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# Tc-99 Adsorption on Selected Activated Carbons

## Batch Testing Results

SV Mattigod  
DM Wellman  
EC Golovich

E Cordova  
RM Smith

December 2010



**Pacific Northwest**  
NATIONAL LABORATORY

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Pacific Northwest National Laboratory  
Richland, Washington 99352



## Summary

CH2M HILL Plateau Remediation Company (CHPRC) is currently developing a 200-West Area groundwater pump-and-treat system as the remedial action selected under the Comprehensive Environmental Response, Compensation, and Liability Act Record of Decision for Operable Unit (OU) 200-ZP-1. This report documents the results of treatability tests Pacific Northwest National Laboratory researchers conducted to quantify the ability of selected activated carbon products (or carbons) to adsorb technetium-99 (Tc-99) from 200-West Area groundwater.

The Tc-99 adsorption performance of seven activated carbons (J177601 Calgon Fitratorb 400, J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, J177612 Norit GAC830, J177613 Norit GAC830, and J177617 Nucon LW1230) were evaluated using water from well 299-W19-36. Four of the best performing carbons (J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830) were selected for batch isotherm testing.

The batch isotherm tests on four of the selected carbons indicated that under lower nitrate concentration conditions (382 mg/L),  $K_d$  values ranged from 6,000 to 20,000 mL/g. In comparison, under higher nitrate (750 mg/L) conditions, there was a measureable decrease in Tc-99 adsorption with  $K_d$  values ranging from 3,000 to 7,000 mL/g. The adsorption data fit both the Langmuir and the Freundlich equations. Supplemental tests were conducted using the two carbons that demonstrated the highest adsorption capacity to resolve the issue of the best fit isotherm. These tests indicated that Langmuir isotherms provided the best fit for Tc-99 adsorption under low nitrate concentration conditions. At the design basis concentration of Tc 0.865  $\mu\text{g/L}$  (14,700 pCi/L), the predicted  $K_d$  values from using Langmuir isotherm constants were 5,980 mL/g and 6,870 mL/g for the two carbons. These  $K_d$  values did not meet the target  $K_d$  value of 9,000 mL/g.

Tests conducted to ascertain the effects of changing pH showed that at pH values of 6.5 and 7.5, no significant differences existed in Tc-adsorption performance for three of the carbons, but the fourth carbon performed better at pH 7.5. When the pH was increased to 8.5, a slight decline in performance was observed for all carbons.

Tests conducted to ascertain the temperature effect on Tc-99 adsorption indicated that at 21°C, 27°C, and 32°C there were no significant differences in Tc-99 adsorption for three of the carbons. The fourth carbon showed a noticeable decline in Tc-99 adsorption performance with increasing temperature.

The presence of volatile organic compounds (VOCs) in the source water did not significantly affect Tc-99 adsorption on either of two carbons tested. Technetium-99 adsorption differed by less than 15% with or without VOCs present in the test water, indicating that Tc-99 adsorption would not be significantly affected if VOCs were removed from the water prior to contact with carbon.

After evaluating these results and performing a cost-benefit analysis, CHPRC personnel have selected carbon J177606 Siemens AC1230AWC as the preferred carbon source for Tc-99 removal based on batch testing. Performing follow-on column testing could be considered because it could provide important estimates of carbon lifetime in the treatment system, the amount of Tc-99 that can be loaded onto the carbon, and the quantity of volatile organic constituents contained in the groundwater that will load onto the carbon. This information is used in design of the treatment system and considerations for waste disposal of the spent carbon.



## Acronyms and Abbreviations

°C	degree(s) Celsius (or Centigrade)
°F	degree(s) Fahrenheit
ALARA	as low as reasonably achievable
CHPRC	CH2M HILL Plateau Remediation Company
COCs	constituents of concern
Cr	chromium
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
GAC	granular activated carbon
ICP-MS	inductively coupled plasma-mass spectrometry
g	gram(s)
$K_d$	distribution coefficient(s)
L	liter
mg/L	milligram(s) per liter
$\mu\text{g/g}$	microgram(s) per gram
$\mu\text{g/L}$	microgram(s) per liter
OU	Operable Unit
pCi/L	picocuries per liter
PNNL	Pacific Northwest National Laboratory
SU	standard unit(s)
Tc	technetium
TSS	total suspended solids
TOC	total organic carbon
VOC	volatile organic compound



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## 1.0 Introduction

CH2M HILL Plateau Remediation Company (CHPRC) is currently developing a 200-West Area groundwater pump-and-treat system as the remedial action selected under the Comprehensive Environmental Response, Compensation, and Liability Act Record of Decision for Operable Unit (OU) 200-ZP- 1, and in accordance with the Hanford Federal Facility Agreement and Consent Order (Ecology, EPA, and DOE 1989). The treatment design is based, in part, on the removal of selected constituents of concern (COCs) using various sorbent media. CHPRC requested that Pacific Northwest National Laboratory (PNNL) perform a treatability test to quantify the ability of selected activated carbon products (or carbons) to adsorb both radioactive (principally technetium-99 [Tc-99]) and nonradioactive COCs from 200-West Area groundwater. The carbon products supplied to PNNL for testing were selected by CHPRC from a larger population of carbon products on which they performed screening tests. This report presents the results of batch adsorption tests where the selected carbons were contacted with 200-West Area groundwater.

## 2.0 Objective

Laboratory sorption experiments included batch tests from which the sorption characteristics of each of the seven tested carbon products were quantified. Results from preliminary tests on all seven activated carbon products were used to select four of the carbons for more extensive testing from which sorption isotherms were developed.

The goals of the batch testing were to determine the following:

- Tc-99 sorption isotherms for each carbon product
- Tc-99 sorption isotherms for each carbon product at twice the initial nitrate concentration
- the effect of solution pH on Tc-99 distribution coefficients ( $K_d$ ) for each carbon product
- the effect of solution temperature on Tc-99  $K_d$  for each carbon product
- the effect of volatile constituents in the test water on Tc-99  $K_d$  for selected carbon products.

This report summarizes the results of all batch test results of activated carbons.

## 3.0 Materials and Methods

The materials and methods used for batch testing of activated carbons are described below.

### 3.1 Materials

#### 3.1.1 Carbons

The initial batch adsorption tests were conducted using the seven activated carbons selected by CHPRC presented in Table 3.1.

**Table 3.1.** Description of Granulated Activated Carbons

Sample #	Vendor	Product Name	Source Material & Treatment
J177 601	Calgon	Filtrisorb 400	Bituminous coal reagglomerated
J177 606	Siemens	AC1230AWC	Coconut shell acid washed
J177 609	Carbon Resources	CR-1240A-AW	Sub-bituminous coal/acid washed
J177 611	General Carbon	GC20X50	Bituminous coal/unwashed
J177 612	Norit	GAC830	Coconut shell unwashed
J177 613	Norit	GAC830	Bituminous coal unwashed
J111617	Nucon	LW 12x30	Coconut shell acid & water washed

#### 3.1.2 Source Water

Groundwater from well 299-W19-36 in the 200-UP-1 OU was used for all of the bench-scale tests. Table 3.2 summarizes the design groundwater composition to be treated.

**Table 3.2.** Design Composition of Influent Water to Tc-99 Ion Exchange System in 200 West Pump-and-Treat Groundwater Treatment Facility

<b>Constituent</b>	<b>Concentration (mg/L)</b>
Sodium	24
Potassium	7
Calcium	75
Magnesium	24
Iron (dissolved)	0.19
Manganese (dissolved)	0.049
Chloride	18
Sulfate	34
Nitrate as N	69
Nitrate as ion	306
Alkalinity (as CaCO <sub>3</sub> )	103
Fluoride	0.37
Total organic carbon	1.3
Total dissolved solids	614
<b>Constituent</b>	<b>Concentration (µg/L)</b>
Hexavalent chromium	161
Uranium	5.9
Carbon tetrachloride	491
Trichloroethene	3.2
Chloroform	0.025
<b>Radionuclides</b>	<b>Activity (pCi/L)</b>
Tc-99	14,700
I-129	0.86
H-3	23,800
Am-241*	0.5
C-14*	13.1
Co-60*	15.2
Cs-137*	5.18
Ni-63*	7.1
Np-237*	0.075
Pu-239/240*	0.5
Se-79*	784
Sr-90*	12.4
Data: CALC 382519-TMEM-003, 200-ZP-1 Basis of Design Memorandum. Rev. 4, August 11, 2009.	
*Data taken from 382519-CALC-017, Rev. 3, April 2010.	

## 3.2 Methods

### 3.2.1 Activated Carbons

All carbons were ground so that at least 95 percent of each passed through a U.S. 325-mesh sieve when wet screened. Each of the ground and sieved carbons were centrifuge washed with deionized water and the wash water was decanted. The centrifuge washing was repeated until the supernate was clear. Then the carbons products were dried in an oven overnight until no further weight loss occurred.

### 3.2.2 Source Water

The source water for these tests was collected from well 299-W19-36. The water was analyzed for volatile organic compounds (VOCs), including acetone, carbon tetrachloride, chloroform, dibromochloromethane, methylene chloride, tetrachloroethene, trichloroethene, 1,1,1-trichloroethane, 1,1 dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene (total), and BTEX (benzene, toluene, ethylbenzene, and xylenes) by gas chromatography. About 20-liter quantities of water were air sparged to remove the VOCs until they were below detection limits. A quantity of unsparged source water was set aside for testing the effect of VOCs on Tc-99 adsorption on activated carbons. If necessary, sodium nitrate was added to the source water to adjust the nitrate concentration to ~375 mg/L. The temperature of stored source water was maintained at  $18.3 (65^{\circ}\text{F}) \pm 2^{\circ}\text{C}$  (ambient temperature in the laboratory). The constituents in the source water (e.g., Tc-99, uranium, Cr (VI), total Cr, nitrate, total organic carbon, and total suspended solids, pH and temperature) were analyzed.

### 3.2.3 Solution Analysis

Concentrations of Tc-99 and total chromium (Cr) were measured by inductively coupled plasma-mass spectrometry (ICP-MS)<sup>1</sup>. Hexavalent chromium was measured using an U.S. Environmental Protection Agency (EPA 2004) method. Nitrate analysis was conducted using ion-chromatography.<sup>2</sup> The total organic carbons (TOC) analyses were performed using a PNNL standard method.<sup>3</sup> The total suspended solids (TSS) in groundwater was measured using a standard method. The pH measurements were conducted using a PNNL standard method.<sup>4</sup> Detailed descriptions are provided in the Appendix.

### 3.2.4 Adsorption Test Method

These batch adsorption tests (in duplicate) were conducted using measured quantities of carbon product and sparged source water. At the beginning of each batch test, the carbon samples were deaerated by pulling a vacuum on each centrifuge tube for 5 minutes. Next, measured volumes of

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<sup>1</sup> PNNL-AGG-415. 2008. "Inductively Coupled Plasma Mass Spectrophotometry (ICP-MS) Analysis." Pacific Northwest National Laboratory, Richland, Washington (unpublished technical procedure).

<sup>2</sup> PNNL-AGG-IC-001. 2004. "Determinations by Ion Chromatography (IC)." Pacific Northwest National Laboratory, Richland, Washington (unpublished technical procedure).

<sup>3</sup> Kutnykov, I. 2004. "Operating of Carbon Analyzer (TOC-V + SSM-5000A + ASI (Shimadzu))." AGG-TOC-001, unpublished PNNL Technical Procedure, Pacific Northwest National Laboratory, Richland, Washington.

