Testing Guidelines for Technetium-99 Adsorption on Activated Carbon

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Contractor for the U.S. Department of Energy
under Contract DE-AC06-08RL14788

CH2MHILL
Plateau Remediation Company

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Richland, Washington 99352

Approved for Public Release;
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Program/Project: S&GRP

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Terms

ASL	CH2M HILL Applied Sciences Laboratory
ASTM	American Society for Testing and Materials
CERCLA	<i>Comprehensive Environmental Response, Compensation, and Liability Act of 1980</i>
CHPRC	CH2M HILL Plateau Remediation Company
COC	contaminant of concern
EPA	U.S. Environmental Protection Agency
ERDF	Environmental Restoration Disposal Facility
GAC	granular activated carbon
gpm	gallons per minute
IX	ion exchange
Kd	distribution coefficient
OU	operable unit
pK	acidity constant
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
QC	quality control
RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
TCE	trichloroethene
Tri-Party Agreement	<i>Hanford Federal Facility Agreement and Consent Order</i>
VOC	volatile organic compound
WAC	<i>Washington Administrative Code</i>

1 Introduction

CH2M HILL Plateau Remediation Company (CHPRC) is currently evaluating the potential use of activated carbon adsorption for removing technetium-99 from groundwater as a treatment method for the Hanford Site's 200 West Area groundwater pump-and-treat system. The current pump-and-treat system design will include an ion-exchange (IX) system for selective removal of technetium-99 from selected wells prior to subsequent treatment of the water in the central treatment system. The IX resin selected for technetium-99 removal is Purolite¹ A530E. The resin service life is estimated to be approximately 66.85 days² at the design technetium-99 loading rate, and the spent resin must be replaced because it cannot be regenerated. The resulting operating costs associated with resin replacement every 66.85 days are estimated at \$0.98 million/year. Activated carbon pre-treatment is being evaluated as a potential cost-saving measure to offset the high operating costs associated with frequent IX resin replacement.

This document is preceded by the *Literature Survey of Technetium-99 Groundwater Pre-Treatment Option Using Granular Activated Carbon* (SGW-43928), which identified and evaluated prior research related to technetium-99 adsorption on activated carbon. The survey also evaluated potential operating considerations for this treatment approach for the 200 West Area. The preliminary conclusions of the literature survey are as follows:

- Activated carbon can be used to selectively remove technetium-99 from contaminated groundwater.
- Technetium-99 adsorption onto activated carbon is expected to vary significantly based on carbon types and operating conditions. For the treatment approach to be viable at the Hanford Site, activated carbon must be capable of achieving a designated minimum technetium-99 uptake.
- Certain radionuclides known to be present in 200 West Area groundwater are also likely to adsorb onto activated carbon.
- Organic solvent contaminants of concern (COCs) will load heavily onto activated carbon and should be removed from groundwater upstream of the activated carbon pre-treatment system. Unless removed upstream, the adsorbed loadings of these organic constituents could exceed the land disposal criteria for carbon.

The literature survey presented the following conceptual approach for technetium-99 pre-treatment using activated carbon (SGW-43928):

- The major process operations, in order of treatment, would be as follows:
  1. Influent equalization tank
  2. Air stripper to remove volatile organic carbon compounds
  3. Granular activated carbon (GAC) system for technetium-99 removal
  4. IX resin system for technetium-99 polishing.

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¹ Purolite® is a registered trademark of The Purolite Company, Bala Cynwyd, Pennsylvania.

² Calculation 382519-CALC-009, *Hanford - 200 West, Phase II - Tc-99 Leakage Analysis from 2-Stage Ion Exchange at 200 West*, Rev. 1, CH2M HILL Plateau Remediation Company, Richland, Washington.

- Effluent water from the IX system would be sent to the central treatment plant for further treatment. Gas from the stripper vent would be sent to a vapor GAC system (likely at the central treatment system) for further treatment. Waste GAC would be stabilized with Ceramicrete³ or another fitting stabilization material with a neutral pH, placed in containers, and transferred to the Environmental Restoration Disposal Facility (ERDF) for disposal.

Recommendations from the literature survey included laboratory testing to confirm the principal conclusions and continuing investigations into the key remaining unknown factors. As such, the purpose of this document is to provide guidelines for the bench-scale testing that was identified as the next step based on the literature survey's recommendations.

This document presents an overview of the 200 West Area groundwater characteristics, applicable test methods that have been used in previous research studies, and recommended test approach to use for testing of technetium-99 adsorption on activated carbon. The recommended testing to be performed consists of three phases:

- Phase 1: Carbon screening and selection
- Phase 2: Batch isotherm adsorption testing
- Phase 3: Continuous-flow column adsorption testing.

A detailed test plan will be prepared for Phase 2 and Phase 3 adsorption testing. At the conclusion of Phase 1 carbon screening, the laboratory will prepare a data report that will be used to determine which carbon materials should be selected for Phase 2 batch isotherm adsorption testing. Similarly at the conclusion of Phase 2 testing, the laboratory will prepare a data report that will be used to determine which carbon materials should be selected for Phase 3 continuous-flow column adsorption testing. At the conclusion of Phase 3 testing, a final report will be prepared to summarize the adsorption testing results and conclusions from Phases 2 and 3.

Key objectives of the bench-scale testing described in this document are as follows:

- Identify commercially available activated carbon products that could be used in a technetium-99 pre-treatment system.
- Establish loading capacity of technetium-99 onto the candidate activated carbons.
- Evaluate difference in technetium-99 loading capacities observed for batch and continuous-flow column testing.

The test results will be used by CHPRC to perform future economic and technical feasibility analyses to determine if the activated carbon pre-treatment option for technetium-99 should be pursued further. If the results of the cost and technical evaluation indicate that the carbon pre-treatment option is feasible, then a pilot-scale demonstration would need to be conducted on a side stream within the radiological building or at a well in order to obtain design data prior to full-scale implementation. In addition, economic and technical evaluations and testing related to spent GAC stabilization and disposal at the ERDF would also be required prior to implementation.

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³ Ceramicrete® is a registered trademark of Argonne National Laboratory and the University of Chicago, Chicago, Illinois. Note that the Ceramicrete trademark only applies to one version of the many possible formulations, namely Mg-K-PO₄ matrix combined with fly ash.

This document does not include testing guidelines for other technologies that could be used for technetium-99 removal from groundwater, such as sulfur-modified iron or other resins. A literature survey and test plans for these other treatment options are being addressed separately.

2 Characteristics of Hanford Site 200 West Area Groundwater

Table 2-1 presents the estimated influent water quality for a hypothetical technetium-99 activated carbon pre-treatment system. This is the same water quality assumed for the influent water to the technetium-99 IX system for the 200 West Area pump-and-treat system design. The water quality data presented in Table 2-1 are discussed in Sections 4.2 and 4.3 when selecting groundwater samples for the testing. A similar table was presented in the literature survey document (SGW-43928), but Table 2-1 has been updated to incorporate up-to-date information. The following table includes revised information for influent concentrations of COCs, non-COC radionuclides, pH, temperature, and flow rate.

Table 2-1. Estimated Source Water Quality for Technetium-99 Pre-Treatment Activated Carbon System

pH (average) ^a	7.7
Raw well water temperature (°F) ^a	65
Recommended influent water design range (°F) ^b	40 to 90
Flow rate (gpm) ^a	670
Technetium-99 (activity)	14,457 pCi/L
Iodine-129 (activity)	0.899 pCi/L
Tritium (activity)	24,071 pCi/L
Uranium	3.93 µg/L
Carbon tetrachloride	506 µg/L
Nitrate (as nitrogen)	84 mg/L
Trichloroethene	3.29 µg/L
Hexavalent chromium	161 µg/L
Total chromium	161 µg/L
Americium-241 (activity)	0.5 pCi/L
Carbon-14 (activity)	13.1 pCi/L
Cesium-137 (activity)	5.2 pCi/L
Cobalt-60 (activity)	15.2 pCi/L
Neptunium-237 (activity)	0.1 pCi/L
Nickel-63 (activity)	7.1 pCi/L
Plutonium-239/240	0.5 pCi/L
Selenium-79 (activity)	783.6 pCi/L
Strontium-90 (activity)	12.4 pCi/L

Table 2-1. Estimated Source Water Quality for Technetium-99 Pre-Treatment Activated Carbon System

a. Calculation 382519-CALC-050, *Integrated Mass Balance Calculation*, Rev. 1, dated February 4, 2010, CH2M HILL Plateau Remediation Company, Richland, Washington (Stream Tc-2 – Influent to Tc-99 IX). Nonradionuclide constituents of interest and common salts reported in the mass balance calculation are not shown. Nitrate value shown in the mass balance calculation was reported as 372,328 µg/L as nitrate. This value was converted to 84 mg/L as nitrogen, as shown in the table.

b. Recommended design temperature range from Table 3 in 382519-TMEM-004, *Basis of Design Technical Memorandum*, Rev. 4.

COC = contaminant of concern

gpm = gallons per minute

The literature survey provided additional points for consideration. Previous research conducted for technetium-99 adsorption onto activated carbon showed a wide range of distribution coefficients (Kds) (in mL/g). The Kds in the technical literature that were deemed most applicable to the 200 West Area source groundwater ranged between 1,204 and 35,476 mL/g. As stated in the literature survey, a target minimum Kd value of 9,008 mL/g was derived based on the assumption that carbon pre-treatment would be attractive if (1) activated carbon could handle the bulk of the technetium-99 removal, and (2) carbon replacement costs were 50 percent of the expected resin replacement costs. Assuming a 50 percent cost savings, the Kd (9,008 mL/g) that would achieve the necessary carbon replacement frequency and costs was calculated. The cost-savings calculation was based only on the assumed activated carbon costs (\$45/ft³) and resin costs (\$300/ft³). Evaluating testing results against this target Kd of 9,008 mL/g will be an important goal of the testing identified in this document. A more detailed cost evaluation will be required after bench-scale testing to evaluate these cost savings based on the technetium-99 adsorption capacity determined experimentally.

The literature survey also evaluated other radionuclides that may adsorb onto activated carbon. Radionuclide constituents assumed to adsorb onto activated carbon included iodine, cobalt, cesium, and strontium. The literature survey results for uptake of uranium and neptunium were not conclusive and will need to be identified during future testing. Americium, carbon-14, and plutonium were not considered in the literature survey because they were not considered to be radionuclide constituents of interest at the time of the survey; therefore, the adsorption characteristics of these three radionuclides onto activated carbon have not yet been investigated. Finally, protactinium was considered to be a constituent of interest in the literature survey but has since been removed as a radionuclide constituent of interest for the project and, as such, this radionuclide will not be addressed as part of testing. Protactinium was replaced by plutonium-239/240 and, as previously stated, was not investigated because it was included after the conclusion of the literature survey. If laboratory batch and column testing results for technetium-99 adsorption are successful, then the evaluation of radionuclide adsorption onto activated carbon should be addressed during future testing.

The literature survey showed that organic, solvent COCs in groundwater will need to be considered. Evaluations in the literature survey showed that solvent COC loadings could exceed disposal criteria after a short period of operation; therefore, the solvents will need to be removed from groundwater prior to treatment with activated carbon. In a full-scale system, an air stripper is recommended for upstream removal of these volatile organic compounds (VOCs). In this testing application, solvent removal from the groundwater test water prior to carbon testing will also be necessary.

Chromium may also adsorb onto activated carbon and may compete with technetium-99 for adsorption, which will also need to be considered during carbon testing, if present in test water.

