (YY) represent a two letter code for the type of sample collected. These should fit into one of six categories, listed below:

SC = pipe scale samples

SO = soil samples

WA = production waste material samples

RP = canisters from radon flux measurements from pipes

RE = radon emanation fraction measurements

GP = gas pipe samples

- ⊘ The next three digits represent sequential numbers assigned in the field to each individual sample of

a particular sample type at a single facility. The sequence should start at 001 for each sample type collected at an individual facility, and should proceed in the order in which the samples are collected.

- ⊚ The final digit (R) represents field replicate samples collected for the purposes of QA/QC, and should be placed only on those samples that have been designated as replicate samples. Non-QA/QC samples should not have any letter or number in the 11th position.

An example of a sample ID number is PF274WA001, representing waste sample number 1 collected at production facility number 274.

2. **Site and Sampling Location:** This information must identify the site or facility at which the sample was collected and should adequately describe the physical location within the facility at which the sample was collected. For pipe scale samples, sampling location must describe the rack, row, and number within the row of the pipe from which the sample was collected. For soil samples and for production facility waste samples, sampling location must include a brief description of where within the facility the sample was collected. The purpose for including this information is to allow for the relocation of the sample site at a later date.
3. **Sample Type:** Applicable types of sample include pipe scale, soil, radon emanation, gas delivery system pipe, and production facility waste.
4. **Time and Date of Collection:** The time the sample was collected must be recorded using a 24-hour clock (e.g. 1445 for 2:45 p.m.). The date of collection must be noted by day-month-year (e.g. 09-AUG-93).
5. **Sampler:** The individual in charge of collecting the sample must be identified so that any questions regarding the collection of the sample may be properly directed.

An example of a sample label is provided in Figure 4.2.

### **4.3.2 Sample documentation and recordkeeping**

Recordkeeping and documentation of field activities is a necessary component of any monitoring and assessment program. A written record of all field data, activities, and observations is important for several reasons, including:

- ⊃ Ensuring the timely, correct, and complete analysis for all parameters required for a given sample;

**Figure 4.2**      **Example Sample Label**

<b>NORM CHARACTERIZATION PROGRAM</b>		
<b>SITE AND SAMPLING LOCATION:</b>		
<b>SAMPLE TYPE:</b>		
<b>DATE COLLECTED:</b>	<b>TIME COLLECTED:</b>	<b>SAMPLER:</b>
<b>SAMPLE IDENTIFICATION NUMBER:</b>  -----		

- ☐ Satisfaction of applicable the QA/QC requirements;
- ☐ Ensuring that all essential information acquired in a consistent manner, and is preserved for current use and future reference;

These records provide the basis for decisions regarding the existence of environmental problems at the sampling areas.

Maintaining standardized records facilitates the collection of the data required to conduct site activities. Because samples will be collected by many different individuals, field logbooks will not be used for this program. However, standardized forms will be used to ensure that the necessary information is recorded consistently for each sample collected. These forms will be completed by the sample collection personnel in the field at the time the samples are collected. As with the sample labels, these forms will be completed using waterproof ink. If weather conditions prohibit the use of ink, a pencil may be used, but the reason for its use must be noted on the form. If an error is made on any of the sample forms, a correction will be made by drawing a single line through the error and entering the correct information. All such corrections must be initialed and dated. An example of the data collection form is provided in Figure 4-3. Serial numbers will not be used on the forms for this program.

Much of the information to be provided on the sample collection forms will be identical to that provided on the sample label. This will include:

1. Name, affiliation, and address of field contact (lead individual in the sample collection process);
2. Names of additional personnel on sampling team;

Figure 4.3 Example Sample Collection Form

NORM CHARACTERIZATION PROGRAM  
SAMPLE COLLECTION FORM

Sample Identification Number \_\_\_\_\_

Collection Date \_\_\_\_\_ Collection Time \_\_\_\_\_  
Day Month Year

Sampler \_\_\_\_\_

Name Affiliation

Company Coordinator \_\_\_\_\_

Name Affiliation

Sample Form Checked By \_\_\_\_\_ Name

Sample collection SOP followed (Ref. #) \_\_\_\_\_

Facility Sampled (Name, Location) \_\_\_\_\_

Specific Location of Sample \_\_\_\_\_

Sample Type \_\_\_\_\_ Sample Quantity \_\_\_\_\_

Pertinent Field Measurements and Observations \_\_\_\_\_

\_\_\_\_\_ Does the sample contain  
oily material? \_\_\_\_\_

If the above question is yes the complete the following (Laboratory only)

