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A Sustainable Choice for Water Treatment/Recycling When Injection is Not an Option or Water Supply is Limited

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Abstract

Limited disposal options and/or water scarcity have led the oil and gas industry in some shale plays to establish goals for minimizing the use of freshwater for drilling and completion activities by maximizing the recycle of wastewaters generated from exploration and production (E&P) activities. Many operators have established a goal of 100 percent recycle for their development activities. Eureka Resources, LLC (Eureka) has developed a business model which gives oil and gas operators a sustainable choice for recycling oil and gas wastewaters when water is scarce or disposal options are limited. The business model is based on operation of centralized facilities that targets recovery of useable by-products. There are many challenges associated with operating a centralized oil and gas wastewater treatment system that includes pretreatment, crystallization, biological treatment, ion exchange and reverse osmosis unit processes. This paper summarizes the challenges and lessons learned during startup and shakedown of a thermal treatment (crystallization) based model. This paper also summarizes the results of sampling and characterization efforts focused on:

- Demonstrating that the treated water from a centralized oil and gas wastewater treatment facility can meet freshwater standards
- Demonstrating that the salt quality generated by the crystallizer is equivalent to manufactured rock salt used for road deicing and other industrial applications
- Establishing characteristics and market value for the concentrated high calcium chloride brine purge generated by the crystallizer

Introduction

One of the most prominent of the Shale Basins in the US is the Marcellus Shale Basin – underlying large swaths of New York, Pennsylvania, Ohio, West Virginia, and Maryland. Due to scarcity of water resources as well as regulatory constraints and expensive treatment technologies, E&P companies in all shale basins must develop and implement innovative methods for re-use and adopt more localized and centralized water management systems. E&P companies in various shale plays across the US are now consistently seeking out sustainable methods to transport, treat, dispose, and reuse development and produced waters to maximize the profitability of shale gas production and remain competitive in the marketplace.

Eureka has been operating centralized oil and gas wastewater treatment facilities in Pennsylvania for over 7 years and has optimized a thermal treatment based model for sustainable treatment and recycle of oil and gas wastewaters. The model involves permitting, designing, constructing and operating comprehensive centralized wastewater treatment facilities utilizing state-of-the-art technologies to achieve the following goals and objectives:

- Continuous improvement in the management/treatment approach for oil and gas wastewaters. Where logistics are favorable, centralized treatment can be cost competitive with UIC disposal.
- Locating centralized treatment facilities in close proximity to major fairways of development activities to minimize hauling and associated costs.
- Providing customers with options for level of treatment with the goal of maximizing recycle of:
    - Pretreated Wastewater - water treated to reduce the suspended solids
    - Distilled Wastewater - water treated to remove dissolved solids
    - Clean Concentrated Brine - heavy (over 10.5 pounds per gallon) water that is very high in dissolved solids
    - De-Wasted Water - water that meets freshwater standards
- Providing a discharge option that returns as much water as possible to the hydrologic cycle.
- Maximizing recovery and beneficial reuse of recoverable by by-products (co-products) such as methanol, sodium chloride (salt) and calcium chloride.
- Reduction of the long-term costs associated with treatment and recycling of oil and gas wastewaters.

HISTORY OF EUREKA

Eureka Resources, LLC (Eureka) currently provides centralized treatment, recycling and/or disposal of flowback/produced waters (wastewaters), as well as waste drilling fluids generated as a result of nonconventional natural gas exploration and development activities in the Marcellus play. Eureka placed its first centralized pretreatment facility in operation in November 2008 on Second Street in Williamsport, PA with a capacity of approximately 4,800 barrels per day (bpd) or 200,000 gallons per day (gpd). The facility was originally permitted by the PADEP Bureau of Waste Management under general permit WMGR119. In 2010, Eureka opened an expanded centralized treatment facility which is currently capable of treating/recycling up to 10,000 bpd (420,000 gallons) of flowback/produced wastewater daily. The facility permit was converted to a WMGR123 general permit at this same time, as authorized by the PADEP Bureau of Waste Management.

In 2012, Eureka obtained WMGR123 permits for two additional facilities – one in Standing Stone Township, Bradford County, PA and one off Reach Road in Williamsport, PA (See Figure 1). All three of the Eureka facilities are permitted as essentially mirror facilities with 10,000 bpd capacities. All of the Eureka facilities are permitted to allow installation of some or all of several unit processes. The Second Street Williamsport Facility has pretreatment, oil recovery, mechanical vapor recompression distillers and methanol rectification. The Standing Stone facility has pretreatment, first phase crystallization (generating commercial/industrial grade dry sodium chloride and liquid calcium chloride brine) and membrane biological reactor/ion exchange/reverses osmosis treatment capability.