<sup>4</sup> Valenta, MM. 2009. "pH Measurements." AGG-pH-00 1, unpublished PNNL Technical Procedure, Pacific Northwest National Laboratory, Richland, Washington.

sparged groundwater were added to centrifuge tubes containing the appropriate masses of carbon. The adsorption tests were conducted for 24 hours. During this time, the centrifuge tubes were agitated continuously to keep the carbon and groundwater well mixed. The tubes were agitated using temperature-controlled ( $\pm 0.5^\circ\text{C}$ ) shaker incubators. After the required contact time ( $\sim 24$  hr), each tube was centrifuged, then contact solution was separated from carbons using  $0.45\text{-}\mu\text{m}$  syringe filters, then Tc-99 concentrations were measured on the sample aliquots.

### 3.2.5 Activated Carbon Screening Tests

An initial set of tests on seven carbons were performed to select the carbon/solution ratios to be used in batch isotherm tests. Sparged source water was used in these tests, in which  $0.1$  g of each carbon was contacted with  $50$  mL of source water (carbon to solution ratio of  $2 \times 10^{-3}$  g/mL). The results of these tests were also used to select four carbons for all subsequent batch tests.

### 3.2.6 Batch Isotherm Tests

Batch isotherm tests were conducted using four of the initial seven carbons that were selected by CHPRC. The CHPRC selection process was based on how each carbon performed in the initial screening tests and a cost analysis from which a desired minimum  $K_d$  value of  $9,000$  mL/g was selected. The four carbons selected for the isotherm tests were J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830. The U.S. Department of Energy (DOE) principle of limiting radiation dose to as low as reasonably achievable (ALARA) was used to minimize waste volume by selecting appropriate masses of carbons and solution volumes. Accordingly, the mass of carbon-to-solution volumes used in each batch contact was reduced while maintaining the same carbon-to-solution (W/V) ratio as specified in Test Plan SGW-47038. These tests were conducted with sparged and pH-adjusted ( $7.7 \pm 0.2$ ) source water. For the second set of tests, sodium nitrate was added to adjust the nitrate concentration in the source water to twice the concentration ( $\sim 750$  mg/L) to be used in the first set of experiments ( $\sim 375$  mg/L). The actual concentrations achieved in the tests are presented with the results of the testing. The following carbon-to-solution ratios were used in all batch isotherm tests:

- $0.01$  g carbon/ $50$  mL solution ( $2 \times 10^{-4}$  g/mL)
- $0.02$  g carbon/ $50$  mL solution ( $4 \times 10^{-4}$  g/mL)
- $0.04$  g carbon/ $50$  mL solution ( $8 \times 10^{-4}$  g/mL)
- $0.1$  g carbon/ $50$  mL solution ( $2 \times 10^{-3}$  g/mL).

The adsorption tests were conducted as described previously in the Adsorption Test Method section. A total of  $64$  batch isotherm tests were conducted (including duplicates) and the test matrix used is listed in Table 3.3.

**Table 3.3.** Batch Isotherm Testing Matrix

Isotherm #	Nitrate Conc (mg/L)	Carbon	Carbon/Soln Ratio (g/mL)	Carbon/Soln Ratio (g/mL)	Carbon/Soln Ratio (g/mL)	Carbon/Soln Ratio (g/mL)
1	~375	J177606 Siemens AC1230AWC	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
2	~375	J177609 Carbon Res. CR-1240-AW	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
3	~375	J177611 General Carbon GC20X50	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
4	~375	J177613 Norit GAC830	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
5	~750	J177606 Siemens AC1230AWC	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
6	~750	J177609 Carbon Res. CR-1240-AW	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
7	~750	J177611 General Carbon GC20X50	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$
8	~750	J177613 Norit GAC830	$2 \times 10^{-4}$	$4 \times 10^{-4}$	$8 \times 10^{-4}$	$2 \times 10^{-3}$

### 3.2.7 Variable pH Batch Tests

Another set of batch tests was set up to evaluate the effects of variable pH on Tc-99 adsorption, as follows. Using the prepared source water (with nitrate concentration adjusted to 375 mg/L) and four carbon products (J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830), three sets of tests were conducted with the pH of the source water adjusted to 6.5, 7.5, and 8.5, respectively. The pH adjustments were within  $\pm 0.2$  standard units (SU) and all the tests were conducted in duplicate at a fixed carbon-to-solution ratio of  $4 \times 10^{-4}$  g/mL. The adsorption tests were conducted as described previously in the Adsorption Test Method section. A total of 24 batch tests were conducted (including duplicates) and the test matrix used is listed in Table 3.4.

**Table 3.4.** Variable pH Test Matrix

pH Curve	Nitrate Conc (mg/L)	Carbon	Carbon/Soln Ratio (g/mL)	Test pH 1	Test pH 2	Test pH 3
1	~375	J177606 Siemens AC1230AWC	$4 \times 10^{-4}$	6.5	7.5	8.5
2	~375	J177609 Carbon Res. CR-1240-AW	$4 \times 10^{-4}$	6.5	7.5	8.5
3	~375	J177611 General Carbon GC20X50	$4 \times 10^{-4}$	6.5	7.5	8.5
4	~375	J177613 Norit GAC830	$4 \times 10^{-4}$	6.5	7.5	8.5

### 3.2.8 Variable Temperature Batch Tests

Another set of batch tests was set up to evaluate the effects of temperature on Tc-99 adsorption, as follows. Using the prepared source water (with nitrate concentration adjusted to 375 mg/L) and four carbon products (J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830), three sets of tests were conducted with the temperature of the test water set to 21°C (69.8°F), 27°C (80.6°F), and 32°C (89.6°F). The set temperatures were controlled within  $\pm 0.5^\circ\text{C}$  using a temperature incubator shaker. All the tests were conducted in

duplicate at a fixed carbon-to-solution ratio of  $2 \times 10^{-3}$  g/mL. The adsorption tests were conducted as described previously in the Adsorption Test Method section. A total of 24 batch tests were conducted (including duplicates) and the test matrix used is listed below (Table 3.5).

**Table 3.5.** Variable Temperature Test Matrix

Temp Curve	Nitrate Conc (mg/L)	Carbon	Carbon/Soln Ratio (g/mL)	Temp 1	Temp 2	Temp 3
				(°C)	(°C)	(°C)
1	~375	J177606 Siemens AC1230AWC	$4 \times 10^{-4}$	21	27	32
2	~375	J177609 Carbon Res. CR-1240-AW	$4 \times 10^{-4}$	21	27	32
3	~375	J177611 General Carbon GC20X50	$4 \times 10^{-4}$	21	27	32
4	~375	J177613 Norit GAC830	$4 \times 10^{-4}$	21	27	32

### 3.2.9 Volatile Organic Carbon Effect Tests

An abbreviated set of tests was conducted to evaluate the effects of VOCs in the source water on Tc-99 adsorption on two activated carbons (J177606 Siemens AC1230AWC and J177613 Norit GAC830). These two carbons were selected by CHPRC based on the adsorption isotherm data. The contact solution consisted of unpurged groundwater with VOCs intact (as described previously in the source water preparation section). The tests were conducted at two selected carbon-to-solution ratios— $4 \times 10^{-4}$  g/mL and  $2 \times 10^{-3}$  g/mL. The adsorption tests were conducted as described previously in the Adsorption Test Method section. A total of eight batch tests were conducted (including duplicates) and the test matrix used is listed below (Table 3.6).

**Table 3.6.** VOC Effects Test Matrix

VOC Test #	Nitrate Conc (mg/L)	Carbon	Carbon/Soln
			Ratio (g/mL)
1	~375	J177606 Siemens AC1230AWC	$4 \times 10^{-4}$
2	~375	J177606 Siemens AC1230AWC	$2 \times 10^{-3}$
3	~375	J177613 Norit GAC830	$4 \times 10^{-4}$
4	~375	J177613 Norit GAC830	$2 \times 10^{-3}$

## 3.3 Results

### 3.3.1 Constituents in Source Water

The results of initial analyses of source water from 299-W19-36 used in these batch experiments are listed in Table 3.7 and Table 3.8.

**Table 3.7.** Concentrations of Specified Constituents in 299-W19-36 Source Water

Constituent	Concentration (µg/L)	Constituent**	Unsparged Conc (µg/L)	Sparged Conc (µg/L)
Barium	113	Acetone	<0.0028	<0.0028
Calcium	122,000	1,1-Dichloroethene	0.01	0.01
Chloride	181,000	Methylene Chloride	0.15	0.15
Total Cr	<17.3	cis1,2-dichloroethene	<0.001	<0.001
Cr(VI)	<0.05	chloroform	0.25	0.01
Magnesium	36,400	1,2 dichloroethane	0.10	<0.002
Molybdenum	65.9	1,1,1 trichloroethane	<0.002	<0.002
Nitrate	317,000	benzene	0.01	0.01
Potassium	7,020	carbon tetrachloride	4.99	0.03
Sodium	118,000	trichloroethene	0.09	0.02
Sulfate**	50,000	toluene	0.02	0.03
Strontium	618	Dibromochloromethane	0.01	0.01
Tin***	216	Tetrachloroethene	<0.001	<0.001
Alkalinity (as CaCO <sub>3</sub> )*	116,000	ethyl benzene	0.03	0.06
Uranium**	174	p/m xylene	0.08	0.19
Total Suspended Solids	607	o-xylene	0.04	0.09
Total Organic Carbon	<5			
pH	8.2 (SU)			

\* Average of duplicate measurements

\*\* Average of four measurements (samples SS-0911410-AIN-ALPH A, SS-0911410-AIN-ALPH B, SW-091140-1400 ALPH, and SW-091140-1400 LPH)

\*\*\* The source of these constituents in the groundwater is unknown.

Because the different batch tests were conducted over a period of several weeks, the technetium concentration in the stored source water was analyzed before each set of batch experiments was initiated. These analyses were conducted to account for any changes in technetium concentration that may have occurred in the stored source water due to adsorption and desorption on suspended solids, growing algae,<sup>1</sup> and container wall. Therefore, the source water was filtered and analyzed for Tc concentration as needed.

**Table 3.8.** Technetium Concentrations of Source Water Used for Batch Experiments

Sample	Tc Conc (µg/L)	Tc Conc (pCi/L)
SW-081610-1, sparged low nitrate	0.433	7,360
SW-082310-1 sparged, high nitrate	0.437	7,430
SW-091510-pH6.5	0.389	6,610
SW-091510-pH7.5	0.389	6,610
SW-091510-pH8.5	0.389	6,600
SW-091510-VOC	0.385	6,540
SW-091510-TEMP	0.444	7,550
SS-0911410-AIN-ALPH-A	0.373	6,340
SS-0911410-AIN-ALPH-B	0.397	6,750
SW-091140-1400-ALPH	0.383	6,510

<sup>1</sup> When the stored source water sample aliquots were filtered before analyses, green staining was observed on the filter that indicated that the presence of algae.

### 3.3.2 Activated Carbon Screening Tests

The results of carbon screening tests are tabulated in Table 3.9 and shown graphically in Figure 3.1. The distribution coefficients ( $K_d$ ) for Tc-99 adsorption for these activated carbons ranged from 5,000 mL/g to 12,000 mL/g. The four activated carbon samples with distribution coefficients near or above the criterion value of 9,000 mL/g were as follows:

- Norit GAC830 (Sample J177-613), an unwashed bituminous coal ( $K_d = 12,000$ ), whose Tc-99 uptake characteristics were unanticipated from the initial acid-base titration curves<sup>1</sup>
- Carbon Resources CR-1240A-AW (Sample J177-609), an acid-washed sub-bituminous coal ( $K_d = 11,200$ ), whose Tc-99 uptake characteristics were expected to be favorable based on its acid-base titration curves
- Siemens AC1230AWC (Sample J177-606), acid-washed coconut shell ( $K_d = 8,920$ ), which was believed to have Tc-99 uptake potential based on acid-base titration curves
- General Carbon GC20x50 (J177-611), an unwashed bituminous coal ( $K_d = 8,700$ ), which was expected to have poor Tc-99 uptake potential based on its acid-base titration curve.

