

technical bulletin

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Treatment of Chromium Contamination and Chromium Ore Processing Residue

1. INTRODUCTION

Chromium (Cr) is a heavy metal which has historically been used in a wide range of industrial applications including steel, pigments, wood preservatives, electroplating, metal finishing, dyes, leather tanning, textiles and chemical manufacture.

In general, the treatment of Cr contamination will focus on the highly toxic hexavalent form of Cr (Cr(VI)), and its transformation into the relatively low toxicity trivalent form (Cr(III)), a process that typically involves chemical reduction and precipitation as Cr(III). Over the last ten years there have been a number of laboratory and field-scale studies that have demonstrated the extension of these approaches from treating industrial waste streams to remediating contaminated soil and groundwater, mostly in the USA. Cr(VI) gained notoriety and world-wide attention, thanks to Julia Roberts' film portrayal of *Erin Brockovich* in 2000, which led to increased public awareness of the issues of Cr(VI) contamination in groundwater.

This bulletin provides information on the treatment of Cr contamination in the environment and summarises a number of remedial methods that have been used to treat it. In addition, it looks at the wider issue of chromium ore processing residue (COPR) contamination in the south-east of Glasgow, potential methods for its remediation and the next steps for the redevelopment of this area. The information in this bulletin has largely been taken from a report prepared by Dr Richard Bewley of URS Corporation Ltd (URS) for Environmental Development and Education Network (EDEN) concerning the development of potential remedial options for COPR in Glasgow.

2. BACKGROUND

Cr is a transition metal of Group VIB of the Periodic Table. The three most common forms of Cr are the 0 or elemental state, the trivalent state [Cr(III)] and hexavalent state [Cr(VI)]. Elemental Cr does not occur naturally and is highly resistant to chemical attack. Both the trivalent and hexavalent forms exist in the environment, the latter being largely anthropogenic in origin. In unpolluted soils, the relatively insoluble and less mobile Cr(III) predominates in the form of hydroxides and oxides and adsorbed onto clay particles, soil organic matter, metal oxyhydroxides and other negatively charged surfaces.

Elevated concentrations of Cr(VI) in soil are most likely to be from pollution. Cr(VI) is often found as chromate (CrO_4^{2-}), although it is typically in pH-dependent equilibrium with other forms including dichromate ($\text{Cr}_2\text{O}_7^{2-}$), and it is considered more soluble and more mobile than Cr(III). Although not readily adsorbed to most mineral surfaces, Cr(VI) is adsorbed by clay minerals that possess exposed inorganic hydroxyl groups, such as iron and aluminium oxides (DEFRA, 2002b).

Most trivalent Cr compounds are insoluble, (except for nitrate and chloride) whilst most hexavalent compounds are soluble (apart from zinc and lead chromates) (DEFRA, 2002a).

The overall toxicity of trivalent Cr is relatively low. Indeed, Cr(III) is an essential element in human metabolism and health benefits from supplementation of diets of subjects with Cr deficiencies have been observed.



Fig.1: Example of chromium ore processing residue

Hexavalent Cr on the other hand is of significant toxicity. It is classified by both the International Agency for Research on Cancer and US Environmental Protection Agency (USEPA) as a known human carcinogen and hexavalent Cr compounds are also known to have mutagenic potential. Toxicity arising from Cr(VI) may occur via inhalation, ingestion or dermal exposure. It has an irritating and corrosive effect on skin and mucous membranes resulting in ulceration and dermatitis. It may also result in skin sensitisation, causing skin allergies to occur in affected individuals (DEFRA, 2002a).

On the basis of its toxicity, the occurrence of Cr(VI) as a contaminant in soil, surface water and groundwater may therefore represent an issue of potential concern. Industrial applications of Cr have resulted in specific cases of Cr(VI) contamination associated with the particular facility of concern.

The Contaminated Land Exposure Assessment (CLEA) guidelines set out Soil Guideline Values (SGVs) for the assessment of risks posed to human health by the presence of contamination in soils. Based upon a toxicological review (DEFRA 2002a), guideline values have been developed for Cr contamination according to end use and these are reported in R&D publication SGV 4 (DEFRA 2002b). These guidelines are summarised in Table 1 and are based on total Cr concentration in soil with the assumption that all Cr present is Cr(VI). It is useful to note that the literature cited in SGV 4 reports background levels of total Cr in UK soils ranging from 0.2-1300 mg/kg.