3 Test Method Discussion

This chapter includes a discussion of test methods used in previous research studies to evaluate technetium-99 adsorption on activated carbon. Section 3.1 presents research findings regarding carbon selection for technetium-99 removal. Section 3.2 presents research findings for batch adsorption testing of technetium-99 on activated carbon. The research findings for continuous-flow methods to evaluate technetium-99 adsorption on activated carbon are discussed in Section 3.3.

3.1 Carbon Media Screening

Previous research has evaluated carbon sources that may be attractive for use in technetium-99 adsorption applications. In “Behavior of Per technetate Ion Adsorption from Aqueous Solutions Shown by Activated Carbon Derived from Different Solutions” (Ito and Yachidate, 1992), it was stated that coconut-derived and oil-pitch-derived carbons would be best suited for technetium-99 removal. The same study found that products derived from coal had slightly lower K_d values and were not as attractive. However, in “Efficient Separation and Recovery of Technetium-99 from Contaminated Groundwater” (Gu et al., 1996), a bituminous coal manufactured product (no longer being produced) was used that showed favorable results for K_d values. In “Control of Per technetate Sorption on Activated Carbon by Surface Functional Groups” (Wang et al., 2006), favorable K_d value results were shown for products derived from lignite coal. Previous research appears to indicate that various grades of coal (i.e., lignite and bituminous), as well as coconut and oil-pitch products, may be advantageous for use in this application. Other lignite coal products by the same carbon manufacturer referenced in the same Wang et al. (2006) study showed much lower K_d values. Therefore, it can be concluded that not all lignite products will behave the same, and activated carbon performance will not be solely a function of the carbon source. Consequently, it was deemed appropriate to select a wide assortment of activated carbon materials for future screening.

The literature survey also concluded that selection of carbon media containing certain surface functional groups could enhance carbon performance for technetium-99 adsorption. According to *Chemistry: The Molecular Nature of Matter and Change* (Silberberg, 2004), a surface functional group is defined as a specific combination of atoms, typically a carbon multiple bond and/or heteroatom bond, that reacts in a characteristic way no matter in which molecule it occurs. Therefore, selecting carbon media with specific surface functional groups could increase the technetium-99 adsorption capacities for those carbons.

Gu et al. (1996) concluded that surface reactions between per technetate anions and C-O (or C-OH) surface functional groups on activated carbon may be the dominant interaction mechanisms. Gu et al. (1996) also concluded that anion-exchange reactions cannot be completely ruled out because an increase in ionic strength appears to reduce per technetate ion (TcO_4^-) adsorption on activated carbon.

Screening of carbon media for surface functional groups was later accomplished by Wang et al. (2006) using titration and data analysis. Wang et al. (2006) performed acid-base titrations for six activated carbons using an auto-titrator. The titrations were performed in three electrolyte solutions: (1) deionized water, (2) 0.01M NaCl, and (3) 0.1M NaCl. Prior to titration, a solution of HCl was added to the sample to start the titration process from an acidic pH point. The NaOH was used as the base titrant. Wang et al. (2006) noted that the titrations were performed with the maximum allowed titration time interval between successive additions (i.e., 90 seconds) to maximize the equilibrium time between incremental titrant additions. It was further noted that these titrations were performed under carbon dioxide-free conditions to avoid carbon dioxide uptake by the base titrant, which could obscure the results.

With the titration curve results, the Wang et al. (2006) study then calculated the distribution of acidity constants using the following condensation approximation (Equation 3-1):

$$f(pK) \approx -dQ/dpH \quad (\text{Equation 3-1})$$

where:

Q = overall degree of protonation of activated carbon surfaces

pK = acidity constant defined as $pK = -\log_{10}K$

$K = ([A^-][H^+]) / [HA]$

Wang et al. (2006) then created acidity constant-distribution curves which showed $f(pK)$ values plotted versus pK values. Peaks occurring at specific pH values, however, have a pK equal to the pH at that point on the curve; therefore, these $f(pK)$ values assumedly were plotted as a function of pH to derive these curves. This allowed for identification of the surface functional groups shown as peaks on the plots. Wang et al. (2006) shows the distribution of activity constants for four carbon products, which is provided below as Figure 3-1.

Source: "Control of Perchlorate Sorption on Activated Carbon by Surface Functional Groups" (Wang et al., 2006).

Figure 3-1. Example of Acidity Constant Distribution Curves

The value of Q was defined in *Characterization of the Surfaces of Activated Carbons in Terms of Their Acidity Constant Distributions* (Bandosz et al., 1993). The methods of this work state the following:

The titration curves, $pH(V_{base})$, consisting of 30-200 experimental points, were transformed into proton binding isotherms, $Q(pH)$, by using the proton balance equation and a theoretical blank reference:

$$Q = (1/m) [V_o \{[H]_i - [OH]_i\} + V_t N_t - (V_o + V_t) \{[H]_f - [OH]_f\}] \quad \text{(Equation 3-2)}$$

where V_o and V_t are the volumes of solution and of the titrant added, N_t is the normality of titrant, m is the weight of the sample, and the subscripts i and f refer to the initial and equilibrium concentrations of H^+ and OH^- ions. (Bandosz et al., 1993)

Bandosz et al. (1993) indicated that the method showed accurate results within a pH window between 3 and 11. Outside of this range, the data become obscured due to slight deviations between the experimental and calculated blanks.

Wang et al. (2006) also tested the adsorption capacities of the six commercial activated carbons for which they analyzed the surface functional groups. Half of the carbon materials showed high sorption capacities and were grouped by Wang et al. (2006) as "Type 1" carbons. The other half of the carbon materials showed relatively low sorption capacities and were grouped as "Type 2" carbons. Wang et al. (2006) then analyzed the carbon materials for presence of surface functional groups as previously described. The study concluded that the presence of specific carboxylic functional groups were responsible for the high sorption capacities for the Type 1 carbon materials. The large fraction of noteworthy carboxylic functional groups, defined by Wang et al. (2006) as carboxylic "Subgroup A" (at approximately $pK = 8.5$) and carboxylic "Subgroup B" (at approximately $5 < pK < 6$), were associated with the high pertechnetate sorption capacities observed in the attractive "Type 1" materials. This conclusion, however, seems counter-intuitive because carboxylic groups are anionic (negatively charged) and would be unlikely to adsorb pertechnetate, an oxyanion (also negatively charged). It is possible that the common acidity constant spikes were actually identifying the presence of another functional group such as a quinone, carbonyl, lactone, or other. These functional groups may look similar to carboxylic groups at the higher end of the pH scale on the titration curve but would be more likely to adsorb pertechnetate.

Wang et al. (2006) showed that the more favorable K_d values in the Type 1 carbons were all obtained using commercial powdered activated carbon. Therefore, powdered activated carbon products will also be considered for testing, if available. It is unclear why the powdered activated carbon products would have all been classified as the high adsorption Type 1 carbons since all of the commercial products used in this study (granular and powdered) were believed to have been crushed to a powdered form before being tested by Wang et al. (2006).

A second relationship for consideration for carbon products was used in the Wang et al. (2006) study. Two of the three attractive Type 1 carbon materials were unwashed products and all three of the less attractive Type 2 carbon materials were acid washed. Therefore, the presence of the characteristic carboxylic surface functional group acidity constant spikes may also be a function of the carbon-washing technique. Consequently, it seems appropriate to select activated carbon materials for screening with an assortment of wash techniques applied (e.g., acid wash, water wash, and unwashed).

Numerous techniques can be used for characterizing the types of surface function groups present on activated carbon ("Surface Chemistry of Activated Carbons and Its Characterization" [Bandosz and Ania, 2006]). The Boehm titration method and the potentiometric titration method both provide information on the composition and concentration of surface functional groups on activated carbon.

The Boehm titration method is a simple, traditional form of chemical analysis. The concept of this method is to neutralize surface functionalities based upon their acid strength, as it is known that a functional group of a given pKa can only be neutralized by a base having a higher value of pKa. Bandosz and Ania (2006), however, found the following using the Boehm method:

...shows some ambiguities and discrepancies in the report results when applied to carbon materials. It is due to the fact that the surface heterogeneity of carbon materials makes extremely difficult any attempt to accurately determine the surface functionalities on carbons, according only to their acidic and base properties. (Bandosz and Ania, 2006)

Therefore, the potentiometric titration method (as referenced in the Wang et al. [2006] study and the Bandosz et al. [1993] study) appears to be best suited for future carbon testing because auto-titration potentiometric methods will allow for smaller, more discrete measurements and computation of the proton binding isotherms.

3.2 Batch Isotherm Testing

Several previous investigations tested the adsorption of technetium-99 onto activated carbon media. These studies were discussed in detail in Chapter 4 of the literature survey (SGW-43982). The technetium-99 adsorption tested in these previous studies occurred in the form of the pertechnetate ion (TcO_4^-). The pertechnetate form is also the applicable technetium-99 ion in 200 West Area groundwater.

Batch experiments in the previously discussed studies used known quantities of activated carbon and added it to solutions of known concentration of pertechnetate. The amount of carbon samples in the previous works usually varied between 0.05 and 0.25 g. The volume of solution varied between 10 mL and 5 L. These studies then measured the distribution of the pertechnetate ion by identifying the concentration of ions adsorbed onto activated carbon and the concentration of ions remaining in solution. The K_d was then calculated based on the batch test data results. A higher K_d value indicated a greater distribution of pertechnetate onto the activated carbon and less remaining in solution. Each study defined the K_d values using slightly different equations. The equations presented for further use as part of these testing guidelines are from "Separation of Technetium with Active Carbon" (Yamagishi and Kubota, 1989) (shown below as Equation 3-3) and Wang et al. (2006) (shown as Equation 3-4). Both of these equations will yield the same results for K_d in terms of mL/g, but the variables defined in each equation are slightly different.

$$K_d = \{(A_i - A_f) / A_f\} \times (V / m) \quad \text{(Equation 3-3)}$$

where:

A_i = constituent activity (i.e., technetium-99) of solution before adsorption

A_f = constituent activity (i.e., technetium-99) of solution after adsorption

m = amount of activated carbon (in g)

V = volume of solution (in mL)

$$K_d = \frac{\text{amount of Tc sorbed on solid/mass of solid (in g)}}{\text{amount of Tc dissolved/volume of solution (in mL)}} \quad \text{(Equation 3-4)}$$

Previous studies also looked at the pertechnetate distribution as a function of mixing time in order to determine the time to reach equilibrium. In "In Situ Sorption of Technetium Using Activated Carbon" (Holm et al., 2000), it was concluded that the test solution reached equilibrium in approximately 5 to 6 hours. "Adsorption of Pertechnetate Ion on Active Carbon from Acids and Their Salt Solutions" (Ito and Akiba, 1991) found that the time to reach equilibrium was approximately 6 to 10 hours. The equilibrium curves shown in these studies appeared to follow a characteristic logarithmic trend. Ito and Akiba (1991) also found that use of high-concentration sulfuric acid test solutions caused a significant change in the equilibrium curve behavior. In these test runs, the K_d value peaked after approximately 4 hours of mixing time. The K_d value then decreased significantly during the 4- to 12-hour mixing time, and then either continued to slightly decrease or reached an equilibrium value. It was assumed that the high concentration of sulfate ions would displace some of the pertechnetate ions over time. Gu et al. (1996) and Wang et al. (2006) used slightly longer equilibrium times, which were overnight and 24 hours, respectively.