The other three granular activated carbon (GAC) samples, Calgon Filtrasorb 400 (Sample J177-601), Norit GAC830 (Sample J177-612), and Nucon LW 12x30 (Sample 111-617) had screening  $K_d$  values that were lower than the four selected carbons and below the target value of 9,000 mL/g.

These results indicate that titration screening of activated carbons may not be an appropriate means to evaluate their Tc-99 adsorption potential and the best way to assess this potential would be to actually conduct batch adsorption experiments.

The four carbon types in the bullets listed above were further evaluated in batch isotherm tests, described in the following section.

### 3.3.3 Batch Isotherm Tests

The results of batch isotherm tests conducted on the four selected activated carbons listed in the preceding section (606, 609, 611, and 613) are listed in Table 3.10 through Table 3.12.

- Siemens AC1230AWC (Sample J177-606) under low nitrate and high nitrate conditions yielded  $K_d$  values that ranged from 9,000 to 10,000 mL/g, and 3,000 to 6,000 mL/g, respectively.
- Carbon Resources CR-1240A-AW (Sample J177-609) under low nitrate and high nitrate conditions yielded  $K_d$  values that ranged from 6,000 to 12,000 mL/g, and 3,000 to 6,000 mL/g, respectively.
- General Carbon GC20x50 (J177-611), under low nitrate and high nitrate conditions yielded  $K_d$  values that ranged from 7,000 to 11,000 mL/g, and 4,000 to 6,000 mL/g, respectively.
- Norit GAC830 (Sample J177-613), under low nitrate and high nitrate conditions yielded  $K_d$  values that ranged from 10,000 to 16,000 mL/g, and 6,000 to 7,000 mL/g, respectively.

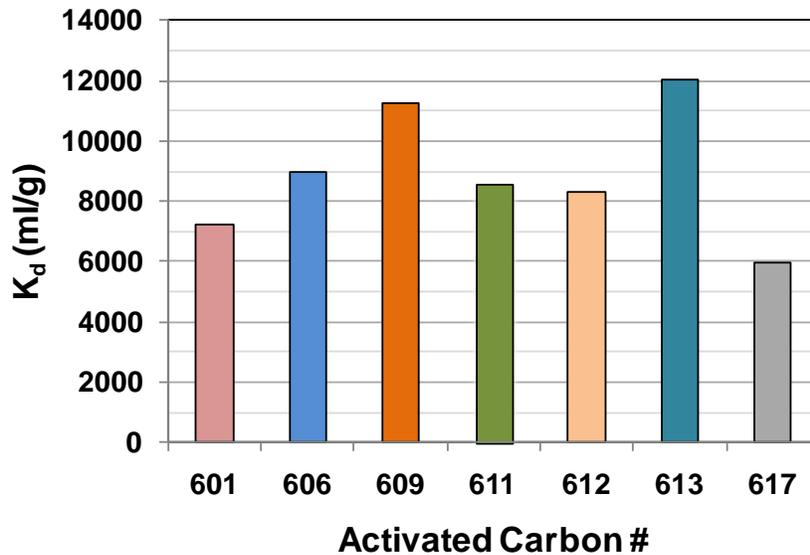
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<sup>1</sup> Technical Memorandum from K Maxey, M Schaerer, and K Perez (CH2M HILL, Inc.) to M Byrnes (CH2M HILL Plateau Remediation Company), Subject: "Titration Screening Results for Various Activated Carbon Samples," dated July 7, 2010.

**Table 3.9.** Technetium-99 Adsorption Data from Screening Batch Tests on Selected Activated Carbons

Sample #	Carbon	Carbon Mass (g)	Soln Vol (mL)	Init Tc Conc (µg/L)	Final Tc Conc (µg/L)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)	Av K <sub>d</sub> (mL/g)	Source Material & Treatment	Titration Evaluation*
J177 601	Calgon Filtrasorb 400	0.096	49.735	0.433	0.028	0.220	7,860	7,410	Bituminous coal reagglomerated	Class A - Strong pH 5 peak. <b>Anticipate Tc-99 uptake</b>
		0.100	49.938	0.433	0.029	0.202	6,970			
J177 606	Siemens AC1230AWC	0.094	49.570	0.433	0.024	0.216	9,000	8,920	Coconut shell acid washed	Class B - weak pH 5 shoulder and/or weak high pH shoulder <b>Possible Tc-99 uptake</b>
		0.092	49.766	0.433	0.025	0.221	8,840			
J177 609	Carbon Resources CR-1240A-AW	0.096	49.893	0.433	0.020	0.215	10,750	11,200	Sub-bituminous coal/acid washed	Class A - strong pH 5.0 peak <b>Anticipate Tc-99 uptake</b>
		0.104	49.588	0.433	0.017	0.198	11,650			
J177 611	General Carbon GC20X50	0.093	50.024	0.433	0.025	0.219	8,760	8,700	Bituminous coal/unwashed	Class C - No unequivocal peaks <b>Anticipate poor Tc-99 uptake</b>
		0.108	49.849	0.433	0.022	0.190	8,640			
J177 612	Norit GAC830	0.097	49.522	0.433	0.024	0.209	8,710	8,380	Coconut shell unwashed	Class C - No unequivocal peaks <b>Anticipate poor Tc-99 uptake</b>
		0.110	49.670	0.433	0.023	0.185	8,040			
J177 613	Norit GAC830	0.096	49.924	0.433	0.018	0.216	12,000	12,000	Bituminous coal unwashed	Class D - Mid-range Plateau <b>Uncertain Tc-99 uptake</b>
		0.109	50.273	0.433	0.016	0.192	12,000			
J111617	Nucon LW 12x30	0.096	49.763	0.433	0.034	0.207	6,090	5,920	Coconut shell acid & water washed	Class E - Equivocal peak at pH >8 <b>Possible Tc-99 uptake</b>
		0.095	49.595	0.433	0.036	0.207	5,750			

\*Titration Evaluation from "Titration Screening Results for Various Activated Carbon Samples," CH2M HILL Technical Memorandum, dated 6-15-2010. Carbon samples ending in numbers 606, 609, 611, and 613 were selected for isotherm testing.



**Figure 3.1.** Technetium-99 Distribution Coefficients for Seven Activated Carbons

These data indicated that technetium adsorption on these carbons was reduced by about an order of magnitude when the nitrate concentration was increased from 382 mg/L to 750 mg/L.

The adsorption data (Table 3.10 – Table 3.13) were fit using both Langmuir and Freundlich isotherms (Figure 3.2 – Figure 3.9) and the resulting constants are listed in Table 3.14. Using these constants and the design Tc concentration of 0.865  $\mu\text{g/L}$ , the predicted distribution coefficients were calculated for each carbon tested under low and high nitrate conditions.

- In all cases, the Langmuir isotherm provided a slightly better fit to the data than the Freundlich isotherm.
- For all carbons under both low and high nitrate concentrations, for the design concentration of Tc 0.865  $\mu\text{g/L}$ , the  $K_d$  values predicted using Freundlich constants were higher than the predicted  $K_d$  values derived from using Langmuir isotherm constants.
- For the design Tc concentration of 0.865  $\mu\text{g/L}$  (14,700 pCi/L), the Freundlich-predicted  $K_d$  values (Table 3.14) for only two carbons (J177606 Siemens AC1230AWC and J177613 Norit GAC830, tested under low nitrate conditions) exceeded the target  $K_d$  value of 9000 mL/g.

To resolve the question of whether the Langmuir or Freundlich isotherms provide a better fit to the data, two additional data points for each carbon were obtained by conducting batch adsorption experiments on J177606 Siemens AC1230AWC and J177613 Norit GAC830 carbons. These tests were conducted under low nitrate conditions using source water spiked at 2.205  $\mu\text{g/L}$  (37,500 pCi/L) of Tc. A solution-to-solid ratio of  $2 \times 10^{-4}$  g/mL was used and the experiment was duplicated for each carbon.

These additional data obtained from the supplemental tests are listed in Table 3.15. These data combined with the low nitrate data listed in Table 3.10 for J177606 Siemens AC1230AWC and Table 3.13 for J177613 Norit GAC830 were used in developing a new set of isotherms (Figure 3.10 and Figure 3.11).

**Table 3.10.** Tc-99 Adsorption Isotherm Data for J177606 Siemens AC1230AWC

Sample Number	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
<b>Nitrate 382 mg/L</b>						
606-18-S2-R1-1	0.437	0.129	0.013	49.136	1.164	9,020
606-18-S2-R1-2	0.437	0.118	0.014	49.203	1.121	9,500
606-18-S2-R2-1	0.437	0.087	0.018	49.354	0.960	11,060
606-18-S2-R2-2	0.437	0.085	0.019	49.075	0.909	10,680
606-18-S2-R3-1	0.437	0.048	0.040	49.616	0.482	10,030
606-18-S2-R3-2	0.437	0.050	0.038	49.065	0.500	10,090
606-18-S2-R4-1	0.437	0.019	0.099	49.828	0.211	11,390
606-18-S2-R4-2	0.437	0.020	0.100	50.611	0.211	10,550
*606-18-S1-R4-1	0.433	0.024	0.094	49.570	0.216	9,070
*606-18-S1-R4-2	0.433	0.025	0.092	49.766	0.221	8,870
<b>Nitrate 750 mg/L</b>						
606-18-S3-R1-1	0.437	0.276	0.010	49.244	0.793	2,870
606-18-S3-R1-2	0.437	0.221	0.009	49.213	1.181	5,340
606-18-S3-R2-1	0.437	0.130	0.020	49.065	0.753	5,790
606-18-S3-R2-2	0.437	0.131	0.021	50.187	0.731	5,580
606-18-S3-R3-1	0.437	0.077	0.039	49.999	0.462	6,040
606-18-S3-R3-2	0.437	0.085	0.039	49.606	0.447	5,250
606-18-S3-R4-1	0.437	0.036	0.099	49.688	0.201	5,570
606-18-S3-R4-2	0.437	0.036	0.100	49.267	0.198	5,500

\*Data from Carbon Screening Tests

**Table 3.11.** Tc-99 Adsorption Isotherm Data for J177609 Carbon Resources CR-1240-AW

Sample Number	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
<b>Nitrate 382 mg/L</b>						
609-18-S2-R1-1	0.437	0.195	0.010	49.950	1.209	6,200
609-18-S2-R1-2	0.437	0.201	0.010	49.079	1.158	5,760
609-18-S2-R2-1	0.437	0.095	0.020	49.062	0.839	8,830
609-18-S2-R2-2	0.437	0.110	0.019	50.776	0.874	7,940
609-18-S2-R3-1	0.437	0.044	0.040	49.574	0.487	11,100
609-18-S2-R3-2	0.437	0.046	0.039	49.034	0.492	10,690
609-18-S2-R4-1	0.437	0.017	0.101	49.345	0.205	12,300
609-18-S2-R4-2	0.437	0.017	0.099	50.176	0.213	12,220
*609-18-S1-R4-1	0.433	0.020	0.096	49.893	0.215	10,900
*609-18-S1-R4-2	0.433	0.017	0.104	49.588	0.198	11,670
<b>Nitrate 750 mg/L</b>						
609-18-S3-R1-1	0.437	0.259	0.009	49.822	0.985	3,810
609-18-S3-R1-2	0.437	0.261	0.010	50.265	0.885	3,390
609-18-S3-R2-1	0.437	0.153	0.021	49.319	0.667	4,360
609-18-S3-R2-2	0.437	0.145	0.019	49.004	0.753	5,190
609-18-S3-R3-1	0.437	0.088	0.039	49.448	0.443	5,050
609-18-S3-R3-2	0.437	0.080	0.040	49.032	0.438	5,480
609-18-S3-R4-1	0.437	0.034	0.100	49.035	0.198	5,870
609-18-S3-R4-2	0.437	0.032	0.099	49.017	0.201	6,330