Sample mass before removal of oily material \_\_\_\_\_ grams

Sample mass after removal of oily material \_\_\_\_\_ grams

Net mass of oily material \_\_\_\_\_ grams

Person responsible for removal of oily material \_\_\_\_\_

Affiliation \_\_\_\_\_

Sample Destination and Hauler \_\_\_\_\_

Name and Address to Return Sample

\_\_\_\_\_  
Name

\_\_\_\_\_  
Address



3. Identification, by reference (section number), of the Sample Collection SOP followed:
4. Location of the facility and the location within the facility at which the sample was collected;
5. Type of sample collected (e.g., soil, sludge, waste water, scale, radon canister, etc);
6. Quantity of sample collected (in terms of approximate sample mass or volume).
7. Time (24-hour) and Date (day-month-year) of Collection;
8. Sample ID Number;
9. Field measurements and observations associated with each sample collected (e.g., counts/min, estimated radium concentration, etc.);
10. Sample destination and mode of transportation (e.g. name of laboratory,hauler);
11. Gamma survey results, including sketch of facility grounds indicating locations of elevated gamma readings;
12. References, such as maps or photographs of the sampling site, if appropriate;
13. Address to which the sample should be sent following analysis.

Additional information will be required for each type of sample (e.g. sample depth for soil samples, volume of the sample, container type, etc. Suspected waste composition, including concentrations.). This information will be stipulated in the Sample Collection SOPs for each specific type of sample. The sample collection form should be signed by the collector and the individual who performs the QA check on the completed form. A copy of this form shall accompany each sample sent to GJPO for analysis. GJPO shall file the form and submit all sample collection forms to EG&G Idaho at the completion of all analytical activities associated with the program.

#### **4.3.3 Sample management:**

Although no formal chain of custody procedures will be used, some basic standard sample handling procedures will be applied, and are described in this section. The purpose of establishing sample management

procedures is to enable the tracking of samples. Each sample tracking system must ensure:

- ☐ that all samples are collected according to the Sampling and Analysis Plan; and
- ☐ that samples can be traced from sample collection through laboratory analysis, and to the point when the data are entered into the Data Management System.

As part of the tracking system, these procedures provide a traceable record from analytical laboratory results back to the proper field sample, through the following sequence of events:

- ☐ Field sampling;
- ☐ Shipping;
- ☐ Receipt of samples at the Analytical Laboratory;
- ☐ Sample preparation;
- ☐ Sample analysis;
- ☐ Disposal of samples and residual waste material;

The sample shipping record identifies the samples shipped to the laboratory for analysis and the return of the samples to their point of origin. The sample shipping form should include the following information:

- ☐ Sample ID Number(s) contained in the shipment;
- ☐ Number of Containers: Indicate the total number of containers used for the respective sample ID number(s).
- ☐ Shipped By/Received By: Indicate the individual who ships the samples and the individual accepting the possession and custody of the sample(s). Also, include the date and time of the transfer.

☐ Maximum surface gamma readings for each container.

An example of the shipping form is provided in Figure 4.4. the same form should be used to send samples back to their origin after analysis.

Shipping of all samples collected as part of the NORM Characterization Program will be by common carrier. None of samples collected need refrigeration or other precautions for the preservation of the samples, since radioactivity is the measure of interest.

Figure 4.4 Sample Shipping Form

NORM CHARACTERIZATION PROGRAM

SAMPLE SHIPMENT FORM

Shipped By:

Name: \_\_\_\_\_

Affiliation: \_\_\_\_\_

Address: \_\_\_\_\_

Received By:

Name: \_\_\_\_\_

Affiliation: \_\_\_\_\_

Address: \_\_\_\_\_

Date Shipped: \_\_\_\_\_

Day Month Year

Date Received: \_\_\_\_\_

Day Month Year

Number of Containers: \_\_\_\_\_ Shipped \_\_\_\_\_ Received \_\_\_\_\_

Sample I.D. Numbers:

_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Maximum Container Surface Gamma Readings: \_\_\_\_\_

Soil samples will be placed in wide mouth plastic jars, and packed with other similar samples in such a manner as to prevent breakage within the shipping container. The shipping container will be sealed, and shipping papers identifying the contents of the container placed within the container. For commercial shipping, the container will be labeled "This End Up" and "Environmental Samples", and transported as required with no Department of Transportation shipping restrictions.

