

Environmental Technology Verification Report

Removal of Uranium in Drinking Water

Brimac Environmental Services, Inc.
Brimac HA 216 Adsorptive Media

Prepared by



NSF International



Under a Cooperative Agreement with
U.S. Environmental Protection Agency

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THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM



U.S. Environmental Protection Agency



NSF International

ETV Joint Verification Statement

TECHNOLOGY TYPE:	ADSORPTIVE MEDIA
APPLICATION:	REMOVAL OF URANIUM IN DRINKING WATER
PRODUCT NAME:	BRIMAC HA 216 ADSORPTIVE MEDIA
VENDOR:	BRIMAC ENVIRONMENTAL SERVICES, INC.
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NSF International (NSF) manages the Drinking Water systems (DWS) Center under the U.S. Environmental Protection Agency's (EPA) Environmental Technology Verification (ETV) Program. The DWS Center recently evaluated the performance of the Brimac Environmental Services, Inc. (Brimac) HA 216 Adsorptive Media. The New Hampshire Department of Environmental Services (NHDES) monitored the operation of the pilot unit containing the media, collected water samples, and provided some laboratory services. NSF also analyzed samples and authored the verification report and this verification statement. The verification report contains a comprehensive description of the test.

EPA created the ETV Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The ETV Program's goal is to further environmental protection by accelerating the acceptance and use of improved and more cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer-reviewed data on technology performance to those involved in the design, distribution, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations, stakeholder groups (consisting of buyers, vendor organizations, and permittees), and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

ABSTRACT

The Brimac HA 216 Adsorptive Media was tested for uranium (U) removal from a drinking water source (well water) at Grappone Toyota located in Bow, New Hampshire. The HA 216 media is a hydroxyapatite-based material. A pilot unit, consisting of a TIGG Corporation Cansorb® C-5 steel drum with 50 pounds (lb) (23 kilograms, 1.3 ft³) of media, was used for this verification test. The pilot unit was operated at a flow rate of approximately two gallons per minute (gpm), resulting in a hydraulic loading rate of 1.04 gpm/ft², and an empty bed contact time (EBCT) of 4 minutes and 54 seconds. The integrity test phase included observation of the operation of the pilot unit. The pilot test unit was simple and easy to operate, particularly since there were no pumps required for this installation and no need for automated controls or backwash systems.

The source water contained a mean uranium concentration of 190 µg/L. The pilot unit produced treated water with uranium concentrations of <1 µg/L at the start of the test. The uranium concentration in the treated water began to increase after two days of operation and exceeded the EPA National Primary Drinking Water Regulation (NPDWR) maximum contaminant level (MCL) of 30 µg/L after approximately 21,400 gallons (gal) of water had been treated, representing 2,200 bed volumes (BV). The uranium concentration in the treated water exceeded the stop-test concentration of 60 µg/L at 33,700 gal (3,500 BV). The test was stopped two days later at 40,500 gal after the uranium results had been received showing that 60 µg/L had been passed. While the treated water uranium concentration increased more quickly than anticipated, the mean concentration for the 15-day monitoring period was 29.7 µg/L, which is below the MCL. Based on the mean source and treated water uranium concentrations (171 µg/L and 12.6 µg/L respectively) for the first ten days of operation before the treated water exceeded 30 µg/L of uranium, the 23 kilograms (kg) of media absorbed 13.1 g of uranium (5.7×10^{-4} g U/g media). For the entire test period, the media adsorbed approximately 24.8 g of uranium (0.001 g U/g media).

TECHNOLOGY DESCRIPTION

The following technology description was provided by the manufacturer and has not been verified.

Brimac HA 216 Adsorptive Media is a hydroxyapatite-based media. The molecular formula for hydroxyapatite is $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$. Hydroxyapatite sequesters uranium by three processes: 1) incorporation within the hydroxyapatite lattice through ion-exchange with calcium, 2) physisorption and chemisorption with reactive phosphate and calcium oxide groups at the mineral surface, and 3) reaction with free phosphate to form solids that precipitate out of solution. The particles are highly porous and capable of adsorbing heavy metals, color forming compounds, trihalomethane (THM) precursor compounds, taste and odor producing compounds as well as other organic and inorganic compounds. The media performs over a wide range of pH and temperature. HA 216 has a Langmuir isotherm capacity of just over 1 g of uranium per g of media.

Uranium adsorption by hydroxyapatite occurs more slowly than contaminant adsorption by activated carbon. The rate-determining step is adsorption, not the rate of diffusion, as with activated carbon. For this reason, Brimac considers uranium adsorption by hydroxyapatite to be more like an ion exchange process. The bed of hydroxyapatite media has a mass transfer zone that moves through the bed in a plug flow manner until the media is exhausted.

HA 216 is certified by NSF to NSF/ANSI Standard 61 for water treatment plant applications and received European Pharmacopeia and UK Drinking Water Inspectorate approvals. Hydroxyapatite is also listed 'Generally Recognized as Safe' by the U.S. Food and Drug Administration.

VERIFICATION TESTING DESCRIPTION

Test Site and Equipment

The verification test was conducted using a pilot unit installed at Grappone Toyota at 514 Route 3A in Bow, New Hampshire. Groundwater was drawn from an 11 gpm capacity well, serving 82 employees. Brimac provided a pilot unit containing HA 216 media installed in a TIGG Corporation Cansorb[®] C-5 steel drum. The drum contains internal schedule 40 PVC plumbing to ensure proper distribution of the feed water onto the media bed. The C-5 is 30 inches (in) high, with a diameter of 19 in. For the verification test, the pilot unit contained 50 lb (23 kg) of media, which equals approximately 1.3 ft³ of media at a depth of 8.2 in. in the C-5 drum. The unit was set up to operate at approximately 2.0 gpm.

The inlet water line was connected to the pressure (bladder) tank that was used to maintain water pressure in the building water supply system. This provided sufficient water pressure to operate the pilot unit, and no additional pumping was required to maintain flow to the test system. Treated water was discharged to the sanitary sewer.

The verification test included two main tasks: system integrity verification and adsorptive capacity verification. System integrity verification was a two-week test of the pilot unit with daily monitoring to ensure the media and pilot unit were functioning properly and to identify any major systemic problems such as channeling, insufficient media, excessive headloss buildup, etc. Adsorption capacity verification evaluated the capability of the media at a set contact time to remove uranium to below the EPA NPDWR MCL of 30 µg/L. As requested by Brimac, the test was continued until at least 60 µg/L of uranium was detected in the treated water.

Methods and Procedures

The testing methods and procedures are detailed in the *Product-Specific Test Plan Removal of Uranium in Drinking Water Brimac HA 216 Adsorptive Media*. The EPA/NSF *ETV Protocol for Equipment Verification Testing for Removal of Radioactive Chemical Contaminants* (April 2002, Chapter 1) and the EPA/NSF *ETV Equipment Verification Testing Plan for Adsorptive Media Processes for the Removal of Arsenic* (September 2003, Chapter 6) provided the basis for the procedures used to develop the test plan and to ensure the accurate documentation of pilot unit performance and treated water quality. NSF and NHDES co-managed verification responsibilities and analytical laboratory efforts. The pilot unit was operated 24 hours a day, seven days a week during the testing period.

For the first 14 days of the integrity test, operational data were collected once per day, Monday through Saturday. These data included cumulative feed water volume, feed water flow rate, treated water pressure, and time on site. Grab samples for on-site and laboratory water quality analyses were collected daily for temperature, pH, turbidity, and uranium. Grab samples were collected weekly for TSS, TOC, TDS, calcium magnesium, sodium, iron, hardness, chloride, sulfate, fluoride alkalinity, phosphorus, nitrate, arsenic aluminum silica, radon 222, alpha radioactivity, and UV₂₅₄. Prior to collecting samples, the sample tap was flushed for at least five seconds. All samples were collected into clean containers.

The analytical laboratories performed the water quality analyses using EPA or Standard Methods procedures. Samples for off-site laboratory analysis were collected and preserved according to *Standard Methods* 3010 B.

VERIFICATION OF PERFORMANCE

System Operation

Brimac coordinated with NHDES and NSF to install the equipment and ready the system for operation. Once ready for operation, Brimac ran initial startup and shakedown tests to determine operating conditions for water treatment. The system started up quickly and without any difficulties. Verification

testing was started on July 10. The two-week integrity test was completed on July 24 and the capacity test phase ended on July 25 after 15 days of operation. The capacity test was stopped because the uranium data showed that the concentration in the treated water had exceeded the stop-test level of 60 µg/L on the 13th day. The pilot unit continued in operation until July 30, while the analyses were being completed.

The average daily flow rate reported for the 19 total days of operation (Days 0-20) was 1.97 gpm and the average flow rate calculated using the total volume treated was 2.03 gpm (54,728 gal over 19 days, as recorded from the flow meter totalizer). The flow rate to the unit cycled between a high to low flow rate, as the pressure in the well system cycled from high to low. The field technician observed several flow rates over several minutes and recorded a range of flow rates on the bench sheet. These flow rate ranges were then used to report an average flow rate for the unit. While the flow rate did change over a range of readings, the average flow rate was close to the target of 2.0 gpm and was consistent during the test. Overall, the frequent change in flow rate did not impact the volume of water treated each day, as shown by comparing the data for the average flow rate and daily volume treated.

The hydraulic loading rate during the test, based on a mean flow rate of 1.97 gpm and a pilot unit surface area of 1.90 ft², averaged 1.04 gpm/ft². The EBCT during the verification test was approximately 4.9 minutes (4 minutes, 54 seconds).

Test Results

The source water had a mean uranium concentration of 190 µg/L. All turbidity measurements were <1 NTU and all TSS concentrations were <2 mg/L. A sediment/particulate pre-filter was not used ahead of the test unit. There was no indication during the test of any problems with particulate accumulation in the media bed. The pH of the source water and treated water was steady throughout the test, with a range of 6.52-6.93 SU and 6.63-7.29 SU, respectively.

Figure VS-1 presents the uranium removal results plotted as a function of the bed volumes treated during the integrity and capacity tests. At the beginning of the verification, the uranium concentration observed in the treated water was near or below 1 µg/L. The uranium concentration observed in the treated water began to increase as the cumulative bed volumes of treated water increased. The concentration exceeded the water quality standard of 30 µg/L after approximately 21,400 gal of water were treated, or 2,200 BV. The capacity test was stopped two days later at 40,500 gal after the uranium results had been received showing that the treated water concentration had exceeded 60 µg/L. While the treated water uranium concentration increased more quickly than anticipated, the mean concentration for the 15-day monitoring period was 29.7 µg/L, which is below the MCL. However, the treated water was below the water quality standard for only the first 10 days of the test.

Considering the mean source and treated water uranium concentrations (171 µg/L and 12.6 µg/L) for the first ten days of data (until breakthrough had occurred at 30 µg/L), the 50 lbs (23 kg) of media adsorbed 13.1 g of uranium (5.7×10^{-4} g U/g media). Over the entire test period, the 23 kg of media adsorbed approximately 24.8 g of uranium (0.001 g U/g media). These data indicate that while the HA 216 media had capacity to adsorb uranium beyond the first 10 days, movement of the mass transfer zone thru the media and the adsorption kinetics were not well predicted for the contactor configuration used in the test, and the media would need to be changed frequently using the current contactor configuration.

Uranium adsorption kinetics of HA 216 media are slow compared to activated carbon, and design EBCT has a significant impact on the final treated water concentration, as the media is loaded with uranium. The size of the mass transfer zone moving through the bed and the equilibrium between the media and the treated water concentrations will vary as a function of EBCT. Particle size can also affect the kinetics of

the adsorption process with smaller particle sizes providing more surface area for adsorption in a given media volume.

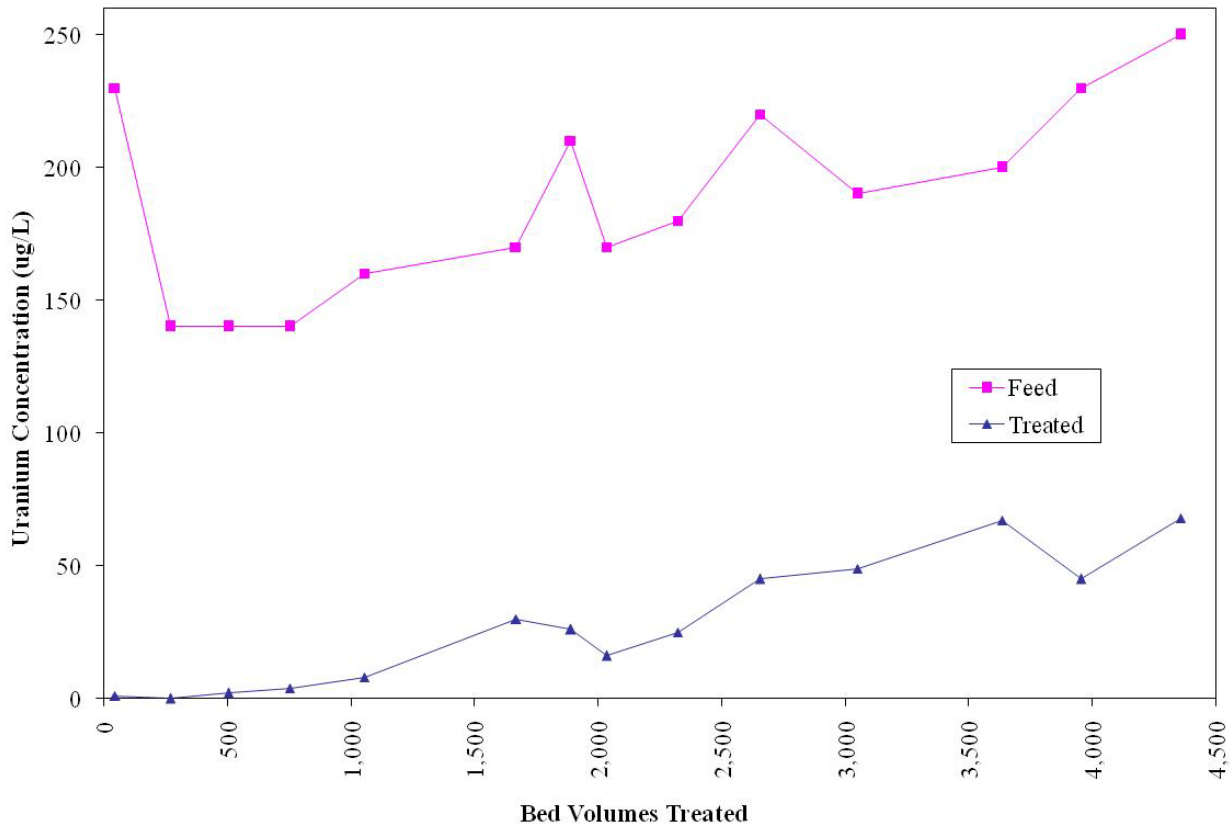


Figure VS-1. Uranium Concentration versus Bed Volumes Treated

Supplemental data provided by Brimac is presented in the report concerning adsorption rates and capacity of the HA 216 media. Their documentation indicates that reducing the particle size of the media increases the adsorption rate. Brimac is currently developing an approach to manufacture a smaller particle size media. Brimac has indicated the need for additional verification testing in the future with a redesigned treatment contactor and media.