Table 1: Soil Guideline Values for total Cr for different land uses (DEFRA, 2002b)

Standard land use	Soil Guideline Value (mg/kg dry weight)
Residential with plant uptake	130
Residential without plant uptake	200
Allotments	130
Commercial/industrial	5,000

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The key water quality standards for total Cr in the UK are the drinking water guideline of 50 µg/l and an environmental quality standard (EQS) ranging from 2 to 10 µg/l (depending on hardness). Assessment of risks to surface water and groundwater from Cr contamination needs to be undertaken on a site-specific basis with reference to recent guidance issued by the Environment Agency (Environment Agency, 2006). This involves consideration of whether the site poses a risk to meeting the objectives of the Water Framework Directive.

3. TREATMENT OF CHROMIUM(VI)

3.1 Introduction

Given the relatively low toxicity of the trivalent form, the overall objective of most forms of treatment of Cr(VI) has been that of chemical reduction. The majority of trivalent Cr salts are also of low solubility, so provided that the pH of the medium is neutral to alkaline, the trivalent Cr may also be expected to be of low mobility in the environment. Consideration should also be given to aquifer plugging, which may be a wanted or unwanted side-effect of the precipitation of Cr compounds.

As will be discussed below there are a number of chemical mechanisms, including some mediated by microbiological action by which chemical reduction of Cr(VI) to Cr(III) will take place and these have been employed in treatment processes. There are three main groups of compounds that can be used to reduce Cr (Su & Ludwig, 2005):

- reduced sulphur compounds such as hydrogen sulphide, iron sulphide, sodium sulphite, sodium metabisulphite and sodium dithionite;
- iron based compounds such as zero valent iron, dissolved ferrous iron (Fe²⁺) or iron-containing minerals hematite, magnetite and biotite;
- Various organic compounds, including constituents of soil organic matter.

In the field, conversion through chemical reduction may take place in highly reducing environments and this can be accelerated by the addition of some of the above compounds. Once reduction has taken place, the Cr is essentially stable since re-conversion from the trivalent to the hexavalent form requires highly oxidising conditions and the presence of manganese as catalyst (Fruchter et al., 2000).

If Cr(VI) can be converted to the trivalent form it is therefore likely to be both stable and of relatively low mobility under neutral to alkaline conditions. The treatment strategy therefore becomes one of minimising total concentrations of Cr in the aqueous or potentially leachable phase in addition to simple chemical reduction.

The following sections examine the various mechanisms of treatment for media contaminated with Cr(VI). Given the broad similarity in terms of treatment objectives, treatment of wastewater, groundwater and soil are considered together. As well as methods based on chemical reduction, consideration is also given to other methods for treatment of soil and groundwater.

3.2 Chemical Treatment

3.2.1 Sulphur dioxide

Sulphur dioxide (SO₂) has traditionally been one of the most widely used methods of treatment of reducing Cr(VI) in metal plating industry effluents (Wild, 1987). Precipitation of the insoluble chromium hydroxide by addition of an alkali follows as a subsequent stage (Kiff, 1987).

There is a report of full-scale *in situ* reduction of Cr(VI) using sulphur dioxide injected into soils within and beneath a pit used for disposal of spent acidic chromate solutions (Yiannakakais et al., 1999). A 1:1 or 3:2 ratio of sulphur dioxide to chromate reduced was estimated as being required, followed by the addition of an alkali. Gas injection took place over a 3-week period and was reported as achieving complete (100%) reduction in the treated area. A capillary barrier soil cover was engineered to minimise water infiltration into the treated zone.

3.2.2 Sodium bisulphite and sodium metabisulphite

Sodium bisulphite (NaHSO₃) has often been used in the metal plating industry for treatment of Cr(VI) in preference to sulphur dioxide (Wild, 1987), sometimes in combination with hydrazine. It has the advantage over ferrous sulphate (see below) in producing less sludge following precipitation with alkali. Sodium metabisulphite (Na₂S₂O₅) has also been employed as a reductant in studies cited by Su & Ludwig (2005). Various sulphur based reductants (unspecified) are also being used for full-scale *in situ* geochemical treatment of Cr(VI)-contaminated groundwater (USEPA, 2000).