## **4.4 Data Management SOPs**

The need for sample and document management procedures is mandated by several areas of concern, including:

- ⊘ To establish the authenticity of the evidence collected;
- ⊘ To facilitating interpretation of sampling and analysis results; and
- ⊘ To standardize data entries for input into the data management system.

Data management and analysis has two major objectives:

- ⊘ To ensure rapid and error-free transmission of data from the point of generation through several layers of verification and validation to the point of placement final report; and
- ⊘ To provide a data management system (DMS) that accommodates the final storage and archiving of the data generated during the program.

Because samples analyzed as part of the NORM characterization program will be of a number of distinct types, and collected from a many different sites, data management issues must be clearly identified and addressed by developing and adhering to data management procedures. Consistent implementation of these data management procedures by all participants of the program will ensure reliable, consistent, and effective flow, treatment, and storage of the data.

The NORM program will include two types of data: field sampling data and analytical laboratory data.

#### **4.4.1 Field sampling data**

Data associated with field sampling includes any measurements conducted in the field, such as external gamma measurements on pipes or pipe scale samples, etc., as well as supplementary information such as the locations of field samples, detailed field conditions, and instrument methods employed. This information must be available to the analytical laboratory for use in interpreting the analytical data. Existing data from the earlier gamma survey are included.

Procedures have been developed for receiving, logging, and entering the appropriate field information. The form and content of all Sample Log Sheets have been determined, and a standard set of measurements will be taken for each sample type consistent with the Sample Log Sheets. The Sample Log Sheets and the Chain-of-Custody (COC) Records must be reviewed and prepared for data entry.

#### **4.4.2 Laboratory data**

Data associated with the analytical laboratory includes any and all data resulting from analyses conducted at GJPO at Grand Junction, as well as any data generated at other analytical laboratories as part of the QA/QC procedures. Where appropriate, procedures for entering or reformatting the instrument output into a standardized format for providing basic analytical data for each of the analytical methods have been developed.

Laboratory and field data will be delivered by GJPO in an ASCII readable format. Copies of all sample forms shall be sent to EG&G Idaho. Analytical results from GJPO shall also be sent to EG&G upon completion for entry into the database.



## **4.5 Waste Disposal SOPs:**

All unused sample material will be returned to its source after completion of the required analysis. It will be the responsibility of GJPO to maintain records regarding the source of each sample, so that the sample may be returned. The appropriate return address and the name of the person responsible for receiving the remaining and used portions of the sample will be provided a sample collection form.





## 5. Responsibilities

The purpose of this section of the Sampling and Analysis Plan is to define the responsibilities of the various organizations involved with the NORM Characterization Program. The primary responsibility for each task within the program (e.g. sample collection, analysis, data management, QA/QC, report writing, program management, etc.) has been assigned, and the key individuals involved from each organization has been identified wherever possible.

### 5.1 Program Development and Overall Responsibility:

EG&G Idaho, Inc., a contractor for the U.S. Department of Energy at the Idaho National Engineering Laboratory, is charged with the overall responsibility for the NORM Characterization Program. This responsibility includes the development of the overall program, including the writing of this Sampling and Analysis Plan, and the coordination between participating organizations. Within EG&G, the following key individuals have been identified:

**EG&G Project Manager:** Dr. Charles Thomas has been identified as the Project Manager from EG&G Idaho. As such, he is responsible for the overall management of the project. His duties include maintaining an interface with DOE-ID, submitting bimonthly progress reports to API, managing and tracking the Program budget, and solving management level problems relating to the Program.

**EG&G Principal Investigator:** Arthur Rood has been designated as the Principal Investigator from EG&G Idaho. The Principal Investigator is responsible for all technical aspects of the project, including technical management of the portion of the program conducted by GJPO. The technical responsibility includes preparation of the Sampling and Analysis Plan, field testing of the Plan, technical coordination with API representatives and with GJPO, managing the data resulting from the Program, and preparation of the final report.

Other key individuals from EG&G Idaho include Gregory White and Alan Crockett, who will be responsible for various technical aspects of the Program.

Responsibilities for program management have also been assigned for the three primary funding organizations, API, GRI and DOE. Program management at API and GRI will be the responsibility of Mark Rubin

and Jim Evens respectively. API technical management of the project is the responsibility of Irwin Supernaw. Program Management at the DOE Fossil Energy office at Metairie, Louisiana has been assigned to Dr. Brent Smith.

