Occurrence of Metastudtite (Uranium Peroxide Dihydrate) at a FUSRAP Site

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ABSTRACT

Uranium concentrations in groundwater in a localized area of a site exceed the USEPA Maximum Contaminant Level (MCL) by a factor of one thousand. Although the groundwater seepage velocity ranges up to 0.7 meters per day (m/day), data indicate that the uranium is not migrating in groundwater. We believe that the uranium is not mobile because of local geochemical conditions and the unstable nature of the uranium compound present at the site; uranium peroxide dihydrate (metastudtite).

Metastudtite [$UO_4 \circ 2(H_2O)$ or $(U(O_2)\,|\,O\,|\,(OH)_2) \circ 3H_2O$] has been identified at other sites as an alteration product in casks of spent nuclear fuel, but neither enriched nor depleted uranium were present at this site. Metastudtite was first identified as a natural mineral in 1983, although documented occurrences in the environment are uncommon.

The U.S. Army Corps of Engineers (USACE) is conducting a remedial investigation at the DuPont Chambers Works in Deepwater New Jersey under the Formerly Utilized Sites Remedial Action Program (FUSRAP) to evaluate radioactive contamination resulting from historical activities conducted in support of Manhattan Engineering District operations. From 1942 to 1947, Chambers Works converted uranium oxides to uranium tetrafluoride and uranium metal. More than half of the production at this facility resulted from the recovery process, where uranium-bearing dross and scrap were reacted with hydrogen peroxide to produce uranium peroxide dihydrate.

The 280-hectare Chambers Works has produced some 600 products, including petrochemicals, aromatics, fluorochemicals, polymers, and elastomers. Contaminants resulting from these processes, including separate-phase petrochemicals, have also been detected within the boundaries of the FUSRAP investigation.

USACE initiated remedial investigation field activities in 2002. The radionuclides of concern are natural uranium (U_{nat}) and its short-lived progeny. Areas of impacted soil generally correspond to the footprints of the former production buildings. U_{nat} concentrations in soil exceed the investigative screening value, 518 Becquerels per kilogram (Bq/kg) [14 picocuries per gram (pCi/g)], to an approximate depth of 2.5 m. This depth corresponds to the depth of buried demolition debris from the uranium processing site. Aqueous-phase uranium has also

been confirmed at the site and appears to coincide with uranium-impacted soils. Soil textures in the impacted area consist mainly of fine-grained silty sand and rubble. The hydraulic conductivities range from 5E-6 to 1E-5 m/s. Groundwater seepage velocity ranges from 0.003 m/day to 0.7 m/day in the impacted area.

Groundwater investigations conducted throughout the FUSRAP site indicate that redox conditions in the shallow groundwater are reducing, with low dissolved oxygen concentrations, as would be expected underlying a petrochemical facility. In contrast, groundwater in the uranium source area is an oxidizing microenvironment, with elevated pH conditions, despite the presence of free-phase liquid hydrocarbons in close proximity.

Dissolved oxygen is elevated in the uranium source area, which may be due to the presence of metastudtite. Metastudtite has been shown to produce hydrogen peroxide through the process of alpha irradiation of water molecules. Uranium peroxide dihydrate is more soluble in water than other hexavalent mineral forms.

The literature suggests that in the absence of hydrogen peroxide, metastudtite is unstable in groundwater. Although the presence of metastudtite in the source area may have caused locally high levels of aqueous-phase uranium to form, the uranium ions may not be mobile outside of this small area because of significant abrupt changes in geochemical conditions.

The ongoing groundwater investigation includes tasks to confirm the presence of metastudtite and hydrogen peroxide, and monitor for seasonal geochemical or hydrogeologic changes.

INTRODUCTION

The U.S. Army Corps of Engineers (USACE)-Philadelphia District is conducting a Remedial Investigation (RI) for radioactively contaminated areas at the DuPont Chambers Works (Chambers Works) in Deepwater, New Jersey. The RI is being performed under the Formerly Utilized Sites Remedial Action Program (FUSRAP). FUSRAP was established in 1974 by the Department of Energy (DOE) to identify the nature and extent of radiological contamination and take appropriate cleanup action at sites where work had been performed as part of the nation's early atomic energy program. A current phase of the RI is being conducted is to address the potential impact of Manhattan Engineering District (MED)-related radionuclides of potential concern (ROPCs) on the local groundwater.