Drivers for Technology Selections

Regulatory requirements are a very important driver for determining treatment requirements for oil and gas wastewaters. Regulatory requirements can vary significantly between states. Eureka has developed and maintained a strong working relationship with the Pennsylvania Department of Environmental Protection (PADEP) in order to adjust our business model as necessary to accommodate the ongoing evolution of regulatory requirements. Prior to 2012, PADEP rules and regulations for disposing of flowback/produced water that is sent off-site for reuse was regulated as a residual waste, requiring permitting under one of three different General Permits applicable to oil and gas operations, as enforced by the PADEP Bureau of Waste Management: WMGR119, WMGR121, and WMGR123.

On March 24, 2012 the PADEP revoked WMGR119 and 121 and revised/reissued General Permit WMGR123**,** which authorizes the processing and beneficial use of processed liquid wastes generated on oil and gas well sites and associated infrastructure. WMGR123, issued under the authority of the PADEP Bureau of Waste Management, replaced the three existing general permits which previously regulated the recycling and reuse of oil and gas wastewaters.

The new general permit removed some current restrictions on the recycling of oil and gas wastewaters, and added some new requirements. For facilities that plan to recycle and reuse relatively dilute waters, the new permit should be helpful. In particular, for wastewaters with low total dissolved solids (TDS) (i.e., less than 500 mg/l) that are in compliance with standards found in Appendix A of the permit (see Table 1), the wastewater will essentially be considered de-wasted and E&P companies will not have to manage the waste as a residual waste, and should be able to utilize existing fresh water designs for impoundments and handling of the water.

However, for high TDS wastewaters which do not comply with the Appendix A standards, both the generators and users of the recycled water have new compliance standards. Until the processed oil and gas liquid wastewater has been transported to a well site and is actually used to develop a well, it must be managed as a residual waste. The requirements to manage the wastewater as a residual waste apply to both the operator generating the waste and the operator reusing the waste. If either the generator of the wastewater, or the party beneficially reusing the wastewater, wishes to store the waste prior to either shipment or reuse, they will need to comply with storage requirements that are generally more stringent than the requirements under the oil and gas regulations.

In addition to regulatory drivers, Eureka established business drivers (objectives) including: maximizing recovery of valuable by-products, applying a level of treatment that allows return of water to the hydrologic cycle, minimizing risks associated with transport/storage of wastewaters by E&Ps and providing a sustainable choice for oil and gas wastewater treatment when injection is not an option or water supply is limited. Selection of the various technologies and technology suppliers employed by Eureka required consideration of our business objectives as well as a variety of factors including Marcellus-specific wastewater characteristics, make up or recycle water requirements and desired by-product characteristics. For example, even when the decision was made to apply thermal technologies to achieve Eureka's business objectives, selection of the appropriate type of thermal technology was a critical step. Eureka considered several thermal technologies for by-product generation before selecting MVR crystallization based on performance drivers including higher energy efficiency, ability to handle a wider range of feed qualities, ability to capture condensate, and the potential for expansion in a sequential/modular fashion to achieve recovery of multiple by-products.

Eureka's centralized treatment business model is illustrated on Figure 2. The unit processes employed by Eureka are summarized below:

- **Pretreatment:**
  - Receiving Water Tanks
  - Primary Settling Clarifiers/Oil Skimmers
  - Methanol Rectification
  - Oil Separation/Recovery Facilities
  - Indoor Raw Water Storage Tanks
- **Chemical Precipitation:**
  - pH Adjustment
  - Chemical Addition (sodium hydroxide, sodium sulfate, soda ash)
  - Secondary Clarifiers
- **Sludge Dewatering:**
  - Sludge Holding
  - Sludge Dewatering (plate and frame, centrifuge)

- **Secondary Treatment - Distillation:**

  - Pretreated Water Holding Tanks
  - NOMAD Units
  - Concentrated Brine Holding Tanks
  - Distillate Water Storage Tanks

- **Secondary Treatment - Crystallization**

  - Pretreated Water Holding Tanks
  - Crystallizer
  - Concentrated Brine Holding Tanks
  - Distilled water Storage Tanks