3.2.3 Ferrous sulphate

Due to its frequent availability as a by-product from pickling, ferrous sulphate (FeSO₄) has provided a further traditional reducing agent for treatment of metal-industry process effluents (Wild, 1987). As with sodium metabisulphite and sulphur dioxide, a two phase approach has been employed involving acidification to around pH 2-3, followed by neutralisation with quicklime or sodium hydroxide to precipitate the trivalent Cr as the hydroxide.

There are examples in the literature of ferrous sulphate having been used for Cr(VI) treatment *in situ*. At a site of a former paper mill on the Delaware River, Cr(VI) concentrations in the perched aquifer were reduced from 85,000 µg/l to 50 µg/l through treatment by reduction and precipitation using an acidified solution of ferrous sulphate heptahydrate. The latter was applied using a combination of wells and trenches, without groundwater extraction and therefore without the need for disposal of treatment sludge (Brown et al., 1998). Additionally application of ferrous sulphate to 1300 m³ of excavated fill material was treated by surface spraying.

3.2.4 Ferrous ammonium sulphate

One alternative to ferrous sulphate is the use of ferrous ammonium sulphate, which has the advantage over the former of reacting sufficiently rapidly over a neutral to alkaline pH range avoiding the requirement for acidification. A treatability study at bench-scale using samples of waste material (of pH 11) reported that appropriate Cr(VI) reduction was achievable using a 1:1 ratio of ferrous ammonium sulphate:waste based on treatment of 6000 mg/kg Cr(VI), with a 10% ratio required for treatment of 600 mg/kg (Jacobs, 1992).

3.2.5 Zero valent iron

Zero valent iron has reportedly been used to treat a plume of Cr(VI) at the US Coast Guard Support Centre in Elizabeth City, North Carolina. The iron was applied in a permeable reactive barrier (PRB) through which the contaminated groundwater was channelled (Puls et al., 1999, cited by Su & Sudwig, 2005). Several other examples of iron particle barriers have been reported as operating successfully in the USA (Fruchter, 2002). In the presence of iron, Cr(III) precipitates as a mixed iron-chromium hydroxide and the high pHs that form in such barriers may result in precipitation of various minerals.

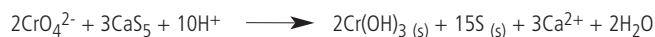
3.2.6 Sodium dithionite

The mechanism of Cr(VI) treatment using sodium dithionite (Na₂S₂O₄) involves the conversion of ferric iron in soils or sediments to ferrous iron which subsequently reduces the Cr(VI) to form the Cr_xFe_{1-x}(OH)₃ solid. This reducing agent has been employed to treat Cr contamination *in situ* through direct injection into the capillary fringe, at the aforementioned site in Elizabeth City (Khan & Puls, 2003). At this site, injection of sodium dithionite caused a significant decrease in redox potential by as much as ~700 mV and Cr(VI) concentrations in the reduced zone were reduced to below the detection limit of 0.01 mg/l.

This chemical has also been reported as being used successfully in a PRB mode via deep well injection into a contaminated aquifer at the US Department of Energy Hanford site in Washington state (USEPA, 2000). The field test was based on using *In Situ* Redox Manipulation (ISRM) to create a 15 m diameter cylindrical treatment zone through the injection of 77,000 litres of buffered sodium dithionite solution over a 17 hour period (Fruchter et al., 2000). Three and a half years after the test the treatment zone is reportedly still anoxic and Cr(VI) levels have been reduced from 0.060 mg/l to below detection limit of 0.008 mg/l. No permeability changes have been detected during any phase of the test.

3.2.7 Calcium polysulphide

Calcium polysulphide (CPS) is used extensively as an agricultural soil amendment and for removal of metals in water treatment systems. It is understood to have recently been approved for *in situ* remediation at several sites in the United States. CPS is more stable and persistent in subsurface environments than other reductants such as sodium dithionite. It is also relatively safe to handle in the field. The chemical reduction that takes place can be simplified as follows:



The fixation of Cr(VI) by CPS is considered to be a permanent remediation technique under most groundwater conditions. Whilst the reaction is theoretically reversible under natural groundwater conditions, the equilibrium condition is dominated by the right side of this reaction.