Feed and treated water concentrations of cations and anions (calcium, magnesium, sodium, iron, silica, chloride, sulfate, alkalinity, fluoride, nitrate, phosphorus) were about the same, with the exception of phosphorus. The phosphorus levels increased from <0.05 mg/L in the source water to a concentration range of 0.08 to 0.19 mg/L in the treated water. The HA 216 adsorptive media contains calcium, phosphorus, and hydroxide. The slight increase in phosphorus could be due to a small amount of dissolution of the phosphorus from the media. The contribution appears small. There was minimal or no increase in calcium or hydroxide (alkalinity) concentrations in the treated water.

System Operation

The test unit was simple and easy to operate, particularly since there were no pumps required for this installation and no need for automated controls or backwash systems. Flow control was maintained by one manual control valve and the source water was fed to the unit using well system pressure. In this application with the treated water discharging by gravity to the sewer system, there was no concern with operating the unit in-line with the water supply system. Time to operate and monitor the system was

minimal with most time being spent for sample collection. Over the testing period, the average time on site was about 40 minutes each day (90 minutes, the first two days).

The feed water contained low turbidity and low TSS concentrations, and pressure buildup due to solids entering the media bed was not observed. Other source waters may require pre-filtration and continuous monitoring of inlet and outlet pressures to address possible media fouling conditions.

QUALITY ASSURANCE/QUALITY CONTROL

NSF provided technical and QA oversight of the verification testing, including an on-site audit of operating and sampling procedures. The NSF QA Department performed a QA review of the analytical data. A complete description of the QA/QC procedures is provided in the verification report.

Original signed by Sally Gutierrez 10/06/10

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NOTICE: Verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA and NSF make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always operate as verified. The end-user is solely responsible for complying with any and all applicable federal, state, and local requirements. Mention of corporate names, trade names, or commercial products does not constitute endorsement or recommendation for use of specific products. This report is not an NSF Certification of the specific product mentioned herein.

Availability of Supporting Documents

Copies of the test protocol, the verification statement, and the verification report (NSF report # NSF 10/33/EPADWCTR) are available from the following sources:

1. ETV Drinking Water Systems Center Manager (order hard copy)
NSF International
P.O. Box 130140
Ann Arbor, Michigan 48113-0140
2. Electronic PDF copy
NSF web site: <http://www.nsf.org/info/etv>
EPA web site: <http://www.epa.gov/etv>

May 2010

Environmental Technology Verification Report

Removal of Uranium in Drinking Water

Brimac Environmental Services, Inc.

Brimac HA 216 Adsorptive Media

Prepared by:

NSF International and Scherger and Associates
Ann Arbor, Michigan 48105

Under a cooperative agreement with the U.S. Environmental Protection Agency

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Notice

The U.S. Environmental Protection Agency, through its Office of Research and Development, funded and managed, or partially funded and collaborated in, the research described herein. It has been subjected to the Agency's peer and administrative review and has been approved for publication. Any opinions expressed in this report are those of the author (s) and do not necessarily reflect the views of the Agency, therefore, no official endorsement should be inferred. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Foreword

The EPA is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permittees, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six environmental technology centers. Information about each of these centers can be found on the internet at <http://www.epa.gov/etv>.

Under a cooperative agreement, NSF International has received EPA funding to plan, coordinate, and conduct technology verification studies for the ETV "Drinking Water Systems Center" (DSWC) and report the results to the community at large. The DWSC has targeted drinking water concerns such as arsenic reduction, microbiological contaminants, particulate removal, disinfection by-products, radionuclides, and numerous chemical contaminants. Information concerning specific environmental technology areas can be found on the internet at <http://www.epa.gov/nrmrl/std/etv/verifications.html>.

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Abbreviations and Acronyms

ANSI	American National Standards Institute
BV	bed volume
Brimac	Brimac Environmental Services, Inc.
cm	centimeter
°C	degrees Celsius
DWSC	Drinking Water Systems Center
EBCT	empty bed contact time
EPA	U. S. Environmental Protection Agency
ETV	Environmental Technology Verification
FNPT	Female National Pipe Thread
FTO	Field Testing Organization
ft	foot
g	gram
gpm	gallons per minute
kg	kilogram
L	liter
lb	pound
LCS	laboratory control sample
LFM	laboratory fortified matrix
MB	method blank
MCL	maximum contaminant level
N/A	not applicable
NA	not analyzed
ND	not detected
NHDES	New Hampshire Department of Environmental Services
NIST	National Institute of Standards and Technology
NRC	United States Nuclear Regulatory Agency
NRMRL	National Risk Management Research Laboratory
NSF	NSF International (formerly known as National Sanitation Foundation)
NTU	Nephelometric Turbidity Units
pCi	picocuries
psi	pounds per square inch
PSTP	product-specific test plan
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
QAPP	Quality Assurance Project Plan
RPD	relative percent difference
SM	<i>Standard Methods for the Examination of Water and Wastewater</i>
SOP	Standard Operating Procedure
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
U	uranium
µg	microgram
µmhos	micromhos

Acknowledgments

NSF International (NSF) and the New Hampshire Department of Environmental Services (NHDES) co-managed the Field Testing Organization (FTO) responsibilities. NHDES staff provided on site services including sample collection, observation and recording of the equipment operating conditions, measurement of pH, temperature, radon, and alpha radioactivity. NHDES also participated in review of the Test Plan and Final Report. NSF provided logistical and communications support for the verification test, and was responsible for data management, data interpretation, and the preparation of this report. The NSF laboratory provided analytical services for uranium testing and related water quality parameters. Brimac Environmental Services (Brimac) personnel installed the equipment and provided technical assistance during the testing.

Field Testing Organizations

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NSF also wishes to acknowledge Bruce Bartley for providing guidance and program management support, and Dale Scherger of Scherger and Associates for help with report preparation.

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Chapter 1

Introduction

1.1 Environmental Technology Verification (ETV) Program Purpose and Operation

The U.S. Environmental Protection Agency (USEPA) has created the ETV Program to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and more cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations; with stakeholder groups consisting of buyers, vendor organizations, and permittees; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders; by conducting field or laboratory testing, collecting and analyzing data; and by preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The USEPA has partnered with NSF International (NSF) under the ETV Drinking Water Systems Center (DWSC) to verify performance of drinking water treatment systems that benefit the public and small communities. It is important to note that verification of the equipment does not mean the equipment is “certified” by NSF or “accepted” by USEPA. Rather, it recognizes that the performance of the equipment has been determined and verified by these organizations under conditions specified in ETV protocols and test plans.

1.2 Purpose of Verification

The DWSC evaluated the performance of the Brimac Environmental Services, Inc. (Brimac) HA 216 adsorptive media for removal of uranium (U) from drinking water. The verification was initially split into two phases. The initial test (the test reported herein) was designed to evaluate the ability of the adsorptive media to remove uranium from a drinking water source to a level at or below the EPA National Primary Drinking Water Regulations (NPDWR) maximum contaminant level (MCL) of 30 µg/L, and to determine the adsorptive capacity of the media. This report presents the verification test results for the initial test of the Brimac HA 216 adsorptive media. The second test was planned to verify the media capacity and adsorptive performance at a second drinking water location. The second test was designed to be performed with changes to the media and test conditions (quantity of media used, residence time, loading and flow rates, media particle size, etc.) based on the findings from this initial verification. Based on these initial findings, the second test has been postponed until Brimac can make changes to the media particle size to address media capacity and the short run times encountered during this test.

1.3 Testing Participants and Responsibilities

The ETV testing of the Brimac HA 216 adsorptive media was a cooperative effort between the following participants:

NSF
New Hampshire Department of Environmental Services (NHDES)
Brimac
USEPA

Figure 1-1 presents the primary participants in the ETV and their organizational relationships.

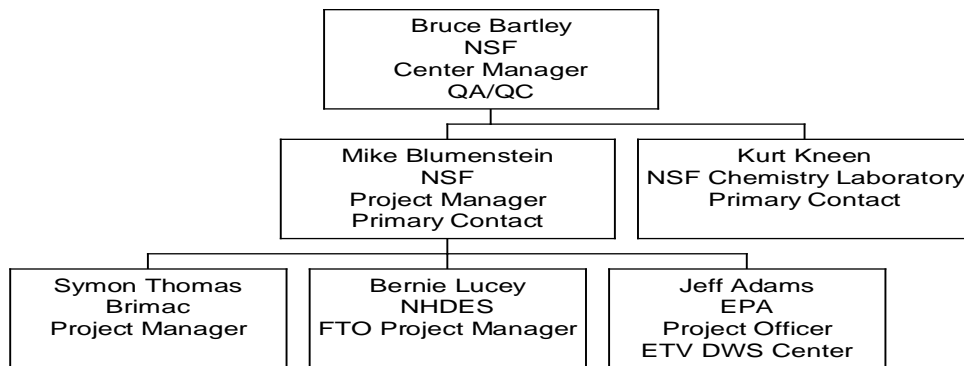


Figure 1-1. ETV test organization chart.

The following is a brief description of each of the ETV participants and their roles and responsibilities.

1.3.1 Field Testing Organizations

NSF and NHDES co-managed the field-testing organization (FTO) responsibilities for this ETV test. The FTO was responsible for conducting verification testing of the pilot unit. Specific responsibilities of the FTO were as follows:

- Provide needed logistical support, establish a communications network, and schedule and coordinate the activities of all verification testing participants (NSF);
- Verify that the locations selected as the test sites have feed water quality consistent with the objectives of the verification testing (NSF); and
- Oversee and conduct the daily testing activities, collecting test samples and delivering those samples to the laboratories for analysis (NSF and NHDES).

1.3.1.1 NSF International

NSF is a not-for-profit organization dedicated to public health and safety, and to protection of the environment. Founded in 1946 and located in Ann Arbor, Michigan, NSF has been instrumental

in the development of consensus standards for the protection of public health and the environment. The USEPA partnered with NSF to verify the performance of drinking water treatment systems through the USEPA's ETV Program. NSF entered into an agreement on October 1, 2000 with the EPA to create a DWSC dedicated to technology verifications. NSF manages an ETV Program within the DWSC for the purpose of providing independent performance evaluations of drinking water technologies. Verified results of product evaluations presented in reports from ETV tests may accelerate a technology's entrance into the commercial marketplace.

For the Brimac HA 216 adsorptive media verification test, NSF prepared the test/QA plan, provided laboratory testing services, managed, evaluated, interpreted, and reported on the data generated by the testing, and reported on the performance of the technology.

The following were specific NSF roles and responsibilities:

- Prepare the Product Specific Test Plan (PSTP) for the verification testing;
- Review the PSTP to insure compliance with the general requirements of the appropriate EPA/NSF ETV Protocols;
- The NSF QA/QC Department conducted an audit at the test site to confirm testing followed the PSTP;
- Manage, evaluate, interpret and report on the test data;
- Coordinate the report reviews; and
- The NSF Chemistry Laboratory analyzed samples throughout the test for uranium and various other water quality parameters.

Contact Information:

NSF International
789 N. Dixboro Road
Ann Arbor, MI 48105
Phone: 734-769-8010
Fax: 734-769-0109
Contact: Bruce Bartley, Project Manager
Email: bartley@nsf.org

1.3.1.2 New Hampshire Department of Environmental Services

NHDES was responsible for the field support for the verification test. NHDES personnel conducted the daily testing and observation activities at the test site in New Hampshire. NHDES staff observed the equipment operation, recorded field measurements for flow, treated water volume, pressure, temperature, etc. NHDES staff was responsible for collecting all water samples and packaging the samples for transport to the NHDES laboratory and for shipment to NSF.

In addition to FTO responsibilities, NHDES was responsible for reviewing the test plan and final report since this testing may also serve as a pilot study component of a water supply permit application for the installation of a full-scale version of this type of process at this site. Also,

since the site was already a permitted public water supply, NHDES needed to be involved with any modifications that may occur.

The NHDES Laboratory was responsible for analyzing samples throughout the test for various water quality parameters, including pH, turbidity, radon, and alpha radioactivity,

Contact Information:

State of New Hampshire Department of Environmental Services
Drinking Water and Groundwater Bureau
29 Hazen Drive
P.O. Box 95
Concord, NH 03301
Phone: (603) 271-2513
Fax: (603) 271-2513
Contact: Bernie Lucey, Project Administrator

1.3.2 Brimac Environmental Services, Inc.

As the equipment manufacturer, Brimac was responsible for installing and removing the pilot unit at the test site. Brimac was also responsible for providing written and verbal instructions for equipment operation. Brimac provided technical assistance to the FTO during testing and during the development of the PSTP. Brimac also reviewed this Verification Report.

Contact Information:

Brimac Environmental Services, Inc.
318 Galake Ave
Ann Arbor, Michigan 48103
Phone: (734) 998-0763
Contact: Symon Thomas
E-mail: symonthomas@brimacservices.com

1.3.3 U.S. Environmental Protection Agency

EPA provides leadership in the nation's environmental science, research, education and assessment efforts. EPA works closely with other federal agencies, state and local governments, and Indian tribes to develop and enforce regulations under existing environmental laws. EPA is responsible for researching and setting national standards for a variety of environmental programs and delegates to states and tribes responsible for issuing permits, and monitoring and enforcing compliance. Where national standards are not met, EPA can issue sanctions and take other steps to assist the states and tribes in reaching the desired levels of environmental quality. The Agency also works with industries and all levels of government in a wide variety of voluntary pollution prevention programs and energy conservation efforts.

The following are specific EPA roles and responsibilities for this ETV:

- Technical review and QA oversight of the PSTP;
- Final approval of lab methods; and
- Technical review of the final report.

Contact Information:

United States Environmental Protection Agency
National Risk Management Research Laboratory
Water Supply and Water Resources Division
26 W. M.L. King Drive
Cincinnati, OH 45268
Phone: (513) 569-7835
Fax: (513) 569-7185
Contact: Jeffrey Q. Adams, Project Officer
E-mail: adams.jeff@epamail.epa.gov

1.4 Verification Test Site Location

This initial test was performed using a pilot unit containing HA 216 media installed at a business served by groundwater drawn from a well deriving water from the fractured bedrock. The site was at Grappone Toyota at 514 Route 3A in Bow, New Hampshire. This well serves 82 employees. The well can draw up to 11 gpm. The treated water was discharged to the sanitary sewer system, which discharges to a municipal treatment plant.

1.5 Raw Water Characterization

The first task (Task A) of the verification test was to obtain a chemical and physical characterization of the raw water. Historical data were needed to confirm that the source water selected for the verification test had chemical constituents that would challenge the treatment system and were also within the specifications required by the treatment system to be tested.

Historical water quality data supplied by NHDES for the test site are presented in Table 1-1. Note that Table 1-1 gives uranium in picocuries per liter (pCi/L). Uranium reported as pCi/L can be estimated in $\mu\text{g/L}$ by multiplying the pCi/L number by 1.5, as specified by USEPA in the Safe Drinking Water Act. This conversion applies to naturally occurring uranium, where the most abundant isotope is U^{238} .