- **Tertiary Treatment [Patent Pending]:**

  - Membrane Biological Reactor (MBR) System
    - Anoxic Tank
    - Aerobic Tanks
    - Membrane Tanks
  - Reverse Osmosis (RO) System
    - RO Feed Tank
    - RO skid
    - Effluent Storage Tank

The actual unit processes installed at each facility depend on customer needs. Eureka recently modified the WMGR123 and air permits for the Second Street facility to include methanol rectification (removal and recovery of methanol for beneficial reuse) and is in the process of modifying the Standing Stone facility WMGR123 and air permits to include methanol rectification. Eureka also obtained a WMGR029 permit for the Second Street facility which allows management of waste oil. Eureka is also in the process of planning additional centralized treatment facilities in Pennsylvania, as well as possible additional centralized treatment facilities in Ohio and West Virginia. Eureka is also involved in discussions with O&G Producers about dedicated centralized or satellite treatment and/or storage facilities.

Crystallization Technology - Lessons Learned

Eureka has gained valuable experience during the selection, design and operation of a crystallizer based oil and gas wastewater treatment facility in the Marcellus Play in Pennsylvania. Many lessons have been learned which can be applied to future applications of crystallizer technologies to oil and gas wastewater treatment:

- **Mechanical Vapor Recompressions (MVR) Crystallizer Technology Vendor Selection:** Application of crystallizer technology for treatment of oil and gas wastewaters is extremely limited. Much of the existing design and operating experience for MVR crystallizers is based on solution mining brine feeds which are nearly saturated with sodium chloride and are lower in variability with regard to other constituents that can complicate crystallizer design and operation (e.g., other chloride salts). Also, much of the non-desalination crystallizer experience is outside the United States (US). There are several factors which must be considered when selecting a crystallizer vendor:
  - **Domestic vs. Foreign Vendors -** Selection of a foreign vendor can complicate various elements along the lifeline of the project, including general travel and communication (time zone) issues, scheduling and shipment of components, potential management of numerous foreign sub-vendors, challenges associated with procurement of replacement/spare parts during operation to minimize downtime, responsiveness of technical support and operator training, obtaining clear and usable design deliverable and O&M documentation, currency conversions, and units of measure challenges.

- **Blowers versus Turbo-compressors –** Capital costs, energy use, safety and operating costs should be considered. The need for a bypass line to assure proper operation should also be considered. Visits to similarly sized operating facilities to verify performance are recommended.
- **Turn Down –** The design capacity and ability of the crystallizer to operate at lower than basis of design (turn down) must be well understood. The turn down capability along with the reliability of raw wastewater delivery rates will dictate the need for and size of storage requirements upstream of the crystallizer.
- **Building Codes/Certifications–**Foreign vendors may not be familiar with local, state and federal building codes and certifications which must be factored into the design of the crystallizer process.
- **Materials of Construction –** The corrosive nature of mixed brine solutions must be considered when selecting the materials of construction for the major equipment, vessels and process piping.
- **Fabrication –** Visits to the various major equipment manufacturers/sub-vendors during fabrication must be performed to verify that fabrication activities are on schedule. Delays in fabrication will delay completion of construction.
- **Boundary Limits -** Attention to boundary limit requirements is important; including confirming that the necessary utilities are available and accessible to support operation of thermal treatment technologies, and awareness of the challenges that may be faced in any given geographic area (e.g., availability of power, uncontrolled power outages, power supply flickers, availability of natural gas, etc.).

