

THE CHEMICAL AND PHYSICAL MAKEUP OF
HORIZONTAL DIRECTIONAL DRILLING
RESIDUALS AND THEIR ENVIRONMENTAL AND
AGRONOMIC IMPACT VIA LAND APPLICATION

By

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Abstract: Type the abstract here. Limit 350 words, single spaced.

There is a growing need for economical disposal of spent horizontal directional drilling (HDD) fluid, or HDD residuals. This study was conducted to physically and chemically characterize HDD residuals from a wide geographic area, and determine if land application is a viable option for disposal. Fifty-eight HDD residuals were received from throughout the U.S. Residuals were analyzed for potential nutrients, trace metals, and salts. Two separate field studies were conducted to study the effects of six application rates of HDD residuals on soil properties and plants. Residuals were applied to established grass plots and to bare soil plots. Forage yield and percent cover were measured respectively. Soil samples were taken at 7, 30, and 90 days after application, and analyzed for potential nutrients, salts, and water soluble metals. After chemical and physical characterization of 58 samples, no trace metals concentrations exceeded EPA Part 503 ceiling for exceptional quality biosolids. Nutrient concentrations were negligible. Salt concentrations were variable. In the established grass field study, there was no significant difference in yield over all treatments. For the bare soil field study, there was no significant difference in percent cover at day 30. At day 63 percent cover at the lowest application rate was significantly higher than the control and all other treatments while percent cover at the highest application rate was significantly lower than the control. Electrical conductivity, pH, SAR, and Mehlich-3 Ca were significant over application rates. Of the 58 residual samples received, nothing was chemically limiting land application. Total solids was the most limiting factor. Land applying HDD residuals at the lowest rate possibly promotes seed germination and application at the highest rate potentially hinders it. Results suggest that land application is a viable option for disposal. However, soil and/or cuttings from the borehole are a large portion of HDD residuals; so boring through a contaminated site could result in a contaminated residual, possibly limiting its potential to be land applied. In this scenario, testing the residual prior to application is recommended. Tillage is advised when applying a residual with a high EC and/or SAR to a bare soil.

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CHAPTER I

INTRODUCTION

Humans have been land applying a wide variety of waste residuals, in many forms, as soil amendments to supplement and improve the soil for over 2000 years (Lederer and Rechberger, 2010). Many residuals are applied for the purpose of waste disposal. Often the sources of these residuals are agricultural, municipal, and industrial wastes or by-products. These residuals, in their broadest sense, vary widely in terms of their overall beneficial and environmental impacts. Information acquired over years of research suggests that sustainable land application systems can be established and maintained under a wide range of conditions (Bastian, 2005). Because of environmental concerns and economic restraints in regards to land-filling and incineration, public interest in land application of wastes is continuing to grow (O'Connor et al., 2005). Probably the most important factor in regards to the land application of agricultural, municipal, and industrial by-products is earning the public's trust with regard to safety. Gaining the public's trust will require waste management in such a way that protects human and animal health, safeguards soil and water resources, and maintains a quality ecosystem for the long term (Larney et al., 2000).

In the last three decades there have been many scientific studies generating a great deal of information on the benefits and environmental impacts associated with the land application of many types of residuals (O'Connor et al., 2005). There are typically two criteria used to assess a

residuals “suitability” for land application and whether or not the intended use will be beneficial: (i) the by-product’s ability to enhance, or have no negative impact, on the productivity of the soil and plant growth (ii) a lack of negative environmental impacts (Whalen et al., 2000). Because residuals differ so much in their physical, chemical, and biological properties (Whalen et al., 2000), each material should be examined independently for potential beneficial or negative impact.

LITERATURE REVIEW

Regulations and Legislation Regarding the Land Application of By-Products

Because land applying by-products can potentially have a negative impact on human health and/or the environment, regulations and legislation govern the rates at which they may be applied to soils. The U.S. Environmental Protection Agency (EPA) regulates the disposal of solid wastes under the Resource Conservation Recovery Act (RCRA). Solid wastes are defined by the EPA as “any garbage or refuse; sludge from a wastewater treatment plant, water supply treatment plant, or air pollution control facility; and other discarded material, including solid, liquid, semisolid, or contained gaseous material resulting from industrial, commercial, mining, and agricultural operations, and from community activities. Solid wastes include both hazardous and nonhazardous waste.” (EPA 1984). Hazardous wastes are defined by the EPA in 40 CFR Part 261. The disposal of hazardous waste is treated different from non-hazardous waste.