Table 1-1. Grappone Toyota Well Monitoring Data		
Parameter	Sample Date	
	11/09/99	10/15/01
Uranium (pCi/L) ⁽¹⁾	187	177
Gross Alpha Radiation (pCi/L)	183	192
Radon (pCi/L)	70,000	57,000
Radium 226 (pCi/L)	ND (0.1)	0.6
pH	6.8	NA
Specific Conductance (µmhos/cm)	1093	NA
Alkalinity (mg/L)	90.2	NA
Hardness (mg/L)	273	NA
Chloride (mg/L)	267	340
Cyanide (µg/L)	ND (50)	NA
Nitrate (mg/L N)	1.76	1.81
Nitrite (mg/L N)	ND (0.05)	ND (0.05)
Sulfate (mg/L)	11	NA
Antimony (µg/L)	ND (2)	NA
Arsenic (µg/L)	9.5	NA
Barium (µg/L)	ND (5)	NA
Beryllium (µg/L)	ND (2)	NA
Cadmium (µg/L)	ND (1)	NA
Chromium (µg/L)	ND (5)	NA
Fluoride (mg/L)	1.19	NA
Iron (mg/L)	ND (0.05)	NA
Manganese (µg/L)	19.3	NA
Mercury (µg/L)	ND (1)	NA
Nickel (µg/L)	ND (5)	NA
Selenium (µg/L)	ND (5)	NA
Silver (µg/L)	ND (5)	NA
Sodium (mg/L)	93.8	NA
Thallium (µg/L)	ND (1)	NA
Zinc (µg/L)	12	NA

(1) Note that uranium here is presented in pCi/L.

NA = Not analyzed

ND(X) = Not detected; (X) is the laboratory reporting limit for the analysis.

Chapter 2 Equipment Description

2.1 Statement of Performance Capabilities

Brimac provided the following statement of performance capability:

“The Brimac Environmental Services, Inc. pilot treatment unit containing Brimac HA 216 adsorptive media is capable of reducing uranium up to 150 µg/L to less than one (1) µg/L when the feed water is treated at a hydraulic loading rate of 1.05 gpm/ft² of media cross-sectional surface area. Under these treatment conditions, approximately 83,000 gallons (gal) of feed water can be treated per ft³ of media before uranium is detected in the treated water.”

Brimac’s statement of performance capabilities was used to establish the data quality objectives for this verification test.

2.2 Equipment Description

2.2.1 Basic Scientific and Engineering Concepts of Treatment

The conceptual treatment process for uranium adsorption is based on passing uranium-contaminated feed water through a bed of adsorptive media that has a strong affinity for uranium.

Uranium occurs in water predominantly as U²³⁴ (0.0057% abundance), U²³⁵ (0.7198%) and U²³⁸ (99.276%). These isotopes are radioactive alpha particle emitters. The isotopes have long half lives (2.33x10⁵ years, 7.04x10⁸ and 4.5x10⁹ years, respectively), so uranium is stable for treatment and disposal.

Brimac HA 216 is a hydroxyapatite-based media. The molecular formula for hydroxyapatite is Ca₅(PO₄)₃(OH). Hydroxyapatite sequesters uranium by three processes: 1) incorporation within the hydroxyapatite lattice through ion-exchange with calcium, 2) physisorption and chemisorption with reactive phosphate and calcium oxide groups at the mineral surface, and 3) reaction with free phosphate to form solids that precipitate out of solution.

According to Sorg (1988), uranium is a very reactive element that can form a variety of complexes. Near pH 7, the common uranyl ion (UO₂⁺²) forms stable complexes with phosphate and carbonate. In waters ranging from pH 7 to 10, and in the presence of carbonate, the predominant soluble uranium complexes are UO₂(CO₃)₂⁻² and UO₂(CO₃)₃⁻⁴. Millard and Hedges (1996) found that the presence of carbonate increases uranyl sorption to hydroxyapatite.

Adsorptive media is normally in a packed bed contained in a pressure vessel. As the water flows through the bed the uranium concentration decreases until it is no longer detectable. As the feed water continues to flow through the treatment bed, the media, which comes in first contact with the feed water, becomes saturated with uranium. A treatment band then progresses through the treatment bed until breakthrough occurs. At that point, traces of uranium appear in the treated

water. As flow continues the treatment band progresses through the media until the bed is saturated. The uranium concentration in the treated water is then the same as that in the feed water

Uranium adsorption by hydroxyapatite occurs slower than contaminant adsorption by activated carbon, such that the rate-determining step is the chemical reaction resulting in adsorption, not the rate of diffusion, as with activated carbon. For this reason, Brimac considers uranium adsorption by hydroxyapatite to be more like an ion exchange process. The bed of hydroxyapatite media has a mass transfer zone that moves through the bed in a plug flow manner until the media is exhausted. Mass transfer zone length can be controlled by controlling the hydraulic loading rate of the media.

2.2.2 Brimac HA 216 Media

As described in Section 2.2.1, Brimac HA 216 is a hydroxyapatite media. The particles are highly porous and capable of adsorbing heavy metals, color forming compounds, trihalomethane (THM) precursor compounds, taste and odor producing compounds as well as other organic and inorganic compounds. The media can perform over a wide range of pH and temperature. HA 216 has a Langmuir isotherm capacity of just over one gram (g) of uranium per g of media. HA 216 specifications are given below in Table 2-1.

HA 216 is certified by NSF to NSF/ANSI Standard 61 for water treatment plant applications. HA 216 also has European Pharmacopeia and UK Drinking Water Inspectorate approvals. Furthermore, hydroxyapatite is listed as ‘Generally Recognized as Safe’ by the U.S. Food and Drug Administration.

Table 2-1. Brimac HA 216 Media Specifications	
Chemical Constituents:	70-76% hydroxyapatite
	7-9% CaCO ₃
	9-11% carbon
Physical Properties:	
Total surface area	100 m ² /g
Bulk density	560 – 720 kg/m ³
Pore size	7.5 – 60,000 nm
Pore volume	0.225 cm ³ /g
Moisture	< 5%

2.2.3 Pilot Unit Containing HA 216 Media

Brimac provides custom-designed treatment systems containing HA 216 media, or can also supply the media alone. For verification testing, Brimac provided a pilot unit containing HA 216. The pilot unit consisted of the media in a TIGG Corporation Cansorb[®] C-5 steel drum. The drum contains internal schedule 40 PVC plumbing to ensure proper distribution of the feed water onto the bed of media. The C-5 is 30 inches (in) high, with a diameter of 19 in. The inner diameter of the vessel was assumed to be 18.7 in. The vessel has an internal volume of

approximately 4.8 ft³. The inlet and outlet openings are 3/4" female national pipe thread (FNPT). The recommended maximum pressure to the vessel is 10 psig.

For this test, the pilot unit contained 50 pounds (lb) (23 kilograms (kg)) of media. Table 2-1 lists a bulk density range of 560-720 kg/m³ (35-45 lb/ft³) for the media. Using the median density of 40 lb/ft³, the volume of 50 lb of media is approximately 1.3 ft³. With an internal diameter of 18.7 in, the media depth in the C-5 drum was approximately 8.2 in. The 18.7 in diameter gives a media surface area of 1.9 ft². The unit was setup to be operated at 2.0 gpm, for a hydraulic loading rate of 1.05 gpm/ft². This flow would yield an empty bed contact time (EBCT) of approximately 4.9 minutes (4 minutes and 54 seconds).

The feed and treated water lines were fitted with sample taps by installing 3/4 inch tees that provided locations for sample collection. A totalizing water flow meter was installed in the inlet line, downstream of the inlet sample tap and upstream of a gate valve. The gate valve was used to control flow to the unit. A pressure gauge was installed on the inlet line downstream of the gate valve to monitor inlet water pressure to the unit. The pressure gage was installed by placing a 3/4 tee in the inlet line and connecting the pressure gauge to the tee. All fittings and meters were easy to install using standard 3/4 inch pipe and fittings. These meters and gauges were supplied by NSF and installed in the field. The feed line was connected to the pressure (bladder) tank that is used to maintain water pressure in the building water supply system. The pressure from the water system, as maintained by the well pump and bladder system was used to feed the pilot unit. No additional pumping was required or used to maintain flow to the test system. Figures 2-1 through 2-3 show pictures of the pilot unit and the installation.

Treated water was discharged to the sanitary sewer. This was accomplished by placing the discharge hose into a toilet tank that flowed into the building sewer system. The potential for cross contamination of the water and wastewater systems with this configuration was recognized, but was considered acceptable for this temporary installation for the verification test. In a permanent installation the treated water line would be piped directly into the water supply system.



Figure 2-1. Photo #1 of the Brimac pilot unit.



Figure 2-2. Photo #2 of the Brimac pilot unit.



Figure 2-3. Photo #3 of the Brimac pilot unit.

2.3 Operator Requirements

Operator attention during the verification test consisted of monitoring the equipment, conducting on-site process water quality analyses, collecting samples for laboratory water quality analyses, and to confirm operation in accordance with the PSTP. The pilot unit did not require daily attention. However, an operator was on site six days per week to collect water samples and record flows and pressures during the first two weeks (14 days) for the system integrity test. The initial plan was to take daily readings and samples for the first 14 days (system integrity test task) and then change to three site visits per week for the continuing capacity test. However, the capacity test was completed shortly after the 14-day integrity test. Therefore, during the verification test, an operator was present to collect samples, take readings, and observe the unit operation on a daily basis, except Sundays.

2.4 Required Consumables

The following consumables were used in the ETV test:

- Brimac HA 216 media: one 50 lb bag.
- No chemicals were added to the water and no electricity was used to pump influent water to the unit for this verification. Therefore, there were no other consumables used for this test.

2.5 Waste Production

The media does not require backwashing, so the only waste produced is spent media. The media needs to be disposed of following United States Nuclear Regulatory Commission (NRC) guidelines. Spent media that has accumulated uranium above 0.05 percent by weight is classified as a source material under The Atomic Energy Act of 1954. Brimac has assumed ultimate responsibility for disposal of the spent media. Brimac has an agreement with a uranium recovery and reprocessing company to recover the adsorbed uranium from the spent media.

2.6 Licensing Requirements Associated with Equipment Operation

States generally require a specific grade of waterworks operator permit in order to operate a filter process on a public water supply. However, this requirement did not apply for the ETV since all of the treated water was discharged to the sewer system.

2.7 Known Limitations of HA 216

Divalent metals, calcium, and lead present at concentrations higher than 1 mg/L may reduce uranium adsorption capacity by competing for reactive sites. General water quality parameters, such as Ca, Mg, Mn, Fe, Alkalinity, SO₄, Cl, F_l, and silica were monitored to determine if any significant adsorption of these common constituents was occurring in the media.

Chapter 3 Methods and Procedures

3.1 Objectives

The objectives of the verification test were to evaluate the pilot unit in the following areas:

- Evaluate the ability of Brimac HA 216 adsorptive media to remove uranium from a drinking water source;
- Determine the adsorptive capacity of the HA 216 media for uranium;
- Determine impacts of any variations in feed water quality or process variation on media performance;
- Report the logistical, human and other resources necessary to operate the equipment; and
- Determine ease of operation of the equipment.

3.2 Quantitative and Qualitative Evaluation Criteria

In order to address the above objectives, the verification test employed the quantitative and qualitative factors listed in Table 3-1 for evaluation of the Brimac pilot unit.

Table 3-1. Quantitative and Qualitative Evaluation Criteria	
Quantitative Criteria	Qualitative Criteria
Feed water flow Feed and treated water quality Hours of operator attention Quantity of spent media Length of operation until uranium exceeds 30 µg/L	Ease of operation Safety Maintenance requirements Impact of operator experience on successful operation

3.3 Operational Data and water quality analyses

Table 3-2 gives operational and water quality parameters monitored during the verification test.

Turbidity and pH were measured at the NHDES Laboratory instead of in the field. The NHDES Laboratory is only a few miles from the test sites, so grab samples for these parameters were collected and immediately transported to the lab for analysis within the allowable holding times.

The radiological analyses (radon and alpha radioactivity) performed by the NHDES were only for informational purposes, because the media is not designed to remove radioactive contaminants as a group, only uranium. As such, the data for these parameters were not included as primary verification parameters during development of the test plan.

Table 3-2. Operational and Water Quality Data Recorded			
On-Site Parameters		Water Quality Laboratory Analyses	
Operational Data	Water Quality Data	NHDES	NSF
Feed water flow Feed water pressure Cumulative volume of water treated Hours operator(s) spent on site	Temperature	pH Turbidity Alpha Radioactivity Radon 222	Alkalinity Arsenic (total) Aluminum Calcium Chloride Total Dissolved Solids (TDS) Fluoride Hardness (total) Iron (total) Magnesium Manganese Nitrate Total Organic Carbon (TOC) Phosphate (total) Silica (total) Sodium Sulfate Total Suspended Solids (TSS) UV ₂₅₄ Uranium

3.4 Field Operations Procedures

The *EPA/NSF ETV Protocol for Equipment Verification Testing for Removal of Radioactive Chemical Contaminants* (April 2002, Chapter 1) and the *EPA/NSF ETV Equipment Verification Testing Plan for Adsorptive Media Processes for the Removal of Arsenic* (September 2003, Chapter 6) specify the procedures to be used to ensure the accurate documentation of pilot unit performance and treated water quality.

NSF and NHDES co-managed the verifications test, sharing the responsibilities of FTO and analytical laboratory. Testing activities were conducted following the procedures described in the PSTP. The pilot unit was operated 24 hours a day, seven days a week throughout the testing period.

The verification test plan included two main tasks: System Integrity Verification and Adsorptive Capacity Verification. System Integrity Verification was a two-week operation of the pilot unit with daily monitoring to ensure the media and pilot unit were functioning properly, and to identify any major systemic problems such as channeling, insufficient media, excessive headloss buildup, etc. Adsorption Capacity Verification was intended to evaluate the capability of the media at a set contact time to remove uranium to below the EPA NPDWR MCL of 30 µg/L.

3.5 Recording Statistical Uncertainty for Water Quality Parameters

For the analytical data obtained during verification testing, 95% confidence intervals were calculated for uranium data and for all other water quality data where the sample set contained eight or more values.

The following formula was employed for confidence interval calculation:

$$\text{Confidence interval} = \bar{X} \pm t_{n-1, 1-\frac{\alpha}{2}} \left(S / \sqrt{n} \right)$$

Where: \bar{X} is the sample mean;
S is the sample standard deviation;
n is the number of independent measures included in the data set;
t is the Student's t distribution value with n-1 degrees of freedom; and
 α is the significance level, defined for 95% confidence as: $1 - 0.95 = 0.05$.

According to the 95% confidence interval approach, the α term is defined to have the value of 0.05, thus simplifying the equation for the 95% confidence interval in the following manner:

$$95\% \text{ confidence interval} = \bar{X} \pm t_{n-1, 0.975} \left(S / \sqrt{n} \right)$$

Results of these calculations are expressed as the sample mean plus or minus the width of the confidence interval.

pH statistics were calculated on a log basis. It should be noted that using mean data and confidence intervals for treated water (effluent) samples for a parameter that is removed in an adsorptive process has only limited application when the test run is short (1-2 months). The concentration of uranium in the treated water was expected to start at a low concentration and then rise as the media was exhausted. Therefore, the mean concentration represented the average of very low and much higher values.

3.6 Verification Testing Schedule

Verification testing activities include equipment set up and shakedown, equipment integrity, adsorptive capacity verification tests, and water quality sampling and analysis. The test schedule was developed to encompass all of these activities.

Testing began in July of 2007. The system integrity and adsorptive capacity verification tests were initiated simultaneously. The system integrity test ran for a two-week (13 full days plus 8 hours) period. The adsorptive capacity test was designed to run until at least 60 $\mu\text{g/L}$ of uranium was detected in the treated water. Initially, it was expected that the capacity test would run for three weeks after the end of the integrity test. However, the capacity ended after 15 days of operation as the effluent concentration of uranium had exceeded the 60 $\mu\text{g/L}$ level.