A report by Jacobs (2001) describes the application of CPS as an effective reducing agent resulting in the conversion of Cr(VI) to chromium hydroxide at a former wood treatment facility in Ukiah, California. Delivery took place using enhanced direct push injection together with a high-pressure lance system. A reduction in

groundwater concentrations of 99% was reportedly achieved over an 18-month period. Fruchter (2002) also refers to a wood treatment facility in California, this time at Turlock, in which injection of CPS was used to enhance treatment using an existing pump and treat scheme. Over a 21 month period the size and mass of the plume was reportedly reduced by 98%.

URS has successfully employed this treatment at a metal plating shop in Arizona for *in situ* geochemical fixation of Cr(VI) in soil and groundwater in alluvial fan sediments contaminated by Cr. Concentrations of Cr(VI) in groundwater at the site exceed 200 mg/L. URS completed vadose zone and groundwater pilot tests using CPS and full-scale vadose zone application in the source area is underway.

Prior to vadose zone treatment, Cr(VI) concentrations in the ~2 m² test zone were as high as 2,190 mg/kg in soil, and 3,600 mg/L in the vadose zone pore water. Over a period of about 24 hours, approximately 2.5 m³ of 29% CPS were applied to infiltration trenches, followed by 9.5 m³ of water to disperse the chemical through the test zone. The wetting front was monitored and sampled with soil lysimeters installed in a basement wall. Results during the first 30 days indicated that eight of the nine lysimeters used to monitor the test were impacted and demonstrated a 90% reduction in Cr(VI) concentrations.

In the groundwater pilot test area, the aquifer at 50 m below ground level was impacted by Cr(VI) concentrations of 240 mg/L. Approximately 34 m³ of 29% CPS were injected through an existing monitoring well, followed by ~300 m³ of water, at an average rate of 117 litres per minute to flush the well and transport the CPS to an observation well located 9.1 m across the regional hydraulic gradient. After 35 to 40 hours from injection, a decrease in redox potential and an increase in pH in the observation well indicated breakthrough of CPS. Concentrations of Cr(VI) in the observation well decreased from 240 mg/L to less than 1 mg/L shortly after the Oxidation Reduction Potential (ORP) became negative, with rebound of Cr(VI) concentrations occurring after 115 days, to at least pre-injection levels. In the injection well, ORP remained negative and Cr(VI) concentrations were below detection after 419 days, representing a decrease from 51 mg/l to 0.015 mg/l.

3.2.8 Metals remediation compound (MRC™)

A proprietary treatment for heavy metal contaminated groundwater has been developed by the company Regenesis, using a compound traded as MRC (Metals Remediation Compound). MRC is supplied as a viscous liquid for direct injection into contaminated and saturated soils. The product is comprised of a polylactate polymer and a benign organosulfur compound allowing for a controlled release of its active component as Cr(VI) is directly precipitated as the hydroxide. Regenesis has provided evidence of both pilot scale and full-scale application of MRC having reduced Cr(VI) to the requisite target concentrations in groundwater at several plating facilities in the USA.

3.3 Microbiological Treatment

Microbial reduction of Cr(VI) may take place by several alternative mechanisms. It may involve the cell gaining energy as say for iron (III) or manganese (IV) reduction, this being mediated by dissimilatory metal reducing bacteria (DMRB) under anaerobic conditions. Alternatively, it could be carried out as a detoxification mechanism both aerobically or anaerobically. A particularly stable form of reduced Cr may be obtained microbiologically through the process of biomineralisation. This refers to the capacity of certain microbial species to form chemically stable minerals. As with the application of a solution of reagent, it has the advantage over fixation processes in that the bioremedial fluids can enter pore spaces and micro fractures to access otherwise unavailable Cr(VI).

No full-scale microbial treatments of contaminated soil in the unsaturated zone were found in the literature, although treatment of groundwater contaminated by Cr(VI) has been reported using the application of readily available carbonaceous substrates such as molasses. This is considered to be an indirect microbial mechanism whereby the ready utilisation of this carbon source causes the redox potential to fall and create highly reducing conditions conducive for Cr(VI) reduction.