The EPA has a list of hazardous wastes found in 40 CFR Part 261.30. If a waste is not considered exempt from RCRA under 40 CFR Part 261.4 or listed on the hazardous waste list, the waste’s potential to be hazardous must be determined. In order for a waste to be considered hazardous, it must exhibit one of four characteristics defined in 40 CFR Part 261 subpart C. The four characteristics include ignitability, corrosivity, reactivity, and toxicity. A waste is ignitable if it creates fire under certain conditions, is spontaneously combustible or has a flash point below 60°C. A waste is considered corrosive if it has a $\text{pH} \leq 2$ or ≥ 12.5 and/or is capable of corroding a metal container. In order to be considered reactive, a waste must be unstable under ‘normal’ conditions i.e. can cause explosions, result in violent reactions or generate toxic gases, fumes, vapors or explosive mixtures when they are heated, compressed or mixed with water. Toxic

wastes are harmful or fatal when absorbed or ingested i.e. contain high levels of heavy metals such as lead, mercury, arsenic etc. (EPA 2009). The toxicity of a waste is defined through a laboratory procedure called the Toxicity Characteristic Leaching Procedure (TCLP). The TCLP helps to identify if a waste has the potential to leach high concentrations of contaminants that can be harmful to the environment or to human health.

Sometimes special legislation is adopted for land application of a single waste, such as biosolids. Guidelines for land applying biosolids can be found in CFR 40 Part 503 Subpart B of the National Sewage Sludge Rule. This rule was released by the EPA under the Clean Water Act (CWA) in 1992. Different provisions within the Part 503 rule apply to the entity that produces the biosolids and the entity applying them. Part 503 sets ceiling concentration limits for 10 “heavy metals”: arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg), molybdenum (Mo), nickel (Ni), selenium (Se) and zinc (Zn). There are also requirements for pathogens as well as site restrictions for the land receiving the biosolids. Biosolids are categorized into one of four groups. These groups are Exceptional Quality (EQ), Pollutant Concentration (PC), Cumulative Pollutant Loading Rate (CPLR) and Annual Pollutant Loading Rate (APLR). All groups are safe for land application. Safety is ensured by the combination of management practices and pollutant limits imposed by each group (EPA 2014).

Types of Residuals used for Land Application

Animal manure

The most common ‘agricultural’ by-product used as a residual for land application is animal manure. In fact, at more than 100 million dry Mg annually, manure is the most widely land applied residual. (O’Connor et al., 2005). Several types of manure can be used, but beef and poultry are the most common. The land applied manure can be slurry, solid, or liquid, each requiring different management practices (Eghball and Power, 1994).

Manure is a valuable residual because it provides organic matter and nutrients to the soil (Eghball and Power, 1994). It is an excellent source of nitrogen, phosphorous, potassium, and secondary nutrients (Risse et al., 2006). The added soil organic matter acts as an ion exchange material, buffering material, chelating agent, and can play a vital role in soil structure (Eghball and Power, 1994). Soil organic matter helps to form aggregates thus improves soil drainage. Manure can also raise or maintain the soil pH, in turn, lowering the bioavailability of harmful metals to plants, this is true especially for poultry litter (Risse et al., 2006). Many studies have shown that crop yields on land where manure has been applied are equivalent or superior to those using inorganic fertilizers (Edmeades, 2003) (Risse et al., 2006) (Penn et al., 2014a). Unlike commercial fertilizers the nutrients in manure take longer to become plant available. This is due to the microbial decomposition that is needed to mineralize the organic forms of nitrogen (N) into ammonium (NH_4) and nitrate (NO_3). This process is highly variable and relies heavily on the carbon (C) to nitrogen ratio (C:N) within the manure, soil moisture and temperature (Eghball et al., 2002). When the C:N ratio of organic matter is above 20:1, microbes actually use or immobilize plant available NO_3 as excess C is consumed. When the ratio falls below 20:1, microbes begin to mineralize the N as NH_4 , making it plant available (Havlin et al., 2005). Depending on the type of manure being applied, the C:N ratio varies. A study done in Ithaca, NY showed the C:N ratio for laying hens, beef cattle, dairy cattle and swine were 6, 19, 16 and 14, respectively (Rynk, 1992). This makes determining application rates more difficult, however, the slower release helps improve plant utilization and lessens the nutrient loss to surface and ground waters (Risse et al., 2006).