Based on the equilibrium data results, these investigators then developed isotherm plots of the technetium-99 adsorbed per mass of carbon versus the technetium-99 concentration in solution at equilibrium. The studies then evaluated the adsorption isotherm behavior. Yamagishi and Kubota (1989) found that for technetium-99 equilibrium concentrations between 10^{-4} and 10^{-2} M (1.7×10^8 and 1.7×10^{10} pCi/L), the Freundlich isotherm equation would apply. However, technetium-99 equilibrium concentrations above 10^{-2} M (1.7×10^{10} pCi/L) showed that the Langmuir equation seemed more applicable. Later research by Ito and Akiba (1991) found the Freundlich isotherm equation best suited for much lower technetium-99 equilibrium concentrations between 10^{-13} and 10^{-11} M (1.7×10^{-1} and 1.7×10^1 pCi/L). Gu et al. (1996) showed that the data results fit to nearly linear isotherms for technetium-99 equilibrium concentrations between 0 and 60,000 pCi/L. The equations for linear, Freundlich, and Langmuir isotherm plots, as shown in *Perry's Chemical Engineers Handbook* (Perry and Green, 1997), are shown in Equations 3-5, 3-6, and 3-7, respectively.

$$n_i = K_i C_i \quad (\text{Equation 3-5})$$

where:

n_i = quantity adsorbed per unit mass

K_i = equilibrium constant

C_i = concentration in solution

$$n_i = K_i C_i^m \quad (\text{Equation 3-6})$$

where the exponent m is positive and is generally not an integer.

$$n_i = (n_i^s K_i C_i) / (1 + K_i C_i) \quad (\text{Equation 3-7})$$

where n_i^s is the monolayer capacity approached at large concentrations.

Examples of two of these characteristic isotherm data plots are shown in Figure 3-2 (note that the Langmuir isotherm not shown).

These studies also determined several key parameters that will affect technetium-99 uptake onto activated carbon during testing. As discussed in Section 3.1, one of these parameters is the type of surface functional groups present on the carbon media. Identification of these surface functional groups will be a primary goal of testing (Section 4.1).

Figure 3-2. Example Isotherm Plots

Another key parameter affecting technetium-99 uptake is the pH of the test solution. Gu et al. (1996) and Wang et al. (2006) conducted batch sorption testing under various pH conditions of between 4.5 and 9.5. These tests showed that (1) technetium-99 adsorption onto activated carbon (e.g., K_d value) will decrease with increases in pH, and (2) the relationship between K_d decrease and pH increase is approximately linear. Figure 3-3 (excerpted from Gu et al. [1996]) shows the K_d dependence on solution pH. Holm et al. (2000) examined pH conditions across a larger spectrum and determined that K_d values reach a peak at approximately pH 4, and the K_d will then begin to decrease as the solution pH is decreased below pH 4. Therefore, testing pH dependence during adsorption experiments appears to be a necessary component of the batch adsorption testing.

Note: pH-dependent adsorption of TcO_4^- by activated carbon at an initial concentration of $\sim 18 \mu\text{Ci/L}$ (for 1.1 mg Tc/L).

Source: "Efficient Separation and Recovery of Technetium-99 from Contaminated Groundwater" (Gu et al., 1996).

Figure 3-3. Example of K_d Dependence on Solution pH

Yamagishi and Kubota (1989) showed that solution temperature is another key parameter affecting technetium-99 uptake onto activated carbon. This research showed that similar to pH, increases in solution temperature resulted in decreased Kd values. Specifically, the uptake of technetium-99 on activated carbon was found to decrease at a constant rate as the temperature increased from 20°C to 60°C (68°F to 140°F). Above 60°C (140°F), the uptake decreased significantly. Figure 3-4 (excerpted from Yamagishi and Kubota [1989]) shows the Kd dependence on solution temperature.

Source: "Separation of Technetium with Active Carbon" (Yamagishi and Kubota, 1989).

Figure 3-4. Example of Kd Dependence on Solution Temperature

Gu et al. (1996) also showed that the ionic background solution composition and concentration constitute yet other key variables affecting technetium-99 adsorption on activated carbon. The study evaluated technetium-99 adsorption in varying background ionic solutions. The Kd values for technetium-99 decreased by varying amounts based on the type of background ionic solution. These solutions, shown in order of highest Kd (i.e., least affected by the ionic solution) to lowest Kd (i.e., most affected by the ionic solution) were as follows: 0.01M CaCl₂, 0.01M Na₂SO₄, 0.01M NaNO₃, 0.1M Na₂SO₄, 1M NaCl, 0.01M Na-salicylate, and 0.01M Na-phthalate. The study also showed that Kd values decreased with increased ionic concentrations. The findings from the study deemed most applicable to Hanford Site testing guidelines pertained to nitrate concentrations. As noted in Table 2-1, the groundwater quality assumed for carbon pre-treatment will be high in nitrate (at 84 mg/L as nitrogen). Therefore, nitrate concentrations in test water must also be considered for the testing guidelines. Sulfate and chloride concentrations in Hanford Site groundwater are expected to be significantly less than the nitrate concentrations. Furthermore, the Kd values in the Gu et al. (1996) study were greater in the presence of sulfate and chloride background solutions than in nitrate background solutions of a similar molar concentration. For these reasons, sulfate and chloride are considered less important than nitrate for the purpose of evaluation in these testing guidelines.

Previous research also tested for recovery of technetium-99 after pertechnetate had adsorbed onto activated carbon. Gu et al. (1996) showed that by using a 0.5M sodium-salicylate solution at pH 8.8, rapid desorption of the pertechnetate ion occurred and a complete recovery (approximately 100 percent) of the previously adsorbed technetium-99 was achieved. Other research shows general agreement with these results. For example, Ito and Akiba (1991) found sodium-salicylate to be the least favorable ion solution for technetium-99 adsorption.

3.3 Continuous-Flow Testing

Research studies focused on continuous-flow testing were not as widely encountered as those involving batch isotherm testing. A summary of previous studies are included in this section.

In "Development of Partitioning Process in JAERI" (Kubota et al., 1990), a flow-through study was conducted using a 0.004M (6.8×10^9 pCi/L) technetium-99 solution at a flow rate of 5 bed volumes/hour. The bed size was 150 mm long by 7.5 mm in diameter. The technetium-99 adsorption on the carbon was reported at 0.237 mmol/g (4.02×10^8 pCi/g) at 50 percent breakthrough, with a corresponding leakage rate of only 1 percent. The breakthrough curves shown in this study indicated that the technetium-99 breakthrough process occurred quickly once initial breakthrough occurred.

In "Recovery of Technetium with Active Carbon Column in Partitioning Process of High-Level Liquid Waste" (Yamagishi and Kubota, 1993), testing was conducted using an activated carbon column. The column size was 7.5 mm by 150 mm.

Gu et al. (1996) studied technetium-99 adsorption capacity on an activated carbon column in a flow-through experiment and did not see breakthrough after 14,000 pore volumes (approximately 31.7 L) of influent water. A full breakthrough of the pertechnetate ion (TcO_4^-) was not observed even after 23,000 pore volumes (approximately 52.1 L). The influent concentration of technetium-99 in the study was 3,000 pCi/L (4.8×10^{-5} mmol/g), and the GAC column size was 6.6 mm by 30 mm. The flow rate varied between 1 and 2 mL/min.

Holm et al. (2000) studied technetium-99 uptake on carbon-impregnated cartridge filters and showed that high technetium-99 uptake on the filters could be accomplished with relatively short contact time. The filter sizes were 6.7 cm by 24.8 cm, the total volumes of filtered water were approximately 700 and 800 L, and the flow rate used was 2 to 6 L/min.

In these experiments, the total volume of treated water was tracked and the column effluent water was regularly analyzed for technetium-99 concentration. Gu et al. (1996) monitored for breakthrough as a function of effluent concentration. These studies presented data results in the form of a breakthrough curve, plotted as effluent concentration versus water volume treated.

4 Recommendations for Technetium-99 Adsorption Testing on Activated Carbon

The following testing guidelines identify the three testing phases to be completed, performance goals, and the required elements for each phase. After each phase of testing, a hold point is established where the testing results will be evaluated against the established criteria prior to moving onto the next phase of testing.

The three testing phases are as follows:

- Phase 1: Activated carbon screening and selection (Section 4.1).
- Phase 2: Batch isotherm adsorption testing (Section 4.2).
- Phase 3: Continuous-flow column adsorption testing (Section 4.3).

The testing phases must be conducted in sequential order. Each phase is discussed in this chapter.

4.1 Phase 1 — Activated Carbon Screening and Selection

Phase 1 testing consists of screening potential activated carbon media and selecting activated carbon for subsequent testing during Phase 2 (Section 4.2). The first phase of testing will be completed at the CH2M HILL Applied Sciences Laboratory (ASL) in Corvallis, Oregon.

4.1.1 Test Performance Goals

The performance goals for Phase 1 testing are as follows:

- Identify a wide variety of carbon products that are commercially available and have a track record.
- Screen a range of activated carbon products to identify the surface functional groups present on each type based on the distribution of acidity constants (pK).
- Select four to eight candidate activated carbons to use in subsequent testing based on the surface functional group characterization.

4.1.2 Test Materials

4.1.2.1 Carbon Samples

Activated carbon samples for testing will be selected by CH2M HILL and CHPRC. Carbon products similar to those used in previous studies (described in Section 3.1) will be the focus when seeking out recommendations from vendors. Products derived from various grade coal sources (i.e., bituminous, sub-bituminous, and lignite), coconut, and oil pitch will be investigated if a commercial product is available. Both granular and powdered carbons will be considered. Products of various wash types (e.g., unwashed, water washed, and acid washed) will also be selected. Ideally, a broad combination of carbon sources from these various sources (in both granular and powdered forms and in various wash types) will be tested.

Only products that are commercially available will be considered. This will allow for a more straightforward, economic analysis at the completion of testing because media replacement costs can be established.

Between 10 and 20 different carbon samples will be selected for screening. The final quantity of carbon products will be determined based on the number of available carbon products recommended by vendors. The carbon products chosen for testing will be purchased by CHPRC. The sample volume required for each carbon product will be established based on testing needs. Material safety data sheets and product cut sheets will be obtained from the carbon manufacturers for each product. Leftover carbon sample materials will not be disposed after testing so the materials can be later viewed by others, if requested. Carbon materials will be stored until CHPRC provides direction to dispose of the materials, CHPRC makes arrangements to collect the materials, or 6 months elapse, whichever occurs first.

4.1.2.2 Laboratory Equipment

Surface functional groups will be identified by potentiometric titration. An auto-titrator capable of dosing the titrant in small increments (e.g., 0.001 mL) will be required. A pH meter with a glass electrode will also be required.

Other standard laboratory equipment (e.g., an oven, glassware, sieves, and mortar and pestle) will also be required. Remaining equipment needs will be determined and coordinated by the laboratory.

4.1.2.3 Stock Solutions

Standard stock solutions for bases and acids to be used in titration testing are available at the laboratory. Titrations will be performed in a NaCl electrolyte solution. Solutions of HCl and NaOH will be required for titrations.

4.1.3 Analytical Methods

Titrations will be performed in accordance with the operating instructions for the laboratory auto-titrator and pH meter. Titrations should be performed with the maximum allowed titration time interval to allow a close approach to equilibrium between incremental titrant additions. Titrations should be performed in a nitrogen-purge environment to prevent carbon dioxide uptake by the solution and drift in standard buffer solutions.