- **Safety:** Oil and gas wastewater treatment facilities typically include multiple technologies and require well trained operators. Inclusion of a crystallization process significantly increases the level of mechanization and incorporates both high temperature and pressure vessels. Safety considerations must be factored into the design of the crystallizer and operators must be educated and trained on safe operating protocols.
- **By-Product Quality Control:** Design and operation of a crystallizer requires a thorough understanding of thermodynamics. Much of the design and operating history for crystallizers is based on sodium chloride brine feeds with consistent characteristics. The characteristics of the mixed brine oil and gas wastewaters can be extremely variable with elevated concentrations of constituents that can complicate treatment, including various alkali earth metal chloride salts (sodium, barium, strontium, calcium, and magnesium chlorides), radionuclides, organics, etc. It is important to understand the range of variability associated with the oil and gas wastewater which will be received at the facility so that the required pretreatment system and residuals management strategies can be selected and designed to generate optimum crystallizer feed quality necessary to achieve and maintain optimum by-product quality. For example, the barium content of the wastewater must be maintained below a certain level relative to the sodium and calcium concentrations so as to achieve optimum crystallizer performance and protect salt quality, purge quantity, and purge quality. Also, the crystallizer design and performance criteria will be based on the assumed sodium chloride content of the crystallizer feed. Variations in the sodium chloride concentration of the raw feed relative to other chloride salts present, and/or modification of wastewater chemistry prior to being fed to the crystallizer system (e.g., recycle related dilution, pH change, etc.) can drastically impact crystallizer operation, salt by-product generation, sodium chloride capture efficiency, and purge quality.
- **Condensate Management:** Management options for the condensate (sometimes referred to as distillate) produced by a crystallizer must be factored into the design of a centralized oil and gas wastewater treatment facility. Options include recycle and/or direct or indirect discharge. All options require consideration of onsite storage requirements. Direct or indirect discharge may require additional treatment following crystallization due to the presence of elevated levels of inorganics (e.g. barium and strontium), volatile organics, ammonia nitrogen and other constituents that can become present in the distillate generated by the system. Application of additional downstream treatment technologies may be needed to reliably achieve discharge limits and/or reuse standards. The high temperature of the condensate may also be a challenge. For example, if biological treatment technologies are required

downstream of the crystallizer, condensate discharge may require cooling prior to biological treatment to protect biomass health.

- **By-Product Market Development**: In order to develop a reliable market for by-products, an understanding of the market for the by-products is required. First, there is the need to demonstrate product quality equivalency which necessarily requires extensive by-product testing. There will also be a need for targeting and engaging potential customers ahead of time, obtaining necessary regulatory approvals for sale of the by-products, and implementing necessary QA/QC procedures and plans.
- **By-Product Management**: In order to develop a reliable market for by-products, investment in equipment to obtain/assure by-product quality may be necessary. Such equipment may include conveyance, processing, packaging, and storage facilities.

De-Wasting Determination

Eureka has a patent pending process for generating "De-Wasted" water from oil and gas wastewater which allows for the storage and reuse of recycled water as fresh water as well as direct discharge under an NPDES permit. The sampling and characterization plan for the demonstration process was developed based on the PADEP's WMGR123 Appendix A requirements. The initial sample collection (MBR/IX/RO effluent) effort for the demonstration occurred from July 21 through September 19, 2014. The effort included: daily flow-proportional composite samples, weekly flow-proportional composite samples and grab samples. The analyte list is summarized in Table 1. Analytical results confirmed that concentrations of all parameters as required by Part C.22.b of the WMGR123 general permit were lower than, equal to, or otherwise not detected when compared to the de-wasting limits included in Appendix A for all samples. The De-Wasting request letter was submitted to PADEP on 10/3/14 and PADEP approval was received 11/24/14.

In addition to the De-Wasting demonstration, Eureka has also worked with the Center for Sustainable Shale Development (CSSD) to characterize the Standing Stone facility effluent in an effort to support CSSD's development of a performance standard for discharging treated wastewater to surface water. The CSSD is an independent 501(c) (3) nonprofit organization whose mission is to support continuous improvement and innovative practices through performance standards and third-party certification. The sampling effort focused on characterizing MBR/IX/RO effluent samples prior to the potential discharge of the effluent through Eureka's NPDES permit. Five sampling events were completed over a three to four week period. Table 2 summarizes the comprehensive CSSD parameter list. Wastewater effluent toxicity (WET) testing was also performed. The testing results demonstrated that the Eureka Standing Stone treatment process was performing as intended and achieving a high quality effluent capable of meeting the overall CSSD surface water discharge performance standard objective of not releasing toxic constituents in toxic amounts to surface water.

Co-Product (By-Product) and Beneficial Reuse Efforts

Methanol: Eureka utilizes a methanol rectification column to recover methanol from compressor station wastewaters that typically contain high concentrations of methanol. The recovered methanol by-product contains 97 percent methanol, less than 1 percent ammonia nitrogen, as well as trace levels of BTEX (benzene, toluene ethyl benzene and xylene), MEK (methyl ethyl ketone), MIBK (methyl isobutyl ketone), acetone, zinc, boron, calcium, aluminum and water. Eureka recycles the recovered methanol primarily for reuse in oil and gas field applications including compressor station operations which does not require a Beneficial Reuse Determination. Eureka is also pursuing reuse of the methanol as a supplemental feed for wastewater treatment plants which will require a Co-Product or Beneficial Reuse Determination.