Manure must be properly managed, similar to inorganic fertilizers. When the nutrients become available, they are subject to loss and can be transported to ground water or into surface waters where eutrophication becomes a problem. The EPA considers agriculture to be the leading cause

of pollution to lakes and streams (EPA 1998). This has led to many best management practices (BMP's) initiatives in the agriculture sector.

Biosolids

Another common residual that is land applied is treated sewage sludge, also known as biosolids. Biosolids are a by-product of the municipal waste water or sewage treatment process. This makes biosolids a 'municipal' by-product. The waste water treatment process reduces the amount of easily degradable organic matter (biological oxygen demand; BOD). It is the insoluble solid residue remaining after this process that is coined sewage sludge, biosolids, or domestic wastewater residuals (Singh and Agrawal, 2008). It is estimated that approximately 7.5 million Mg of biosolids are produced each year in the United States (Basta, 2000). Disposal options include landfilling, incineration, land application and dumping at sea (Bright and Healey, 2003). It is suggested that landfilling and land application are the most economical methods of biosolids disposal (Metcalf and Eddy, 2003).

Waste water treatment plants receive domestic sewage as well as industrial effluents and storm water runoff. Industrial effluents and storm water runoff can contain many toxic substances ranging from detergents to pesticides (Singh and Agrawal, 2008). However, like manure, biosolids contain both organic and inorganic plant available nutrients as well as soil conditioning properties, giving incentive for its use as a source of fertilizer (Singh and Agrawal, 2008). A study done by Sommers (1977) showed the median N, phosphorus (P) and potassium (K) percentages in biosolids were 3.3, 2.3 and 0.3 respectively. The same study showed median levels of Pb, Zn, Cu, Ni, Cd and Cr to be 500, 1740, 850, 82, 16 and 890 mg/kg respectively. Because biosolids have the potential to be both beneficial and detrimental to the environment, there is need for regulation. The land application of biosolids is federally regulated by the EPA under 40 CFR Part 503 (EPA 2007). This law governs the use of biosolids for land application

by putting limits on heavy metal concentration, pathogens present, and site and harvesting restrictions in order to minimize threats to public health and environmental degradation (Guo, 2012). Biosolids are also regulated on the state level; these regulations vary by state.

Fly ash

Another common land applied residual is fly ash. Fly ash is the by-product that is formed during the process of coal combustion, thus it is coined an 'industrial' waste. Over half of the energy produced in the United States comes from the combustion of coal, and consumption is only expected to increase as we strive to be an energy independent nation (Sajwan et al., 2003). This will only increase the need for viable disposal options for the waste materials from energy production.

Fly ash generally contains an amorphous ferroaluminosilicate mineral that's chemical and physical properties are dependent on the parent coal, combustion temperature, type of emission control device being used, and methods of storing and handling (Adriano and Weber, 2001). According to the American Coal Ash Association there were nearly 52 million tons of fly ash produced in the United States in 2012 (American Coal Ash Association, 2012). The most common methods of disposal for this by-product are storage in settling ponds, stockpiling, landfilling, and applying to open land (Adriano and Weber, 2001); (Jala and Goyal, 2006).

In the past fly ash was considered solely as a waste material but, thanks to numerous detailed studies conducted in the latter half of the 20th century, we now know its nature and composition. Due to the abundance of basic mineral elements resembling the Earth's crust, fly ash is an excellent replacement for natural materials (Jala and Goyal, 2006). The United States utilizes roughly 44% of its annual production of fly ash (American Coal Ash Association, 2012). It can be used as a partial substitute for Portland cement, road fills and bases, in asphalt, and for waste solidification and stabilization (Adriano and Weber, 2001).