4.1.4 Test Procedures

The following test procedure should be followed but is not intended to identify all of the detailed steps necessary to perform Phase 1 testing:

1. Prepare activated carbon products received from vendors. Reduce activated carbon samples in particle size so 95 percent passes through a U.S. 325-mesh sieve by wet screening (or equivalent). Wash the products with deionized water. Activated carbon products should then be centrifuged to separate carbon media from the washwater. Continue procedures for washing, centrifuging, and pouring off supernate until the supernate is clear. Dry the carbon products in an oven overnight until no further weight loss occurs.
2. Weigh a sample of the dried carbon media and add to it a known volume of 0.01M NaCl solution.
3. Continuously stir the carbon suspension for the remaining steps and protect it from air contact by a continuous stream of nitrogen gas.
4. Allow the pH of the electrolyte solution to equilibrate.
5. Deaerate the carbon by pulling a vacuum for 5 minutes.

6. Add sufficient volume of an HCl standard solution to bring the test solution to pH 2 at equilibrium. This will allow for the titration process to begin at an acidic pH range.
7. Measure the initial conditions of solution, including pH, volume of HCl added, and total initial volume of the test solution (including the carbon).
8. Complete the auto-titration by dosing small increments (e.g., 0.01 mL) of NaOH into the solution, with maximum time delay between successive additions. The increments of NaOH addition must be recorded, as well as the equilibrium pH of the solution after each titration increment. The molarity of the NaOH solution used as the titrant in this step and the molarity of the HCl solution added in step 4 should be similar.
9. Continue titration until the pH of the solution is 12.
10. Repeat titration steps 2 through 9 for each type of carbon product.

4.1.5 Data Results

Titration results will be analyzed and presented, as described below. Following completion of testing and data analysis, the laboratory will prepare a summary report that includes data plots and findings from Phase 1 testing.

4.1.5.1 Titration Plots

The laboratory will prepare standard titration curves for all runs showing the solution pH as a function of the volume of base added. A separate titration plot should be provided for each carbon product.

4.1.5.2 Proton Binding Isotherms

The titration data results will be used to calculate a proton-binding isotherm $Q(\text{pH})$ using Equation 3-2 (Section 3.1). Isotherm plots of $Q(\text{pH})$ (in mmol/g) versus pH will be provided for each carbon product.

4.1.5.3 Distributions of Acidity Constants

The proton-binding isotherms will be used to further calculate $f(\text{pK})$ using Equation 3-1 (Section 3.1). The results of $f(\text{pK})$ will be plotted versus pK (same as pH) for the range of pK values between 3 and 11. A distribution plot for acidity constants will be provided for each carbon product. Smoothing of the plots will likely be required to allow for easier interpretation of results.

The distribution plots for the acidity constants will then be evaluated for identification of carboxylic functional groups on activated carbon. Plots that contain carboxylic acidity constant spikes at approximately pK 8.5 and $5 < \text{pK} < 6$ will be flagged by the laboratory. This will allow CH2M HILL to identify carbon products with the characteristic Subgroup A and B carboxylic surface functional groups that were identified as having high sorption capacities for technetium-99 in the Wang et al. (2006) study.

4.1.5.4 Data Collection

The laboratory will maintain a logbook/notebook of all notes taken during testing. Auto-titration results will be produced in the form of a data file. The data file will be downloaded to a spreadsheet system (i.e., Microsoft® Excel) to facilitate data reduction and plot development.

Copies of applicable logbooks and quality assurance (QA)/quality control (QC) documents used in testing will be provided by the laboratory to CHPRC.

Microsoft® Excel is a registered trademark of Microsoft Corporation, Redmond, Washington.

4.1.6 Testing Hold Point for Results Interpretation

Following completion of Phase 1 testing, CHPRC and CH2M HILL will review the summary report provided by the laboratory and will interpret results for next test steps. The carbon products flagged during data analysis will be evaluated and considered for subsequent testing, as described in Section 4.2. The basis for selection of short-listed candidate activated carbons is as follows:

- Between two and four of the activated carbon products that were flagged as containing characteristic acidity constant spikes for Subgroup A and B carboxylic surface functional groups will be retained for future testing. Carbon products with a greater amplitude spike, $f(pK)$, for carboxylic Subgroups A and B will be favored.
- Between two and four of the activated carbon products that were not flagged as containing characteristic acidity constant spikes will also be retained for future testing.
- Selecting carbons that have both types of characteristics will provide (1) confirmation whether those carbons with surface functional groups identified as Type 1 in the Wang et al. (2006) study do show higher adsorption capacities during Phase 2 testing, and (2) a greater assortment of carbons for testing in Phase 2 if the results of the Wang et al. (2006) study do not hold true.

4.1.7 Quality Control and Quality Assurance Measures

The laboratory will perform two titrations for each carbon product to allow for duplicate results as a QC measure. A blank titration run with no activated carbon will also be performed.

Other QC measures will be designated by the laboratory based on the standard titration QC measures. For example, the pH probe will be calibrated regularly and calibration records will be provided.

The laboratory must establish procedures for maintaining QA throughout testing. For example, the laboratory shall use reagent-grade chemicals that are used within the product shelf life.

4.1.8 Waste Management

The laboratory will be responsible for disposing all laboratory wastes associated with testing. The wastes derived from Phase 1 testing will not consist of radioactive or mixed wastes.

4.1.9 Qualifications

The laboratory manager overseeing Phase 1 testing will be Mr. Timothy Maloney. Mr. Maloney is a principal technologist and the Treatability/Process Evaluation Group Manager at the CH2M HILL ASL. Mr. Maloney is a recognized expert with over 30 years of experience with treatability and process-simulation testing.

4.1.10 Safety

The laboratory will be responsible for addressing all safety hazards anticipated for this work. A job hazard analysis will be prepared and included in the test documentation.

4.2 Phase 2 — Batch Isotherm Adsorption Testing

Phase 2 testing consists of adsorption testing using batch isotherm methods and the activated carbon products selected during Phase 1 testing (Section 4.1). Phase 2 testing will be completed by Pacific Northwest National Laboratory (PNNL) in Richland, Washington.

4.2.1 Test Performance Goals

The performance goals for Phase 2 testing include the following:

- Determine which of the activated carbon products exhibit the greatest adsorption of technetium-99 from a representative water sample at equilibrium.
- Determine the difference in technetium-99 adsorption using two different nitrate concentrations in the test water.
- Evaluate the technetium-99 adsorption dependence on solution pH.
- Evaluate the technetium-99 adsorption dependence on solution temperature.
- Evaluate the technetium-99 adsorption dependence on VOC water concentrations.

4.2.2 Test Materials

4.2.2.1 Source Water for Testing

Available sources of test water were evaluated against the assumed water quality information presented in Table 2-1. It was determined that using Hanford Site groundwater would be preferred over systematically and artificially creating a test solution with spiked quantities of technetium-99 and other constituents of interest to attempt matching the water quality shown in Table 2-1. Creating an artificial test solution would be cumbersome, and using actual Hanford Site groundwater is much simpler and introduces fewer uncertainties. It is questionable whether an artificial test solution would contain all of the trace or unidentified constituents of groundwater and, therefore, whether subsequent test results would be representative of future adsorption capacities that would be experienced with actual Hanford Site groundwater. It is also questionable whether the form of the technetium-99 spiked would be the same as that present in Hanford Site groundwater.

Groundwater from 200-ZP-1 Operable Unit (OU) extraction well 299-W15-765 was originally selected for test water for the following reasons:

- Water will be supplied from an existing groundwater extraction well on the Hanford Site from the 200-ZP-1 OU. The current technetium-99 IX system, which is part of the 200 West Area pump-and-treat design, will treat groundwater that is mostly from the 200-ZP-1 OU.
- Extracted groundwater from well 299-W15-765 has the highest average technetium-99 concentration among all of the accessible 200-ZP-1 OU groundwater extraction wells.
- Average nitrate concentrations in well 299-W15-765 are similar to those identified in Table 2-1.

Prior to testing, however, it was determined that the pump for extraction well 299-W15-765 could no longer produce water. Therefore, groundwater from 200-UP-1 OU extraction well 299-W19-36 was selected for use in this testing. Well water will still be supplied from an existing Hanford Site extraction well with average technetium-99 concentrations similar to previously identified well 299-W15-765. Minor changes associated with using groundwater from well 299-W19-36, as opposed to well 299-W15-765, include the following:

- Detectable uranium concentrations in well 299-W19-36 that were not recorded in well 299-W15-765
- Average nitrate concentrations in well 299-W19-36 that are lower than those identified in Table 2-1.

Table 4-1 provides the average sample results over the past 6 years (2004 through 2010) for well 299-W19-36. These sample results are compared to the design water quality concentrations for the same constituents shown in Table 2-1.

Groundwater obtained from well 299-W19-36 will need to be prepared before it is used in testing. The groundwater is expected to contain carbon tetrachloride and trichloroethene (TCE) (and possibly other VOCs) that need to be removed before the water is used in testing. The nitrate concentration will also need to be adjusted. Procedures for preparing the source water are described in Section 4.2.4 for batch testing and Section 4.3.4 for column testing.

Table 4-1. Groundwater Quality at Well 299-W19-36

Technetium-99 (activity)	5,947 pCi/L	14,457 pCi/L
Iodine-129 (activity)	0.952 pCi/L	0.899 pCi/L
Tritium (activity)	No data	24,017 pCi/L
Uranium	359 µg/L	3.93 µg/L
Trichloroethene	2.82 µg/L	3.29 µg/L
Carbon tetrachloride	269 µg/L	506 µg/L
Nitrate (as nitrogen)	48.3 mg/L	84 mg/L
Hexavalent chromium	No data	161 µg/L
Total chromium	No data	161 µg/L
pH	7.8 SU	7.7 SU

a. Data query results for well 299-W19-36 for time period 2004 through 2010 provided by L.C. Swanson (CH2M HILL Plateau Remediation Company, Richland, Washington), email dated July 9, 2010. Only sample results reported at detectable levels were used to calculate the average values are shown in Table 4-1.

b. Design source water assumed as previously shown in Table 2-1.

c. Information in this table includes groundwater well 299-W19-36 sample data results for COCs only (as shown in Table 2-1).

COC = contaminant of concern

SU = standard units

4.2.2.2 Carbon Samples

Four to eight carbon products will be identified for this phase of testing (Section 4.1). Additional sample volume of the candidate activated carbon products from vendors will be obtained and sent to the laboratory.

4.2.2.3 Sodium Salicylate

Sodium salicylate should be made available during testing because it may be required for removing (desorption) technetium-99 loaded onto the activated carbon samples. The procedures for using sodium salicylate are included in Section 4.2.4.

4.2.2.4 Stock Solutions

Standard stock solutions for NaOH and HCl will be required to perform pH adjustments of the source water.

4.2.2.5 Laboratory Equipment

Standard laboratory equipment (e.g., glassware, heating elements, mortar and pestle, sieves, and an oven) will also be required. Detailed equipment needs will be determined by the laboratory and included in the test plan.

4.2.3 Analytical Methods

The standard analytical methods to be used for the analyses described in this section will be addressed in the laboratory's test plan (i.e., American Society for Testing and Materials [ASTM]).

4.2.3.1 Test Water Initial Concentrations

The source water will be analyzed before beginning adsorption tests. Test water will be analyzed for the following:

- Technetium-99
- Carbon tetrachloride
- TCE
- Hexavalent chromium
- Total chromium
- Nitrate
- Total organic carbon
- Total suspended solids
- VOCs
- pH and temperature.