The ROPCs at Chambers Works are uranium in its natural isotopic ratios (U_{nat}) and its short-lived progeny. Previous RI activities have confirmed the presence of uranium in both soil and groundwater sampled in the vicinity of Operable Unit 1 (OU1), which is the site of a former uranium processing facility. Filtered and unfiltered groundwater samples collected during a previous investigation were analyzed for gross alpha, gross beta, radium, and uranium. A filtered groundwater sample from OU1 had a uranium concentration of 1200 micrograms per liter (µg/L). Based on these findings, additional investigation into the nature and extent of groundwater contamination in OU1 was recommended. In order to supply information to conduct a risk assessment and feasibility study, it is necessary to understand the nature of the uranium (e.g., its chemical form and the factors either enhancing or inhibiting its mobility), as

well as its extent. The investigation approach has been to install monitor wells in the three uppermost aquifers, conduct slug tests, and sample the groundwater, surface water, and sediments.

SITE BACKGROUND

Chambers Works site is a 280-hectare active chemical plant located in western New Jersey on the shore of the Delaware River, near Wilmington, Delaware. Chambers Works has a rich manufacturing history dating back to 1892 and currently produces some 600 products in 44 manufacturing buildings. These products include organic intermediates, aromatics, petroleum chemicals, fluorochemicals, polymers, elastomers, and specialty chemicals.

Beginning in 1942, the MED contracted with Chambers Works to perform several uranium refinement activities. Chambers Works converted uranium oxides to uranium tetrafluoride and small quantities of uranium metal. The conversion processes included:
- Brown oxide process
- Green salt process
- Recovery process
- Hexafluoride process
- Metal process

DOE has estimated that more than half of the Chambers Works production was from uranium peroxide dihydrate obtained by processing uranium-bearing scrap [1]. Chambers Works converted uranium scrap and dross into uranium peroxide dihydrate in Building 845. Uranium peroxide and oxides were further processed in Building 708. No enrichment or depletion processes occurred at Chambers Works.

MED activities were transferred to the Atomic Energy Commission (AEC) in 1946. Chambers Works continued its research for AEC until late 1947. In 1948 and 1949, AEC conducted radiological surveys and decontamination of building surfaces at the site. Oak Ridge National Laboratory (ORNL) conducted a radiation survey of the Chambers Works site in 1977 on behalf of DOE [2]. Bechtel National, Inc. performed an additional radiation survey in 1983 [3].

When USACE assumed responsibility for the FUSRAP program from DOE, USACE divided the investigation areas into three operable units. Operable Unit 1 (OU1), shown in Fig. 1., consists of the areas where uranium refinement took place.

A remedial investigation of OU1 was conducted in 2002 [4]. Uranium oxides were encountered in an area approximately 3 m in diameter at the location of former Building 845. Uranium concentrations above the investigative screening value (ISV) were measured in surface soils throughout the area of former Building 845. Locations of the former buildings and the existing source zones are indicated in Fig. 1.

Shallow soils in the area of Building 708 also exceeded the ISV. Within the former building footprint, U_{nat} exceeded the ISV to an approximate depth of 2.5 m, which corresponds to the depth of buried debris. Groundwater samples collected within this source area exceeded the

MCL for uranium. A filtered groundwater sample from the area of former Building 708 indicated a uranium concentration of 1,200 micrograms per liter (µg/L).