3.7 Product Specific Test Plan

The ETV protocol and PSTP prepared for this verification test divided the work into three main tasks (A, B, C) with Task C, the verification test itself, divided into five sub-tasks. These tasks were:

Task A: Raw Water Characterization

Task B: Initial Test Runs

Task C: Verification Test

Task 1: System Integrity Verification

Task 2: Adsorptive Capacity Verification

Task 3: Documentation of Operating Conditions and Treatment Equipment Performance

Task 4: Data Management

Task 5: Quality Assurance/Quality Control

Shakedown testing was conducted during Task B to assure the equipment was functioning as intended. There were no changes made to the PSTP after the shakedown period as the media and pilot equipment was found to be working properly.

3.8 TASK A: Raw Water Characterization

The objective of this task was to obtain a chemical and physical characterization of the raw water. Historical water quality data was supplied by NHDES for the test site. These data provided sufficient information to determine that the water source were compatible with the HA 216 adsorptive media and present a fair challenge to the media. The data for the test site is presented in Table 1-1 in Section 1.5.

The first feed water samples from the system integrity test were compared to the historical data to ensure there were no significant changes in the source water quality.

3.9 TASK B: Initial Test Runs

3.9.1 Objectives

The primary objective of this task was to install and operate the test unit to check system integrity and ensure the unit was functioning properly for the verification test. A Brimac representative and an NSF testing technician performed all startup and shakedown testing activities.

3.9.2 Work Plan

Brimac staff coordinated with the FTO to install the equipment and ready the test system for operation. A Brimac representative was on-site to direct final connections and the startup of the equipment. Once ready for operation, Brimac ran the initial startup and shakedown tests to determine the proper operating conditions for water treatment. The system started without any difficulties and no sampling and analysis was performed during the one-day startup period.

3.10 TASK C: Verification test

3.10.1 Introduction

There are five sub-tasks to be performed to achieve a successful verification test. Each of these tasks is described in this section.

3.10.2 Experimental Objectives

The objective of this task is to assess the ability of the Brimac HA 216 media to remove uranium from the feed water, and to assess the media's capacity for uranium adsorption. The tasks described herein are designed to assess uranium removal, monitor equipment operation, and also monitor other water quality parameters. Statistical analysis (standard deviation and confidence intervals) was performed on all analytes with eight or more discrete samples collected over the verification period.

3.10.3 Task 1: System Integrity Verification

3.10.3.1 Experimental Objectives

The objectives of Task 1 were as follows:

- Establish equipment operational reliability under field conditions; and
- Collect operational and water quality data under field conditions that can be related to the operating time, throughput and water quality objectives stated by the manufacturer.

The System Integrity Verification testing was designed to demonstrate the initial ability of the adsorptive media to remove the feed water uranium concentration to below the EPA MCL of 30 µg/L in the treated water. During Task 1, the FTO also evaluated the reliability of pilot unit operation under the environmental and hydraulic conditions at the test site, and determined whether performance objectives stated in 2.1 could be achieved for uranium removal at the set operating parameters for the pilot unit.

3.10.3.2 Operating Conditions

The pilot unit was operated for 320 hours (13 full days plus eight hours) during Task 1 to collect data on equipment performance and water quality for pilot unit and media performance verification. The pilot unit was operated continuously, within the target flow of 2.0 ± 0.5 gpm. Note that the wide tolerance for the flow was necessary due to the water pressure fluctuations at the test site.

3.10.3.3 Operational Measurements and Analytical Schedule

Operational Measurements

Operational data was collected once per day, Monday through Saturday. The dealership was closed on Sundays, so the pilot unit could not be accessed, but the flow was maintained on a continuous basis at approximately 2 gpm. The data collection schedule is summarized in Table 3-3.

Parameter	Monitoring Frequency	Monitoring Method
Feed water cumulative volume	Record once per day	Feed water totalizer meter
Feed water flow	Check & record once per day (adjust if 3 minute running average flow is below 1.5 gpm, or above 2.5 gpm.)	Feed water flow meter
Treated water pressure	Check & record once per day	Treated water pressure gauge
Operating hours	Record once per day in log the total hours of operation since last site visit.	Note operation/downtime in logbook.
Labor Hours	Determine labor hours required.	Record time on-site daily in logbooks.

Water Quality Measurements

Grab samples for on-site and laboratory water quality analyses were collected based on the sampling schedule presented in Table 3-4.

Parameter	Sampling Frequency	Streams to be Sampled
<i>On-Site Analysis</i>		
Temperature	Daily, Mon.-Sat.	Feed and Treated
<i>Laboratory Analyses</i>		
pH	Daily, Mon.-Fri.	Feed and Treated
Turbidity	Daily, Mon.-Fri.	Feed and Treated
Uranium	Daily, Mon.-Sat.	Feed and Treated
Arsenic (total)	Weekly	Feed and Treated
Radon 222	Weekly	Feed and Treated
Alpha Radioactivity	Weekly	Feed and Treated
Alkalinity	Weekly	Feed and Treated
Aluminum	Weekly	Feed and Treated
Calcium	Weekly	Feed and Treated
Chloride	Weekly	Feed and Treated
TDS	Weekly	Feed and Treated
Fluoride	Weekly	Feed and Treated
Total Hardness (as CaCO ₃)	Weekly	Feed and Treated
Iron (total)	Weekly	Feed and Treated
Magnesium	Weekly	Feed and Treated
Manganese	Weekly	Feed and Treated
Nitrate	Weekly	Feed and Treated
TOC	Weekly	Feed and Treated
Phosphate (total)	Weekly	Feed and Treated
Silica (total)	Weekly	Feed and Treated
Sodium	Weekly	Feed and Treated
Sulfate	Weekly	Feed and Treated
TSS	Weekly	Feed and Treated
UV ₂₅₄	Weekly	Feed and Treated

3.10.4 Task 2: Adsorptive Capacity Verification

3.10.4.1 Experimental Objective

The objective of this task was to determine the media's capacity to remove uranium from the feed waters to concentrations less than 30 µg/L. The performance of the media is a function of the feed water quality and contact time. Uranium breakthrough is highly dependent on the source water's uranium concentration, and the adsorptive characteristics of the media. While the water quality standard for uranium is 30 µg/L, Brimac requested that the pilot units be operated until the treated water uranium concentration reached 60 µg/L.

The adsorptive capacity test was designed to provide quality operating and water quality data relative to Brimac's statement of performance capabilities, which was as follows:

“The Brimac Environmental Services, Inc. pilot treatment unit containing Brimac HA 216 adsorptive media is capable of reducing uranium up to 150 µg/L to less than one µg/L when the feed water is treated at a hydraulic loading rate of 1.05 gpm/ft² of media cross-sectional surface area. Under these treatment conditions, approximately 83,000 gal of feed water can be treated per ft³ of media before uranium is detected in the treated water.”

3.10.4.2 Operating Conditions

The Task 2 Adsorption Capacity Verification began simultaneously with Task 1: System Integrity Verification Testing. Based on the performance statement above, Brimac estimated that the pilot unit would need to operate for approximately 36 days until uranium was detected in the treated water.

During the verification test, the unit was operated at the target flow of 2.0 ± 0.5 gpm, as for Task 1. The unit was operated until the treated water uranium concentration rose to 60 µg/L. This occurred sooner than anticipated and the Adsorptive Capacity test ended on July 25 after 15 days of operation.

Test unit operation was monitored, and operational data was collected as described below.

3.10.4.3 Operational and Analytical Schedule

Operational Measurements

The original planned data collection schedule for Task 2 is summarized in Table 3-5. System operation monitoring was similar to that for Task 1, the main difference being the monitoring frequency.

Parameter	Monitoring Frequency	Monitoring Method
Feed cumulative volume	Record at each site visit	Feed totalizer meter
Feed flow	Check & record at each site visit (adjust when 5% above or below target; record before and after adjustment)	Feed flow meter
Feed pressure	Check & record at each site visit	Feed pressure gauge
Operating hours	Record once each site visit the total hours of operation since last site visit. Note any operation downtime in the log book	Note any operation downtime in the log book
Labor Hours	Record number of hours on site at each visit	Record time in logbooks

The original test schedule assumed that the integrity test would end before the adsorptive capacity of the media was reached. The plan at that time was to reduce the monitoring frequency and continue the capacity test. However, since the uranium concentration reached the 60 µg/L level within two days of completion of the integrity test, the monitoring frequency for the capacity test was the same as for the integrity test.

Water Quality Measurements

As discussed above in 3.10.4.2, Task 2 began simultaneously with Task 1. For the duration of Task 1, the Task 1 analytical schedule in Table 3-4 was followed. Once Task 1 was completed, the Task 2 sampling schedule presented in Table 3-6 was going to be followed for the duration of Task 2. The uranium sampling frequencies were intended to provide sufficient water quality data to effectively characterize the breakthrough profile of uranium.

However, as discussed above, the uranium concentration in the treated water had reached 60 µg/L at the end of the integrity test. Therefore the capacity test was stopped after 15 operating days (Days 0-15) and the original plan to reduce sampling frequency was not implemented. The test unit continued to operate for 20 days (Days 0-20). Flow data and operational data were collected on Days 17 and 20, but uranium analyses were not performed.

Table 3.6. Adsorptive Capacity Test Water Quality Sampling Schedule		
Parameter	Sampling Frequency	Streams to be Sampled
<i>On-Site Analyses</i>		
Temperature	M, W, F	Feed and Treated water
<i>Laboratory Analyses</i>		
pH	M, W, F	Feed and Treated water
Turbidity	M, W, F	Feed and Treated water
Uranium	M, W, F	Feed and Treated water
Arsenic (total)	Weekly	Feed and Treated water
Radon 222	Weekly	Feed and Treated water
Alpha Radioactivity	Weekly	Feed and Treated water
Alkalinity	Weekly	Feed and Treated water
Aluminum	Weekly	Feed and Treated water
Calcium	Weekly	Feed and Treated water
Chloride	Weekly	Feed and Treated water
TDS	Weekly	Feed and Treated water
Fluoride	Weekly	Feed and Treated water
Total Hardness (as CaCO ₃)	Weekly	Feed and Treated water
Iron (total)	Weekly	Feed and Treated water
Magnesium	Weekly	Feed and Treated water
Manganese	Weekly	Feed and Treated water
Nitrate	Weekly	Feed and Treated water
TOC	Weekly	Feed and Treated water
Phosphate (total)	Weekly	Feed and Treated water
Silica (total)	Weekly	Feed and Treated water
Sodium	Weekly	Feed and Treated water
Sulfate	Weekly	Feed and Treated water
TSS	Weekly	Feed and Treated water
UV ₂₅₄	Weekly	Feed and Treated water

3.10.5 Task 3: Documentation of Operating Conditions and Treatment Equipment Performance

3.10.5.1 Task 3 Objective

The objective of this task was to accurately and fully document the operating conditions and performance of the equipment. The task was performed in conjunction with both Task 1: System Integrity Verification and Task 2: Adsorptive Capacity Verification.

During each site visit (daily except Sunday), system operating conditions were documented. The volumetric flow through adsorptive media is a critical parameter, and was monitored and documented. Adsorptive media performance is affected by the EBCT, which varies directly with the volumetric flow through the vessel.

3.10.5.2 Work Plan and Analytical Schedule

During each site visit for both Tasks 1 and 2, the treatment equipment operating parameters were monitored and recorded as described in 3.10.3.3 and 3.10.4.3.

3.10.6 Task 4: Data Management

3.10.6.1 Task 4 Experimental Objectives

The objective of this task was to establish an effective protocol for data management at the field operations site, and for data transmission and sample shipment between NHDES and NSF DWSC staff. Development of a viable structure for the recording and transmission of field-testing data by the NHDES was important to ensure NSF received sufficient and reliable data for verification purposes.

The data management system used for this verification involved the use of computer spreadsheet software and manual recording of system operating parameters.

3.10.6.2 Work Plan

The following outline was used for data handling and data verification by the FTO:

- The field technicians recorded operating and water quality data and calculations by hand on custom-designed data sheets bound in a three-ring binder.
- All logbook pages were numbered.
- The logbook indicated the starting and ending dates that apply to entries in the logbook.
- All logbook entries were made in blue or black water-insoluble ink.
- All corrections in the logbook were made by placing one line through the erroneous information and initialed by the field-testing operator.
- Pilot operating logs included a description of the adsorptive media equipment, description of test run(s), names of visitor(s), description of any problems or issues, etc; such descriptions were provided in addition to experimental calculations and other items.

The original logbook was stored on site. The original logbook pages were periodically faxed to the NSF Project Manager.

The database for this verification-testing program was set up in the form of custom-designed spreadsheets. The spreadsheets were capable of storing and manipulating each monitored water quality and operational parameter from each task, each sampling location, and each sampling time. All data from the laboratory notebooks and data log sheets were entered into the appropriate spreadsheets. NSF DWSC staff conducted the data entry offsite. All recorded calculations were checked at this time. Following data entry, the spreadsheet was printed out and another individual checked the printout against the handwritten data sheet. Any corrections were noted on the hard copies and corrected on the screen, and then a corrected version of the spreadsheet was printed out.

3.10.7 Task 5: Quality Assurance/Quality Control (QA/QC)

3.10.7.1 Experimental Objectives

The objective of this task was to maintain strict QA/QC methods and procedures during verification testing. Maintenance of strict QA/QC procedures is important so that if a question arises when analyzing or interpreting data collected, it is possible to verify exact conditions at the time of testing.

3.10.7.2 Work Plan

Equipment flow was verified and recorded during each site visit. The items listed below are in addition to any specified checks outlined in the analytical methods.

It is extremely important that system flow is maintained at a set value, and monitored frequently. Doing so allows a constant and known EBCT to be maintained in the pilot units. Therefore, an important QA/QC objective was the maintenance of a constant volumetric flow rate through the adsorptive media by frequent monitoring and documentation. Documentation included an average and standard deviation of recorded flows through the adsorptive media.

The flow meter and pressure gauges used for verification testing are subject to periodic calibrations as part of the NSF testing laboratory's QA/QC program. The flow meter and pressure gauges used for this test were calibrated within the six months previous to the start of testing.

Weekly QA/QC Verifications:

- In-line flow meter (clean any fouling buildup as needed, and verify flow volumetrically);
- In-line totalizer meter (clean any foulant buildup as needed and verify production rate volumetrically); and
- Tubing/piping (verify good condition of all tubing and connections, replace as necessary).

3.10.7.3 Analytical Methods

The analytical methods used for on-site and laboratory analyses are listed in Table 3-7. The analytical laboratories performed the water quality analyses using EPA or Standard Methods procedures. All of the required QA/QC procedures were performed in accordance with the published methods, and as described in the Quality Assurance Project Plan (QAPP) in Chapter 6 of the PSTP.