Salt: Eureka has completed a Co-Product Determination for the salt (sodium chloride) generated by the crystallizer at the Standing Stone Facility in accordance with 25 PA Code §287.1 and §287.8. The Co-Product Determination included characterization of the following:

- Eureka's crystallized salt
- Bulk stockpiled road salt samples collected from various storage stockpiles maintained by Penn DOT

suppliers for dry application as a road deicing agent
- Bulk stockpiled “solar salt” samples collected from various storage stockpiles maintained by Penn DOT suppliers for wet application as a “pre-wetting” road deicing agent
- Samples of bagged sodium chloride deicing rock salt collected from three different retail outlets within Pennsylvania
- Collection of sodium chloride material from a commercial/industrial that uses salt as a raw material with operations in Southwestern Pennsylvania

The salt samples were analyzed for: a suggested list of parameters provided by the PADEP Bureau of Waste Management; the list of parameters included in the research paper released by the PADEP and Bureau of Water Standards and Facility Registration in May 2011 (authored by R. Titler and P. Curry); the list of parameters included in the draft WMGR128 general permit previously released by the PADEP for the beneficial use of crystallized sodium chloride and liquid calcium chloride from the processing of oil and gas liquid waste; selected hazardous waste parameter from the list included in 40 CFR Part 261, Appendix VIII; and selected parameters taken from material specifications provided to potential future users of Eureka’s salt by their existing sources and/or suppliers. The results of this characterization effort indicated that Eureka’s salt was chemically equivalent to commercially available deicing salts based on the following:

- The concentration/activity level of all analytical constituents in the co-product salt produced at Eureka’s Standing Stone, PA facility, with the exception of barium, were either lower, or comparable to, concentrations/activity levels in the comparative samples of sodium chloride products collected in 2013.
- The concentration of barium in Eureka’s salt sample (approximately 19 ppm) is well below the level of 100 pm set forth in more restrictive deicing salt quality specifications applied by the Pacific Northwest Snowfighters Association (PNSA).
- Based on comparison to salt/brine quality data published in a reputable textbook in the sodium chloride production industry (Kaufman, 1968), the concentration of barium found in Eureka’s salt sample is comparable to levels naturally found in rock salt. The concentration of barium in Eureka’s salt sample (approximately 19 ppm) is at the lower end of the published range of 10 to 100 ppm.
- The concentration of barium found in Eureka’s salt sample is also comparable to levels published in the research paper released by the PADEP and Bureau of Water Standards and Facility Registration in May 2011 (authored by R. Titler and P. Curry). Eureka’s salt barium level (19.15 ppm) falls within the documented barium concentration range of 13.3 to 25.2 ppm.
- The concentration of barium in Eureka’s salt sample (approximately 19 ppm) is well below the lowest Medium-Specific Concentration (MSC) statewide health standard for barium in residential soils (40,000 ppm; direct contact), and below the lowest MSC for barium in soil-to-groundwater health standards (200 ppm), as published by the PADEP Land Recycling Program in the PA Bulletin in January 2011.
- The concentration of barium in Eureka’s salt sample (approximately 19 ppm) is below published mean barium levels in soils measured in the Eastern US (arithmetic mean = 420 ppm; range = 10 to 1,500 ppm), as published by the USGS in a 1984 study.

In addition to chemical equivalency, Eureka has demonstrated that driers, compactors and crushers can be installed to generate a graded rock salt equivalent in physical composition to commercially available rock salt. Eureka is also in the process of pursuing a Co-Product Determination for use of the crystallized salt as pool and/or water softening salt.

Calcium Chloride Brine: The brine purge by-product generated in the Standing Stone crystallizer has a calcium chloride concentration of 18 to 22 percent along with varying concentrations of barium, sodium, strontium and lithium chlorides. After extensive research and market assessment, the best end use of this mixed brine purge is in the oil and gas industry to formulate drilling and development fluids. Eureka is also evaluating and pilot testing additional treatment of this mixed brine to recover other useable by-products and/or to generate a higher purity/higher concentration calcium chloride by-product.