\* Data from Carbon Screening Tests

**Table 3.12.** Tc-99 Adsorption Isotherm Data for J177611 General Carbon GC20X50

Sample Number	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
<b>Nitrate 382 mg/L</b>						
611-18-S2-R1-1	0.437	0.175	0.010	49.024	1.284	7,340
611-18-S2-R1-2	0.437	0.184	0.009	49.773	1.399	7,600
611-18-S2-R2-1	0.437	0.086	0.021	49.695	0.831	9,660
611-18-S2-R2-2	0.437	0.103	0.020	50.164	0.838	8,130
611-18-S2-R3-1	0.437	0.040	0.043	49.064	0.453	11,290
611-18-S2-R3-2	0.437	0.047	0.041	49.594	0.472	10,160
611-18-S2-R4-1	0.437	0.019	0.103	49.987	0.203	10,450
611-18-S2-R4-2	0.437	0.019	0.098	49.939	0.213	10,970
*611-18-S1-R4-1	0.433	0.025	0.093	50.024	0.219	8,740
*611-18-S1-R4-2	0.433	0.022	0.108	49.849	0.190	8,460
<b>Nitrate 750 mg/L</b>						
611-18-S3-R1-1	0.437	0.223	0.011	49.578	0.965	4,330
611-18-S3-R1-2	0.437	0.209	0.012	50.506	0.960	4,590
611-18-S3-R2-1	0.437	0.136	0.021	50.120	0.718	5,280
611-18-S3-R2-2	0.437	0.132	0.021	49.026	0.712	5,390
611-18-S3-R3-1	0.437	0.077	0.041	49.413	0.434	5,610
611-18-S3-R3-2	0.437	0.083	0.040	49.475	0.438	5,260
611-18-S3-R4-1	0.437	0.039	0.101	49.070	0.194	5,030
611-18-S3-R4-2	0.437	0.038	0.100	49.075	0.196	5,110

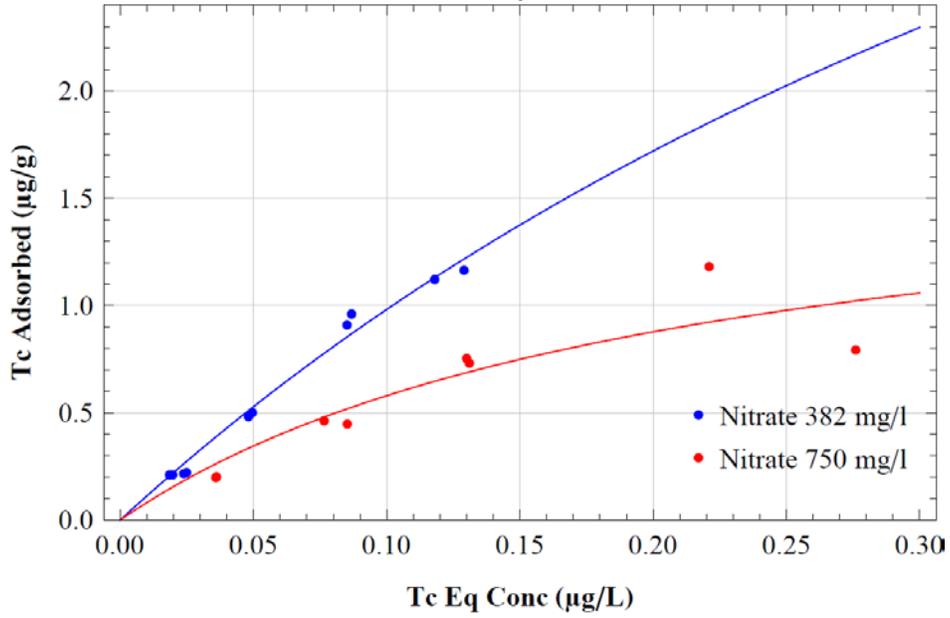
\* Data from Carbon Screening Tests

**Table 3.13.** Tc-99 Adsorption Isotherm Data for J177613 Norit GAC830

Sample Number	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
<b>Nitrate 382 mg/L</b>						
613-18-S2-R1-1	0.437	0.141	0.010	49.276	1.459	10,340
613-18-S2-R1-2	0.437	0.125	0.009	49.439	1.714	13,710
613-18-S2-R2-1	0.437	0.062	0.022	49.081	0.837	13,490
613-18-S2-R2-2	0.437	0.071	0.022	49.804	0.829	11,670
613-18-S2-R3-1	0.437	0.034	0.040	49.299	0.497	14,700
613-18-S2-R3-2	0.437	0.036	0.040	50.815	0.509	13,980
613-18-S2-R4-1	0.437	0.014	0.102	49.148	0.204	14,450
613-18-S2-R4-2	0.437	0.013	0.102	49.676	0.206	15,880
*613-18-S1-R4-1	0.433	0.018	0.096	49.924	0.216	12,060
*613-18-S1-R4-2	0.433	0.016	0.109	50.273	0.192	12,020
<b>Nitrate 750 mg/L</b>						
613-18-S3-R1-1	0.437	0.171	0.011	50.848	1.230	7,190
613-18-S3-R1-2	0.437	0.180	0.011	49.083	1.147	6,370
613-18-S3-R2-1	0.437	0.126	0.019	49.057	0.803	6,370
613-18-S3-R2-2	0.437	0.128	0.019	49.074	0.798	6,240
613-18-S3-R3-1	0.437	0.067	0.040	49.193	0.455	6,830
613-18-S3-R3-2	0.437	0.066	0.039	49.026	0.466	7,050
613-18-S3-R4-1	0.437	0.033	0.099	49.287	0.201	6,200
613-18-S3-R4-2	0.437	0.030	0.100	49.223	0.200	6,700

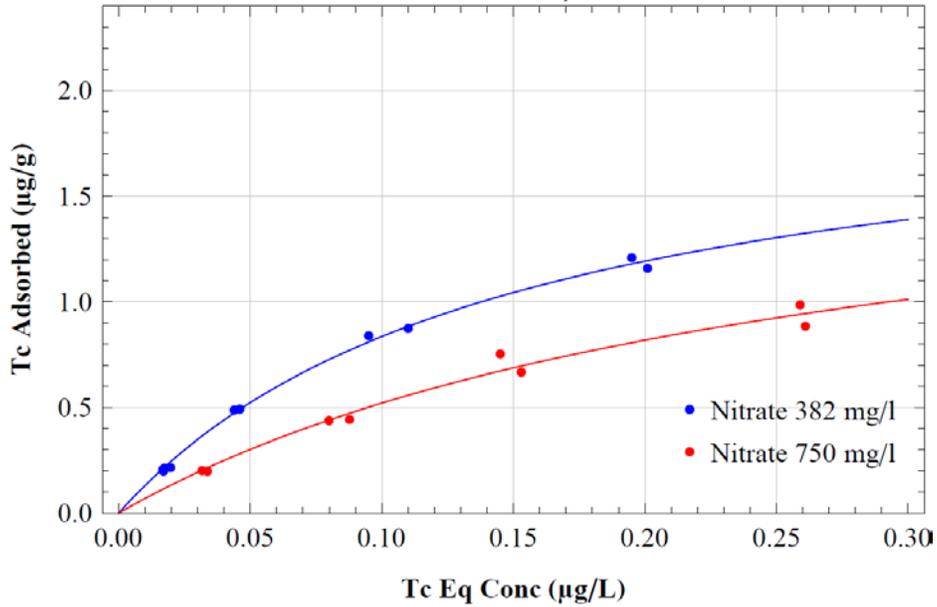
\* Data from Carbon Screening Tests

**J177606 Siemens AC1230AWC  
Coconut Shell/Acid Washed**



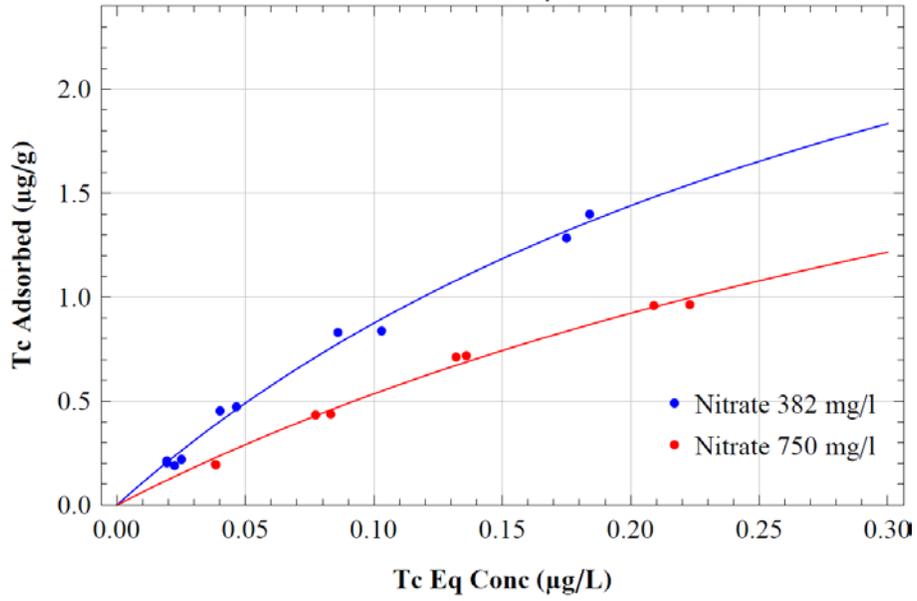
**Figure 3.2.** Langmuir Adsorption Isotherms for J177606 Siemens AC1230AWC

**J177609 Carbon Resources CR-1240A-AW  
Subbituminous Coal/Acid Washed**



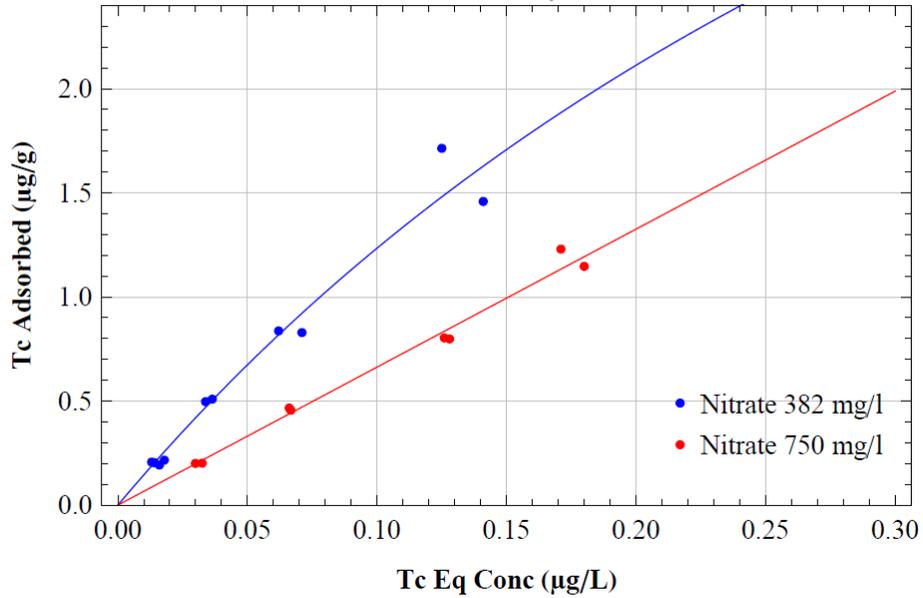
**Figure 3.3.** Langmuir Adsorption Isotherms for J177609 Carbon Resources CR 1240A AW