Table 3-7. Water Quality Analytical Methods

Parameter	Analytical Method	Method Detection Limit
pH	EPA 150.1	N/A ⁽¹⁾
Temperature	SM ⁽²⁾ 2550	N/A
Turbidity	EPA 180.1	1.0 NTU
Uranium	EPA 200.8	1 µg/L
Arsenic (total)	EPA 200.8	2 µg/L
Radon 222	EPA 913	200 pCi/L
Gross Alpha Radioactivity	EPA 900	4 pCi/L
Alkalinity	EPA 310.2	5 mg/L
Aluminum	EPA 200.7	10 µg/L
Calcium	EPA 200.7	20 µg/L
Chloride	EPA 300.0	0.5 mg/L
TDS	SM 2540 C	5 mg/L
Fluoride	SM 4500-F C	0.1 mg/L
Total Hardness (as CaCO ₃)	SM 2340 B	2 mg/L
Iron (total)	EPA 200.7	20 µg/L
Magnesium	EPA 200.7	20 µg/L
Manganese	EPA 200.8	1 µg/L
Nitrate	EPA 300.0	50 µg/L
TOC	SM 5310 C	0.1 mg/L
Phosphorous (total)	SM 4500-P E	0.1 mg/L
Silica (total)	EPA 200.7	0.2 mg/L
Sodium	EPA 200.7	0.5 mg/L
Sulfate	EPA 300.0	0.5 mg/L
TSS	SM 2540 D	2 mg/L
UV ₂₅₄	SM 5910 B	0.000 Absorbance/cm (A/cm)

(1) Not applicable

(2) SM = *Standard Methods for the Examination of Water and Wastewater*

3.10.7.4 Samples Shipped Offsite for Analysis

Samples for off-site laboratory analysis were collected and preserved in accordance with *Standard Methods* 3010 B, paying particular attention to the sources of contamination as outlined in *Standard Methods* 3010 C. The samples were kept cool, in the range of 2°C to 8°C immediately upon collection, shipped in a cooler, and maintained at a temperature of 2°C to 8°C. Any samples collected Friday through Sunday were kept at 2°C to 8°C until they could be shipped on Monday. Temperature blanks accompanied all samples shipped to NSF. The temperature of each blank was measured and recorded for each sample set.

Chapter 4 Results and Discussion

4.1 Introduction

The ETV test of the Brimac HA 216 adsorptive media was designed with two primary tasks, the Integrity Verification Test and the Adsorption Capacity Test. The pilot unit, which contained the media, was installed at the Grappone Toyota location in Bow New Hampshire in early July 2007. A totalizing flow meter and feed pressure gauge, calibrated and supplied by NSF, were installed when the equipment was setup. The well system pump and bladder tank provided sufficient pressure to easily achieve the target flow. The pressure in the bladder tank did vary with water use at the business and cycled based on the minimum and maximum pressure set by the well control system. While the changing pressure did cause some flow fluctuation during a well pump cycle, the flow to the test unit remained within the test specifications of 2.0 gpm \pm 0.5 gpm. Overall the system startup went smoothly and quickly with the unit being ready for the start of verification testing within a two days. No samples were collected during the startup/shakedown period.

The Integrity Test began on July 10 and ran continuously through July 24. This met the target of operating the unit for two weeks (13 days plus 8 hours). The Adsorption Capacity Test began simultaneously with the Integrity Test and continued until July 25. By this date, the concentration of uranium in the treated water had risen to 68 $\mu\text{g/L}$, which exceeded the target for the capacity test. Therefore the capacity test was stopped after only 15 days of operation.

This section of the ETV report presents the results of the Integrity Verification Test and Adsorption Capacity Verification Test, and a discussion of these results. The results and discussion include the concentration of uranium in the feed and treated water, the operational and other water quality data collected during the tests, and an assessment of the equipment operation.

4.2 System Integrity Verification Testing

The Integrity Test was started on July 10 and ran without interruption through the scheduled test period. Monitoring and on site data collection were performed as scheduled to verify equipment performance. There were no significant difficulties encountered during the test. Table 4-1 summarizes the operational data collected during the test. Table 4-1 also includes the additional operating data collected as part of the Adsorption Capacity Test. These data were combined into one table for ease of presentation.

Day	Date	Flow⁽¹⁾ (gpm)	Cumulative Volume from Flow Totalizer (gal)	Feed Pressure (psi)	Time On Site (hrs)
0	10-Jul	1.89	0	0	n/r
1	11-Jul	2.00	2,101	0.7	1.50
2	12-Jul	1.58	4,307	8.5 ⁽²⁾	0.50
3	13-Jul	1.65	6,638	1	0.75
4	14-Jul	1.90	9,489	1	0.42
5	15-Jul	n/m	n/m	n/m	n/s
6	16-Jul	1.85	15,243	1	0.50
7	17-Jul	1.90	17,330	1	1.25
8	18-Jul	1.98	18,694	1	0.33
9	19-Jul	1.90	21,402	1	0.75
10	20-Jul	2.17	24,503	1	0.50
11	21-Jul	2.16	28,231	1	0.50
12	22-Jul	n/m	n/m	n/m	n/s
13	23-Jul	2.15	33,729	1	0.33
14	24-Jul	2.16	36,731	1	0.75
15	25-Jul	n/m	n/m	n/m	n/s
17	27-Jul	2.10	45,726	1	0.33
20	30-Jul	2.10	54,728	n/m	0.67
Number		15	—	14	14
Mean		1.97	—	1.4	0.65
Max.		2.17	—	8.5	1.50
Min.		1.58	—	<0.5	0.33
Std. Dev.		0.18	—	2.1	0.35
95% Conf. Interval		1.88 - 2.06	—	0.4 - 2.5	0.47 - 0.83

(1) The reported flows are the averages of several flows recorded over several minutes.

(2) The field notes indicate the outlet line was kinked, which most likely caused this high pressure reading. Pressure dropped to more normal range after the kink was removed

n/m – not measured

n/s – not on site

n/r – not recorded

4.2.1 Integrity Test Flow and Treated Water Volume

Flow and volume of water treated were identified as critical parameters, because residence time (EBCT) and total volume of water treated directly relate to the performance of adsorptive media. The goal for flow control was to maintain the flow at 2.0 gpm ± 0.5 gpm during the verification test. As shown by the data in Table 4-1, the flow was steady during the test with a mean value of 1.97 gpm, and a range of 1.58 to 2.17 gpm.

The flow to the unit cycled as the pressure in the well system cycled from high to low, varying by up to 0.4 gpm. The well pump would activate every few minutes when the pressure in the bladder tank reached the low-pressure set point and then the pump would shut off when the system reached the high-pressure set point. Therefore, the field technician observed several flow rates over several minutes and recorded a range of flows on the bench sheet. These flow ranges were then used to report an average flow for the unit. While the flow did change over a range of readings, the average flow was close to the target of 2.0 gpm and was consistent during the test.

Overall, the frequent change in flow rate did not impact the volume of water treated on a daily basis, as shown by comparing the data for the average flow and daily volume treated. The mean flow reported for the 20 days of operation (Days 0-20) was 1.97 gpm, while the mean flow calculated using the total volume treated was 2.03 gpm (54,728 gal over 20 days as measured by the totalizer on the flow meter (449.6 hours of operation)).

The hydraulic loading rate during the test, based on a mean flow of 1.97 gpm and a pilot unit surface area of 1.90 ft², averaged 1.04 gpm/ft². This was very close to the Brimac recommended test condition hydraulic loading rate of 1.05 gpm/ft². The Pilot Unit contained 50 lb of media with an approximate volume of 1.3 ft³. The EBCT during the verification test was approximately 4 minutes and 54 seconds.

4.2.2 Integrity Test Uranium Results

The primary objectives of the System Integrity Test were to demonstrate that the source water contained uranium at levels needed for the capacity test and that the test unit could achieve the targeted concentration for uranium (30 µg/L) in the treated water. Table 4-2 shows the uranium results for the entire duration of the verification test (Integrity and Capacity Tests). The uranium concentration in the source water had a mean concentration of 190 µg/L, which was within the target range for the verification test. The pilot unit produced treated water with uranium concentrations of <1 µg/L at the start of the test. Thus, the goals of the Integrity Test were met and the verification test proceeded for the duration of the Integrity Test period (minimum of 13 days, 8 hours). The uranium concentration in the treated water began to increase after two days of operation and exceeded the water quality standard of 30 µg/L on Day 10. As will be discussed further in the Capacity Test Section 4.3, this indicated that the capacity of the media was less than expected. From an Integrity Test perspective, the objectives to demonstrate that the unit could remove uranium to levels below the water quality standard and that the source water concentrations were sufficient to challenge the unit were achieved.

The mean source water uranium concentration was 190 µg/L, which was somewhat lower than the historical concentrations of 260 to 280 µg/L. Note these historical concentrations were estimated based on the concentrations of 177 and 187 pCi/L, as shown in Table 1-1. The conversion factor used for this estimate was 1.5. The somewhat lower concentrations of uranium in the source water were sufficient to provide a challenge to the test unit, as Brimac's performance claim was based on a source water concentration of 150 µg/L.

While the treated water uranium concentration increased more quickly than anticipated, the mean concentration for the 15 day monitoring period was 29.7 µg/L. However, the treated water was only below the water quality standard for the first 10 days of the test.

Figure 4-1 in Section 4.3 presents the uranium results in a time series graph.

Table 4-2. Verification Test Uranium Results			
Day	Date	Uranium Concentration	
		Feed (µg/L)	Treated (µg/L)
0	10-Jul	230	1
1	11-Jul	140	<1
2	12-Jul	140	2
3	13-Jul	140	4
4	14-Jul	160	8
5	15-Jul	n/m	n/m
6	16-Jul	170	30
7	17-Jul	210	26
8	18-Jul	170	16
9	19-Jul	180	25
10	20-Jul	220	45
11	21-Jul	190	49
12	22-Jul	n/m	n/m
13	23-Jul	200	67
14	24-Jul	230	45
15	25-Jul	250	68
	Number	14	13
	Mean	190	29.7
	Max.	250	68
	Min.	140	<1
	Std. Dev.	36.6	23.5
	95% Conf. Interval	170 -210	16.9-42.5

n/m – not measured

4.2.3 Integrity Test Water Quality Results

Several water quality parameters were monitored on a weekly basis during the test. Tables 4-3 and 4-4 show the results for these parameters.

The source water did not contain any noticeable suspended sediment as shown by the turbidity data presented in Table 4-3 and the total suspended solids (TSS) data presented in Table 4-4. All turbidity measurements were <1 NTU and all TSS concentrations were <2 mg/L. A sediment/particulate pre-filter was not used in front of the test unit. There was no indication during the test of any problems with particulate accumulation in the media bed. However, in applications where the source water contains particulate matter, it may be necessary to use a pre-filter to protect the media from accumulating solids, which could result in plugging the media bed.

Table 4-3. Verification Test Temperature, pH and Turbidity Results

Day	Date	pH (SU)		Temperature (°C)		Turbidity (NTU)	
		Feed	Treated	Feed	Treated	Feed	Treated
0	10-Jul	6.68	7.29	13.7	14.5	<1	<1
1	11-Jul	6.69	6.79	14.5	14.5	<1	<1
2	12-Jul	6.68	6.88	14.5	14.5	<1	<1
3	13-Jul	6.63	6.68	14.5	14.5	<1	<1
4	14-Jul	6.93	6.74	14.5	14.5	<1	<1
5	15-Jul	n/m	n/m	n/m	n/m	n/m	n/m
6	16-Jul	6.60	6.64	14.5	14.5	<1	<1
7	17-Jul	6.68	6.63	14.5	14.5	<1	<1
8	18-Jul	6.52	6.69	15.0	15.0	<1	<1
9	19-Jul	6.67	6.68	15.0	15.0	<1	<1
10	20-Jul	6.66	6.63	14.5	14.5	<1	<1
11	21-Jul	6.74	6.63	14.5	15.0	<1	<1
12	22-Jul	n/m	n/m	n/m	n/m	n/m	n/m
13	23-Jul	6.69	6.64	14.5	14.5	<1	<1
14	24-Jul	6.73	6.67	14.5	14.5	<1	<1
15	25-Jul	6.56	6.65	n/m	n/m	<1	<1
17	27-Jul	6.64	6.65	14.5	15.0	<1	<1
20	30-Jul	6.72	6.71	15.0	15.0	<1	<1
Number		16	16	15	15	16	16
Mean		6.68	6.72	14.5	14.7	<1	<1
Max.		6.93	7.29	15.0	15.0	<1	<1
Min.		6.52	6.63	13.7	14.5	<1	<1
Std. Dev.		0.09	0.17	0.31	0.24	N/A	N/A
95% Conf. Interval		6.64-6.72	6.64-6.80	14.3-14.7	14.6-14.8	N/A	N/A

n/m – not measured

The temperature and pH of the source water were steady over the entire test. This was expected, as groundwater sources generally do not vary over short periods of time. The source water pH had mean value of 6.68, and the temperature averaged 14.6 °C. No treatment chemicals were added to the system and the media did not impact the pH or temperature of the water. The treated water pH had a mean value of 6.72, and the outlet temperature averaged 14.7 °C.

The water quality data for normal cations and anions (calcium, magnesium, sodium, iron, silica, chloride, sulfate, alkalinity, fluoride, nitrate, phosphorus), as shown in Table 4-4, indicate that the source water and treated water concentrations are basically the same, with the exception of phosphorus. The phosphorus levels increased from <0.05 mg/L in the source water to a concentration range of 0.08 to 0.19 mg/L in the treated water. The HA 216 adsorptive media is a material that contains calcium, phosphorus, and hydroxide. The slight increase in phosphorus is most likely due to a small amount of dissolution of the phosphorus in the media. The contribution appears small. There was minimal or no discernable increase in calcium or hydroxide (alkalinity) concentrations in the treated water. Calcium and hydroxide are the other two components of the media (Ca₅(PO₄)₃(OH)).

Date	Calcium (mg/L)		Magnesium (mg/L)		Sodium (mg/L)		Iron (mg/L)	
	Feed	Treated	Feed	Treated	Feed	Treated	Feed	Treated
10-Jul	130	120	20	30	170	170	<0.02	<0.02
17-Jul	360	390	97	100	230	200	<0.02	<0.02
25-Jul	130	130	160	170	170	170	<0.02	<0.02
Max	360	390	160	170	230	200	<0.02	<0.02
Min	130	120	20	30	170	170	<0.02	<0.02
Date	Chloride (mg/L)		Sulfate (mg/L)		Alkalinity (mg/L CaCO ₃)		Hardness (mg/L CaCO ₃)	
	Feed	Treated	Feed	Treated	Feed	Treated	Feed	Treated
10-Jul	430	440	13	9.4	82	120	420	430
17-Jul	480	460	14	13	81	82	450	490
25-Jul	400	400	12	13	91	85	960	1,000
Max	480	460	14	13	91	120	960	1,000
Min	400	400	12	9.4	81	82	420	430
Date	Fluoride (mg/L)		Nitrate (mg/L N)		Phosphorus (mg/L P)		Arsenic (mg/L)	
	Feed	Treated	Feed	Feed	Feed	Treated	Feed	Treated
10-Jul	0.90	<0.1	1.7	0.007	0.007	0.08	0.007	<0.002
17-Jul	0.90	0.5	1.9	0.007	0.007	0.19	0.007	0.006
25-Jul	0.98	0.8	1.6	0.007	0.007	0.12	0.007	0.007
Max	0.98	0.8	1.9	0.007	0.007	0.19	0.007	0.007
Min	0.90	<0.1	1.6	0.007	0.007	0.08	0.007	0.006
Date	Aluminum (mg/L)		Manganese (mg/L)		Silica (mg/L SiO ₂)		TDS (mg/L)	
	Feed	Treated	Feed	Treated	Feed	Treated	Feed	Treated
10-Jul	<0.01	<0.01	0.074	<0.01	<0.01	0.074	1,000	1,000
17-Jul	<0.01	<0.01	0.190	0.01	<0.01	0.19	1,000	1,100
25-Jul	<0.01	<0.01	0.053	<0.01	<0.01	0.053	1,100	1,100
Max	<0.01	<0.01	0.190	<0.01	<0.01	0.19	1,100	1,100
Min	<0.01	<0.01	0.053	<0.01	<0.01	0.053	1,000	1,000
Date	TOC (mg/L)		TSS (mg/L)					
	Feed	Treated	Feed	Treated				
10-Jul	0.6	0.3	<2	<2				
17-Jul	0.6	0.4	<2	<2				
25-Jul	0.6	0.5	<2	<2				
Max	0.6	0.5	<2	<2				
Min	0.6	0.3	<2	<2				
Date	Radon 222 (pCi/L)		Alpha Radioactivity (pCi/L)		UV ₂₅₄ (absorbance)			
	Feed	Treated	Feed	Treated	Feed	Treated		
10-Jul	n/m	n/m	n/m	n/m	0.0109	0.9981		
11-Jul	60,000	65,000	170	10	n/m	n/m		
17-Jul	57,000	64,000	n/m	n/m	0.0113	0.0033		
19-Jul	n/m	n/m	n/m	36	n/m	n/m		
25-Jul	64,000	71,000	230	73	0.0139	0.0048		
Max	64,000	71,000	230	73	0.0139	0.9981		
Min	57,000	64,000	170	10	0.0109	0.0033		

n/m – not measured

There was no indication that the adsorption of metals, arsenic, manganese, or aluminum impacted the media or its capacity. Aluminum was not detected in the source water. Arsenic was present at 0.007 mg/L in the source water. On the first sampling day, the treated water arsenic concentration was <0.002 mg/L indicating that some adsorption of arsenic may have occurred. However, the next two sample results (Day 7 and 15) showed that the treated water had similar concentrations to the source water. If some arsenic adsorption did occur it was only for the first few days. Manganese concentrations on Day 1 and 7 were lower in the treated water indicating some adsorption may be occurring. However, the concentration in the treated water was higher on Day 15. Any adsorption that may have occurred did not appear to be at levels high enough to impact the media.