## 5.2 Sample Collection

Because most of the samples collected as part of the NORM Characterization Program will be collected at facilities operated by American Petroleum Institute member companies, sample collection will largely be the responsibility API. Exceptions include the collection of radon flux samples, which will be the responsibility of GJPO personnel, and the collection of gas delivery pipe samples for  $^{210}\text{Pb}$  analysis, which will be the responsibility of GRI personnel.

Responsibility for determining sampling site locations will be shared between the different organization. The initial determination of the general geographic locations at which samples will be collected will be determined by EG&G Idaho. Specific facilities at which the samples will be collected within the geographic areas identified by EG&G will be determined by API for oil production facilities and pipe cleaning and storage yards, and by GRI gas pipe samples. Sampling locations within the facilities will be determined by API (or GRI) personnel using guidelines described in the Sampling and Analysis Plan.

The following individuals have been identified as having the primary responsibility for coordinating sample collection for their respective organizations:

API	Mark Rubin 1201 Main Street Suite 2535 Dallas, TX 75202-3994 (214) 748-4436
EG&G	Arthur Rood PO Box 1625 Idaho Falls ID 83415 (208) 526-1678
GRI	James Evens 8600 West Bryn Mawr Avenue Chicago IL 60631 (312) 399-8329

GJPO Ron Chessmore (Analytical Laboratory)  
2597 B 3/4 Road  
Grand Junction CO 81503  
(303) 248-6166

GJPO Tom Kendrick (Radon Laboratory)  
2567 b 3/4 Road  
Grand Junction CO 81503  
(303) 248-6273

### **5.3 Sample Analysis**

All analysis of samples collected for the NORM Characterization Program, electronic form, ASCII and hardcopy of results, will be the responsibility of the GJPO, operated by Chem-Nuclear Geotech. The Program Manager at GJPO will be Ron Chessmore.

### **5.4 Equipment and Materials**

The purpose of this section of the Sampling and Analysis Plan is to identify the equipment and materials needed to collect each type of sample, and the organization responsible for providing it. This information is provided below in Table 5.1. Equipment and materials needed to analyze the samples are not included in Table 5.1, as all analytical needs are the responsibility of Chem-Nuclear Geotech.

### **5.5 Training**

The lead person responsible for the collection of samples at any given site will be trained prior to collecting samples. This training will include:

- ! application of the appropriate SOPs;
- ! conducting the required gamma surveys
- ! filling out the appropriate paperwork

! packing and shipping the samples

**Table 5.1.** Equipment and materials needed to collect samples for the NORM Characterization Program, and organization responsible for providing them

<u>Item</u>	<u>Responsible Organization</u>
Exposure rate survey meters and check sources	API member company
Equipment for removing scale from pipe	API member company
Soil Core Samplers	API member company
Coliwasa Sampler and core tubes	API member company
Sample Collection Jars	API member company
Shipping Containers	API member company
Tape measures and compass	API member company
Sample labels	EG&G
Sample Collection Forms	EG&G
Sample Shipping Forms	EG&G

EG&G Idaho will be responsible for providing written instructions for the collection of soil, scale, and waste samples, conducting facility gamma surveys, documentation and shipping. Because of the potentially large number of individuals that could be involved in the sample collection nationwide, training by EG&G personnel may be limited to a one-time training session for key API personnel. Subsequent training of additional individuals would then become the responsibility of API.

## **5.6 Data Management**

With the exception of any information determined by the funding agencies to be proprietary, all data management activities for the NORM Characterization Program will be the responsibility of EG&G Idaho. The Principal Investigator (Arthur Rood) will be the individual responsible for this task. Analytical data management will be coordinated with GJPO (Ron Chessmore and Tom Kendrick).

Proprietary information including the specific locations of facilities at which sampling will be conducted, identifies, and maintained by API.

## **5.7 Reporting Requirements**

Bimonthly progress reports for the NORM Characterization Program will be submitted to API and GRI. The responsibility for the preparation of these reports will be with the EG&G Project Manager (Charles Thomas). These reports will describe the current status of the Program, accomplishments achieved since the previous report, expected accomplishments prior to the next report, and the program budget. The bimonthly reports will include input from GJPO, which will be coordinated by the EG&G Principal Investigator. Copies of the bimonthly progress reports will be provided to GJPO and to the DOE Fossil Energy office in Metairie, Louisiana.