The application of fly ash to agricultural land cannot supply crops with the macronutrients nitrogen N or P, but it could supply crops with a sufficient amount of other nutrients such as potassium (K), sulfur (S), Mo, calcium (Ca), boron (B), as well as others such as Zn (Sajwan et al., 2003). Fly ash varies in pH which is largely dependent on the amount of sulfur in the parent coal (Jala and Goyal, 2006). Land application of alkaline fly ash can reduce the acidity of a soil, making certain toxic metals unavailable for plant uptake. Unfortunately, fly ash can also contain different amounts of trace metals such as Cu, Ni, Pb, and Cr which does put a limit on its potential use as a soil amendment. Analysis of fly ash from three coal based thermal power plants in India showed average levels of Cu, Ni, Pb and Cr to be 66, 43, 37 and 93 mg/kg respectively (Sushil and Batra, 2006). Currently, individual states regulate the disposal of fly ash, but the EPA is proposing two federal regulatory options, Subtitle C and Subtitle D, under the nation's primary law for regulating solid waste, RCRA (EPA 2013). Each option will better protect the environment and allow both federal and state enforcement.

Oil & gas drilling mud

Another 'industrial' by-product with a growing need for disposal is the spent drilling fluid from the oil and natural gas industry. The United State is the world's leading producer of oil and natural gas with 1,043,101 oil and gas wells operating onshore and 4,625 oil and natural gas platforms operating offshore, producing 2.4 billion barrels of oil and approximately 29.8 trillion cubic feet of natural gas annually (American Petroleum Institute 2013). The exploration and production of this oil and natural gas generates large volumes of waste in the form of drilling mud (Clements et al., 2010), second only to return water in total volume (Onwukwe and Nwakaudu, 2012). In 1995 the American Petroleum Institute estimated about 150 million barrels of drilling mud was produced from onshore wells located in the U.S. alone (Veil, 2002). As the oil and natural gas industry continue to grow, so will the amount of this by-product and the need for proper disposal.

Current disposal methods for oil and gas drilling mud include landfilling, land application, and slurry injection (Onwukwe and Nwakaudu, 2012). Land application is the most attractive option to the industry due to the minimization of hauling distances to disposal sites (Clements et al., 2010). It is the chemical makeup and physical properties of drilling mud that influence its hazardous characteristics and its ability to impact the environment (Onwukwe and Nwakaudu, 2012). Drilling muds generally contain bentonite clays, synthetic organic polymers, production water, barite, formation cuttings, and salts (Bauder et al., 2005), but may have other constituents such as petroleum hydrocarbons, heavy metals and patent protected ingredients. Drilling mud is classified depending on the base fluid. If the base fluid is water then the mud is called “water based mud” and if the base fluid is diesel then it is called “oil based mud” (Penn et al., 2014b).

Federally, oil and natural gas drilling muds are subject to regulation under the EPA RCRA Subtitle D. Each state also has regulating authority. In Oklahoma, the Oklahoma Corporation Commission (OCC) regulates disposal of drilling wastes including the practice of land applying oil and gas drilling mud. Restrictions vary based on the type of mud, the mud constituents and the chemical and physical characteristics of the soil and landscape receiving the mud (Oklahoma Register Title 165:10-7-9). Adverse environmental impacts to both soil and plant growth after the land application of oil and gas drilling fluid may occur due to soluble salts, which are found primarily in water based mud, as well as high levels of plant available trace metals, which are found primarily in oil based mud (Bauder et al., 2005; Miller et al., 1980; Nelson et al., 1984). Analysis of the water based drilling mud used in a study in Colorado indicated that sodium (Na), Cd and Mo were the only constituents found in higher concentrations than a typical soil. Ranges for Na, Cd and Mo were 9900-20000, 1.1-4.7 and 0.2-9.3 mg/kg respectively. However, following the guidelines set by the OCC, land application can be a sustainable solution for the disposal of oil and natural gas drilling muds, with minimal long term impact to the receiving soil.