A USEPA report on Cr treatment technologies (USEPA, 2000) describes two field applications of molasses in reactive zone mode, one pilot scale, the other full-scale both of which achieved clean up objectives for Cr(VI) in groundwater. In the first of these in the mid-west USA, reduction in Cr(VI) from 15 mg/l to below 0.2 mg/l was achieved within a month and over the subsequent five months of monitoring. The second example was a full-scale application at a site in central Pennsylvania. Injection of molasses resulted in a reduction from 1.95 mg/l to 0.01 mg/l and the plume shrunk to a quarter of its size over 18 months.

3.4 Physical Remediation Methods

3.4.1 Introduction

Soil and groundwater remediation encompasses a variety of physical remediation techniques some of which may be applicable to sites contaminated by Cr. These can include both treatment and containment systems.

3.4.2 Electrokinetic treatment

Electrokinetic treatment is based on the principle that when a low voltage direct current is passed through contaminated soil, ions such as heavy metals will migrate to a place where they can be collected and removed. This is performed by placing electrodes within the soil and energising these using a DC power source to form H⁺ ions at the anode and OH⁻ ions at the cathode. Negatively charged chromate anions will therefore migrate to the anode where they pass through a membrane into the electrode solution, from where they can be collected. The process is reported to be applicable to both the saturated and unsaturated zone.

The USEPA report of October 2000 on *in situ* treatment of soil and groundwater contaminated with Cr classifies electrokinetics as having 'demonstrated effectiveness' for treatment of groundwater and 'potential effectiveness' for soil (USEPA, 2000).

Alkaline soils were reported to be problematic during the early development of the technology although appropriate methods can be used to modify the electrolyte solution to address this. A more significant problem is the presence of solid metallic contamination, so that the presence of metallic underground services will act as preferential current paths.

3.4.3 Soil washing

Soil washing systems separate the pollutants from the soil matrix and can include both high-pressure systems and surfactant enhanced washing. After washing, the liquid effluent can be treated in a wastewater treatment plant, and the pollutants can be recovered as oil flotation sludge and filter cake.

Soil washing is a volume reduction technique, but consideration should be given to the fact that whilst some of the more granular material may be treated appropriately for use as subsequent backfill, there can be a significant proportion of fines, in which the contaminants have been concentrated, that requires off-site disposal.

3.4.4 Containment

Containment systems include both horizontal systems (i.e. capping) alone or in combination with vertical systems to limit lateral flow of contaminated groundwater. The latter may be engineered to form funnel and gate systems whereby a permeable reactive barrier can be used to effect treatment of the groundwater within the gate (as discussed above, using ISRM, either through dosing of a reductant or installation of a specific barrier (zero valent iron)).

3.5 Pump and Treat

Pump and treat schemes can be designed with various above ground treatment systems to address a range of chemicals for which groundwater remediation can be applied, including Cr. These could include any of the chemical reduction processes, referred to above, applied in batch mode for example. A key issue here would be the volume of sludge generated by chemical reaction.

Alternatively treatment of the abstracted groundwater may take place via an absorptive medium, of which Granular Activated Carbon (GAC) is the most widely used example. A cheaper alternative to the latter is 'White Karbon'. This is a granulated sodium aluminium silicate that has been activated by the addition of aluminium oxide at high temperature. One particular product in this line, RME 40 can reportedly absorb 3 - 50% of its bodyweight in heavy metals including Cr.

4. CHROMIUM ORE PROCESSING RESIDUE (COPR) IN GLASGOW

4.1 Introduction

Cr contamination in the south-east of Glasgow has long been recognised as a significant regional issue. The contamination has originated from a chemical works in Shawfield that was one of the world's largest producer of Cr, operational from the mid 19th century until 1968. During this period, an estimated 1.5M m³ of chromium ore processing residue (COPR) were landfilled in various sites located in the Rutherglen and Cambuslang areas of the city. Some of this waste was deposited in clay pits or in mounds, for example under football terracing (see Fig. 2). While most areas of Shawfield are covered in buildings or hardstandings which prevent people being exposed directly to the contamination, it has been leaching into groundwater and into culverted streams flowing into the River Clyde.



Fig.2: Visible chromium contamination from COPR in the stands at a stadium in Shawfield.