Brief monthly progress reports will also be submitted to the Idaho Operations Office of the DOE, as required by contract. These reports will be the responsibility of the EG&G Project Manager. These reports are provided to the DOE-ID project monitor responsible for overseeing the Program.

The Final Report containing the data and resulting conclusions will be prepared by EG&G, as described in the Scope of Work. Input to the report will be provided by API, GRI, DOE, and GJPO. The overall responsibility for preparing this report has been assigned to the EG&G Project Manager.

## **5.8 Waste Disposal**

All waste materials generated during the NORM Characterization Program, including counted sample and unused (excess) sample material, will be returned to the location from which the sample was collected. The responsibility for this will be with the analytical laboratory at GJPO. All applicable state and federal regulations regarding shipment of these materials will be complied with, as described in Section 4.5 (Waste Disposal SOPs) of the Sampling and Analysis Plan.

## **5.9 Summary of Responsibilities**

The responsibilities for each aspect of the NORM Characterization Program are summarized by organization in Table 5.2 below.

**Table 5.2 Summary of responsibilities for the NORM Characterization Program.**

<u>Activity</u>	<u>Responsible Organization</u>
<b>Sample Collection:</b> (includes field selection for sampling sites, filling out all appropriate forms and shipping the samples to the analytical laboratory).	
Pipe scale samples:	APT*
Production facility waste samples:	APT*
Soil samples:	APT*
Pipe to be measured for radon release:	APT*
Gas transmission line samples:	GRI*
Facility Gamma Surveys:	APT*
Facility Maps/Sketches:	APT*
<b>Sample Analysis:</b>	
Soil and scale samples for radium:	Chem-Nuclear, Analytical Services
Radon release from pipe	Chem-Nuclear, Radon Laboratory
Radon emanation fraction	Chem-Nuclear, Radon Laboratory
Return of samples to point of origin	Chem-Nuclear, Analytical Services
Electronic transfer of data from analysis	Chem-Nuclear, Analytical Services
<b>Program Development:</b>	
Data Management and Statistical Analysis	EG&G
Providing Equipment for Sample Collection	API
Training of Field Collection Personnel	EG&G and API (See Section 5.5)
Writing Final Report	EG&G

\* Personnel to come from GRI and API member companies, not API and GRI staff.





## 6. Schedule

The schedule for the completion of the NORM Characterization Program is as shown in Table 6-1.

**Table 6.1. Sampling and analysis schedule for the NORM Characterization Program.**

<b>TASK</b>	<b>START DATE</b>	<b>COMPLETION DATE</b>
Complete Draft Sampling and Analysis Plan	05/01/92	04/01/93
Review and Field Test Sampling and Analysis Plan	04/01/93	06/01/93
Receive Samples from API	06/01/93	10/01/93
Sample Analysis	06/01/93	12/01/93
Conduct Radon Work	06/01/93	10/01/93
Prepare Final Report	12/01/93	05/01/94



## 7. References

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**Appendix B**

**RADIONUCLIDE CONCENTRATION DATA**



Table B-1. Radium concentrations and isotopic ratios for pipe scale samples.

Sample ID #	<sup>226</sup> Ra (pCi/g)	<sup>228</sup> Ra (pCi/g)	Ratio <sup>226</sup> Ra / <sup>228</sup> Ra
PI002SC001	7400	4296	1.72
PF002SC001	895	1508	0.59
PF002SC002	2150	2955	0.73
PF002SC003	1978	2992	0.66
PF002SC004	1941	2789	0.70
PF002SC005	2156	2999	0.72
PF002SC006	398	513	0.78
PF002SC007	425	534	0.80
PF002SC008	2748	4064	0.68
PF002SC009	1042	1623	0.64
PF002SC010	2322	3619	0.64
PF002SC011	2224	3818	0.58
PF002SC012	2629	4039	0.65
PF002SC013	717	1313	0.55
PF002SC014	2338	3605	0.65
PF002SC015	2762	4050	0.68
PF002SC016	707	1295	0.55
PF002SC017	2437	3974	0.61
PF002SC018	658	1364	0.48
PF002SC019	2257	3359	0.67
PF002SC020	592	1503	0.39
PF002SC011R	2225	3682	0.60
PF002SC017R	2620	4125	0.64
PF010WA001	139	46	2.99
380 DR8	5	1	3.21
381 DR21	214	46	4.66
384 DR204	1143	271	4.22
390 DR11	67	16	4.18
393 DR10	15	3	4.33
394 DR11	27	4	7.30
396 DR60-a	293	82	3.56
396 DR60-b	757	156	4.85
399 DR70	532	108	4.94
400 DR34	290	77	3.78
406 DR9	6	2	3.51
<b>MEAN</b>	<b>1403</b>	<b>1852</b>	<b>2.00</b>
<b>STD</b>	<b>1435</b>	<b>1658</b>	<b>2.00</b>



Table B-2. Radium concentrations and isotopic ratios for soil samples.