NHDES monitored radon and alpha radioactivity for informational purposes. Verification testing did not include radon and alpha radioactivity as key or primary parameters. The radon results shown in Table 4-4 indicate that the media had no effect on radon levels in the water. These results were expected, as there is no indication that the HA 216 media provides any treatment for radon in groundwater. The alpha radioactivity was lower in the treated water. The source water ranged from 170 to 230 pCi/L and the treated water ranged from 10 to 73 pCi/L. These data would seem to suggest that the HA 216 media removes some of the materials contributing to the alpha radioactivity.

4.2.4 Integrity Test Operational Observations and Findings

The objectives of the Integrity Test included establishing the equipment reliability and observing factors related to ease of operation.

The pilot test unit was simple and easy to operate, particularly since there were no pumps required for this installation and no need for automated controls or backwash type systems. As described earlier, flow control was maintained by one manual control valve and the source water was fed to the unit using well system pressure. In this application with the treated water discharging by gravity to the sewer system there was no concern with operating the unit in-line with the water supply system.

Time to operate and monitor the system was minimal with most time being spent for sample collection. Table 4-1 shows the time that was spent on site to monitor the system and collect samples. The average time on site was about 40 minutes. Time on site was longer the first two days of the test, 90 minutes, and then decreased for the remainder of the test period.

As noted in the water quality discussion (Section 4.2.3), this source water had very low turbidity and suspended solids levels. Therefore, concerns about plugging the media due to solids accumulation and possible pressure buildup on the inlet side of the unit were not real issues in this application. One of the initial measurements in the PSTP was to monitor the pressure differential across the media by monitoring the inlet and outlet pressure. Measurement of the inlet pressure was not performed, as it was not possible to maintain a steady inlet pressure to compare with the outlet pressure. The inlet pressures varied as the well pump cycled between the high and low set points on the bladder tank. Manual measurements once per day would not account for the variation and could yield biased pressure differential data. Further, based on the

low turbidity and low TSS concentrations, it was not expected that any pressure buildup would occur due to solids entering the bed. While solids accumulation and the need for pre-filtration was not a concern for this source water, other source waters may present a need for pre-filtration and continuous monitoring of inlet and outlet pressure.

4.3 Capacity Verification Test

The Capacity test was started on July 10 and ran until July 25, a total of 15 days. The capacity test was stopped early when it was discovered that the uranium concentration in the treated water had exceeded the stop-test level of 60 µg/L. As discussed below, the test ended sooner than the expected 36 days. Monitoring and on site data collection were performed as scheduled in the test plan or at a greater frequency. There were no significant difficulties encountered during the test, other than unit reached capacity sooner than predicted. Table 4-1 summarized the operational data collected during the test.

4.3.1 Flow, Volumetric Loading, and Bed Volumes

As described in Section 4.2.1, the pilot unit operated on steady basis around the targeted flow of 2.0 gpm during the verification test. The mean flow of 2.0 gpm yielded a hydraulic loading rate of 1.05 gpm/ft². The media volume was estimated to be 1.3 ft³, which gives an EBCT of 4 minutes and 54 seconds.

Table 4-5 shows the flow data for the capacity test and also shows the bed volumes (BV) treated during the capacity test. The test was stopped on July 25th, after 40,941 gal had been treated or approximately 4,400 BV.

Date	Flow (gpm)	Cumulative Volume from Flow Totalizer (gal)	Bed Volumes	Uranium (µg/L)	
				Feed	Treated
10-Jul	1.89	0	0	230	1
11-Jul	2.00	2,101	220	140	<1
12-Jul	1.58	4,307	440	140	2
13-Jul	1.65	6,638	680	140	4
14-Jul	1.90	9,489	980	160	8
16-Jul	1.85	15,243	1,600	170	30
17-Jul	1.90	17,330	1,800	210	26
18-Jul	1.98	18,694	1,900	170	16
19-Jul	1.90	21,402	2,200	180	25
20-Jul	2.17	24,503	2,500	220	45
21-Jul	2.16	28,231	2,900	190	49
23-Jul	2.15	33,729	3,500	200	67
24-Jul	2.16	36,731	3,800	230	45
25-Jul	n/m	40,511 ⁽¹⁾	44,200	250	68
27-Jul	2.10	45,726	4,700	n/m	n/m

n/m – not measured – (Note: test stopped)

(1) Estimated based on flow for period July 24-27.

4.3.2 Capacity Test Uranium Results

The objective of the capacity test was to determine the media capacity to remove uranium to meet the EPA MCL of 30 µg/L. Brimac requested that the capacity test be planned to continue until the uranium concentration in the treated water reached 60 µg/L.

The Brimac performance claim based on their evaluation of the HA 216 adsorptive media was as follows:

“The Brimac Environmental Services, Inc. pilot treatment unit containing Brimac HA 216 adsorptive media is capable of reducing uranium up to 150 µg/L to less than 1 µg/L when the feed water is treated at a hydraulic loading rate of 1.05 gpm/ft² of media cross-sectional surface area. Under these treatment conditions, approximately 83,000 gal of feed water can be treated per ft³ of media before uranium is detected in the treated water.”

Based on the Brimac performance claim, Brimac estimated that the pilot unit, operated at 2.0 gpm with 1.3 ft³ of media (50 lb) and an EBCT of 4.9 minutes, would treat approximately 108,000 gal of source water before any uranium is detected in the treated water. This equates to approximately 36 days of operation and 11,000 BV.

As shown in Table 4-5, the uranium concentration in the treated water consistently was above the water quality standard after 21,402 gal of water was treated, or approximately 2,200 BV. These data indicate that at the loading rate used in the capacity test and a mean source water concentration of 190 µg/L, the media had a lower capacity than expected. Using the mean source and treated water uranium concentrations (171 µg/L and 12.6 µg/L) for the first ten days of data (until breakthrough had occurred at 30 µg/L), the 50 lbs (23 kg) of media had absorbed 13.1 g of uranium (0.00057 g U/g media). For the entire test period, the average the uranium concentration in the treated water was 29.7 µg/L, just under the MCL. Using the entire test period, the 23 kg of media had adsorbed approximately 24.8 grams of uranium (0.0011 g U/g media).

Figures 4-1 and 4-2 show the uranium concentration plotted as a time series and as a function of bed volumes treated. While the media capacity to adsorb uranium and meet the water quality standard was lower than expected, the media was able to remove uranium to levels below the MCL for a period of time. Under the conditions of the verification test, namely a source concentration of 190 µg/L and an EBCT of 4 minutes and 54 seconds, the media would need to be changed frequently or a larger amount of media used per volume being treated. However, with sufficient media in a treatment unit, and frequent media change out, these data show that the water quality standard can be achieved.

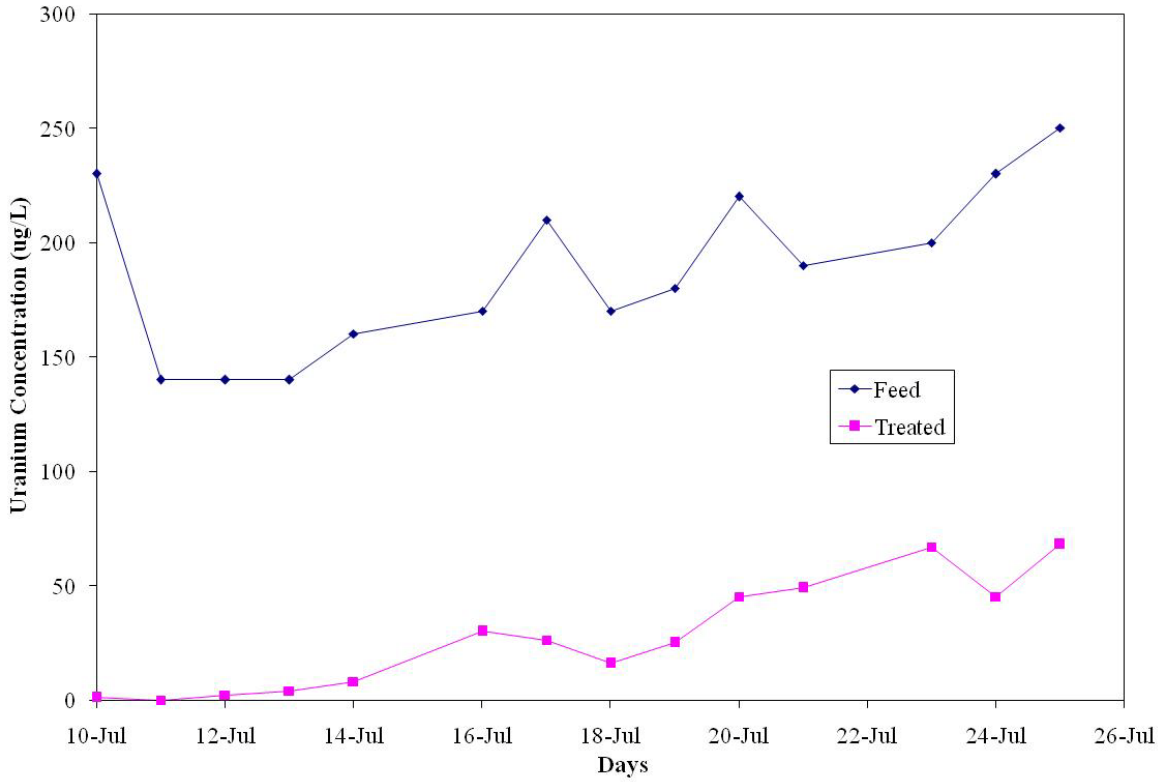


Figure 4-1. Uranium concentration versus time.

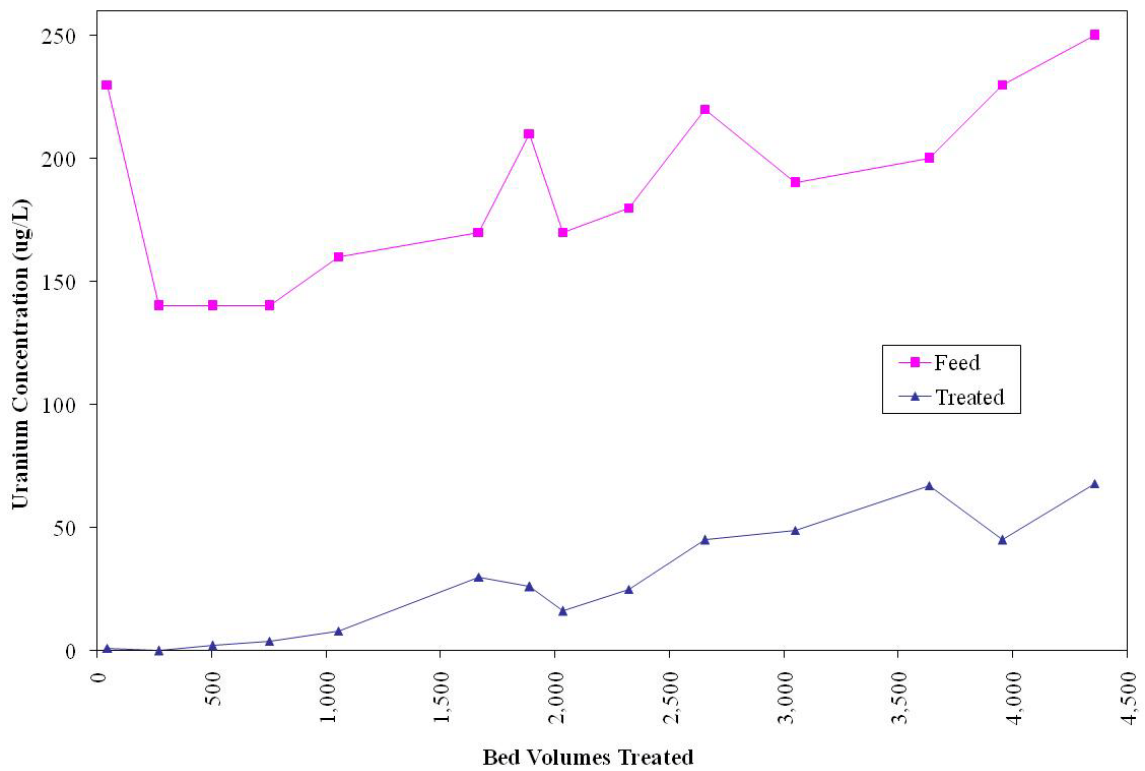


Figure 4-2. Uranium concentration versus bed volumes treated.

4.3.3 Capacity Test Uranium Removal Discussion

The performance of the media is a function of feed water quality and contact time. Uranium breakthrough is highly dependent on the source water concentration and the adsorption characteristics of the media. Thus, there are several factors that can impact the performance and the capacity of the media to adsorb uranium.

The source water quality as described in section 4.2.3 indicates the source water quality did not change significantly through the treatment unit. Adsorption of “other” chemicals can reduce the capacity of an adsorptive media, but the water quality data collected during the verification would seem to indicate that it was not the case for this source water. The concentration of uranium in the source water was close to the expected concentrations and within a reasonable range based on the Brimac specification (150 µg/L). Therefore source water quality does not appear to have had a major impact on the capacity of the media.

The adsorption process for uranium is a slow process and the EBCT will have an impact on the final treated water concentration, as the media is loaded with uranium. The size of the mass transfer zone moving through the bed and the equilibrium between the media and the treated water concentrations will change as bed contact time changes. Particle size can also effect the kinetics of the adsorption process with smaller particle sizes providing more surface and pore area for adsorption, thus increasing overall capacity at a given EBCT.

It would appear that the conditions (media size and EBCT) during this verification test resulted in the treated water reaching the breakthrough target (30 µg/L) long before the actual full capacity of the media was utilized. This was probably due to slow kinetics of the uranium adsorption process.