Investigation of the drainage ditch adjacent to and down-gradient from OU1 indicated no impact to groundwater above MCLs. The maximum U_{nat} activity in filtered groundwater samples from beneath the ditch was 10 µg/L.
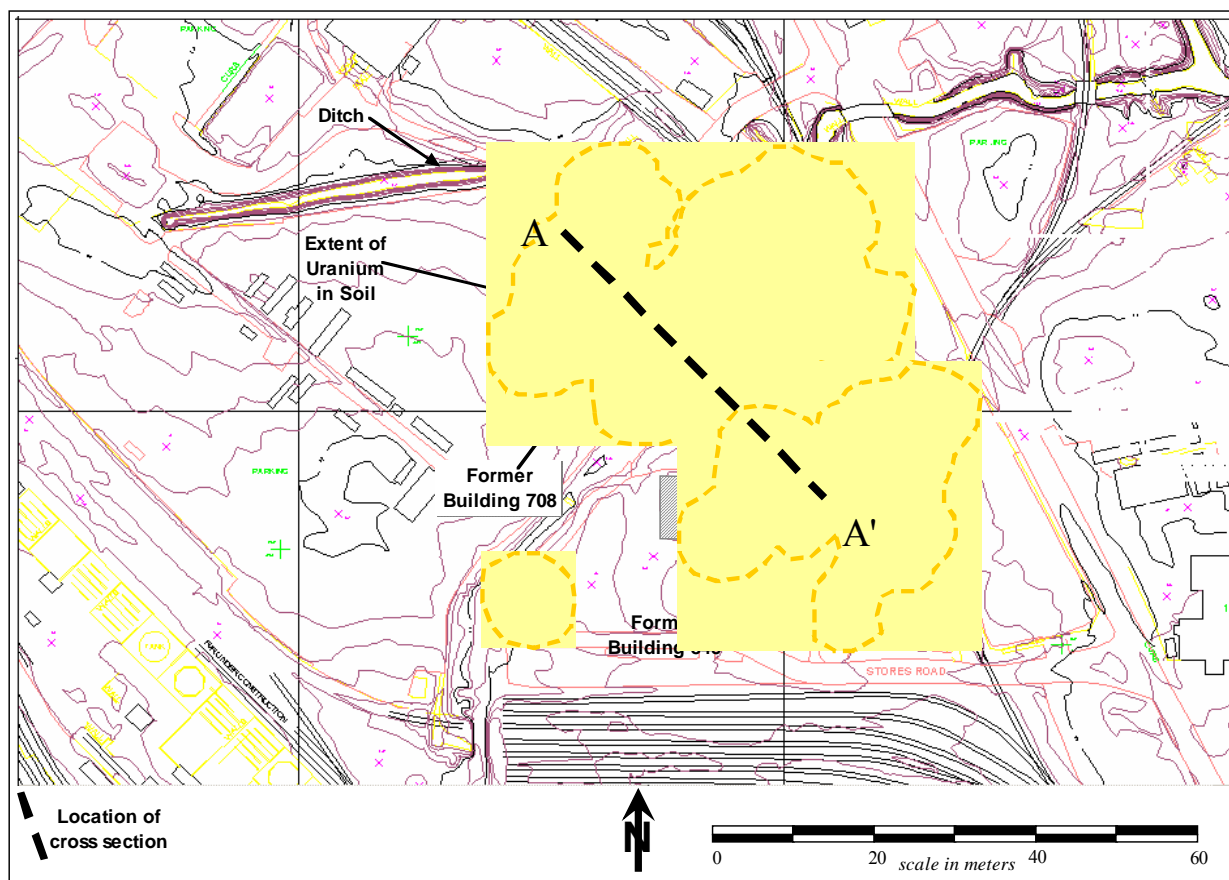

Fig. 1. Operable unit 1

The Chambers Works site lies within the floodplain of the Delaware River. The shallow soils are of alluvial origin but have been substantially modified by landfilling and construction activities. The land along the shoreline either has been accreted as point-bar deposits from the Delaware River or, less likely, consists of over-bank deposits resulting from periodic flooding, which have created a natural levee. Behind these shoreline deposits of sands and silty sands, there once existed a tidal marsh consisting of silty clays, with an elevation near sea level. Over time, the Chambers Works site was gradually enlarged by adding fill material to the marsh areas. Generally, at a distance of more than 200 m inland from the river's edge, the soils existing at sea-level elevation and below are the naturally occurring marsh deposits, while the sediments occurring above sea level are fill material [5].

The Delaware River is tidal at Deepwater and brackish for much of the year; therefore, it is not used as a potable water source. OU1 lies approximately 300 m inland from the bank of the Delaware River. A drainage ditch that traverses the eastern edge of the site was used for

drainage of both runoff and wastewater during the period of MED production. Water elevation in the ditch is approximately at sea level. During August 2005, the average water depth was approximately 30 cm. The water flow is perennial, and flow direction is eastward toward a catchment basin.