Within this part of Glasgow, the Clyde Gateway initiative is one of the largest and most ambitious regeneration projects in Scotland, with a core development area covering 2,095 acres (838 ha). The initiative is aimed at exploiting strategic economic development opportunities created by construction of the M74 motorway and East End Regeneration Route (EERR) to contribute to the growth of the metropolitan area and stimulate regeneration of the East End of Glasgow, Shawfield and Rutherglen. The public sector partners are Glasgow City Council, South Lanarkshire Council, Scottish Enterprise, Communities Scotland and the Scottish Government.

Within the Clyde Gateway area, the 73 ha Shawfield industrial area is planned as the main employment centre, with direct access to the M74 motorway, excellent rail connections and an attractive setting next to the River Clyde and Richmond Park.

During the early 1990s 15 sites in the south-east area of Glasgow were investigated and found to have soil concentrations of Cr in the thousands and tens of thousands of mg/kg. A significant proportion of this was Cr(VI), which also exceeded 10,000 mg/kg at several sites and was commonly present at concentrations in the hundreds or thousands of mg/kg.

The Cr contamination at these sites may exist in soil contaminated with COPR both above and below the water table. Ongoing leaching of Cr(VI) from the former and slow dissolution from the latter provide an ongoing source of contamination to groundwater. Groundwater has been shown to be highly contaminated with total Cr as a result: in one extreme case, a total concentration of 3425 mg/l of Cr(VI) was identified, several orders of magnitude greater than water quality guidelines (e.g. an Environmental Quality Standard (EQS) of 0.01mg/l).

4.2 COPR Remediation

COPR is a geochemically highly complex material and initial field trials conducted on a site contaminated with such material in the 1990s highlighted the limitations of some of the conventional approaches for Cr(VI) remediation (Bewley et al., 2000). Subsequent research has helped to understand these complexities and demonstrate why such treatments met with limited success (Farmer et al., 2006). For example, electron microscopy of COPR particulates, combined with X-ray diffraction, indicated that Cr existed in several minerals present within the COPR with an inner, relatively immobile phase containing Cr(VI) diffusing very slowly to the outside solution.

A study undertaken in the late 1980s by Dames & Moore at a site in Camellia, Australia in collaboration with the University of Wollongong, New South Wales (Ellis, 1988) evaluated the application of sodium metabisulphite for COPR treatment in laboratory trials. When added at 20 g per 100 g of pH adjusted wet waste this achieved a leachate concentration of 20 ppm, representing an improvement of greater than 98% on the leachate from the raw waste. The treatment proposed involved addition of sulphuric acid together with 20% (w/w) of dissolved sodium metabisulphite, followed by addition of sodium hydroxide. If required, addition of 120 g of a 5:1 fly ash: cement mixture per 100 g of treated waste produced a suitably solidified product for landfill disposal.

With respect to potential treatment of COPR, bench-scale studies undertaken by the University of Edinburgh in collaboration with the Macaulay Institute indicated that calcium polysulphide (CPS) was highly effective in converting concentrations of 3000 to 7500 mg/kg Cr(VI) associated with COPR, to insoluble Cr(III) (Graham et al., 2006). Following treatment, Cr(VI), was not detected in either equilibration solution (<0.01 mg/l) or solid phase (<10 mg/kg). CPS was also shown to be effective in

bench and field trials for the treatment of Cr(VI) in New Jersey where similar issues relating to COPR deposition exist as in the Glasgow conurbation. A CPS dosage twice the molar stoichiometric requirement proved effective in meeting Toxicity Characteristic Leaching Procedure (TCLP) requirements at bench-scale and when scaled up to a 1000 kg treatment using a pugmill mixer, the same concentration met TCLP standards following a curing period of 15 months (Wazne et al., 2007).

Sodium dithionite, in combination with ferrous sulphate has also been applied as a treatment for COPR (Su & Ludwig, 2005).

4.3 Next Steps

It is proposed that a planning-based development approach is taken by public agencies towards Clyde Gateway, in consultation with the appropriate local authority, to enable remediation to progress without designation of land as contaminated. The approach aims to engage and secure market confidence to ensure remediated sites achieve business use land values. Due to the extent of Cr waste deposits, a successful approach is likely to require:

- A remediation strategy being developed for the whole of Shawfield; and
- A remediation process being found to remediate COPR.

URS Corporation Ltd has been appointed as technical consultant to prepare a remediation strategy for Shawfield, which is likely to take up to three years to complete. In delivering the strategy, they shall be assisted by MWH Corporation.

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