**J177611 General Carbon GC20X50  
Bituminous Coal/Unwashed**



**Figure 3.4.** Langmuir Adsorption Isotherms for J177611 General Carbon GC20X50

**J177613 Norit GAC830  
Bituminous Coal/Unwashed**



**Figure 3.5.** Langmuir Adsorption Isotherms for J177613 Norit GAC830

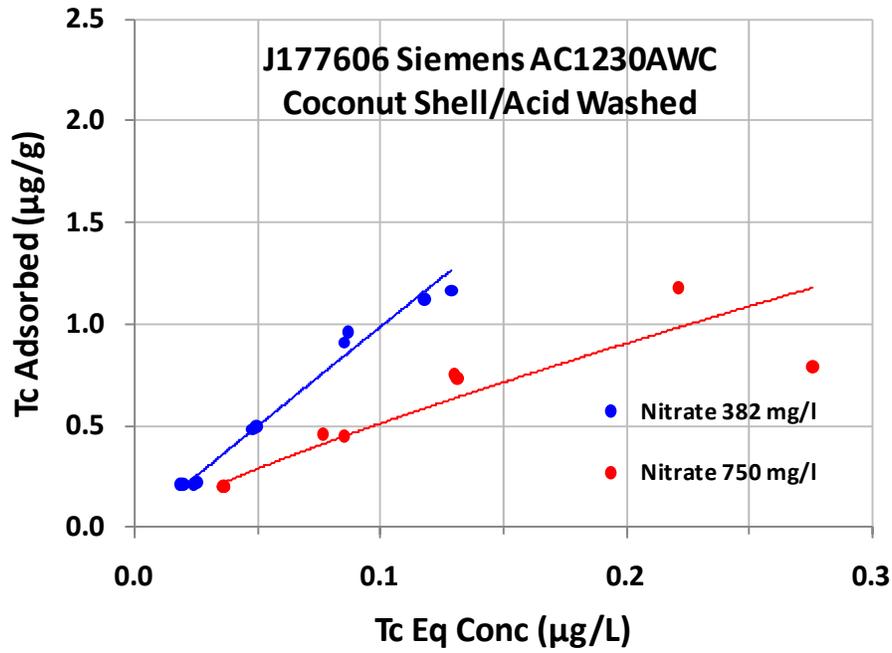


Figure 3.6. Freundlich Adsorption Isotherms for J177606 Siemens AC1230AWC

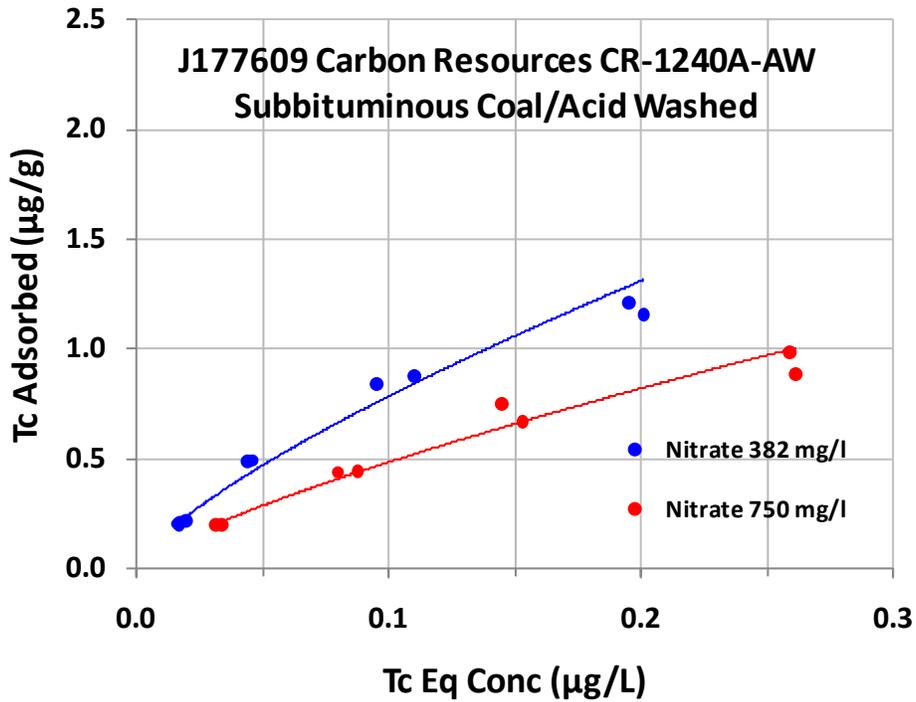


Figure 3.7. Freundlich Adsorption Isotherms for J177609 Carbon Resources CR 1240A AW

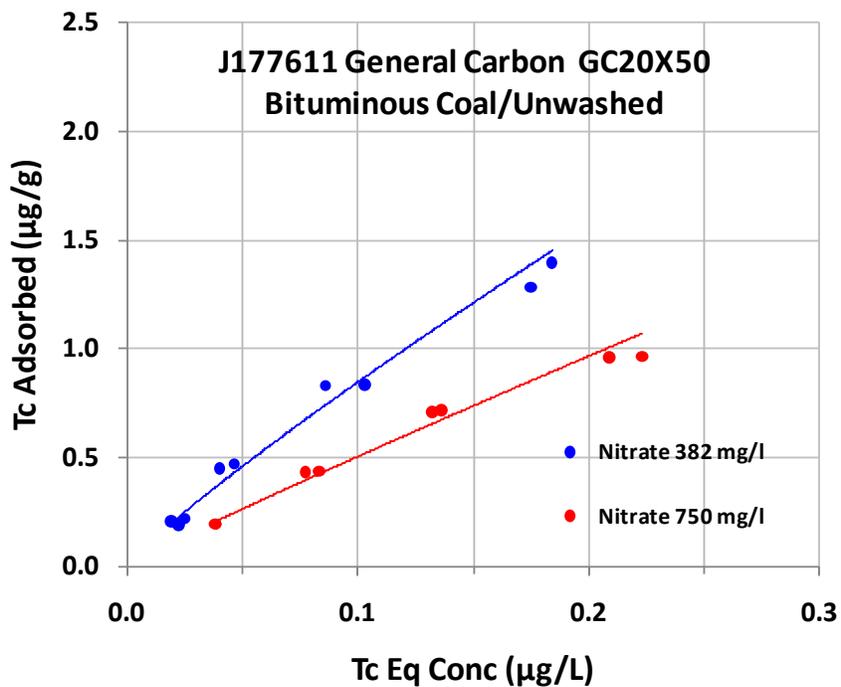


Figure 3.8. Freundlich Adsorption Isotherms for J177611 General Carbon GC20X50

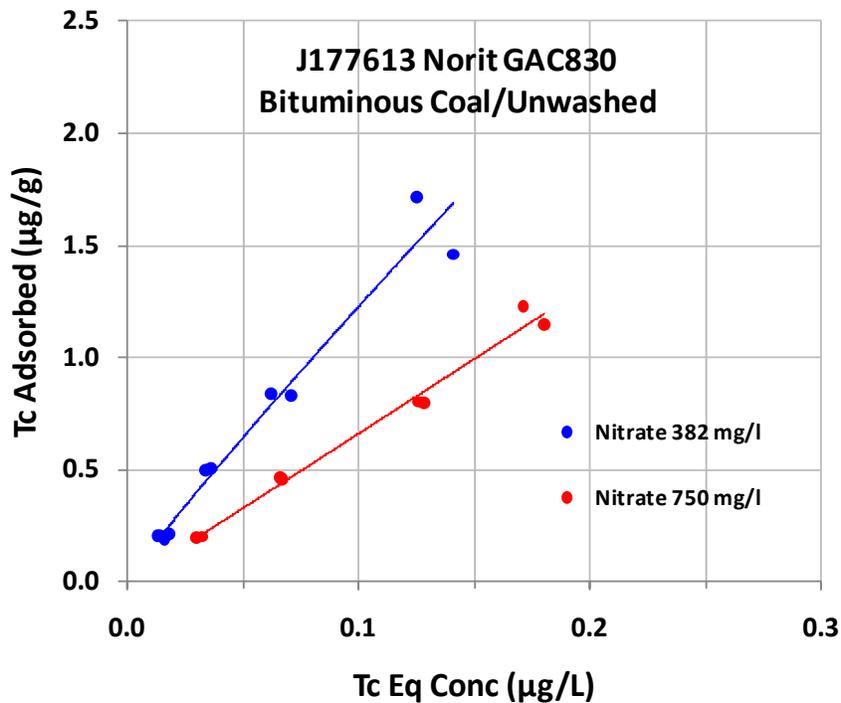


Figure 3.9. Freundlich Adsorption Isotherms for J177613 Norit GAC830

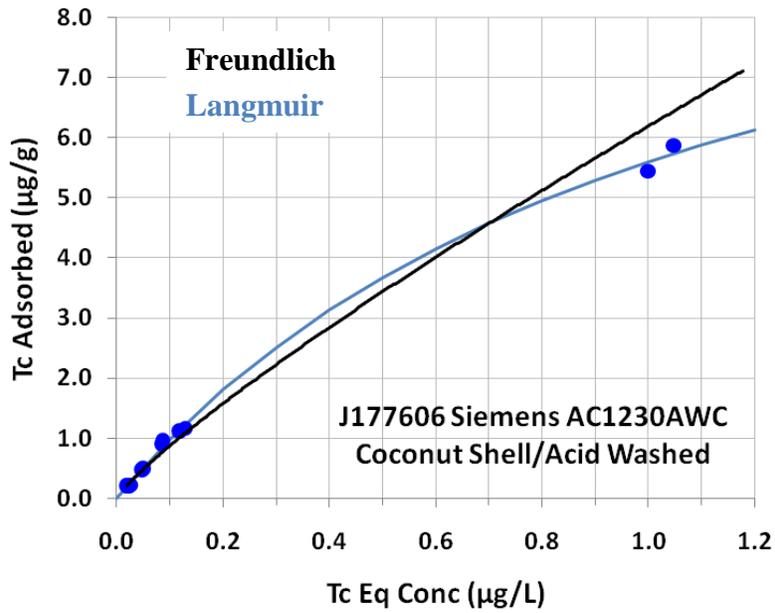
**Table 3.14.** Adsorption Constants and Predicted  $K_d$  for Tc-99 Adsorption on Four Selected Activated Carbon Samples.