The sedimentary deposits beneath the Chambers Works are divided into five major sequences [6]. The uppermost major sequence consists of the A and B aquifers, and the middle ("AB") and lower ("BC") aquitards, as shown in Fig. 2. These units are Holocene and Pleistocene-age sediments consisting of fill, marsh sediments, and Delaware River deposits. The deposits are hydraulically connected to the Delaware River and comprise a significant source of recharge to the underlying Potomac-Group aquifer. These deposits are not widely developed as a groundwater source, although yields of up to 8,200 m^3/day have been reported [7]. The A Aquifer consists of fill material composed of silty sands and rubble. The AB aquitard consists of organic silt, clay, and peat. The AB aquitard is discontinuous and thins to its absence in places [6]. The B aquifer is a fining-upward sand-and-gravel unit and is interpreted as Delaware River alluvium. The unit has an average thickness of 6 m.
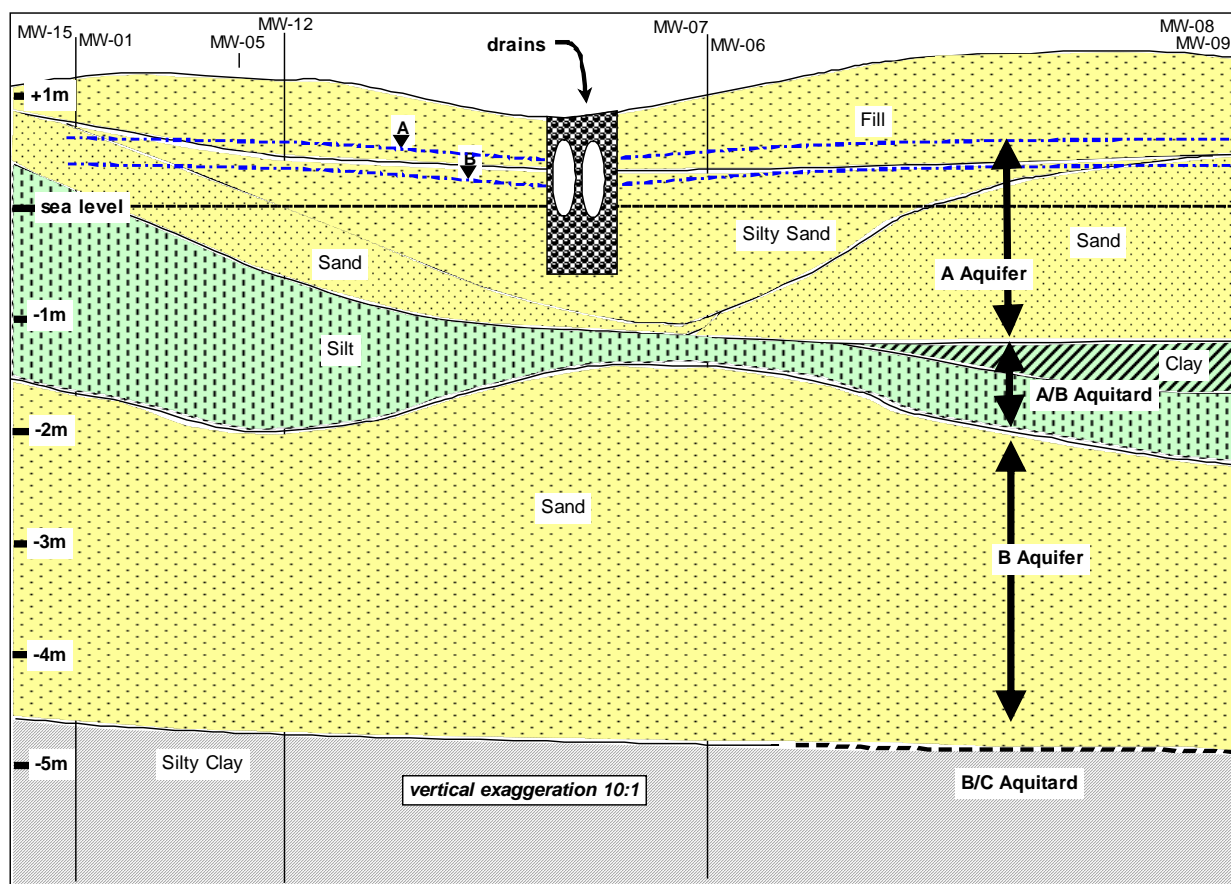

Fig. 2. Geologic cross section

At OU1, groundwater in the B aquifer has a northeastward flow direction, toward a well-point extraction system. Across Chambers Works, the average hydraulic conductivity in the A aquifer is approximately 7E-6 m/s, and in the B aquifer is approximately 1E-4 m/s [8]. Below the B

Aquifer are four separate aquifers that comprise the Cretaceous Age Potomac Group. Chambers Works has delineated source zones and aqueous-phase plumes of chlorinated solvents in the B Aquifer in the vicinity of OU1. Other groundwater contaminants in the B Aquifer include organic lead and arsenic [5].

URANIUM CHEMISTRY

Uranium is found in six oxidation states ranging from U(1+) to U(6+), with tetravalent uranium [U(4+)] and hexavalent uranium [U(6+)] being the most common oxidation states of uranium in nature. The tetravalent form ordinarily occurs in reducing environments, while the hexavalent form is prevalent in oxidizing environments [9]. One researcher determined that the ratio of U(4+) to U_{nat} ranges from 3 to 7% in oxidizing ground waters and from 60 to 90% in anoxic conditions [10].

Uranium occurs in many different compounds, the most common of which include uranium oxides. The uranium oxide compounds pitchblende (U_3O_8) and uraninite (UO_2) are the most common ore minerals of uranium [11]. These uranium compounds have been detected at OU1; however, Chambers Works did not process raw uranium ores. Most likely, these compounds were deposited at OU1 as intermediate byproducts of the uranium refinement –process to produce compounds called "brown oxide" (UO_2) and "black oxide" (U_3O_8). The more mobile U(6+) compounds metastudtite and uranophane (a calcium-uranyl silicate) were encountered in the "Yellow Oxide Area," which is at the former loading dock of Building 845 [4].