Potential Constituents of Concern in By-Products

Trace Metals

The term 'trace metals' identifies the fact that they are metals found in the soil in minute amounts. These metals occur naturally in the soil at various concentrations and are derived from the soil's parent material or lithogenic source (Alloway and Alloway, 1995). In a study by (Richards et al., 2012), naturally occurring concentrations of trace metals were quantified in order to establish a background or baseline level. In this experiment, 28 Oklahoma benchmark soils were digested and analyzed for concentrations of the trace metals As, Cd, Cr, Cu, manganese (Mn), Mo, Ni, Se, Pb and Zn. The concentrations of trace metals found were (mg kg⁻¹): As (0.75-33.6), Cd (0.13-0.80), Cr (4.30-69.7), Cu (1.94-32.7), Mn (27.4-2165), Mo (0.18-1.80), Ni (2.41-57.3), Pb (2.60-31.7), Se (BDL) and Zn (15.3-142). It should be noted that As and Se are actually metalloids rather than metals (Pierzynski et al., 2005), but will be referred to as trace metals throughout this document.

Anthropogenic sources of trace metals may also be found in the soil. Urban areas generally have higher levels of Pb, Zn, Cd and Cu due to concentrated automobile use (Alloway and Alloway, 1995). Due to regulation, concentrated contamination from a single anthropogenic source is not as common as it was in the past. Research on historical trace metal contamination and accumulation of the urbanized region of the Lake Champlain watershed in Burlington, Vermont examined anthropogenic sources of metals over a 350 year period. The research showed metal concentrations above background levels beginning in the early to mid-1800's and maximum concentrations and accumulation rates were seen in the late 1960's. Decreases in concentration and accumulation rates were seen for almost all metals from 1970-present (Mecray et al., 2001). Regulations set by the EPA and various state agencies limit the addition of trace metals to the

soil. Municipal and industrial by-products must meet certain criteria (nontoxic) before being land applied.

Trace metals are found in different forms in the soil. *Cationic* metals such as Cd^{2+} and Pb^{2+} are predominately found as cations in soils and soil solutions. Trace metals that bond with oxygen are called *oxyanions*, and have an overall negative charge. Arsenate (AsO_4^{3-}) and chromate (CrO_4^{2-}) are examples of oxyanions. It is the soil's chemical conditions that determines the chemical species present; and it's the species present that dictate the element's behavior in regards to leachability, bioavailability, and risk to human health (Pierzynski et al., 2005).

There are several chemical processes that govern the speciation of any given trace metal. These processes include precipitation-dissolution, complexation-dissociation, and adsorption-desorption; all of which are affected by pH (He et al., 2005). It is the type of bond formed in the solid-solution interface of the soil that determines the retention of a trace metal.

Precipitation can be defined as a phase-distribution process within a solution where insoluble solids are formed and then separate from a solution. Dissolution is the changing from a gaseous or solid phase into the aqueous phase (Boulding and Ginn, 2003). Both processes rely heavily on a soil solution's chemical properties, predominately ionic activity and pH (Essington, 2004).

When a trace metal is in solid form or precipitated it is not plant available or leachable. However soil consumption is a pathway to consider regarding human health. In regards to environmental and human health, dissolution and transport to drinking water is a more viable pathway. When in solution, free ions are available for plant uptake, but sometimes the dissolved ions will form complexes that remain in solution and may or may not be plant available depending on the plant and complex. A complex consists of one central cation with one or many anions, or ligands, attached. Ligands can be organic or inorganic. There are two types of complexes, inner sphere and outer sphere. Inner sphere complexes are the direct bonding of a central cation to a ligand(s).

These are covalent bonds and are very strong. Simple complexes (monodentate) and chelates (polydentate) are both inner sphere complexes. Certain chelates have the ability to remove various metals from contaminated soil via phytoremediation or soil washing (Leštan et al., 2008). Sometimes chelating agents in conjunction with lowering pH can be even more effective. A study in New Jersey demonstrated that the application of EDTA and acetic acid led to a two-fold accumulation of Pb in Indian mustard shoots compared with the application of EDTA alone (Blaylock et al., 1997). Outer sphere complexes usually consist of ion pairs bonded electrostatically, with water molecules separating the two. Outer sphere complexes are very weak and easily broken.

Adsorption is a general term that can be divided into two categories, specific and non-specific adsorption. Divalent cations such as Cu and Pb are commonly associated with specific adsorption. Specific adsorption occurs when a cation forms a covalent bond(s) with an oxygen atom(s) on the surface of a variable charged soil mineral or humus, forming an inner sphere surface complex (Essington, 2004). This is a very strong bond and complete desorption is highly unlikely. Specific adsorption is pH dependent and increases as pH rises. A trace metal that is specifically absorbed is not bioavailable or leachable. Non-specific or outer sphere adsorption relies on electrostatic forces or cation exchange sites. This is a much weaker bond associated mostly with permanently charged soil minerals and is easily reversible making it more likely to become plant available or leachable under varying conditions.