Conclusions/Takeaways

Actual experience treating development and produced wastewaters generated in Pennsylvania as a result of the Marcellus Shale Gas play has indicated the following:

- Eureka has demonstrated that locating centralized treatment facilities (employing crystallization and a group of supporting technologies that maximize potential for recycle and beneficial reuse) in close proximity to major fairways of development activities to minimize logistics and associated costs is a sustainable business model especially when injection is not an option or water supply is limited.
- Eureka has learned many valuable lessons through actual design, construction and operation of a crystallizer on oil and gas wastewater which can streamline future application of crystallization to treatment of oil and gas wastewater.
- Eureka has over seven years of successful experience treating complex unconventional Marcellus Shale Gas Play flowback and produced waters utilizing a business model that provides:

    - A wide array of treatment options.
    - E&P companies with options for selecting the level of treatment necessary to optimize reuse options and reducing the impact on the hydrologic cycle.
    - Beneficial reuse opportunities for Co-Products (By-Products) extracted from oil and gas wastewaters.
    - A forward-thinking strategic position designed to stay at the forefront of emerging regulatory requirements; promoting a close dialogue with state regulators regarding the development of regulations and standards.

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Figure 1

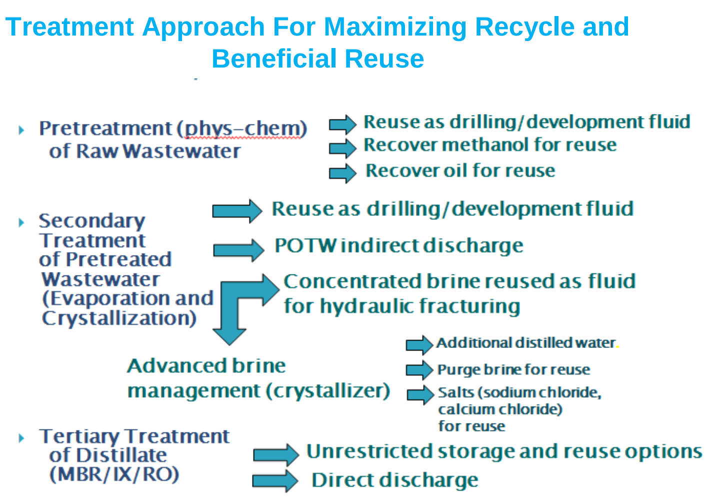

Figure 2

Table 1
PADEP WMGR Appendix A De-Wasting Standards

Constituent	Limit	Constituent	Limit
Aluminum	0.2 mg/L	Manganese	0.2 mg/L
Ammonia	2 mg/L	MBAS (Surfactants)	0.5 mg/L
Arsenic	10 µg/L	Methanol	3.5 mg/L
Barium	2 mg/L	Molybdenum	0.21 mg/L
Benzene	0.12 µg/L	Nickel	30 µg/L
Beryllium	4 µg/L	Nitrite-Nitrate Nitrogen	2 mg/L
Boron	1.6 mg/L	Oil & Grease	ND
Bromide	0.1 mg/L	pH	6.5-8.5 SU
Butoxyethanol	0.7 mg/L	Radium-226 + Radium-228	5 pCi/L(Combined)
Cadmium	0.16 µg/L	Selenium	4.6 µg/L
Chloride	25 mg/L	Silver	1.2 µg/L
COD	15 mg/L	Sodium	25 mg/L
Chromium	10 µg/L	Strontium	4.2 mg/L
Copper	5 µg/L	Sulfate	25 mg/L
Ethylene Glycol	13 µg/L	Toluene	0.33 mg/L
Gross Alpha	15 pCi/L	TDS	500 mg/L
Gross Beta	1,000 pCi/L	TSS	45 mg/L
Iron	0.3 mg/L	Uranium	30 µg/L
Lead	1.3 µg/L	Zinc	65 µg/L

Table 2
CSSD Effluent Characterization Analyte List

Analysis	Method Name
TOC	EPA 415.1
Aldehydes	SW-846 8315
VOCs	SW-846 8260B
SVOCs	SW-846 8270C
Pentanoic and Hexanoic Acids	Targeted Library Search 8270C
Pesticides	SW-846 8081A
PCBs	SW-846 8082
Organic Acids	SW-846 8015B
Alcohols	SW-846 8015B
Glycols	SW-846 8015B
2 Butoxyethanol	SW-846 8270C
TPH C8-C40	SW-846 8015B
30 ICP Metals	SW-846 6010B
Mercury	SW-846 7470A
Hexavalent Chromium	SW-846 7196A
Trivalent Chromium	SW-846 6010B
Sulfate, Chloride, Fluoride, Bromide	EPA 300

Ammonia	EPA 350.2
TDS	SM 2540D
Ra 226 and Ra 228	EPA 903.0 and 904.0