Metastudtite [$UO_4 \cdot 2(H_2O)$ or $(U(O_2)\vert O \vert (OH)_2) \cdot 3H_2O$] has been identified at other sites as an alteration product in casks of spent nuclear fuel [12]. Metastudtite and the associated mineral studtite are the only peroxide-containing minerals. Documented occurrences of metastudtite in the environment are uncommon; it was first identified as a naturally occurring mineral in 1983 [13]. It is thought to be formed naturally on the surface of uranyl oxides by the alpha-radiolysis of water [12], and it forms readily in solutions in the presence of hydrogen peroxide and dissolved uranium [14]. Metastudtite is thermodynamically unstable in the absence of dissolved hydrogen peroxide [14].

Migration

There are several environmental parameters affecting uranium migration. The most important of these parameters include redox state, pH, ligand concentrations (carbonate, fluoride, sulfate, phosphate, and dissolved carbon), aluminum- and iron-oxide mineral concentrations, and uranium concentrations [9]. The propensity of uranium to form complexes and sorb onto colloids enhances uranium mobility in groundwater [15].

Sorption

Naturally occurring organic matter can serve as a possible sink for U(6+) in soils and sediments, and the sorption process may not be significantly reversible. This process may occur by the formation of stable complexes or by the reduction of U(6+) ions to more immobile U(4+) ions [9]. Radionuclides can preferentially sorb onto clay-sized particles [16]. However, uranium

adsorption onto clay minerals is complicated and involves many factors. Sorption can also be correlated with pH values [17, 9]. With all other factors held constant, the maximum sorption to aquifer solids occurs within the pH range of 5 to 8, with uranium becoming more mobile above and below this neutral range [9].

In low ionic strength solutions with low U(6+) concentrations, dissolved uranyl concentrations will likely be controlled by cation exchange and adsorption processes [9]. The uranyl ion and its complexes adsorb onto clays, organics, and oxides. As the ionic strength of an oxidized solution increases, other ions (notably Ca(2+), Mg(2+), and K+) can displace the uranyl ion from soil exchange sites, forcing it into solution [14]. For this reason, the uranyl ion is particularly mobile in high ionic strength solutions. Not only will other cations dominate over the uranyl ion in competition for exchange sites, but carbonate and sulfate ions can form soluble complexes with the uranyl ion, further lowering the activity of these ions while increasing the total amount of mobile uranium.

Dissolution

Some of the secondary phases of uranium may form when sufficient uranium is leached from uranium oxides. In the presence of lignite and other sedimentary carbonaceous substances, uranium enrichment is believed to be the result of uranium reduction to form insoluble precipitates, such as uraninite. USEPA reports that $UO_2(2+)$-phosphate complexes can be important in aqueous systems with a pH between 6 and 9 where the total concentration ratio of PO_4(total) / CO_3(total) is greater than 0.1 [9].

Uranyl acetates, carbonates, and sulfates are among the most soluble. Uranyl nitrates are moderately soluble, while uranyl oxides, hydrides, and carbides are relatively insoluble. Complexes with sulfate, fluoride, and chloride are potentially important uranyl species where concentrations of these anions are high [9]. In sulfate-rich oxidizing environments with low pH, uranyl sulfides are soluble [18]. The Piper diagrams show that the groundwater at OU1 is relatively depleted in bicarbonate ion and that the dominant cation is sulfate.

Colloid Formation

Colloids are fine-grained particles ranging in size from 1 micron to 1 nanometer (i.e., clay-size and smaller), which can be easily suspended [19]. Colloids in natural systems include a range of particle sizes, but they tend to be larger than 0.45 microns. Colloids behave in many respects like solutions, or even dense phase-separate fluids. For instance, they can exhibit density-driven flow. Colloids originate from: (1) the dispersion of clays or iron-oxide coatings on mineral grains, (2) decementation of secondary mineral phases, and (3) homogeneous precipitation of groundwater constituents [9]. Colloidal particles can become mobile in aquifers as a result of changes in the groundwater chemistry, such as a decrease in ionic strength or changes in ionic composition from a calcium- to sodium-dominated chemistry [15]. Colloids can be mobilized by increases in groundwater flow rate. Increases in solution pH have been demonstrated to induce the formation of uranium oxide colloids [9].

FIELD INVESTIGATION

The field investigation was designed to identify the full extent of uranium in groundwater and to discern those factors that may allow for uranium migration. Field work consisted of a combination of on-site measurements of hydrogeologic properties and off-site laboratory analysis of soil and groundwater samples.