Type of Granulated Activated Carbon	Langmuir Constants				Freundlich Constants			
	$K_L$ (L/ $\mu$ g)	M ( $\mu$ g/g)	$R^2$	Predicted* $K_d$ (mL/g)	$K_f$ (L/g)	1/n	$R^2$	Predicted* $K_d$ (mL/g)
J177606 Siemens AC1230AWC (Coconut Shell/Acid Washed) – low nitrate medium	1.646	6.945	0.9959	4,720	9.494	0.983	0.9856	9,510
J177606 Siemens AC1230AWC (Coconut Shell/Acid Washed) – High nitrate medium	4.735	1.803	0.9617	1,670	3.405	0.823	0.9078	3,500
J177609 Carbon Resources CR-1240-AW (Sub-Bituminous Coal/Acid Washed) – low nitrate medium	6.717	2.080	0.9991	2,050	4.308	0.740	0.9854	4,480
J177609 Carbon Resources CR-1240-AW (Sub-Bituminous Coal/Acid Washed) – High nitrate medium	3.735	1.914	0.9956	1,700	2.781	0.758	0.9814	2,880
J177611 General Carbon GC20X50 (Bituminous Coal/Unwashed) – low nitrate medium	2.745	4.062	0.9973	3,300	6.463	0.882	0.9794	6,570
J177611 General Carbon GC20X50 (Bituminous Coal/Unwashed) – high nitrate medium	1.904	3.345	0.9981	2,400	4.378	0.938	0.9865	4,420
J177613 Norit GAC830 (Bituminous Coal/Unwashed) – low nitrate medium	2.025	7.330	0.9865	5,400	10.476	0.9313	0.9817	10,590
J177613 Norit GAC830 (Bituminous Coal/Unwashed) – high nitrate medium	The best fit Constant partition isotherm: $Y = 6.628 x$				$R^2 = 0.9965$			

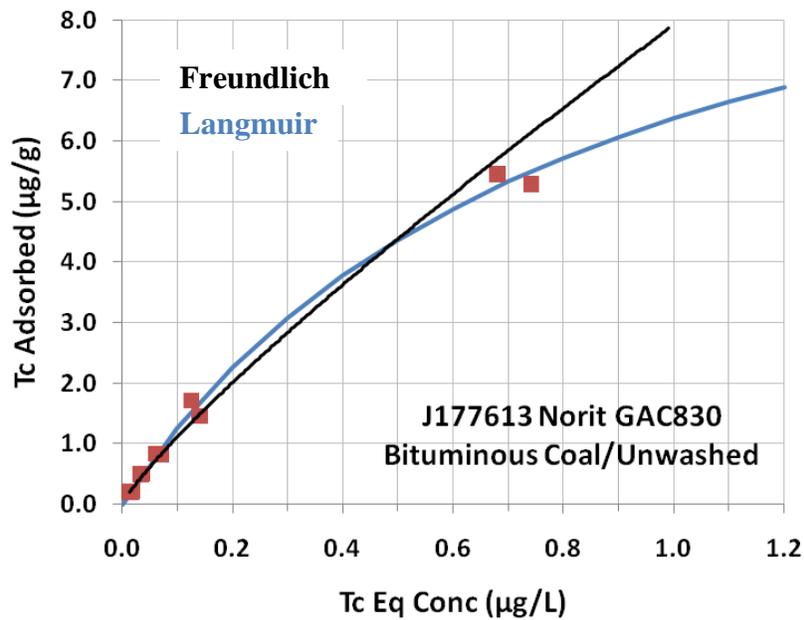
Tc Concentration in groundwater used for isotherm tests: 0.437  $\mu$ g/L  
 $Q = MK_L C / (1 + K_L C)$ , Q = Tc adsorbed/unit mass of carbon, C = Eq. Tc Concentration, M = Adsorption Maximum,  $K_L$  = Langmuir affinity parameter.  
 Freundlich Equation:  $Q = K_f C^{1/n}$ , Q: Tc-99 adsorbed/unit mass of carbon,  $K_f$ : Freundlich Constant, C = Equilibrium Tc-99 Conc  
 \*Predictions based on design Tc-99 concentration of 0.865  $\mu$ g/L (Activity: 14,700 pCi/L)

**Table 3.15.** Supplemental Tc-99 Adsorption Data

Sample Number	Initial Tc Conc ( $\mu\text{g/L}$ )	Final Tc Conc ( $\mu\text{g/L}$ )	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed ( $\mu\text{g/g}$ )	$K_d$ (mL/g)
<b>J177606 Siemens AC1230AWC Nitrate 412 mg/L</b>						
606-18-SX-RX-1*	2.205	1.047	0.010	50.648	5.865	5,600
606-18-SX-RX-2*	2.205	1.000	0.011	49.663	5.443	5,450
<b>J177613 Norit GAC830 Nitrate 412 mg/L</b>						
613-18-SX-RX-1*	2.205	0.741	0.014	50.592	5.289	7,130
613-18-SX-RX-2*	2.205	0.681	0.014	50.098	5.455	8,020



**Figure 3.10.** Langmuir and Freundlich Adsorption Isotherms for J177606 Siemens AC1230AWC with Supplemental Data



**Figure 3.11.** Langmuir and Freundlich Adsorption Isotherms for J177613 Norit GAC830 with Supplemental Data

The results with the supplemental data indicated that Langmuir isotherm best defined the adsorption characteristics of J177606 Siemens AC1230AWC and J177613 Norit GAC830 carbons (Table 3.15, Figure 3.10 and Figure 3.11). These data indicated the following:

- Langmuir isotherms provided the best fit for the Tc-99 adsorption data by the two carbons (606 and 613) under low nitrate concentration conditions.
- For the design concentration of Tc 0.865 µg/L (14,700 pCi/L), the  $K_d$  values predicted using Langmuir isotherm constants were ~5,980 mL/g for carbon 606 and ~6,870 mL/g for carbon 613 (Table 3.16).
- The Tc-99 adsorption performance of 606 and 613 carbons indicated that they do not meet the target  $K_d$  value of 9000 mL/g.

### 3.3.4 Effects of pH, Temperature, and VOCs on Tc-99 Adsorption

The effects of variable pH, temperature, and the presence of VOCs on Tc-99 adsorption on the selected carbons were evaluated. Table 3.17 through Table 3.20 present the results of those tests. Each of these effects is discussed in the following sections

**Table 3.16.** Adsorption Constants and Predicted  $K_d$  for Tc-99 Adsorption on Two Activated Carbons.

Type of Granulated Activated Carbon	Langmuir Constants				Freundlich Constants			
	$K_L$ (L/ $\mu$ g)	M ( $\mu$ g/g)	$R^2$	Predicted* $K_d$ (mL/g)	$K_f$ (L/g)	1/n	$R^2$	Predicted* $K_d$ (mL/g)
*J177606 Siemens AC1230AWC	0.91	11.74	0.9991	5,980	6.19	0.8496	0.9874	6,330
*J177613 Norit GAC830	1.19	11.72	0.9971	6,870	7.92	0.8521	0.9881	8,090

Langmuir Equation:  $Q = \frac{MK_L C}{1 + K_L C}$ , Q: Tc-99 adsorbed/unit mass of carbon, M: Predicted maximum Tc-99 adsorption,  $K_L$  :  
Langmuir constant,  
C = Equilibrium Tc-99 Concentration  
Freundlich Equation:  $Q = K_f C^{1/n}$ , Q: Tc-99 adsorbed/unit mass of carbon,  $K_f$  : Freundlich Constant, C = Equilibrium Tc-99 Conc  
\*Predictions based on design Tc-99 concentration of 0.865  $\mu$ g/L (Activity: 14,700 pCi/L)

**Table 3.17.** Effect of pH, Temperature and VOC on Tc Adsorption for J177606 Siemens AC1230AWC

Sample Number	pH (SU)	Initial Tc	Final Tc	Carbon Mass (g)	Soln Vol (mL)	Tc	K <sub>a</sub> (mL/g)
		Conc (µg/L)	Conc (µg/L)			Adsorbed (µg/g)	
606-18-S4-6.5-1	6.5	0.389	0.130	0.023	49.466	0.556	4,260
606-18-S4-6.5-2	6.5	0.389	0.125	0.023	49.048	0.563	4,520
606-18-S4-7.5-1	7.5	0.389	0.119	0.019	49.036	0.695	5,820
606-18-S4-7.5-2	7.5	0.389	0.123	0.022	49.129	0.593	4,800
*606-18-S2-R2-1	7.8	0.437	0.087	0.018	49.354	0.960	11,060
*606-18-S2-R2-2	7.8	0.437	0.085	0.019	49.075	0.909	10,680
606-18-S4-8.5-1	8.5	0.389	0.136	0.021	49.020	0.591	4,360
606-18-S4-8.5-2	8.5	0.389	0.157	0.019	49.031	0.599	3,820
Sample Number	Temp (°C)	Initial Tc	Final Tc	Carbon Mass (g)	Soln Vol (mL)	Tc	K <sub>a</sub> (mL/g)
		Conc (µg/L)	Conc (µg/L)			Adsorbed (µg/g)	
*606-18-S2-R2-1	18	0.437	0.087	0.018	49.354	0.960	11,060
*606-18-S2-R2-2	18	0.437	0.085	0.019	49.075	0.909	10,680
606-21-S5-1	21	0.444	0.142	0.020	49.023	0.741	5,230
606-21-S5-2	21	0.444	0.137	0.021	49.050	0.718	5,260
606-27-S5-1	27	0.444	0.153	0.022	49.068	0.648	4,230
606-27-S5-2	27	0.444	0.161	0.021	49.086	0.662	4,120
606-32-S5-1	32	0.444	0.166	0.021	49.056	0.650	3,920
606-32-S5-2	32	0.444	0.166	0.020	49.205	0.684	4,130
Sample Number	VOC	Initial Tc	Final Tc	Carbon Mass (g)	Soln Vol (mL)	Tc	K <sub>a</sub> (mL/g)
		Conc (µg/L)	Conc (µg/L)			Adsorbed (µg/g)	
606-18-S6-R1-1	Unsparged	0.385	0.069	0.023	49.400	0.678	9,820
606-18-S6-R1-2	Unsparged	0.385	0.079	0.021	49.173	0.715	9,010
606-18-S6-R2-1	Unsparged	0.385	0.018	0.100	49.009	0.180	10,220
606-18-S6-R2-2	Unsparged	0.385	0.017	0.097	49.401	0.187	10,740
*606-18-S2-R2-1	Sparged	0.437	0.087	0.018	49.354	0.960	11,060
*606-18-S2-R2-2	Sparged	0.437	0.085	0.019	49.075	0.909	10,680
*606-18-S2-R4-1	Sparged	0.437	0.019	0.099	49.828	0.211	11,390
*606-18-S2-R4-2	Sparged	0.437	0.020	0.100	50.611	0.211	10,720
*606-18-S1-R4-1	Sparged	0.433	0.024	0.094	49.570	0.216	9,070
*606-18-S1-R4-2	Sparged	0.433	0.025	0.092	49.766	0.221	8,870

\*Data from Adsorption Isotherm Experiments

**Table 3.18.** Effect of pH and Temperature on Tc Adsorption for J177609 Carbon Resources CR-1240-AW

Sample Number	pH (SU)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
609-18-S4-6.5-1	6.5	0.389	0.137	0.019	49.015	0.6486	4,720
609-18-S4-6.5-2	6.5	0.389	0.124	0.022	49.045	0.5896	4,740
609-18-S4-7.5-1	7.5	0.389	0.136	0.019	49.098	0.6540	4,820
609-18-S4-7.5-2	7.5	0.389	0.137	0.019	49.150	0.6504	4,730
*609-18-S2-R2-1	7.8	0.437	0.095	0.020	49.062	0.8390	8,830
*609-18-S2-R2-2	7.8	0.437	0.110	0.019	50.776	0.8740	7,940
609-18-S4-8.5-1	8.5	0.389	0.157	0.021	49.020	0.541	3,440
609-18-S4-8.5-2	8.5	0.389	0.145	0.019	49.031	0.629	4,340
Sample Number	Temp (°C)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
*609-18-S2-R2-1	18	0.437	0.095	0.020	49.062	0.839	8,830
*609-18-S2-R2-2	18	0.437	0.110	0.019	50.776	0.874	7,940
609-21-S5-1	21	0.444	0.153	0.021	49.201	0.680	4,430
609-21-S5-2	21	0.444	0.158	0.019	49.261	0.741	4,690
609-27-S5-1	27	0.444	0.176	0.020	49.182	0.658	3,730
609-27-S5-2	27	0.444	0.186	0.019	50.164	0.681	3,660
609-32-S5-1	32	0.444	0.186	0.021	49.021	0.602	3,240
609-32-S5-2	32	0.444	0.198	0.019	49.079	0.636	3,220