Total concentration of any given trace metal in a soil is equal to:

$$C_{\text{Total}} = C_{\text{Fixed}} + C_{\text{Adsorbed}} + C_{\text{water}}$$

Where:

C_{Fixed} = the concentration of a trace metal that is fixed within the structure of a clay mineral.

C_{Adsorbed} = the concentration of a trace metal adsorbed to the surface of a soil mineral and onto the exchange sites of soil organic matter.

C_{Water} = the concentration of a trace metal in the water fraction of the soil in equilibrium with C_{Adsorbed} .

The C_{Fixed} portion is the immobile fraction, and the sum of C_{Adsorbed} and C_{Water} is the potentially mobile portion and C_{Water} is the plant available portion (Dragun, 1988).

Soluble Salts

Salts that naturally occur in soils or soil solution are derived from many sources, including weathered parent material, saline water bodies, and atmospheric deposition. There are many anthropogenic sources as well, such as long term irrigation, leaking of saline waste storage pits, and the land application of byproducts such as water based drilling mud and fly ash among others. The salts that are commonly found in soil consist of the basic cations Na^+ , K^+ , Mg^{2+} and Ca^{2+} , and the anions Cl^- , CO_3^{2-} , SO_4^{2-} , NO_3^{1-} and HCO_3^- (Essington, 2004).

There is a classification system for soils with excessive salt. These classifications are based on values of the sodium adsorption ration (SAR) and the electrical conductivity (EC) of the soil solution as well as pH. A soil can be considered *sodic*, *saline* or *saline-sodic*. The condition or quality of a soil also vary by classification. SAR is the ratio of cationic charge from Na to the cationic charge from Ca and Mg. SAR is calculated using the equation $\text{Na} / \sqrt{(\text{Ca} + \text{Mg})} / 2$ where values for Na, Ca and Mg are in meq/L. EC is the measurement of the soils solution's ability to conduct electricity which is directly correlated to the concentration of ions in solution. EC is usually measured using a standard EC probe. Both SAR and EC are quantified using the analyte, or water portion, of a saturated paste extraction (Havlin et al., 2005).

Sodicity can have profound effects on a soil's physical structure. Na ions have a very large hydraulic radius. Therefore, high Na concentrations lead to the increased swelling of Smectitic

2:1 clay minerals within a soil. This decreases a soil's overall porosity which, in turn leads to poor soil structure and increased erosion, poor permeability, low infiltration rate, and a reduction in hydraulic conductivity (Pierzynski et al., 2005). Sodicity and Salinity can also effect plant growth, however tolerance varies by plant species. In one study, tomatoes grown in soils with EC's of 1.6, 3.8, 6.0 and 10.2 mmhos/cm, yield of tomatoes were 59.5, 55.2, 50.7 and 43.9 kg/plot respectively. In the same study, overall fruit weight also declined with an increase in EC at 68.5, 59.5, 55.8 and 51.9 g/unit respectively (Shalhevet and Yaron, 1973). Poor plant growth may be a result of the soils poor structure or could be a result of a decrease in osmotic potential, reduction of plant available nutrients and elemental toxicities; all of which are possible in a sodic or saline sodic soil (Corwin et al., 2003; Pierzynski et al., 2005).

Due to the soluble nature of salts, leaching occurs rapidly with precipitation or irrigation (unless irrigating with saline water). This can be a concern when a large salt plume of anthropogenic source threatens a ground water source. However, on a smaller scale, this make remediation fairly easy. A sodic soil is usually remediated with the addition of water and gypsum (CaSO_4). The process can take many years, but the Ca ion concentration eventually exceeds the concentration of Na ions as they leach through the soil profile (Davis et al., 2003). CEC sites within the soil have a greater affinity for divalent Ca ions than monovalent Na ions as seen in the lyotropic series: $\text{Al}^{3+} > \text{Ca}^{2+} > \text{Mg}^{2+} > \text{K}^+ = \text{NH}_4^+ > \text{Na}^+$ (Havlin et al., 2005)