Initially, a second test was designed to use the same media particle size, flow rates, and hydraulic loading rates as this first test. The depth of the bed was to be increased, which would provide a longer EBCT (11.7 minutes versus 4.9 minutes). The second test, under the planned conditions, was expected to run for approximately 90 days. Brimac estimated that pilot unit #2, if operated at 2.0 gpm would treat approximately 259,000 gal of source water. However, based on this verification data, the planned second test would be projected to run for only 25 to 30 days. It is possible depending on the length and shape of the mass transfer wave, the deeper bed would actually run longer, but it is not expected that it would approach the desired 90 days.

Brimac has provided additional information and data on the adsorption rates and capacity of the HA 216 media. This information, presented in Appendix A, shows that reducing the particle size of the media increases the adsorption rate. Brimac is currently developing the best approach to working with a smaller particle size media that can be used in an additional test. Once this work is complete, a new set of flow rates, EBCT, hydraulic loading will be developed for the additional test.

Chapter 5 QA/QC

5.1 Introduction

An important aspect of the verification testing was the QA/QC procedures and requirements. Careful adherence to the procedures ensured that the data presented in this report were of sound quality, defensible, and representative of the equipment performance. The primary areas of evaluation were representativeness, accuracy, precision, and completeness.

A Quality Assurance Project Plan (QAPP) was developed as part of the PSTP. The full details of the QAPP can be found in the Chapter 6 of the approved PSTP. All of the NSF laboratory activities were conducted in accordance with the provisions of the *NSF International Laboratories Quality Assurance Manual* (NSF 2004).

Both the NSF and NHDES Laboratories are NELAP accredited drinking water laboratories. Furthermore, the NHDES Laboratory is the NELAP accrediting authority for the State of New Hampshire.

5.2 Test Procedure QA/QC

All of the analytical tests performed by the NSF testing laboratory followed the USEPA-approved test/QA plan created specifically for this verification. The NSF QA Department staff reviewed the test procedures and results as part of the normal audit procedure to ensure the proper procedures were followed.

The NHDES Laboratory provided to NSF the daily calibration logs for the pH meter and turbidimeter used for the pH and turbidity measurements. The NSF QA Department reviewed these records and the results to ensure proper procedures were followed. The audit of the data showed that it was acceptable. As specified in the PSTP, the NHDES Laboratory did not provide copies of the raw data logs or QA/QC summaries for the radiological analyses, radon and alpha radioactivity. These data were provided only for informational purposes, since the HA 216 media was not designed to remove these contaminants.

5.3 Sample Handling

All samples analyzed by the NSF and NHDES Chemistry Laboratories were labeled with unique ID numbers. These ID numbers appear in the laboratory reports for the tests. All samples were analyzed within allowable holding times.

5.4 Chemistry Analytical Methods QA/QC

The calibrations of all analytical instruments and the analyses of all parameters complied with the QA/QC provisions of the *NSF International Laboratories Quality Assurance Manual*. The

NSF QA/QC requirements are all compliant with those given in the USEPA method or Standard Method for the parameter. Also, each analytical instrument has an NSF Standard Operating Procedure governing its use.

The field parameters analyzed by the NHDES laboratory (pH, turbidity) were analyzed in accordance with the methods and procedures established in the QAPP. These data were reviewed by the NSF QA Department and found to be acceptable.

5.5 Documentation

All laboratory activities were documented using specially prepared laboratory bench sheets or NSF laboratory reports. Data from the bench sheets and laboratory reports were entered into Excel spreadsheets. These spreadsheets were used to calculate average, maximum, minimum values and other statistics. One hundred percent of the data entered into the spreadsheets was checked by a reviewer to confirm all data and calculations were correct.

5.6 Data Quality Indicators

The quality of data generated for this ETV was established through four indicators of data quality: representativeness, accuracy, precision, and completeness.

5.6.1 Representativeness

Representativeness refers to the degree to which the data accurately and precisely represent the expected performance of the equipment tested. Representativeness was ensured by consistent execution of the test protocol, including timing of sample collection, sampling procedures, and sample preservation. Representativeness was also ensured by using each analytical method at its optimum capability to provide results that represent the most accurate and precise measurement it is capable of achieving.

5.6.2 Accuracy

Accuracy was quantified as the percent recovery of the parameter in a sample of known quantity. Accuracy was measured through use of both matrix spikes of a known quantity, and certified standards during calibration of an instrument. The following equation was used to calculate percent recovery:

$$\text{Percent Recovery} = 100 \times [(X_{\text{known}} - X_{\text{measured}})/X_{\text{known}}]$$

where: X_{known} = known concentration of the measured parameter
 X_{measured} = measured concentration of parameter

Table 5-1 shows the accuracy limits and checks that were established for all analytical parameters. The percent recoveries of all matrix spikes and standards were within the allowable limits for all analytical methods.

Table 5-1. Laboratory Analyses– Accuracy and Precision

Parameter	LFM ⁽¹⁾ (spike sample) Frequency	LFM Acceptance Limits (% Recovery)	MB ⁽²⁾ Frequency	MB Acceptance Limits	LCS ⁽³⁾ (standards) Frequency	LCS Acceptance Limits	LFM Duplicate or Sample Duplicate Frequency	Duplicate Precision Acceptance Limits
pH	N/A	N/A	N/A	N/A	N/A	N/A	10%	30%
Turbidity	N/A	N/A	N/A	N/A	N/A	N/A	10%	30%
Uranium	10%	70-130%	10%	< RL ⁽⁴⁾	10%	85-115%	10%	20%
Alkalinity	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Alpha Radioactivity	10%	70-130%	10%	< RL	10%	80-120%	10%	20%
Aluminum	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Arsenic (Total)	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Calcium	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Chloride	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
TDS	10%	90-110%	10%	< RL	10%	85-115%	10%	20%
Fluoride	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Total Hardness	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Iron (total)	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Magnesium	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Manganese	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Nitrate	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
TOC	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Phosphate (total)	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Radon 222	10%	70-130%	10%	< RL	10%	80-120%	10%	20%
Silica (total)	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Sodium	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
Sulfate	10%	70-130%	10%	< RL	10%	85-115%	10%	20%
TSS	10%	N/A	10%	< RL	10%	85-115%	10%	20%
UV ₂₅₄	N/A	N/A	N/A	N/A	(5)	(5)	N/A	N/A

(1) Laboratory Fortified Matrix

(2) Method Blank

(3) Laboratory Control Sample

(4) Laboratory Reporting Limit

(5) QC Standard is analyzed daily, acceptance limits are assigned by standard manufacturer.

Analyses for pH, temperature, and turbidity do not lend themselves to spike samples and percent recovery or blank analyses. Accuracy for pH, turbidity, and temperature were assured by calibration procedures.

Instrument Calibration Procedures

- pH – Daily 2-point calibration of the pH meter using certified pH buffers of 4.0 and 10.0.
- Temperature – The thermometer used was calibrated within the six months previous to the start of testing using a NIST-traceable certified reference thermometer.
- Turbidimeter – Reagent-grade ultra-pure water was used as a blank on a daily basis to verify proper operation of the instrument. The turbidimeter was calibrated weekly according to the manufacturer's instructions using primary turbidity standards. Secondary standards were used daily to verify the primary standard calibration.

5.6.2.1 Field Equipment Accuracy and Calibration

Equipment operating parameters must be accurate and verifiable for use in evaluating system operating conditions. Routine calibrations and checks of the key operating meters and gauges ensured accurate readings.

Water flow – the difference between the reported flow indicated by a flow meter and the flow as actually measured on the basis of known volumes of water and carefully defined times are standard practices in hydraulics laboratories or water meter calibration shops. The "bucket and stopwatch" technique was used to determine the accuracy of the accessory flow meter and totalizer meter. A stainless steel 5-gallon Seraphin® container was used for the “bucket”. A Seraphin® container is a calibrated container that provides high accuracy for volume measurements. The time to fill the 5-gallon Seraphin® container was measured with a stop watch and then the flow calculated in gpm.

Pressure measurement – accuracy was determined based on a current (within the last six months) manufacturer’s calibration certification.

Meters and gauges were checked at the frequencies presented in Table 5-2 for accuracy. The flow/totalizer meter maintained acceptable accuracy through the test period.

Table 5-2. Field Instrument Calibration Check Schedule			
Instrument	Calibration Check Method	Frequency	Acceptable Accuracy
Pressure Gauge	manufacturer’s certification	once before testing	± 10%
Flow Meter	volumetric using a calibrated 5-gallon Seraphin® container	weekly	± 10%
Totalizer Meter	volumetric using a calibrated 5-gallon Seraphin® container	weekly	± 1.5%
Thermometer	calibration against NIST traceable reference thermometer	monthly	± 5%

5.6.3 Precision

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Analytical precision can be a measure of how far an individual measurement may be from the mean of replicate measurements, or it may be measured as the percent difference between duplicate measurements.

Field duplicate samples were collected to quantify precision. In addition, the samples analyzed by the NSF laboratory were subject to NSF’s laboratory duplicate analysis requirements for verifying precision of the analytical method. The NSF requirement is that at least one out of every ten samples, or one out of every batch of samples, be analyzed in duplicate. The precision of duplicate analyses is calculated using relative percent difference (RPD). The precision control limits are listed in Table 5-1.

RPD is measured using the following equation:

$$RPD = \frac{|S_1 - S_2|}{S_1 + S_2} \times 200$$

where:

S_1 = sample analysis result; and

S_2 = sample duplicate analysis result.

The RPD calculations for the field duplicates are shown in Table 5-3. As can be seen all results are <20% relative percent deviation, except two treated water uranium sample pairs.

The NSF laboratory duplicate data met NSF's internal RPD limits shown in Table 5-1. The data is not displayed here because the samples for this project were batched with samples from other NSF testing activities, therefore, the laboratory duplicate analyses may have been on samples that were not from this project.

Date	Source	Parameter	Base Result	Duplicate Result	RPD
7/10/2007	Feed	pH (SU)	6.68	6.71	0.45
7/20/2007	Feed	pH (SU)	6.66	6.6	0.90
7/11/2007	Feed	Radon 222 (pCi/L)	60,000	64,000	6.45
7/10/2007	Feed	Temperature (°C)	13.7	13.6	0.73
7/10/2007	Feed	Turbidity (NTU)	<1	<1	n/c
7/20/2007	Feed	Turbidity (NTU)	<1	<1	n/c
7/10/2007	Feed	Alkalinity (mg/L CaCO ₃)	82	84	2.41
7/10/2007	Feed	Aluminum (mg/L)	<0.01	<0.01	n/c
7/10/2007	Feed	Arsenic (mg/L)	0.007	0.007	0.00
7/10/2007	Feed	Calcium (mg/L)	130	130	0.00
7/10/2007	Feed	Chloride (mg/L)	430	440	2.30
7/10/2007	Feed	Fluoride (mg/L)	0.9	0.9	0.00
7/10/2007	Feed	Hardness (mg/L CaCO ₃)	420	410	2.41
7/10/2007	Feed	Iron (mg/L)	<0.02	<0.02	n/c
7/10/2007	Feed	Mg (mg/L)	20	18	10.5
7/10/2007	Feed	Mn (mg/L)	0.074	0.084	12.7
7/10/2007	Feed	Nitrate (mg/L N)	1.7	1.7	0.00
7/10/2007	Feed	Phos. (mg/L P)	<0.05	<0.05	n/c
7/10/2007	Feed	Silica (mg/L SiO ₂)	20	20	0.00
7/10/2007	Feed	Sodium (mg/L)	170	170	0.00
7/10/2007	Feed	Sulfate (mg/L)	13	13	0.00
7/10/2007	Feed	TDS (mg/L)	1,000	1,100	9.52
7/10/2007	Feed	TOC (mg/L)	0.6	0.6	0.00
7/10/2007	Feed	TSS (mg/L)	<2	<2	n/c
7/10/2007	Feed	Uranium (ug/L)	230	230	0.00
7/16/2007	Treated	Uranium (ug/L)	30	17	55.3
7/20/2007	Feed	Uranium (ug/L)	220	190	14.6
7/25/2007	Treated	Uranium (ug/L)	68	49	32.5
7/10/2007	Feed	UV ₂₅₄ (Abs)	0.0109	0.009	19.1

n/c = not calculated

5.6.4 Completeness

Completeness refers to the amount of data collected from a measurement process compared to the amount that was expected to be obtained. Completeness is the proportion of valid, acceptable data generated using each method.

The completeness objective for data generated during verification testing was based on the number of samples collected and analyzed for each parameter. Table 5-4 illustrates the completeness objectives for the performance parameters based on the sample frequency.

Number of Samples per Parameter	Percent Completeness
0-10	80%
11-50	90%
>50	95%

Completeness is defined as follows for all measurements:

$$\%C = (V/T) \times 100$$

where:

%C = percent completeness;

V = number of measurements judged valid;

T = total number of measurements.

All completeness objectives were met during the verification test.

5.7 Sampling, sample handling, and preservation

5.7.1 Sampling Locations

Feed and treated water samples were collected from the sample taps outfitted on the feed and treated water lines. The sample taps were installed as close as possible to the feed and treated water ports on the pilot unit drum.

5.7.2 Sample Collection

Prior to collecting samples, the tap was flushed for at least five seconds. All samples were collected into clean containers. Samples requiring 1 liter of volume were collected directly into the 1-liter container. For samples requiring less than 1-liter of volume, a 1-liter container was filled from the tap and then aliquots of the sample were immediately poured into the required containers for laboratory or on-site analysis. Sample times were recorded for all samples collected. Samples for parameters requiring immediate analysis, such as pH and turbidity, were collected separately and transported to the NHDES laboratory as quickly as possible.

The sample bottles for laboratory analysis were prepared ahead of time with the appropriate preservative.

5.7.3 Sample Storage and Transport

Samples for off-site laboratory analysis were collected and preserved in accordance with *Standard Methods* 3010 B, paying particular attention to the sources of contamination as outlined in *Standard Methods* 3010 C. The samples were kept cool, in the range of 2°C to 8°C immediately upon collection, shipped in a cooler, and maintained at a temperature of 2°C to 8°C. Any samples collected Friday through Sunday were kept at 2°C to 8°C until they could be shipped on Monday. Temperature blanks accompanied all samples shipped to NSF. The temperature of each blank was measured and recorded for each sample set.

All samples were analyzed within the Standard Methods or EPA method recommended holding times. The sample collection and preservation details for each parameter are presented in Table 5-5.

Parameter	Bottle Type	Preservation	Holding Time	Notes
pH	1 L polyethylene	none	48 hours	
Temperature	N/A	none	N/A	Immediate analysis in the field
Turbidity	1 L polyethylene	none	48 hours	
Uranium	125 mL polyethylene	nitric acid	180 days	
Alkalinity	1 L polyethylene	none	14 days	no headspace
Gross Alpha Radioactivity	1 L polyethylene	nitric acid	180 days	
Aluminum	125 mL polyethylene	nitric acid	180 days	
Arsenic (Total)	125 mL polyethylene	nitric acid	180 days	
Calcium	125 mL polyethylene	nitric acid	180 days	
Chloride	40 ml glass vial	none	28 days	
TDS	1 L polyethylene	none	7 days	
Fluoride	1 L polyethylene	none	28 days	
Total Hardness	N/A	N/A	N/A	no sample, calculated from calcium and magnesium results
Iron (total)	125 mL polyethylene	nitric acid	180 days	
Magnesium	125 mL polyethylene	nitric acid	180 days	
Manganese	125 mL polyethylene	nitric acid	180 days	
Nitrate	40 mL glass vial	none	48 hours	
TOC	40 mL glass vial	phosphoric acid	28 days	
Phosphate (total)	250 mL amber glass	sulfuric acid	28 days	
Radon 222	4 40 mL glass vials	none	4 days	
Silica (total)	125 mL polyethylene	nitric acid	180 days	
Sodium	125 mL polyethylene	nitric acid	180 days	
Sulfate	40 mL glass vial	none	28 days	
TSS	2 1L polyethylene	none	7 days	
UV ₂₅₄	125 mL amber glass	none	48 hours	

Chapter 6 References

NSF International (2004). *NSF International Laboratories Quality Assurance Manual*. Ann Arbor, NSF International.