The source test water will be analyzed for the presence of other organic solvents identified as project constituents of interest and that could be present in well 299-W19-36 groundwater. In addition to the COCs carbon tetrachloride and TCE, these other VOCs could also potentially adsorb onto activated carbon. For this reason, test water will be analyzed for VOCs in accordance with the standard method. The VOCs to be analyzed include acetone, carbon tetrachloride, chloroform, dibromochloromethane, methylene chloride, tetrachloroethene, TCE, 1,1,1-trichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene (total), and BTEX (benzene, ethylbenzene, toluene, and xylenes). These specific VOCs were selected for analysis because either (1) the compound was included in the 200 West Area pump-and-treat system mass balance (SGW-45097, *Integrated Mass Balance for the 200 West Pump and Treat Facility*, Rev. 3), or (2) the historical data from well 299-W19-36 indicated that the compound was detected above the method detection limit.

4.2.3.2 Test Water Final Concentrations

At the conclusion of every discrete batch test, the test solution will be analyzed for technetium-99.

4.2.4 Test Procedures

The laboratory will develop a detailed test plan for Phase 2 and Phase 3 testing (Sections 4.2 and 4.3). Therefore, the following test procedures should be used as guidelines only and are not intended to identify all of the detailed steps required to perform this testing.

Standard practices for determination of the adsorptive capacity of activated carbon by isotherm techniques should be followed throughout this testing. Refer to ASTM standard practices, where applicable.

4.2.4.1 Material Preparations

Material preparation will be conducted as follows:

1. Prepare activated carbon products received from vendors. Reduce activated carbon samples in particle size so 95 percent passes through a U.S. 325-mesh sieve by wet screening (or equivalent). Wash the products with deionized water. Activated carbon products should then be centrifuged to separate the carbon media from the washwater. Continue procedures for washing, centrifuging, and pouring off supernate until the supernate is clear. Dry carbon products in an oven overnight until no further weight loss occurs.
2. Prepare source water for testing:
  - a. The source water will be initially analyzed for carbon tetrachloride, TCE, and other VOCs as discussed in Section 4.2.3.1.
  - b. Remove VOCs (carbon tetrachloride, TCE, and others, if applicable) from the source water using air sparging methods. Care should be taken to not volatilize water during this process and therefore concentrate the constituents in the groundwater sample.
  - c. The source water should be analyzed after VOC removal to confirm that carbon tetrachloride and TCE (and others, if applicable) have been sufficiently removed. The remaining concentration in the source water needs to be low enough such that 100 percent of the mass of these remaining constituents loaded onto the activated carbon during testing would not exceed the applicable and respective disposal limit. The disposal limit is 6 parts per million (in mg/kg) for carbon tetrachloride and TCE. For example, using a 0.1 g carbon sample and 250 mL of solution, the concentration of carbon tetrachloride in the test water would need to be reduced to less than 2 µg/L prior to carbon testing.
  - d. Adjust the pH of the source water to 7.7 (which is the average pH shown in Table 2-1).
  - e. Adjust the temperature of the source water to 18.3°C (65°F), which is the raw well water temperature shown in Table 2-1.
  - f. For the first set of test runs (as shown in Table 4-2 and described below), spike the source water with sodium nitrate as needed to adjust the nitrate concentration in the source water to 375 mg/L (or approximately 84 mg/L as nitrogen) and, therefore, match the expected nitrate concentration in the 200 West Area pump-and-treat design. For the second set of test runs, spike the source water with sodium nitrate as needed to adjust the nitrate concentration in the source water to 750 mg/L as nitrate (or approximately 169 mg/L as nitrogen), which is twice the concentration of the original test runs.
3. After the source water is prepared for testing, analyze the source water for the remaining constituents identified in Section 4.2.3.1.

4.2.4.2 Batch Isotherm Tests

At the beginning of batch isotherm testing, the laboratory will perform initial contact tests to determine the relative adsorption capacity of each carbon product. A duplicate set of contact tests should be performed for each carbon product at a fixed solids/solution ratio. The results of these initial tests will be used to confirm that the selected carbon/solution test ratios are appropriate (see step 1 below). At the conclusion of these initial tests, the laboratory will hold work and discuss the initial test results

with CHPRC. These results will then be used to select four carbon products for the remaining batch isotherm testing described below:

1. Prepare test jars for the batch adsorption tests using known quantities of carbon product and prepared source water. For each type of carbon product, prepare four test jars of varying amounts of carbon. The suggested carbon and solution combinations are as follows:

- 0.05 g carbon/250 mL solution (test jar #1)
- 0.1 g carbon/250 mL solution (test jar #2)
- 0.2 g carbon/250 mL solution (test jar #3)
- 0.5 g carbon/250 mL solution (test jar #4)

These ratios were established to allow detection of K_d values as low as approximately 1,000 to as high as 35,000. The four ratios of carbon and solution should be adjusted, based on the initial contact test results, in order to obtain a maximum of 85 percent and a minimum of 10 percent technetium-99 removal from solution. This will allow for data-point collection across the isotherm curve.

2. The first batch test (test jar #1) would then include 0.05 g of carbon media. Proceed to the prescribed 0.1 g carbon for jar test #2 and subsequently continue to increase the carbon-to-water ratio in tests jars #3 and #4. Based on the jar test data results, the amount of carbon in the subsequent test jars could also be decreased if necessary (i.e., 0.01 g for the second jar test). The water volume could also be adjusted, providing that four distinct carbon-to-water ratios are tested.
3. At the beginning of each batch test, deaerate the carbon by pulling a vacuum on the test jar for approximately 5 minutes.
4. Conduct each adsorption test for 24 hours to allow equilibrium to occur. Test bottles should be continuously agitated during the 24-hour holding time. The test bottles should then be centrifuged or filtered as necessary to separate the filtrate from the carbon.
5. Analyze the test water for the final technetium-99 concentration (Section 4.2.3.2).
6. Repeat the batch testing procedures for each candidate carbon product. Four jar tests will be completed for each carbon product.
7. Repeat all adsorption batch tests using source water with a higher NO_3 concentration. Procedures for preparing the source water to use in these test runs was previously described under materials preparation (step 2).
8. Duplicate all jar tests as a QC measure per Section 4.2.7.

An example testing matrix is provided in Table 4-2. The test matrix shows that 32 test runs will be completed (64 test runs, including duplicates). Based on the data results, eight isotherm plots would be created in this example scenario.

Table 4-2. Batch Isotherm Example Testing Matrix

1	375 mg/L as nitrate	Carbon 1	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
2	375 mg/L as nitrate	Carbon 2	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
3	375 mg/L as nitrate	Carbon 3	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
4	375 mg/L as nitrate	Carbon 4	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
5	750 mg/L as nitrate (double conc.)	Carbon 1	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
6	750 mg/L as nitrate (double conc.)	Carbon 2	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
7	750 mg/L as nitrate (double conc.)	Carbon 3	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4
8	750 mg/L as nitrate (double conc.)	Carbon 4	Carbon/solution ratio #1	Carbon/solution ratio #2	Carbon/solution ratio #3	Carbon/solution ratio #4

For each isotherm, three of the four jar test results should have a detectable amount of technetium-99 remaining in solution at equilibrium, which will allow for direct calculation of a K_d value in three of the four tests. In some cases, however, the uptake of technetium-99 onto activated carbon cannot be directly calculated based on the final water concentration (i.e., the final concentration in solution is below the method detection limit). If the technetium-99 uptake on activated carbon cannot be directly calculated for two or more of the isotherm jar tests, then additional testing should be performed. This can be accomplished (1) by performing another jar test with a new carbon/solution ratio, or (2) by technetium-99 desorption from activated carbon using a sodium-salicylate solution (as previously described in Section 3.2) in order to avoid running another test.

At the conclusion of the batch isotherm testing described in this section, the laboratory will contact CHPRC and discuss results. CHPRC and the laboratory will discuss whether all four carbon products should be retained for subsequent batch testing as described in Sections 4.2.4.3 and 4.2.4.4.

4.2.4.3 Variable pH Batch Tests

1. Conduct a second set of batch tests to evaluate the effects of variable pH on technetium-99 adsorption.
2. Perform batch adsorption tests using the prepared source water (step 2 in Section 4.2.4.1) with nitrate adjustment to 375 mg/L. For each carbon product, prepare three test jars and adjust the water pH to 6.5, 7.5, and 8.5, respectively. This pH range was selected because it is the pH operating range that could be reasonably expected for a full-scale system.

3. Use the stock solutions of NaOH and HCl to adjust the pH of the test water before adding carbon (sulfuric acid should not be used for pH adjustment).
4. Procedures for the pH batch tests should be the same as those presented for the isotherm batch tests above. For each carbon type, select one carbon/solution ratio to use for all pH batch tests. The selected carbon/solution ratio should have had approximately a 50 percent removal of technetium-99 from solution (Section 4.2.4.2). Conduct each adsorption batch test for 24 hours to allow equilibrium to occur. The concentration of technetium-99 in the test water after the adsorption batch test will be analyzed (Section 4.2.3.2).
5. Duplicate all jar tests as a QC measure per Section 4.2.7.
6. An example testing matrix, assuming four candidate carbon products are selected from Section 4.2.4.2 is provided in Table 4-3. This test matrix shows that 12 tests will be completed (24 tests total, including duplicates). Based on the data results, four pH curves (one for each media type) would be created in this example scenario. The original data results at pH 7.7 determined in Section 4.2.4.2 testing should also be included in each pH curve.

Table 4-3. pH Example Testing Matrix

1	375 mg/L as nitrate	Carbon 1	Use one from Section 4.2.4.2	6.5	7.5	8.5
2	375 mg/L as nitrate	Carbon 2	Use one from Section 4.2.4.2	6.5	7.5	8.5
3	375 mg/L as nitrate	Carbon 3	Use one from Section 4.2.4.2	6.5	7.5	8.5
4	375 mg/L as nitrate	Carbon 4	Use one from Section 4.2.4.2	6.5	7.5	8.5

4.2.4.4 Variable Temperature Batch Tests

1. Conduct a third set of batch tests to evaluate the effects of variable temperature on technetium-99 adsorption.
2. Procedures for performing the temperature batch tests will be the same as the pH batch tests except that pH will not be varied and instead the temperature of the test water will be varied. Use test water with a nitrate concentration of 375 mg/L and the same carbon/solution ratios selected in Section 4.2.4.3.
3. The previous study by Yamagishi and Kubota (1989) showed that the K_d for technetium-99 on activated carbon was found to decrease as the temperature increased from 20°C (68°F). As shown in Table 2-1, the raw well water temperature is assumed to be 18.3°C (65°F). Consequently, only temperatures greater than the raw well water temperature of 18.3°C (65°F), and up to the maximum temperature of the recommended influent water design range of 32.2°C (90°F), will be considered for testing.
4. Duplicate all jar tests as a QC measure per Section 4.2.7.

- An example testing matrix, assuming four candidate carbon products are selected from Section 4.2.4.2 is provided in Table 4-4. This test matrix shows that 12 tests will be completed (24 tests total, including duplicates). Based on the data results, four temperature curves would be created in this example scenario. The original data results at 18.3°C (65°F) determined in Section 4.2.4.2 testing should also be included in each temperature curve.