\*Data from Adsorption Isotherm Experiments

**Table 3.19.** Effect of pH and Temperature on Tc Adsorption for J177611 General Carbon GC20X50

Sample Number	pH (SU)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
611-18-S4-6.5-1	6.5	0.389	0.132	0.019	49.241	0.666	5,040
611-18-S4-6.5-2	6.5	0.389	0.118	0.021	49.221	0.635	5,380
611-18-S4-7.5-1	7.5	0.389	0.121	0.021	49.099	0.626	5,180
611-18-S4-7.5-2	7.5	0.389	0.113	0.022	49.081	0.616	5,470
*611-18-S2-R2-1	7.8	0.437	0.086	0.021	49.695	0.8310	9,660
*611-18-S2-R2-2	7.8	0.437	0.103	0.020	50.164	0.8380	8,130
611-18-S4-8.5-1	8.5	0.389	0.134	0.023	49.166	0.546	4,080
611-18-S4-8.5-2	8.5	0.389	0.138	0.022	49.099	0.560	4,060
Sample Number	Temp (°C)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>d</sub> (mL/g)
*611-18-S2-R2-1	18	0.437	0.086	0.021	49.695	0.831	9,660
*611-18-S2-R2-2	18	0.437	0.103	0.020	50.164	0.838	8,130
611-21-S5-1	21	0.444	0.143	0.020	49.161	0.739	5,150
611-21-S5-2	21	0.444	0.145	0.019	49.175	0.774	5,350
611-27-S5-1	27	0.444	0.158	0.019	49.091	0.738	4,670
611-27-S5-2	27	0.444	0.164	0.020	49.295	0.690	4,210
611-32-S5-1	32	0.444	0.171	0.021	49.084	0.639	3,740
611-32-S5-2	32	0.444	0.157	0.021	49.039	0.670	4,270

\*Data from Adsorption Isotherm Experiments

**Table 3.20.** Effect of pH, Temperature and VOC on Tc Adsorption for J177613 Norit GAC830

Sample Number	pH (SU)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>a</sub> (mL/g)
613-18-S4-6.5-1	6.5	0.389	0.111	0.022	49.071	0.620	5,600
613-18-S4-6.5-2	6.5	0.389	0.108	0.021	49.031	0.656	6,090
613-18-S4-7.5-1	7.5	0.389	0.102	0.020	49.048	0.703	6,870
613-18-S4-7.5-2	7.5	0.389	0.079	0.023	49.096	0.662	8,440
*613-18-S2-R2-1	7.8	0.437	0.0620	0.022	49.081	0.837	13,490
*613-18-S2-R2-2	7.8	0.437	0.0710	0.022	49.804	0.829	11,670
613-18-S4-8.5-1	8.5	0.389	0.107	0.023	49.103	0.601	5,600
613-18-S4-8.5-2	8.5	0.389	0.105	0.022	49.221	0.635	6,060

Sample Number	Temp (°C)	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>a</sub> (mL/g)
*613-18-S2-R2-1	18	0.437	0.062	0.022	49.081	0.837	13,490
*613-18-S2-R2-2	18	0.437	0.071	0.022	49.804	0.829	11,670
613-21-S5-1	21	0.444	0.111	0.021	49.248	0.782	7,070
613-21-S5-2	21	0.444	0.105	0.019	49.214	0.877	8,320
613-27-S5-1	27	0.444	0.122	0.019	49.078	0.832	6,830
613-27-S5-2	27	0.444	0.122	0.021	49.066	0.753	6,190
613-32-S5-1	32	0.444	0.155	0.019	49.071	0.747	4,840
613-32-S5-2	32	0.444	0.146	0.021	49.479	0.702	4,800

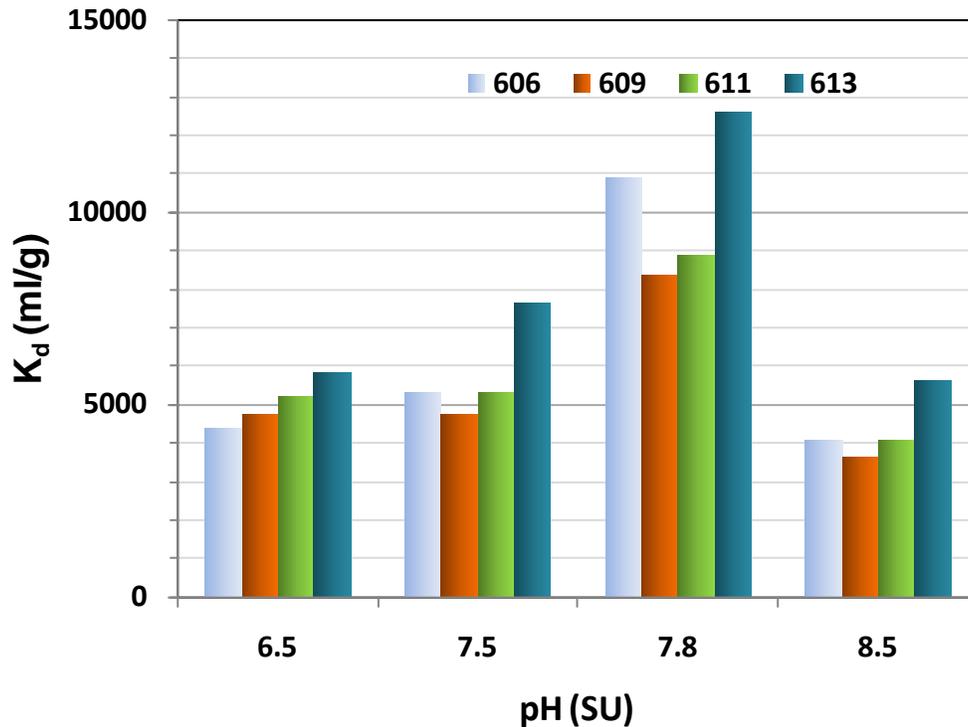
Sample Number	VOC	Initial Tc Conc (µg/L)	Final Tc Conc (µg/L)	Carbon Mass (g)	Soln Vol (mL)	Tc Adsorbed (µg/g)	K <sub>a</sub> (mL/g)
613-18-S6-R1-1	Unsparged	0.385	0.061	0.021	49.239	0.760	12,500
613-18-S6-R1-2	Unsparged	0.385	0.069	0.021	49.209	0.740	10,750
613-18-S6-R2-1	Unsparged	0.385	0.014	0.097	49.007	0.187	13,150
613-18-S6-R2-2	Unsparged	0.385	0.017	0.098	49.020	0.184	10,910
*613-18-S2-R2-1	Sparged	0.437	0.062	0.022	49.081	0.837	13,490
*613-18-S2-R2-2	Sparged	0.437	0.071	0.022	49.804	0.829	11,670
*613-18-S2-R4-1	Sparged	0.437	0.014	0.102	49.148	0.204	14,450
*613-18-S2-R4-2	Sparged	0.437	0.013	0.102	49.676	0.206	15,880
*613-18-S1-R4-1	Sparged	0.433	0.018	0.096	49.924	0.216	12,060
*613-18-S1-R4-2	Sparged	0.433	0.016	0.109	50.273	0.192	12,020

\*Data from Adsorption Isotherm Experiments

### 3.3.5 Variable pH Batch Tests

The results of the pH effects on Tc-99 adsorption is listed in Table 3.17 through Table 3.18 and displayed in Figure 3.12. At pH values of 6.5 and 7.5, no significant differences in Tc-adsorption performance was observed for the three of the carbons (606, 609, and 611), whereas carbon 613 performed better at pH 7.5. When the pH was increased to 8.5, a slight decline in performance was observed for all carbons. At pH 7.8, the adsorption data for all carbons were anomalously high due to the higher initial Tc-99 concentrations measured in the source water (Table 3.8, Table 3.17, and Table 3.18). As discussed earlier, these batch experiments were conducted over a period of approximately 3 months and aliquots of source water were drawn and analyzed before each set of batch experiment to account for

any changes in the Tc-99 concentration in the source water due to adsorption and desorption on suspended solids, on growing algae, and on the container wall.



**Figure 3.12.** The Effect of pH on the Tc-99 Adsorption on Selected Activated Carbons

### 3.3.6 Variable Temperature Batch Tests

The results Tc-99 adsorption tests as a function of temperature are listed in Table 3.17 and Table 3.18 and are shown graphically in Figure 3.13. At 18°C, the  $K_d$  values for all carbons were anomalously high due to the higher initial Tc-99 concentration measured in the source water. These sets of data were generated at an earlier time with a batch of source water that had a higher measured Tc-99 concentration. At 21°C, 27°C, and 32°C, there were no significant differences in Tc-99 adsorption performance between the three other carbons namely, J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, and J177611 General Carbon GC20X50. Comparatively, J177613 Norit GAC830 carbon showed a noticeable decline in Tc-99 adsorption performance with increasing temperature. Among the four carbons tested, the J177613 Norit GAC830 carbon exhibited the best Tc-99 adsorption performance at all temperatures.

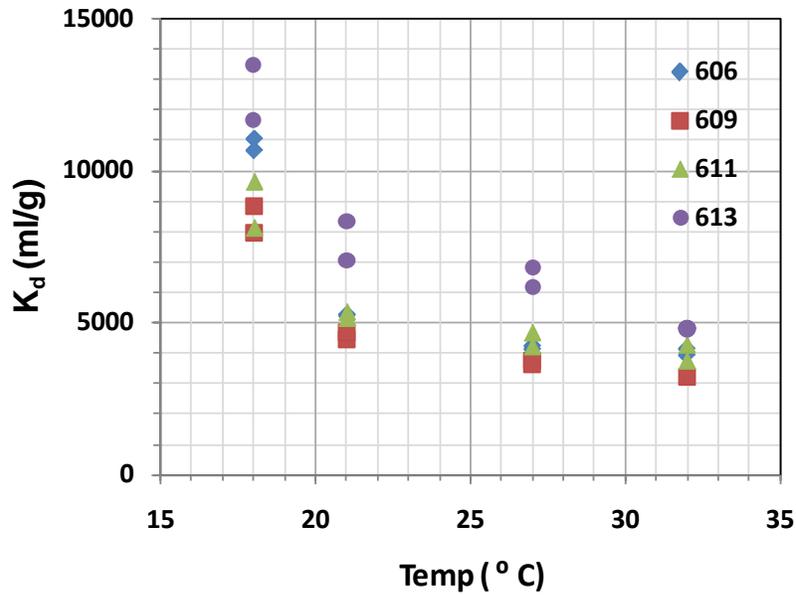


Figure 3.13. The Effect of Temperature on the Tc-99 Adsorption on Selected Activated Carbons

### 3.3.7 Volatile Organic Carbon Effects

The results of VOC effect testing on Tc-99 adsorption on carbons are tabulated in Table 3.17 and Table 3.20 and graphically displayed in Figure 3.14. The data indicated that the presence of VOCs in the source water did not significantly affect Tc-99 adsorption on both of the tested carbons (J177606 Siemens AC1230AWC and J177613 Norit GAC830). The distribution coefficients ( $K_d$ ) with and without VOCs differed by less than 15%, indicating that at the concentrations of VOCs present in the source water would not significantly affect Tc-99 adsorption by these two activated carbons.