Sample ID #	<sup>226</sup> Ra (pCi/g)	<sup>228</sup> Ra (pCi/g)	Ratio <sup>226</sup> Ra / <sup>228</sup> Ra
PF001SO001	6.7	4.3	1.5
PF001SO002	154.7	29.9	5.2
PI002SO001	7.8	1.0	7.8
PI002SO002	8.1	59.6	0.1
PI002SO003	14.4	10.3	1.4
PI002SO004	2.5	0.4	6.3
PF007S0001	<mdc	0.7	
PF007S0002	<mdc	<mdc	
PF008S0001	1.1	1.1	1.0
PF008S0002	<mdc	0.3	
PF101S0001	1.2	0.9	1.3
PF101S0002	0.8	1.0	0.8
PF102S0001	173.7	8.3	21.0
PF102S0002	149.0	11.9	12.5
PF103S0001	103.8	25.2	4.1
PF103S0002	178.8	32.2	5.6
PF011S0001	64.3	<mdc	
PF011S0002	39.6	<mdc	
PF012S0001	2.9	1.4	2.0
PF012S0002	3.7	2.0	1.9
PF013S0001	709.8	30.4	23.3
PF013S0002	562.6	23.9	23.6
PF270S0001	1.6	2.0	0.8
PF270S0002	1.8	2.6	0.7
PF006S0001	1.2	1.7	0.7
PF006S0002	0.4	1.1	0.4
Hackberry S-1	1.9	1.6	1.2
Hackberry S-2	30.5	20.0	1.5
Thornwell S-1	24.5	11.7	2.1
Thornwell S-2	72.3	35.4	2.0
PF014WA002	286.4	8.4	34.1
PF014WA003	72.6	3.1	23.2
PF014SO001	18.4	2.2	8.4
PF014S0002	1.3	0.6	2.3
PF014S0003	13.1	2.0	6.4
PF014S0004	3.0	1.0	3.0
PFxxxS0001	<mdc	1.6	
PFxxxS0002	<mdc	1.8	
PFxxxS0003	<mdc	1.8	
PFxxxS0004	<mdc	1.9	
PFxxxS0005	<mdc	1.6	
PFxxxS0006	<mdc	1.5	
<b>Mean</b>	<b>82.3</b>	<b>8.9</b>	<b>6.7</b>
<b>STD</b>	<b>159.8</b>	<b>13.3</b>	<b>8.9</b>

Table B-3. Radium concentrations and isotopic ratios for waste samples.

Sample ID #	<sup>226</sup> Ra (pCi g <sup>-1</sup> )	<sup>228</sup> Ra (pCi g <sup>-1</sup> )	Ratio <sup>226</sup> Ra / <sup>228</sup> Ra
PF004WA001	96	39.8	2.40
PF004WA002R	105	37.6	2.78
PF004WA003	122	51.6	2.37
PF008WA001	<mdc	<mdc	
PF101WA001	1	4.7	0.27
PF103WA001	264	70.6	3.74
PF011WA001	532	16.8	31.70
PF12WA001	229	111.6	2.05
PF014WA001	207	123.5	1.68
PF251SO001	124	1.2	103.51
PF264WA003	148	48.3	3.06
PF264WA002	97	29.7	3.27
PF264WA001	200	54.5	3.67
PF264WA004	309	97.3	3.18
351 DR8	<mdc	<mdc	
355 DR8	2	<mdc	
358 DR100	331	69.2	4.79
363 DR13	67	14.6	4.61
366 DR8	15	9.9	1.47
368 DR245	1560	443.2	3.52
374 DR25	123	31.1	3.96
375 DR220	1103	250.3	4.41
383 DR65	238	54.6	4.36
387 DR8	6	1.8	3.29
388 DR8	2	1.0	2.43
389 DR8	2	0.2	9.50
398 DR55	70	17.6	3.99
405 DR46	237	67.9	3.50
Mean	238	65.9	8.54
STD	352	95.4	20.64