Table 4-4. Temperature Example Testing Matrix

1	375 mg/L as nitrate	Carbon 1	Same as Section 4.2.4.3	70°F	80°F	90°F
2	375 mg/L as nitrate	Carbon 2	Same as Section 4.2.4.3	70°F	80°F	90°F
3	375 mg/L as nitrate	Carbon 3	Same as Section 4.2.4.3	70°F	80°F	90°F
4	375 mg/L as nitrate	Carbon 4	Same as Section 4.2.4.3	70°F	80°F	90°F

4.2.4.5 Volatile Organic Compound Batch Tests

- Conduct a fourth set of batch tests to evaluate the effects of VOC concentrations on technetium-99 adsorption. Batch adsorption tests will be performed using the original source groundwater without VOC removal.
- Prepare the carbon media (described in Section 4.2.4.1, step 1).
- Adjust the pH of the source water to 7.7, which is the average pH shown in Table 2-1. Adjust the temperature of the source water to 18.3°C (65°F), which is the raw well water temperature shown in Table 2-1. Adjust the nitrate concentration of the source water to 375 mg/L.
- Analyze the source water for the constituents identified in Section 4.2.3.1.
- Select two carbon types from Section 4.2.4.2 for testing. The carbon products selected for this testing should have high K_d results in the previous batch tests (Section 4.2.4.2). The laboratory may need to contact CHPRC to confirm the carbon products to be used for these tests.
- For each carbon type, select two carbon/solution ratios that were used during batch isotherm testing (Section 4.2.4.2). The selected carbon/solution ratios should have had at least 50 percent removal of technetium-99 from solution. Conduct each adsorption batch test for 24 hours to allow equilibrium to occur. The concentration of technetium-99 in the test water after each adsorption batch test will be analyzed (Section 4.2.3.2).
- Duplicate all jar tests as a QC measure per Section 4.2.7.
- An example testing matrix is provided in Table 4-5. This test matrix shows that four tests will be completed (eight tests total, including duplicates).

Table 4-5. VOC Example Testing Matrix

1	375 mg/L as nitrate	Carbon A	Select one from Section 4.2.4.2	Select second from Section 4.2.4.2
2	375 mg/L as nitrate	Carbon B	Select one from Section 4.2.4.2	Select second from Section 4.2.4.2

4.2.5 Data Results

The batch testing results will be analyzed and presented as described below. Following completion of testing and data evaluation, the laboratory will prepare a summary report of the data results, isotherm and trend plots, and the findings from Phase 2 testing. The results will be evaluated as described in Section 4.2.6.

4.2.5.1 Calculation of Distribution Coefficients

1. Evaluate data results for each batch test and calculate the corresponding K_d values for technetium-99 based on Equation 3-3 (Section 3.2). Determine which activated carbons exhibit the greatest adsorption of technetium-99 based on the K_d results.
2. Tabulate all test runs and the corresponding K_d values calculated.

4.2.5.2 Isotherm Plots

Each set of test data as shown in Table 4-2 will be plotted and then fit to an appropriate isotherm. The best-suited specific isotherm will be determined based on empirical results. Isotherm information and equations are provided in Section 3.2. Provide the corresponding isotherm plot and equation for each carbon product type and at both of the NO₃ concentrations.

4.2.5.3 pH Data Results

1. Evaluate data results for the batch tests and calculate the corresponding K_d values for technetium-99. Determine which activated carbons show the greatest change in K_d value with a change in solution pH.
2. Tabulate all test runs and the corresponding calculated K_d values.
3. Provide data plots of technetium-99 K_d values versus pH for each carbon type.

4.2.5.4 Temperature Data Results

1. Evaluate data in a similar manner to the pH data results.
2. Tabulate all test runs and the corresponding calculated K_d values.
3. Provide a plot of technetium-99 K_d values versus temperature for each carbon type.

4.2.5.5 Volatile Organic Compound Data Results

1. Evaluate data results for the batch tests and calculate the corresponding Kd values for technetium-99.
2. Compare these results to jar test results using VOC-stripped test water (Section 4.2.5.1). Indicate if the Kd value for technetium-99 changed using test water with the higher concentration of VOCs.

4.2.5.6 Data Collection

The laboratory will maintain a logbook/notebook of all notes taken during testing. Data sheets may be used for each batch test. The laboratory standard data reports for analytical results will be provided. The laboratory should use a spreadsheet tool (i.e., Microsoft Excel) for data reduction and isotherm plot development.

Copies of applicable logbooks and QA/QC documents used in testing will be provided by the laboratory to CHPRC.

4.2.6 Testing Hold Point for Results Interpretation and Quit Criteria for Future Testing

The final laboratory testing hold point is established at the conclusion of Phase 2 batch testing. At the conclusion of Phase 2 batch testing, the laboratory will prepare a summary report containing the information described in Section 4.2.5. The evaluation matrix to be used for results interpretation and decisions is presented in Table 4-6. The laboratory will use these evaluation criteria to recommend which carbon media should be retained for further testing. Following completion of testing, CHPRC, CH2M HILL, and PNNL will review the summary report and interpret the results for the next test steps. The team will determine which carbon media will be retained for further testing during Phase 3 continuous-flow column testing.

Table 4-6. Batch Isotherm Results Evaluation Criteria

1	Kd >9,008 mL/g	Retain carbon product for Phase 3 testing.
2	6,856 mL/g <Kd <9,008 mL/g	If two or more carbon products already meet the conditions of criterion #1, then reject this carbon for consideration for Phase 3 testing. If one or none of the carbon products meets the conditions of criterion #1, then evaluate further and discuss whether this carbon product should be retained.
3	Kd <6,856 mL/g	Reject this carbon for consideration in Phase 3 testing.
Kd = distribution coefficient		

If more than two different carbon products meet the conditions of criterion #1, then CHPRC and CH2M HILL will need to select only two carbon types that should be retained for additional testing under Phase 3 (Section 4.3). The carbons ultimately selected for Phase 3 testing would assumedly be those with the highest Kd values, but other considerations may be appropriate based on the Phase 2 data results.

Criterion #2 was established to prevent carbon product results that are slightly under the established target Kd of 9,008 mL/g from being rejected for further testing under Phase 3 (if none of the isotherm tests gave Kd values at or above the economic target value). As stated in the literature survey and Chapter 2 herein, this target was derived assuming that carbon pre-treatment would be attractive if a 50 percent cost savings could be accomplished. Assuming a 50 percent cost savings relative to IX resin replacement costs, the required carbon replacement costs and target Kd value were back-calculated. The low-end Kd value for

criterion #2 of 6,856 mL/g was established by similar means assuming only a 33 percent cost savings. The Kd value of 6,856 mL/g is 76 percent of the target Kd value 9,008 mL/g.

Carbon products showing Kd values within the range of criterion #2 will need to be discussed among team members, and the team will determine if these carbons should be considered for Phase 3 testing. As noted in Table 4-4, however, if a sufficient number of carbon products pass criterion #1, then the criterion #2 carbon products will likely not be considered.

Any carbon products that exhibit Kd values significantly lower than the target Kd (less than 6,856 mL/g) will not be considered for additional testing in Phase 3.

Therefore, the three possible outcomes at the conclusion of Phase 2 testing are as follows:

- Conduct Phase 3 continuous-flow column testing with two selected carbon products, OR
- Conduct Phase 3 continuous-flow column testing with only one selected carbon product, OR
- Discontinue testing and do not conduct Phase 3 continuous-flow column testing. This outcome would occur if all carbon products either (1) meet the conditions of criterion #3, or (2) meet the conditions of criterion #2 but all carbons are rejected after consideration.

4.2.7 Quality Control and Quality Assurance Measures

The laboratory will perform two identical test runs for each carbon product to allow for duplicate results as a QC measure. Assuming the number of tests identified in Tables 4-2 through 4-5, a total of 60 QC duplicate tests may be required.

Other QC measures will be designated by the laboratory in their test plan based on the laboratory's standards of practice and analysis methods.

The laboratory must establish procedures for maintaining QA throughout testing. For example, the laboratory shall use reagent-grade chemicals that are used within the product shelf life.

4.2.8 Waste Management

The laboratory will be responsible for managing all laboratory waste, including all radioactive waste, hazardous waste, and mixed wastes. Waste management will be in accordance with PNNL standards of practice. Procedures for laboratory waste management will be addressed in the laboratory test plan.

Possible laboratory wastes may include spent test water, activated carbon media adsorbed with mixed wastes (e.g., radionuclide, nonradionuclide COCs, solvents, etc.), unused carbon samples, spent stock solutions, rinsates, and/or sodium-salicylate/technetium-99 concentrated liquid waste. A list of possible laboratory wastes will be defined in the laboratory test plan.

CHPRC will provide the groundwater and carbon samples to be used for testing.

CHPRC will coordinate with PNNL to arrange the transport and appropriate disposal of all investigation-derived waste. CHPRC will pursue having all waste materials returned for management onsite. The appropriate *Comprehensive Environmental Response, Compensation, and Liability Act of 1980* (CERCLA) decision documents will need to be identified by CHPRC for waste designation and disposal management. All effluent water used in testing may be potentially treatable onsite. All other wastes generated during laboratory testing may be potentially disposed onsite.

At the end of testing, unused test water can be returned to CHPRC if designated as a "sample return."

The laboratory will be responsible for segregating wastes during testing. At the end of testing, the laboratory will need to sample each waste type for the complete list of COCs (as specified in the waste data quality objectives document), as well as any chemicals that were added as part of the testing (e.g., HCl or Na-salicylate). These sample results will be sent to CHPRC for review and determination of how each waste type will be handled. The laboratory will be responsible for proper storage of the waste until a waste designation is provided by CHPRC. The laboratory will also be responsible for transportation of the laboratory wastes to the CHPRC-designated waste or treatment facility.

4.2.9 Qualifications

The project manager for Phase 2 testing will be Ron Smith. Mr. Smith is a senior project manager with PNNL. Since 1977, Mr. Smith has worked on environmental assessments of mixed waste disposal facilities associated with nuclear fuel reprocessing and with the U.S. Department of Defense on investigation and remediation of CERCLA and *Resource Conservation and Recovery Act of 1976* (RCRA) facilities.

The technical manager for Phase 2 testing will be Dr. Shas Mattigod. Dr. Mattigod is a Scientist V with PNNL. Dr. Mattigod's professional experience is related to characterizing and developing remediation methods for liquid and solid effluents, leachates, RCRA wastes, contaminated soil, and groundwater from CERCLA sites.

The laboratory operations manager for Phase 2 testing will be Dr. Dawn Wellman. Dr. Wellman is a staff scientist with PNNL. Her research combines results of experimental investigations using variably saturated column testing and single-pass flow-through and static dissolution tests under complex conditions with spectroscopic, hydrodynamic, and geochemical thermodynamic modeling to quantify fundamental processes and interactions occurring at fluid/solid interfaces and predict the coupled effects of hydro-geochemical interactions controlling the longevity and long-term fate of metal and radionuclide contaminants in various aqueous media including groundwater, produced water, and process effluents. Other key personnel for Phase 2 testing are as follows:

- Kent Parker is a Scientist II within the Field Hydrology and Geochemistry group at PNNL. Mr. Parker will perform the batch isotherm tests.
- Mike Lindberg is an analytical chemist within the Applied Geology and Geochemistry group at PNNL. Mr. Lindberg will perform laboratory analyses of solutions.
- Steven Baum is a Grade 5 technician within the Applied Geology and Geochemistry group at PNNL. Mr. Baum will also perform laboratory analyses of solutions.

4.2.10 Safety

The laboratory will be responsible for addressing all safety hazards anticipated for this work. Radiological safety hazards must be addressed. Health and safety procedures will be included in the laboratory test plan. Job hazard analysis forms will be prepared and included in the test documentation.

4.3 Phase 3 — Continuous-Flow Column Adsorption Testing

Phase 3 testing consists of adsorption testing using continuous-flow column methods and the activated carbon products retained during Phase 2 testing (Section 4.2). Phase 3, which is third and final phase of testing, will be completed by PNNL.