APHA, AWWA, and WEF (1999). *Standard Methods for the Examination of Water and Wastewater*, 20th edition. Washington D.C., APHA, AWWA, and WEF.

EPA and NSF (2000). *EPA/NSF ETV Protocol for Equipment Verification Testing for Removal of Radioactive Chemical Contaminants*.

EPA and NSF (2003). *EPA/NSF ETV Protocol for Equipment Verification Testing for the Removal of Arsenic* (Chapter 6).

Millard, A. R. and R. E. M. Hedges (1996). A diffusion-adsorption model of uranium uptake by archeological bone. *Geochimica Et Cosmochimica Acta* 60(12): 2139-2152.

Sorg, T. J. (1988). Methods for removing uranium from drinking water. *Journal of the American Waterworks Association*. July: 105-111.

APPENDIX A

Additional Data on HA 216 Media Adsorption Rates and Capacity

Provided by Brimac

(Note that this information is provided for informational purposes and was not verified as part of the ETV test)

Adsorption of Uranium onto Adsorbent from Brimac

Brimac has been manufacturing the adsorbent media Hydroxyapatite (HA) for over 180 years. Originally this media was exclusively used in the sugar industry for its ability to adsorb very large molecular weight organic compounds (color) and adsorb / exchange / retain 'ash' or inorganic salts such as sulfates, the two combined together being the first process for the manufacture of white sugar. Over the past 50 years, Brimac has expanded the application of HA into the areas of heavy metals removal (with a specifically tailored media) as well as broad drinking water treatment, under the NSF 61 standard.

With the ever increasing need for drinking water, the reducing availability of fresh water sources and the increasing awareness of the need to remove harmful contaminants, the opportunity for novel adsorption solutions to low level contamination issues is gaining momentum. Along with the removal of arsenic and other heavy metals, Brimac sees a market in the remediation of radio nucleotides, especially Uranium and in particular from residential scale borehole sources.

Early work on the adsorption of Uranium onto HA media showed a capacity of 1 g/Uranium per g/HA (potentially dangerous, in that fission conditions might be possible if this capacity was realized in real world conditions and scale).

Due to the difficulty surrounding laboratory uranium availability and testing above batch isotherm scale, the ETV process has been the vehicle for the next stage of this project. The evaluation of a small scale filter was not designed or intended to be a rigorous evaluation of a 'finished product', but a real world development project in progress.

Brimac is a company with an extensive background in design and utilization of media adsorption systems, we knew that while total theoretical capacity is important, the achievable kinetics of adsorption is by far the most important criteria when judging if a system is economically feasible. Hydroxyapatite is not an activated carbon and a number of application errors have been made by third parties in trying to fit this media into the accepted Empty Bed Contact time 'rules of thumb' used for GAC applications. In this case, we knew from experience that the adsorption of metals by HA can have very long 'effective' contact time requirements and this study was to be that first rough cut evaluation of 'column based' Uranium removal.

It is evident from this first test that capacity is present and adsorption is possible, but a systematic work up of the bed depth requirements based on these results indicated a system scale that is not economically practical. Again from previous experience, we understood the relationship between particle size and effective utilization of readily available surface area and it was with this knowledge we approached Arizona State University and the Rapid Small Scale Column Test (RSSCT) team to evaluate the dynamic relationship between the available capacities to the available surface area.

Without a doubt, the conclusions of this test indicate that a granular system would never meet the real world requirements of contact time and only a powder based technology would realize the potential of the media.

In the continuation of our ETV project, the goal of our Stage two study will be the application of the media using block filter technology. Brimac is currently in a development program with KX Technologies to manufacture a nominal 10 micron block filter, comprising of 100% drinking water grade Hydroxyapatite. It is hoped that the enormous increase in readily available surface area provided by the block filter technology will provide the means to utilize the capacity demonstrated in the RSSCT results.

Stage one of our ETV project has been an informative journey, even if our initial expectations may not have been met. I hope you will agree that the conclusions reached point in a logical manner to a new product in a developing field of application. The distribution of this report and the media's potential will hopefully lead to more opportunities to partner with other organization, local and State authorities etc. in the real word application of solving problems and providing cleaner drink water for us all.

1 Adsorption experiment

Faster mass transfers can be obtained by reducing the size of the adsorbents. The adsorbent were grinded and sieved. Adsorbent with the particle size from 75 μm to 90 μm were collected. Adsorption capacities of uranium onto the adsorbent both at the original size and 75 μm to 90 μm were accessed.

Adsorption kinetics and isotherm were determined using duplicate batch experiments. In each experiment, certain amounts of adsorbent were added to a series of 50 ml PTEE tubes. Uranium contaminated water was added into the tube. The tubes were put in a shaker and kept at 25 °C.

Kinetic data were collected by adding 0.045, 0.225, 0.450, 1.35, 2.250 g adsorbent to 45 mL uranium contaminated water. Residual aqueous uranium concentrations at 0, 10, 30, 60, 180 and 360 min were analyzed using ICP-MS after the suspensions were centrifuged for 5 min at 6,000 rpm using centrifuge.

Isotherm tests were conducted for 180 min by changing the concentration of adsorbent at a fixed uranium concentration. The adsorbed amount of uranium onto the adsorbent was calculated from the initial and final concentrations of uranium. Blank experiments were also conducted demonstrated that uranium adsorption onto the walls of the flasks was negligible.

2 Experimental results

2.1 Adsorption kinetics (adsorbent in original size, 250~1000 μm)

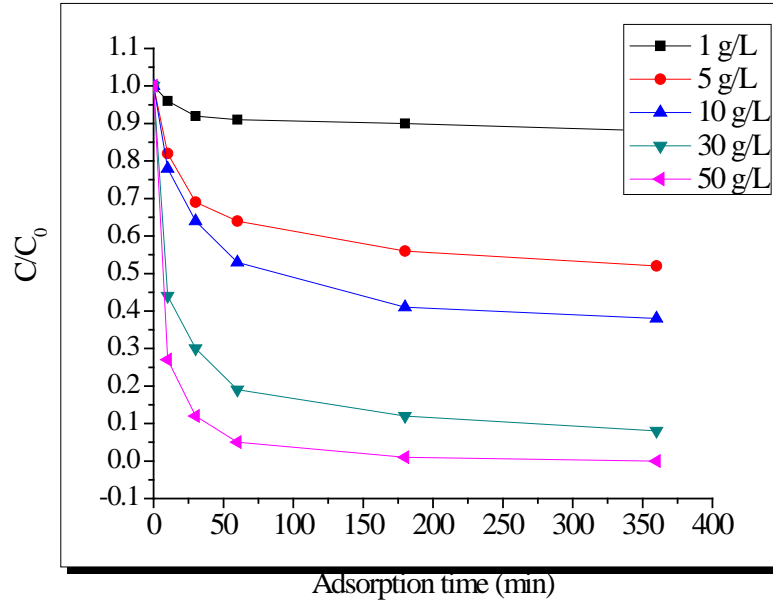


Fig 1 Adsorption kinetics of uranium onto adsorbent with original size

Adsorption kinetics was observed for 360 min, and the results are shown in Fig. 1. Uranium was adsorbed onto adsorbent quickly, and equilibrium was reached within 180 min. Increasing adsorption rate was observed with the increasing of adsorbent concentration. Only 10% of the initial uranium was adsorbed at the adsorbent concentration of 1 g/L, However, 100% adsorption was reached when the adsorbent concentration increased to 50 g/L.

2.2 Adsorption kinetics (grinded adsorbent, 75~90 μm)

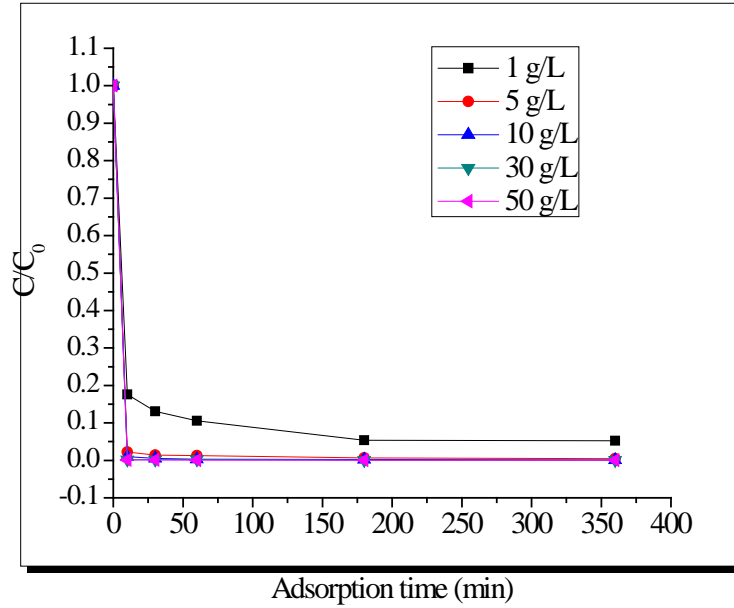


Fig 2 Adsorption kinetics of uranium onto adsorbent within 75 μm to 90 μm

Due to the higher surface area, the adsorption ability of the adsorbent with smaller particle size was much higher than that of at the original particle size. 90% of the initial uranium was adsorbed at the adsorbent concentration of 1 g/L, and, 100% adsorption was reached when the adsorbent concentration was higher than to 5 g/L.

2.3 Adsorption isotherms

The adsorption isotherm data were analyzed using Freundlich (eq 1) and Langmuir (eq 2) adsorption expressions:

$$\log Q = \log K_F + \frac{1}{n} \log C_e \quad (1)$$

$$\frac{1}{Q} = \frac{1}{bQ_{\max}} \times \frac{1}{C_e} + \frac{1}{Q_{\max}} \quad (2)$$

where Q_m (mg g^{-1}) is the maximum adsorption capacity, Q (mg g^{-1}) is the amount of adsorbed uranium, C_e is the equilibrium uranium concentration in solution (mg/L), K_F and n are the

Freundlich constants, and b is the Langmuir constant. The Freundlich and Langmuir parameters were obtained by nonlinear least-squares regression analysis.

Table 1 The Freundlich and Langmuir parameters of adsorption data

Size (μm)	Langmuir isotherm			Freundlich isotherm		
	Q_{max} (mg/g)	b	R^2	K_F (mg/g)	n	R^2
Original size (250~1000)	0.010	263.6	0.754	0.030	0.372	0.890
Grinded (75~90)	1.647	24.6	0.983	7.800	0.809	0.990

As shown in Table 1, the correlation coefficient (R^2) values of the Freundlich isotherm for the adsorbents with original size and at the range of within 75 μm to 90 μm are 0.890 and 0.990, while those of the Langmuir isotherm are 0.745 and 0.983, respectively. The Langmuir and Freundlich plot of the two adsorbents are shown in Fig 3 and Fig 4 respectively. Experimental data of uranium adsorption onto the adsorbents fit Freundlich isotherm well, indicating that heterogeneity of the surface due to involvement of both strong and weaker binding sites for adsorption, thus resulting in a multisite adsorption processes for these adsorbate ions.

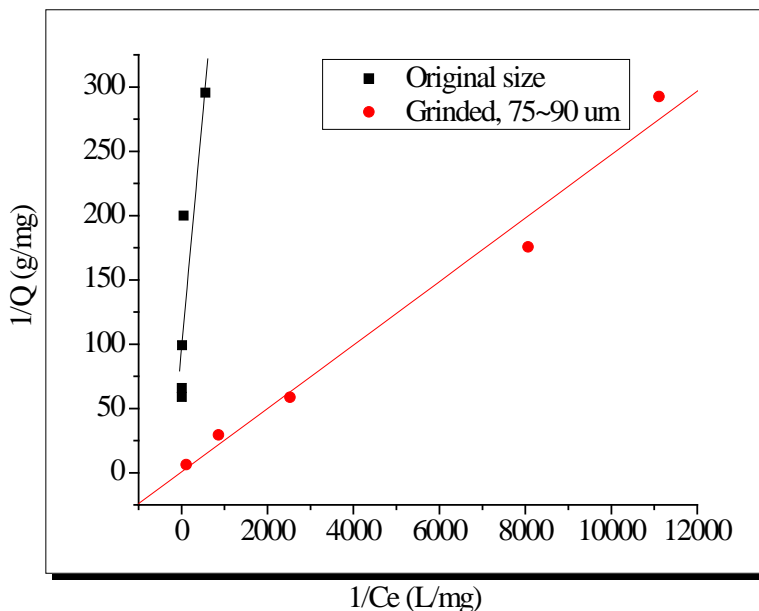


Fig 3 Langmuir plot of adsorption of uranium onto the two adsorbents

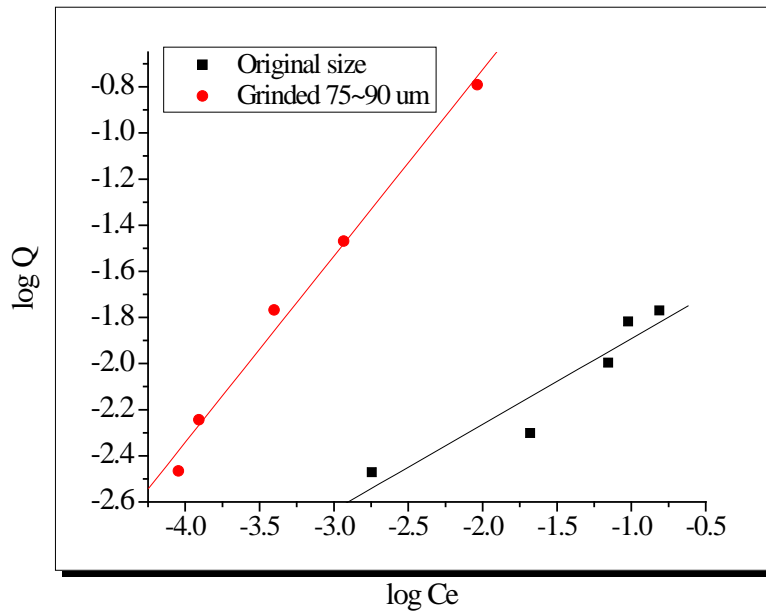


Fig 4 Freundlich plot of adsorption of uranium onto the two adsorbents

3 Conclusions

3.1 Adsorption process is fast; equilibriums can be reached in about 3 hours.

3.2 Grinded particles with the smaller particle size have much stronger adsorption capability for uranium.

3.3 Freundlich isotherm is better to describe the adsorption of uranium onto the adsorbents.

3.4 Faster mass transfers can be obtained by crushing adsorbents to much smaller sizes and using smaller column, higher loading rate, and thus the pilot scale tests can be scaled in a short period.