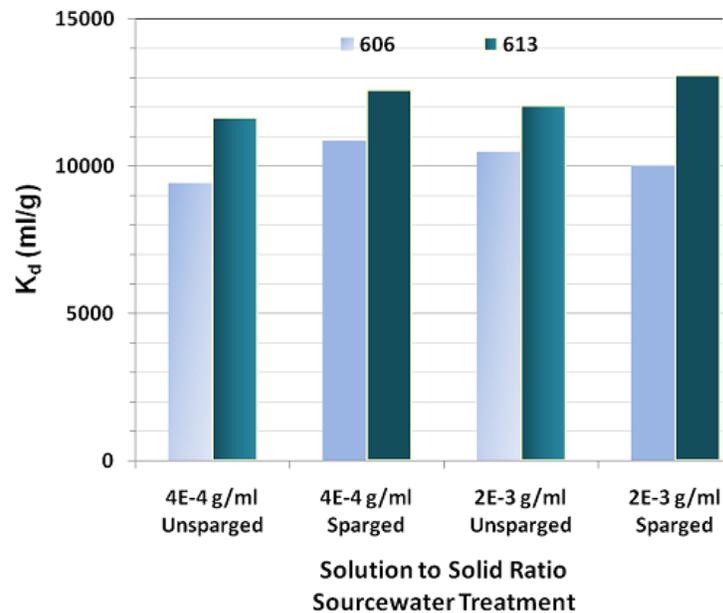


Figure 3.14. The Effect of VOCs on the Tc-99 Adsorption on Selected Activated Carbons

## 4.0 Conclusions

The Tc-99 adsorption performance of seven activated carbons (J177601 Calgon Fitratorb 400, J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, J177612 Norit GAC830, J177613 Norit GAC830, and J177617 Nucon LW1230) were evaluated using water from well 299-W19-36. Based on the results of screening tests, four of the carbons (J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830) were selected for batch isotherm testing, and for determination of their adsorption characteristics under variable pH and temperature conditions. The effect of VOCs in source water on Tc-99 adsorption characteristics of two of the carbon samples (J177606 Siemens AC1230AWC and J177613 Norit GAC830) was also tested. The results of these tests are as follows:

1. Screening tests of the seven activated carbons indicated that the distribution coefficients ( $K_d$ ) for Tc-99 adsorption ranged from 5,000 mL/g to 12,000 mL/g. The four of the activated carbon samples—J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, J177611 General Carbon GC20X50, and J177613 Norit GAC830—that exhibited highest  $K_d$  values were derived from acid-washed coconut shell, acid-washed sub-bituminous coal, and the last two, unwashed bituminous coal, respectively.
2. The results of the batch isotherm tests on four of the selected carbons indicated that under lower nitrate concentration conditions (382 mg/L), Tc-99 adsorption for these carbon samples typically ranged from 0.190 to 1.459  $\mu\text{g/g}$  with corresponding  $K_d$  values ranging from 6,000 to 20,000 mL/g. In comparison, under higher nitrate (750 mg/L) conditions, there was a measureable decrease in Tc-99 adsorption. The Tc-99 adsorption under these conditions ranged from 0.196 to 1.230  $\mu\text{g/g}$  and the corresponding  $K_d$  values ranging from 3,000 to 7,000 mL/g. The adsorption data were fit to the Langmuir and the Freundlich equations, and the adsorption affinity parameters and the predicted  $K_d$  values for the design Tc-99 concentration (0.865  $\mu\text{g/g}$ , 14,700 pCi/L) were tabulated.
3. Supplemental data were collected for 606 and 613 carbons with source water spiked at 2.205  $\mu\text{g/L}$  of Tc to resolve the issue of best fit isotherm. Langmuir isotherms provided the best fit for the composited Tc-99 adsorption data by the two carbons (606 and 613) under low nitrate concentration conditions. For the design concentration of Tc 0.865  $\mu\text{g/L}$  (14,700 pCi/L), the  $K_d$  values predicted from using Langmuir isotherm constants were ~5,980 mL/g for carbon 606 and ~6,870 mL/g for carbon 613. These  $K_d$  values indicated that they do not meet the target  $K_d$  value of 9,000 mL/g.
4. At pH values of 6.5 and 7.5, no significant differences in Tc-adsorption performance was observed for three of the carbons (606, 609, and 611), whereas carbon 613 performed better at pH 7.5. When the pH was increased to 8.5, slight decline in performance was observed for all carbons. At pH 7.8, the  $K_d$  data for all carbons were anomalously high due to the higher initial Tc-99 concentrations measured in the source water. As discussed earlier, these batch experiments were conducted over a period of approximately 3 months and aliquots of source water were drawn and analyzed before each set of batch experiments to account for any changes in the Tc-99 concentration in the source water due to adsorption and desorption on suspended solids, on growing algae, and on the container wall.
5. At 18°C, the  $K_d$  values for all four (606, 609, 611, and 613) carbons were anomalously high due to the higher initial Tc-99 concentration measured in the source water. These f data were generated at an earlier time with a batch of source water with higher measured Tc-99 concentration. At 21°C, 27°C, and 32°C, there were no significant differences in Tc-99 adsorption performance between three other

carbons namely—J177606 Siemens AC1230AWC, J177609 Carbon Resources CR-1240-AW, and J177611 General Carbon GC20X50. Comparatively, J177613 Norit GAC830 carbon showed a noticeable decline in Tc-99 adsorption performance with increasing temperature. Among the four carbons tested, the J177613 Norit GAC830 carbon exhibited the best Tc-99 adsorption performance at all temperatures.

6. The presence of VOCs in the source water did not significantly affect Tc-99 adsorption on both of the tested carbons (J177606 Siemens AC1230AWC and J177613 Norit GAC830). The distribution coefficients ( $K_d$ ) with and without VOCs differed by less than 15%, indicating that at the concentrations of VOCs present in the source water Tc-99 adsorption by these two activated carbons would not be significantly affected.

## 5.0 References

Clesceri LS, AE Greenberg, and AD Eaton. 1998. *Standard Methods for the Examination of Water and Wastewater*, 20th Edition. American Public Health Association, American Water Works Association, and Water Environment Federation, Washington, D.C.

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**Appendix**  
**Chemical Analyses**

# Appendix

## Chemical Analyses

### A.1 Analytes and Analytical Methods

The analytes and analytical methods for the reported testing are described in this section.

#### A.1.1 Analyte List

Table A.1 provides the list of analytes for the batch testing.

**Table A.1.** Technetium-99 (Tc-99) Adsorption on Activated Carbons – Well 299-W19-36

Test Phase	Media	Analyses
Batch	Influent Water	Tc-99, uranium, Cr (VI), VOC, total Cr, nitrate, TOC, TSS, pH and temperature
	Batch contact solution	Tc-99

#### A.1.2 pH Analysis

Approximately 3-mL aliquots of the unfiltered groundwater/test solution will be used for pH measurement following the Pacific Northwest National Laboratory (PNNL) procedure AGG-pH-001, “pH Measurements,”<sup>1</sup> which is similar to EPA’s SW-846, *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Third Edition; Final Update IV-B*, Method 9040C (EPA 2004a). Solution pH values will be measured with a glass calomel pH electrode and a pH meter calibrated with appropriate buffers at 4, 7, and 10.

#### A.1.3 Trace Metals Analysis

Uranium and Tc-99 analyses of the groundwater/test solution will be performed by inductively coupled plasma-mass spectrometer (ICP-MS) following procedure PNNL-AGG-415, “Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Analysis,”<sup>2</sup> which is similar to EPA SW-846, Method 6020A (EPA 1996). High-purity single element standards traceable to the National Institute of Standards and Technology (Ultra Scientific, Kingston, RI and Inorganic Ventures, Lakewood, NJ) were used to generate calibration curves and to verify continuing calibration during the analytical run. A serial dilution will be made of select samples to investigate and correct for matrix interferences. Typical instrument detection limits for the ICP-MS are in the range of parts per trillion.

<sup>1</sup> PNNL-AGG-pH-001. 2009. “pH Measurements,” Pacific Northwest National Laboratory, Richland, Washington (unpublished technical procedure).

<sup>2</sup> PNNL-AGG-415. 2008. “Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Analysis,” Pacific Northwest National Laboratory, Richland, Washington (unpublished technical procedure).

#### **A.1.4 Alkalinity**

The alkalinity of the groundwater will be measured by titrimetry in accordance with *Standard Methods for the Examination of Water and Wastewater*, Method 2320B (AWWA et al. 1998).

#### **A.1.5 Nitrate and Sulfate Analysis**

Nitrate and sulfate analyses of the groundwater/test solution will be performed using PNNL procedure AGG-IC-001, “Determinations by Ion Chromatography (IC),”<sup>1</sup> which is similar to EPA SW-846, Method 9056A (EPA 2007). Nitrate will be separated on a Dionex AS18 column with a sodium hydroxide gradient elution and measured using a conductivity detector. The only modification to the EPA ion chromatography method is the use of sodium hydroxide for gradient elution. High-purity calibration standards will be used to generate calibration curves and to verify continuing calibration during the analytical run.

#### **A.1.6 Total Organic Carbon Analysis**

Total organic carbon (TOC) analyses of the groundwater/test solution will be performed using AGG-TOC-001, “Operating of Carbon Analyzer (TOC-V + SSM-5000A + ASI)” (Shimadzu) (unpublished PNNL technical procedure), which is similar to “Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods SW-846 9060A” (EPA 2004b). Sample aliquots are analyzed for TOC by first acidifying a sample aliquot with 3 M HCL to a pH less than 3. The acidified sample is introduced into a combustion chamber with an oxidation catalyst and heated to 680°C. The released carbon from the combustion is converted to CO<sub>2</sub>, swept from the combustion chamber by ultra pure oxygen, dehumidified, and scrubbed to remove halogens. The carrier gas then delivers the sample combustion products to the cell of a non-dispersive infrared gas analyzer where the carbon dioxide is detected and measured. The amount of CO<sub>2</sub> measured is proportional the TOC content of the sample. High-purity calibration standards will be used to generate calibration curves and verify continuing calibration during the analytical run.

#### **A.1.7 Total Suspended Solids**

The total suspended solids (TSS) analyses of the groundwater/test solution will be performed using *Standard Methods for the Examination of Water and Wastewater Method 2540D* (Clesceri et al. 1998). A well-mixed solution is filtered through a weighed glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105°C. The increase in weight of the filter represents the TSS.

#### **A.1.8 Hexavalent Chromium Analysis**

Hexavalent chromium analyses of the groundwater/test solution will be performed using “Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods SW-846 7196A” (EPA 1992). Dissolved hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color is produced and its absorbance is measured photometrically at 540 nm.

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<sup>1</sup> Lindberg, MJ. 2004. “Determinations by Ion chromatography (IC).” AGG-IC-001, unpublished PNNL Technical Procedure, Pacific Northwest National Laboratory, Richland, Washington.

High-purity NIST-traceable standards (NIST SRM 136f, and Alfa Aesar) calibration standards will be used to generate calibration curves and verify continuing calibration during the analytical run.

### **A.1.9 Carbon Tetrachloride, Trichloroethene, and VOC Analysis**

Carbon tetrachloride, trichloroethene, and VOC analysis will be performed via gas chromatography-mass spectrometry (GC-MS). The GC-MS analyses will be conducted according to standard technical procedures developed by PNNL. The water samples will be diluted 4 to 500 times in boiled Milli-Q water and analyzed with a Hewlett Packard 5890 gas chromatograph fitted with a purge and trap system (P&T, 0.1. Analytical, Model 4660) with photoionization (PID, Model 4430) and electrolytic conductivity (ELCD, Model 5320) detectors. Solute compounds will be separated on a 105-m by 0.53-mm megabore capillary column (Restek Corporation) and quantified using a four-point calibration. Calibration standards will be prepared from a commercial standard in Restek 502.2 Calibration Mix #2.

## **A.2 References**

EPA 1996. "Method 6020A, Inductively Coupled Plasma - Mass Spectrometry," Rev. 1. In: *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. EPA SW 846, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. Accessed June 1, 2010. Available at: <http://www.ppa.izov.epawaste/hazard/testrmethods/sw846/pdfs/6020a.pdf>.

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