Well Installation and Soil Sampling

A total of 23 wells were installed over the course of two field seasons. Wells were installed using both conventional hollow-stem augers and by rotasonic drilling. Wells installed to investigate the A Aquifer were completed to total depths of 2.5 m below ground surface, while those accessing the B Aquifer were completed to total depths of 6 m below ground surface.

During drilling, soil textures were logged, and the cores were scanned using a jig-mounted Bicron Model G5 Field Instrument for Detection of Low- Energy Radiation (FIDLER) coupled with a Ludlum Model 2221 rate meter. (A FIDLER was used rather than a Geiger-Mueller detector because of its greater sensitivity to the low-energy gamma radiation.) In addition, soil samples were collected for analysis by gamma spectroscopy, evaluation of soil partitioning coefficients, and sequential extraction testing to assess uranium mobility.

Groundwater Sampling

Groundwater samples were collected from all newly installed wells for off-site laboratory analysis. Water-quality indicator parameters (e.g., pH, turbidity, dissolved oxygen, oxidation reduction potential, and temperature) were monitored and recorded during micropurge sampling using an in-line flow-through cell [20]. Dedicated sampling equipment, constructed of inert materials, was used whenever possible.

Samples were collected using low-flow sampling protocols [20]. Purging flow rates were less than 200 mL/min in the B Aquifer wells and less than 100 mL/min in the A Aquifer wells. Field-filtered and unfiltered duplicates were collected using dedicated in-line high-volume 0.45 micron filters. The use of 0.45-micron filters may overestimate the dissolved load and underestimate the mobile load since some colloids are smaller than 0.45 microns [17], but comparison of filtered to unfiltered results can indicate whether ROPCs are present on mobile colloids. The reactive ions ferrous iron, sulfide, and nitrite were analyzed onsite in unfiltered groundwater samples using colorimetric-reaction methods. Unfiltered samples were also analyzed for chloride, fluoride, nitrate, orthophosphate sulfate, and alkalinity concentrations by an offsite laboratory.

Aquifer Tests

Aquifer testing was initiated after the wells were developed. Pneumatic slug tests were used because these tests generate less waste than standard slug tests and result in higher quality data [21]. Field procedures for the pneumatic slug test are comparable to standard slug test methods [22].

Water level changes were monitored at 15-minute intervals in a number of A Aquifer and B Aquifer wells and in the drainage ditch using pressure transducers and a data logger. Weather conditions were monitored with an onsite weather station, while tidal-stage information for the Delaware River was collected from published sources.

INVESTIGATION RESULTS

Soil Radiochemistry Results

Uranium concentrations in soil agreed with the results of earlier studies. U_{nat} results ranged from less than 300 Bq/kg to 287,000 Bq/kg. Approximately half the U_{nat} results were below their respective minimum detectable concentrations. U_{nat} results higher than 4,000 Bq/kg were encountered beneath the former locations of both Building 845 and Building 708.

Groundwater Analysis Results

Thirteen of the 18 wells could be stabilized to final turbidities of less than 20 nephelometric turbidity units. Values for pH ranged from 6 to 9, except at wells MW-08 and MW-11, which indicated pH values of 13.7 and 11.2, respectively. Oxidation reduction potentials indicated reducing conditions at all wells except MW-03 (B Aquifer), MW-08 (A Aquifer), and MW-10 (A Aquifer).   Light non-aqueous phase liquid was encountered in B Aquifer wells at the same locations where dissolved uranium was encountered (wells MW-01B, MW-03B, and MW-05B).

U_{nat} concentrations in groundwater ranged from 0.6 to 29,552 µg/L. In the A Aquifer, U_{nat} results exceeded the MCL of 30 µg/L at the former locations of Buildings 845 and 708, and also in one up-gradient well (MW-18), as shown in Fig. 3. In Aquifer B, U_{nat} in groundwater exceeded the MCL only at the former location of Building 708, where concentrations were 45 µg/L and 22,388 µg/L in wells MW-05 and MW-03, respectively (see Fig. 4.). Comparison between filtered and unfiltered sample splits showed high correlation of the results; U_{nat} results had a correlation coefficient of 0.998.