4.3.1 Test Performance Goals

The performance goals for Phase 3 testing are as follows:

- Determine the technetium-99 effluent concentration from test columns for the activated carbon product(s) as a function of water volume through columns.
- Determine when technetium-99 breakthrough occurs and the shape of the breakthrough curve.
- Determine the activated carbon loading capacity for technetium-99.
- Determine what other constituents are removed by activated carbon.
- Evaluate VOC loadings on activated carbon and compare to disposal limits.
- Determine whether hot water/air stripping regeneration methods could be used to remove VOCs from activated carbon to concentrations below the disposal limits.
- Determine whether technetium-99 would be desorbed from activated carbon during hot water/air stripping and, if so, the amount of technetium-99 that is desorbed.

4.3.2 Test Materials

4.3.2.1 Source Water for Testing

Groundwater extracted from well 299-W19-36 will also be used for Phase 3 testing. As described in Section 4.2, the source water must be prepared prior to testing. The laboratory will need to prepare a sample request form for a special study using well 299-W19-36 sample water. CHPRC and PNNL will work together to define requirements for collecting samples, manifesting, and shipping. The laboratory will designate the required water quantity for Phase 3 testing.

4.3.2.2 Carbon Samples

Up to two carbon products shall be retained for this phase of testing, as specified in Section 4.2. Additional sample volume of the candidate activated carbon products from vendors should be obtained if necessary.

4.3.2.3 Laboratory Equipment

Small test columns will need to be acquired for GAC testing. Available sizing and material options will be discussed with CHPRC and PNNL. The selected column size, material, and other parameters will be included in the laboratory proposed test plan. The laboratory should provide a flow diagram for the test apparatus to be used for Phase 3 testing.

For a laboratory-conducted column test, it is assumed that each test column diameter will be at least 10 mm and no more than 25 mm. The test column will need to be relatively small to maintain a low column flow rate and to limit the required groundwater volume that must be transported into the laboratory. The internal diameter of the test column should be a minimum of 50 times the average particle diameter of the tested carbon media. Carbon column depths should be maintained within a reasonable depth for bench-scale testing and will need to be sized by the laboratory. As a general guideline, the ratio of carbon column depth to column diameter is typically a minimum of 4:1 and a maximum of 12:1. Three columns should be installed in series with sample ports at the effluent of each column. Test data can be collected showing the constituent loading in each bed with the installation of multiple columns. The time for carbon bed exhaustion can also be predicted more easily during the test.

If a powdered activated carbon product is retained for testing, then discussions with CHPRC and PNNL will be required to determine the filter device that would be most appropriate for use during laboratory testing. Potential options include cartridge-type filters or ceramic candle-type filters.

In addition to the test column, a source-water holding tank and an effluent water-collection tank will be required. A small pump and flow meter will also be required along with tubing, fittings, valves, and other miscellaneous parts. Details for the test column apparatus will be provided by the laboratory in their test plan.

4.3.3 Analytical Methods

The standard analytical methods to be used will be addressed by the laboratory in their test plan (i.e., ASTM).

4.3.3.1 Test Water Initial Concentrations

Source water will be analyzed prior to column testing. The test water will be analyzed for the following:

- Technetium-99
- Uranium (total)
- Carbon tetrachloride
- TCE
- VOCs
- Hexavalent chromium
- Total chromium
- Nitrate
- Total organic carbon
- Total suspended solids
- pH and temperature.

The source test water will be analyzed for the presence of other VOCs that are identified as project constituents of interest and could be present in well 299-W19-36 groundwater. In addition to the COCs carbon tetrachloride and TCE, these VOCs could also potentially adsorb onto activated carbon. For this reason, test water will be analyzed for VOCs in accordance with standard method. The VOCs to be analyzed include: acetone, carbon tetrachloride, chloroform, dibromochloromethane, methylene chloride, tetrachloroethene, TCE, 1,1,1-trichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene (total), and BTEX (benzene, ethylbenzene, toluene, and xylenes). These specific VOCs are the same constituents analyzed in the initial test water in Section 4.2.3.1.

4.3.3.2 Column Effluent Water Concentrations

Test column effluent water will be analyzed for technetium-99. As part of test plan preparation, the laboratory will identify the minimum turnaround times available for analysis of technetium-99. This will be critical during Phase 3 continuous-flow testing because the data will be needed quickly when breakthrough in the carbon filter begins to occur. The laboratory should consider use of an auto-sampler, which would assist with turnaround-time issues.

4.3.3.3 Final Carbon Media

After continuous-flow column testing, the spent carbon media will be analyzed to determine whether other constituents were adsorbed onto the activated carbon. Carbon media will be analyzed for the following:

- Technetium-99
- Uranium (total)
- Nitrate
- Hexavalent chromium
- Total chromium.

4.3.4 Test Procedures

As previously stated, the laboratory will develop a detailed test plan for Phase 3 column testing. Therefore, the following test procedures should be used as guidelines only.

4.3.4.1 Column Testing for Technetium-99 Adsorption

The continuous-flow column testing will be conducted using standard practices for rapid small-scale column testing for GAC and aqueous-phase systems. Refer to ASTM standard practices for testing procedures.

Once the carbon products are selected for Phase 3 testing, the laboratory will need to calculate the empty-bed contact time, hydraulic loading rate, test column carbon depth, and flow rate for the small-scale columns to be used in testing. The laboratory will need to scale these parameters based on the small test column average carbon particle diameter, the "as-received" average carbon particle diameter that would be used in a full-scale system and the assumed characteristics for a full-scale system. Column parameters should be scaled based on a full-scale, empty-bed contact time of 15 minutes, a flow rate of 335 gallons per minute (gpm) and a hydraulic loading rate of 4.3 gpm/ft² (corresponds to a 10-ft diameter vessel).

Small-scale column tests can be designed (scaled) based on assumptions of constant surface diffusivity (ASTM D6586-03, *Standard Practice for Prediction of Contaminant Adsorption on GAC in Aqueous Systems Using Rapid Small-Scale Column Tests*) or proportional surface diffusivity ("Design of Rapid Fixed-Bed Adsorption Tests for Non-Constant Diffusivities" [Crittenden et al., 1987]). The appropriate method is often determined based on experimental knowledge of the adsorption mechanism for the target compounds to be adsorbed, whereas rapid small-scale column testing selection is determined by considering those mechanisms that cause spreading in the mass transfer zone and breakthrough curve ("Predicting GAC Performance with Rapid Small-Scale Column Tests" [Crittenden et al., 1991]). Since this information is not readily available for pertechnetate, the constant diffusivity model is preferred over the proportional diffusivity model because the former has a smaller empty-bed contact time and test results can be obtained more quickly. The constant diffusivity model, however, may result in high head losses through the column. This factor should be evaluated during column test design and discussed with CHPRC and CH2M HILL, as appropriate. The superficial velocity through the test column could be lowered if needed.

Methods for calculating the column test parameters will be included in the laboratory test plan.

1. Prepare the first type of carbon media. Reduce activated carbon samples in particle size to a U.S. 60- by 80-mesh sieve size. Wash products with deionized water. Continue washing the carbon and pouring off supernate until the supernate is clear. Dry the carbon products in an oven overnight until no further weight loss occurs. Experimentally determine and record the moisture content, apparent density, and average particle diameter of the prepared carbon. Preparing the GAC and calculating the final carbon sample parameters is very important for good test results.
2. Prepare source water (described in Section 4.2.4.1, steps 2a through 2f). Adjust the nitrate concentration to 375 mg/L.

3. After the source water is prepared for testing, analyze the source water for the constituents identified in Section 4.3.3.1.
4. Set up the three-column test apparatus for the first type of candidate activated carbon.
5. Deaerate test carbon and slurry carbon into the column. Record the carbon bed depths.
6. Begin feeding groundwater into the column. Water should flow through the column continuously and at a constant flow rate. Record the flow rate and any interruption to the flow.
7. Regularly collect water from the lead, middle, and lag carbon column effluent sample taps during the column test. To accomplish this, the sampling interval will need to be established based on the estimated column run time. The estimated column run time before breakthrough can be calculated based on the laboratory selected flow rate, the technetium-99 source water influent concentration and the Kd value established for the carbon during the Phase 2 batch adsorption testing. Assuming that the run time for each column would be approximately 3 days, the column effluent water should be regularly sampled a minimum of four times daily.
8. Analyze for the technetium-99 concentration, in the lead column effluent only, until technetium-99 concentrations begin to increase in the lead column effluent (i.e., breakthrough begins to occur). Once breakthrough begins in the lead column, start analyzing the water from the middle column effluent. When the technetium-99 concentrations begin to increase at the middle column effluent sample port, begin analyzing water in the lag column effluent. Analyzing the samples in this manner will reduce the total number of samples that must be analyzed; however, water volumes will still be collected and held. A minimum of 20 samples should be analyzed during each continuous-flow column test. The laboratory will need to determine the final number of samples analyses needed to create complete breakthrough curves.
9. As the effluent concentration of technetium-99 begins to increase (i.e., when breakthrough begins), increase the frequency of effluent sample collection and analysis for the column experiencing breakthrough and minimize turnaround times for data results. (Previous studies have shown that breakthrough of technetium-99 has occurred quickly.)
10. At the end of the column test, the spent carbon from the lead test column only should be analyzed for the constituents identified in Section 4.3.3.3.
11. If two carbon products were selected for Phase 3 testing, repeat steps 1 through 10 for the second carbon product.

4.3.4.2 Column Testing for Volatile Organic Compound Adsorption

One of the carbon products tested in Section 4.3.4.1 column testing will be further tested to evaluate VOC loadings with the original groundwater VOC concentrations. If two carbon products were selected for Phase 3 testing, then the laboratory should confirm with CHPRC regarding which carbon product should be used for additional testing.

1. Prepare the carbon media (described in Section 4.3.4.1, step 1).
2. Adjust the pH of the source water to 7.7, which is the average pH shown in Table 2-1. Adjust the temperature of the source water to 18.3°C (65°F), which is the raw well water temperature shown in Table 2-1. Adjust the nitrate concentration to 375 mg/L.
3. Analyze the source water for the constituents listed in Section 4.3.3.1.

4. Set up a one-column test apparatus for the activated carbon. Use one column from the test column apparatus used in Section 4.3.4.1 testing.
5. Follow procedures for preparing the carbon column in Section 4.3.4.1, step 5.
6. Begin feeding groundwater into the column. Maintain the same flow rate as used in the previous column test in Section 4.3.4.1. Water should flow through the column continuously and at a constant flow rate. Record the flow rate and any interruption to the flow.
7. The column run time before technetium-99 breakthrough, as determined during Section 4.3.4.1 testing, should be used as a guideline for the expected run time for this column test. Begin sampling and analyzing the column effluent water for technetium-99 before breakthrough is expected to begin. As the effluent concentration of technetium-99 increases (i.e., as breakthrough is occurring), increase the frequency of effluent sample collection and analysis in order to predict the point of 100 percent breakthrough.
8. Discontinue testing once technetium-99 breakthrough reaches 100 percent.
9. Analyze the spent carbon from the test column for the constituents listed in Section 4.3.3.3. Also analyze the spent carbon for the following VOCs: acetone, carbon tetrachloride, chloroform, dibromochloromethane, methylene chloride, tetrachloroethene, TCE, 1,1,1-trichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene (total), and BTEX (benzene, ethylbenzene, toluene, and xylenes).
10. The disposal limit is 6 parts per million (in mg/kg) for carbon tetrachloride and TCE. If the concentration of both of these constituents on the spent carbon media is less than the disposal limit, then discontinue the remaining testing described in Section 4.3.4. If the concentration of either of these constituents on the spent carbon media is equal to or greater than the disposal limit, then proceed to the testing steps in Section 4.3.4.3.