Major Ions in Groundwater

Wells in the B Aquifer have elevated chloride concentrations compared to wells in the A Aquifer. Average chloride concentrations in B Aquifer were approximately 200 mg/L, while average chloride concentrations in A Aquifer were approximately 20 mg/L. Sulfate concentrations varied widely but averaged approximately 160 mg/L. Total alkalinity (as $CaCO_3$) averaged 350 mg/L. Comparison of dissolved uranium concentrations with either major ion concentrations or well-stabilization parameters showed no correlations. The average balance between cations and anions at each well location was within 10%. The diagram shows that sodium and potassium dominate among cations, and chloride and sulfate dominate among anions. Generally, a sodium-potassium-sulfate facies dominates.
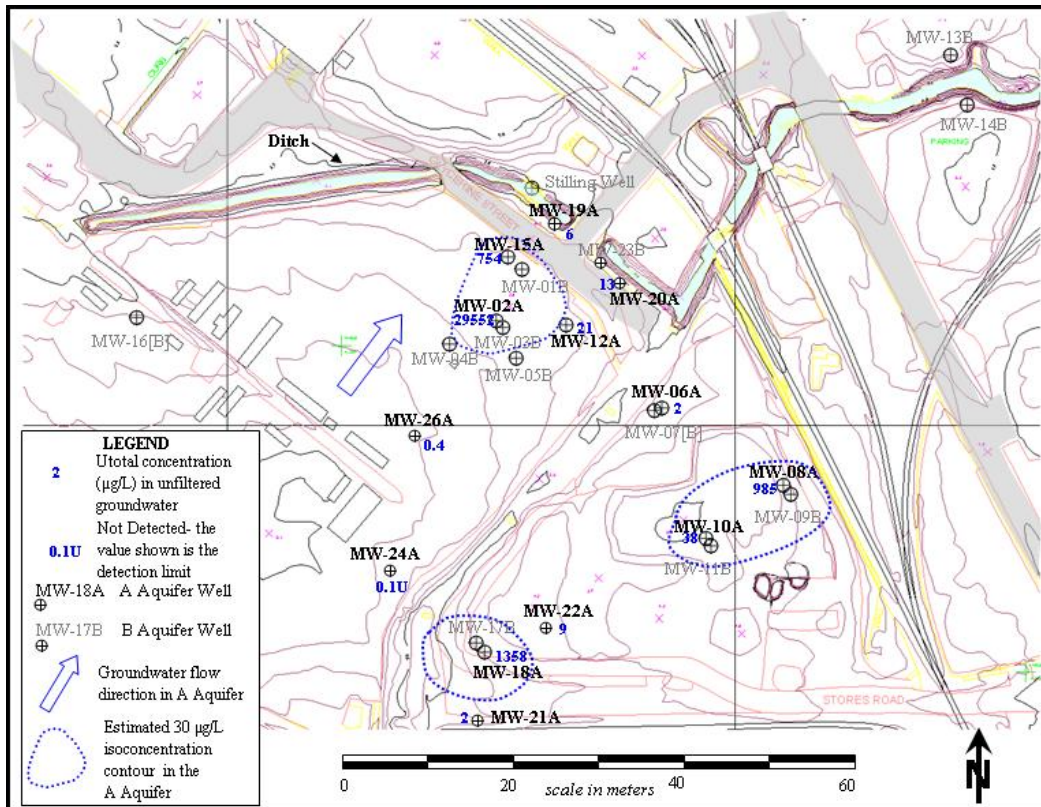
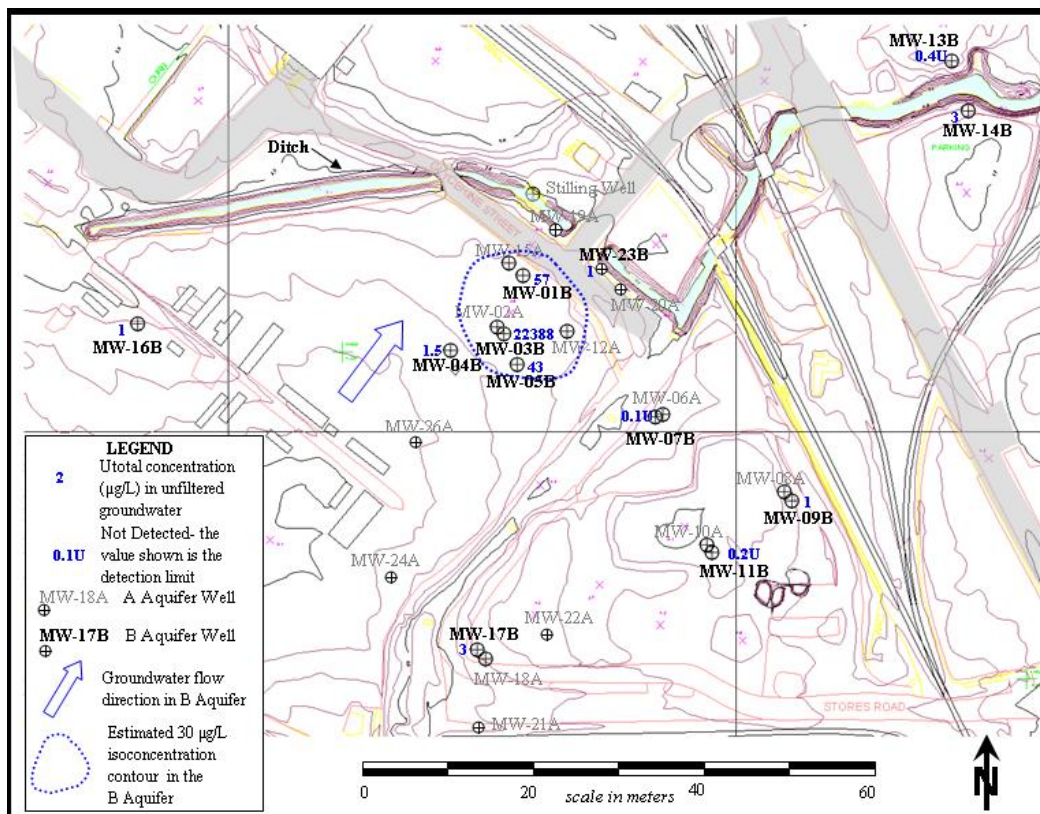Fig. 3. Aqueous-phase uranium concentrations in the A aquifer

Fig. 4. Aqueous-phase uranium concentrations in the B aquifer

Groundwater Flow

On average, the piezometric surface in the A Aquifer is one foot higher in elevation than in B Aquifer B throughout OU1. The water elevations in both aquifers are higher than the water elevation in the drainage ditch. Long-term monitoring of water levels in the A Aquifer, B Aquifer, and CDD wells shows that B Aquifer wells are affected by tidal fluctuations but A Aquifer wells are not.

Groundwater flow direction in the A Aquifer appears to be toward the drainage ditch, but in the B Aquifer, the flow direction is toward a recovery well system in the northeast. The groundwater flow gradient in the A Aquifer appears to be largely dependent on the distance from the ditch, although in the former location of Building 708, the gradient is 1%. The groundwater flow gradient in the B Aquifer is much lower than in the A Aquifer. For instance, the gradient in the B Aquifer at this same location is 0.2%.

Pneumatic slug tests were performed in 13 of the wells. Based on slug test results, the average hydraulic conductivity was calculated to be 9E-6 m/s in the A Aquifer and 5E-6 m/s in the B Aquifer. These values fall within the range expected for silty fine sands [19, 23]. For the A Aquifer, the average seepage velocity was estimated to be approximately 250 meters per year. For the B Aquifer, the average seepage velocity was estimated to be approximately 1 meter per year.

CONCLUSIONS

Several lines of evidence indicate that the original form of uranium released in the source zones was uranium peroxide dihydrate, and that while the uranium solubility is relatively high, the instability of the mineral form causes the uranium to be immobile in groundwater.

Uranium peroxide dihydrate was produced in larger volumes onsite than other uranium compounds. The area of highest aqueous-phase uranium concentrations is at the former location a building which accepted uranium peroxide dihydrate and further processed it into brown oxide. Aqueous-phase uranium appears to be present in the A Aquifer in locations where uranium-impacted soils are present, but has not migrated to the open ditch at concentrations above MCLs.

Aqueous-phase uranium is present in the B Aquifer in a single location beneath the former location of Building 708. Uranium has been detected in a well directly downgradient from the uranium occurrence in the B Aquifer, although this detection is below the MCL. There is no evidence that uranium has been mobilized and transported any significant lateral distance within the B Aquifer.

Relatively high concentrations of aqueous-phase uranium are likely due to the greater solubility of peroxide compounds compared to oxide compounds. The source zones display higher pH, higher dissolved oxygen concentrations, and higher oxidation-reduction potentials, which are characteristic of oxidizing microenvironments. These conditions are incongruous with expectations, since there are free-phase hydrocarbons present, yet consistent with the presence of peroxides. The lack of plume migration indicates either the absence of groundwater flux, strong sorption of uranium to soil, and/or reactivity of the uranium species. Since there is very little difference between uranium concentrations in filtered and unfiltered groundwater samples, uranium mobility due to colloid formation appears unlikely.

In the B Aquifer, the source zone is an oxidizing microenvironment, which is unusual considering the presence of free-phase liquid hydrocarbons in close proximity (at B Aquifer wells MW-01, MW-03, and MW-05). Redox conditions are normally reducing and anoxic in the presence of liquid hydrocarbons. It is possible that oxygen in the zone has been replenished by the alpha-hydrolysis of water caused by the radioactive decay of uranium peroxide. Uranium peroxide dihydrate is more soluble in water than other hexavalent mineral forms, with a comparatively low solubility product. However, in the absence of dissolved hydrogen peroxide, metastudtite is unstable [14]. It therefore appears that although metastudite has created locally high levels of aqueous-phase uranium, the uranium ions do not persist in a mobile form because of local geochemistry.

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