4.3.4.3 Carbon Testing for Volatile Organic Compounds and Technetium-99 Desorption

If the concentrations of carbon tetrachloride or TCE on the spent carbon exceed the disposal limits, then the laboratory will attempt to strip the spent carbon of VOCs.

1. Heat high-purity water in a test container to 60°C (140°F).
2. Place spent carbon material from Section 4.3.4.2 column testing into the hot water. Maintain the hot water temperature at 60°C (140°F). Air sparge the carbon/hot water mixture for approximately 6 hours. This procedure is intended to simulate the hot water air-stripping process that will be used for VOC stripping from spent resin materials in the 200 West Area pump-and-treat facility.
3. After the hot water air sparging is complete, separate the water from the carbon media.
4. Analyze the hot water treated carbon media for technetium-99 and the VOCs listed in Section 4.3.4.2 (step 9).
5. Analyze the stripping water for technetium-99 and the VOCs listed in Section 4.3.4.2 (step 9).

4.3.5 Data Results

Following completion of testing and data evaluation, a formal report providing the methods, data results, and interpretation of results for both Phase 2 batch isotherm testing (Section 4.2) and Phase 3 continuous-flow column testing (Section 4.3) will be prepared. The report will require formal PNNL review, as well as review by CHPRC and CH2M HILL.

4.3.5.1 Breakthrough Curves

Plot data results for technetium-99 column effluent concentrations versus the water volume through each column. Fit the data results to a sigmoid-type breakthrough curve. Establish points on the curve showing where the effluent concentration is 5, 10, 25, 50, 75, 90, and 100 percent of the feed concentration. Differentiate the actual data points from the curve fitting.

4.3.5.2 Total Technetium-99 Loading Capacity

Report the final technetium-99 concentration on the carbon media from each lead column. Calculate the total amount of technetium-99 that was theoretically loaded onto each activated carbon column based on the data results and the shape of the breakthrough curve. Calculate the total treatable water volume at 100 percent breakthrough.

4.3.5.3 Other Constituent Loading

Report the final concentration of uranium, nitrate, total chromium and hexavalent chromium (Section 4.3.3.3) that were loaded onto the carbon media of each lead column. Flag which of the constituents adsorbed onto the activated carbon.

4.3.5.4 Volatile Organic Compound Loading

Report the final concentration of carbon tetrachloride, TCE, and other VOCs loaded onto the spent carbon media for testing performed in Section 4.3.4.2. Indicate whether the concentration of carbon tetrachloride or TCE exceeded the disposal limit.

4.3.5.5 Volatile Organic Compound and Technetium-99 Desorption

Report the final concentration of technetium-99, carbon tetrachloride, TCE, and other VOCs on the spent carbon media after hot water air stripping was performed (Section 4.3.4.3). Indicate whether the concentrations of carbon tetrachloride and/or TCE on the hot water treated carbon media were reduced below the disposal limit. Report the final concentration of technetium-99 and VOCs in the air stripper water. Calculate the mass of technetium-99 removed from the spent carbon during the air-stripping process, if applicable.

4.3.5.6 Data Collection

The laboratory will maintain a logbook/notebook of all notes taken during testing. Data sheets may be used for data collection during column testing. The laboratory standard data reports for analytical results will be provided. The laboratory should use a spreadsheet tool (i.e., Microsoft Excel) for data reduction and development of breakthrough curves.

Copies of applicable logbooks and QA/QC documents used in testing will be provided by the laboratory to CHPRC.

4.3.6 Quit Criteria

For each column test in Section 4.3.4.1, continue introducing the source water into the test column apparatus until the one of the following occurs:

- The column effluent concentration of technetium-99 measured in the second column is 100 percent of the influent concentration (i.e., 100 percent breakthrough), OR
- After 3 weeks of continuous column testing

For the column test in Section 4.3.4.2, continue introducing source water into the test column apparatus until the column effluent concentration of technetium-99 is 100 percent of the influent concentration (i.e., 100 percent breakthrough).

Phase 3 testing must conclude no later than December 31, 2010.

4.3.7 Quality Control and Quality Assurance Measures

It is assumed that duplicate column runs will not be completed. However, if only one carbon type is selected in Section 4.2, then CHPRC and CH2M HILL may elect to perform two column tests of the same carbon media. This would allow for collection of a duplicate data set.

Other QC measures will be designated by PNNL in their test plan based on their standards of practice and analytical methods.

The laboratory must establish procedures for maintaining QA throughout testing. For example, the laboratory shall use reagent-grade chemicals that are used within the product shelf life.

4.3.8 Waste Management

The waste management guidelines for Phase 3 testing are the same as those for Phase 2 testing (Section 4.2.8).

4.3.9 Qualifications

The project manager for Phase 2 testing will be Ron Smith. Mr. Smith is a senior project manager with PNNL. Since 1977, Mr. Smith has worked on environmental assessments of mixed waste disposal facilities associated with nuclear fuel reprocessing and with the U.S. Department of Defense on investigation and remediation of CERCLA and RCRA facilities.

The technical manager for Phase 2 testing will be Dr. Shas Mattigod. Dr. Mattigod is a Scientist V with PNNL. Dr. Mattigod's professional experience is related to characterizing and developing remediation methods for liquid and solid effluents, leachates, RCRA wastes, contaminated soil, and groundwater from CERCLA sites.

The laboratory operations manager for Phase 2 testing will be Dr. Dawn Wellman. Dr. Wellman is a staff scientist with PNNL. Her research combines results of experimental investigations using variably saturated column testing and single-pass flow-through and static dissolution tests under complex conditions with spectroscopic, hydrodynamic, and geochemical thermodynamic modeling to quantify fundamental processes and interactions occurring at fluid/solid interfaces and predict the coupled effects of hydro-geochemical interactions controlling the longevity and long-term fate of metal and radionuclide contaminants in various aqueous media including groundwater, produced water, and process effluents.

Other key personnel for Phase 3 testing include the following:

- Kent Parker is a Scientist II within the Field Hydrology and Geochemistry group at PNNL. Mr. Parker will perform the column tests.
- Mike Lindberg is an analytical chemist within the Applied Geology and Geochemistry group at PNNL. Mr. Lindberg will perform laboratory analyses of solutions.
- Steven Baum is a Grade 5 technician within the Applied Geology and Geochemistry group at PNNL. Mr. Baum will also perform laboratory analyses of solutions.

4.3.10 Safety

The laboratory will be responsible for addressing all safety hazards anticipated for this work. Radiological safety hazards must be addressed. Health and safety procedures will be included in the laboratory test plan. Job hazard analysis forms will be prepared and included in test documentation.

4.4 Criteria/Constraints

This testing is performed in support of CERCLA cleanup of the 200-ZP-1 OU at the Hanford Site. The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for remediation of the 200-ZP-1 OU, as described in the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) (Ecology et al., 1989). Should unforeseen conditions warrant, EPA (as the responsible regulatory agency) in consultation with the U. S. Department of Energy, Richland Operations Office, has the authority to terminate the treatability test. Testing will be subject to all CERCLA requirements, as well as all applicable or relevant and appropriate requirements, unless exempted by a waiver under CERCLA Section 121(d)(4).

Treatability testing will be conducted at the ASL and PNNL.

Work at the ASL will not involve contaminated water or other materials for the 200-ZP-1 OU. Testing at the ASL is not expected to generate hazardous or radioactive wastes, and radioactive substances will not be involved in the testing. Solid waste generated during testing will be managed and disposed in accordance with standard laboratory operating procedures.

Work at PNNL will be conducted using groundwater from the 200-UP-1 OU, which is both radioactive (contains technetium-99, uranium, iodine, and variety of other radioactive isotopes) and hazardous (listed RCRA waste codes "F001" through "F005" are applied). Transportation of water from the 200-UP-1 OU moreover is subject to U.S. Department of Energy orders and U.S. Department of Transportation requirements for the transport of hazardous waste and radioactive materials. Testing at PNNL will generate both solid waste (including spent activated carbon) and wastewater (including both treated water and excess untreated water).

All treatability test residuals generated at PNNL will be evaluated and managed in compliance with appropriate waste regulations. *Washington Administrative Code* (WAC) 173-303, "Dangerous Waste Regulations," requires the identification and appropriate management of dangerous wastes. WAC 173-304, "Minimum Functional Standards for Solid Waste Handling," requires the identification and appropriate management of solid wastes that are not dangerous wastes. Solid wastes generated during testing (i.e., unused GAC) will be disposed, as required, at the ERDF, which is designed to meet the substantive standards for disposal of solid and dangerous wastes. Purgewater generated during sampling of wells will be managed in accordance with Hanford Site requirements. CHPRC will

coordinate with PNNL to arrange for the transport and appropriate disposal of all investigation-derived waste.

4.5 Organization and Function Responsibilities

The CHPRC's Soil and Groundwater Remediation Project has primary responsibility for 200-ZP-1 OU pump-and-treat operations. CHPRC is responsible for preparing test plans, preparing work orders and subcontracts, ordering necessary supplies, and ensuring that testing begins properly and safely. Proper and active communications will be maintained with all other involved groups and functions involved in testing (i.e., safety, environmental, QA, ASL, and PNNL). CHPRC will collect the required water samples to support this work under existing procedures. Water will be transported to PNNL using existing procedures. The GAC will be purchased and shipped to ASL and PNNL by CHPRC. Trained crafts personnel will assist with sample collection, as specified in applicable procedures.

The ASL and PNNL are responsible for appropriate storage of materials supplied for testing (including radioactive and hazardous water used in batch and column testing). These laboratories are also responsible for checking and verifying proper operation of test equipment prior to testing and for following procedures specified for testing. Organizational responsibilities are summarized in Table 4-7.

Table 4-7. Organization Responsibilities

CHPRC Soil and Groundwater Water Remediation Project	• Prepare test guidelines. • Prepare and implement run plans. • Provide personnel to collect water/transport to PNNL. • Submit waste designation requests if necessary. • Approve detailed test plan. • Approve test reports. • Prepare statement of work and select laboratories. • Approve test report. • Approve work packages.
U.S. Department of Energy, Richland Operations Office	• Review planning documents. • Review reports.
CHPRC Safety Engineering	• Approve work packages for sampling and transportation.
CHPRC Radiological Control	• Approve work packages for sampling and transportation.
CH2M HILL Applied Sciences Laboratory	• Provide detailed plans for the characterization of functional groups of GAC. • Perform Phase 1 testing activities to characterize GAC. • Prepare test report from Phase 1 containing all relevant data and preliminary conclusions, and documentation. • Provide copies of controlled laboratory logbooks, procedures, and other test documentation for Phase 1.
PNNL	• Prepare detailed plan for Phase 2 and Phase 3 batch and column testing. • Perform testing activities for Phases 2 and 3. • Prepare test report from Phases 2 and 3 containing all relevant data and preliminary conclusions, and documentation. • Provide copies of controlled laboratory logbooks procedures, and other test documentation. • Provide copies of procedures used for Phases 2 and 3.

Table 4-7. Organization Responsibilities

CHPRC Waste Management	• Determine the options for management and disposition of waste products. • Specify requirements for hazardous waste. • Determine waste packaging and disposal requirements.
CHPRC = CH2M HILL Plateau Remediation Company	
GAC = granular activated carbon	
PNNL = Pacific Northwest National Laboratory	

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