.Nutrients

There are sixteen elements that are essential to plant growth. Three of them, C, H and O are obtained from the atmosphere and water. The remaining thirteen consist of the primary nutrients N, P and K; secondary nutrients Ca, Mg and S; and the micronutrients iron (Fe), Mn, Zn, Cu, B, Mo and Cl. These remaining thirteen are mineral nutrients and are supplied to the plant by the soil and/or fertilizers (Zhang and Raun, 2006). In a natural ecosystem, the plants that are present

is ultimately dictated by weather and available nutrients that naturally occur in the soil. The origin of these nutrients are primarily from the parent material in which the soil was formed, animal waste as well as atmospheric deposition.

Approximately 10,000 years ago, or the Neolithic age, humans began cultivating plants, marking the dawn of agriculture. When the addition of fertilizers to the soil by man began is not known, but was written about by Homer in his poem the Odyssey around 800 B.C. Homer mentioned the manuring of vineyards, and references a manure heap, suggesting manure was stored for this purpose (Havlin et al., 2005). Approximately 2800 years later, man has certainly come a long way in terms of fertilization, but by-products such as manure and biosolids are still used as fertilizers to this day.

It is understood that the primary nutrients N, P and K are the most important mineral nutrients needed for plant growth. Of these, N is the most important and the most limiting due to its mobility in the soil. NO_3 and NH_4sw are the two forms of N taken up by plants. Sources of these forms of N originate from symbiotic and non-symbiotic N_2 fixation by plants and microbes, atmospheric deposition via lightening and rainfall, the microbial decomposition of plant and animal residues (nitrification) and industrial fixation. Due to its negative charge, NO_3 is easily repelled by the overall negatively charged soil (CEC), and in conjunction with precipitation or irrigation, it is lost due to leaching. Another loss of N occurs via volatilization after the aminization and ammonification processes in which bacteria and/or fungi transform the amine functional groups of soil organic matter to NH_4 . Volatilization can occur when plants are stressed and release gaseous NH_3 . Under anaerobic conditions denitrification occurs and N is lost to the atmosphere as N_2O , NO and N_2 (Havlin et al., 2005; Pierzynski et al., 2005; Sylvia et al., 2005; Vu Tran, 2008).

As previously stated, the EPA considers agriculture to be the leading cause of pollution to lakes and streams, therefore fertilization rates and manure application must be properly managed. Nitrogen based manure applications can lead to an increase in soil test P beyond what plants need. The over-enrichment of mineral nutrients, such as N and P, is subject to loss via water transport to ground waters and surface waters. The discharge of N and P to surface waters from cropland are estimated to be approximately 3,204,000 and 615,000 Mg/yr respectively (Carpenter et al., 1998). In surface waters these nutrients are often chemically limiting to algal growth (Correll, 1999), so once they become readily available algae thrive . As the algae die decomposition occurs. This decomposition process uses the dissolved oxygen in the water eventually making it anoxic. Anoxic conditions can result in fish kills among other various detriments to the ecosystem.

INTRODUCTION TO THE HORIZONTAL DIRECTIONAL DRILLING INDUSTRY

The horizontal directional drilling (HDD) industry is certainly on the rise worldwide. In North America alone, the number of drilling units grew from 12 units in 1984 to more than 2,000 units in 1995 (Kirby et al., 1996). HDD is a trenchless technology and its popularity is likely a direct result of its noninvasive nature and the reduced project cost that is a result thereof (Allouche et al., 2000). It is usually used for the installation of underground utilities for municipalities ranging from 50 mm utility conduits to 900 mm sewer lines (Allouche et al., 2000).

Spent HDD mud is an 'industrial' by-product resulting from the drilling or boring process, which utilizes drilling fluid. Drilling fluid is used in the boring process as a means to stabilize the bore hole, float cuttings to the surface, and keep the bit cool. Once the drilling fluid's density reaches 9-10 lbs/gallon it is no longer viable and needs to be disposed of (Ariaratnam and Eng, 2001). At this point, the by-product is known as "mud". HDD drilling fluid constituents are usually water,

bentonite (montmorillinite) and/or polymers (Ariaratnam and Eng, 2001) as well as surfactants and soda ash when needed. These additives may or may not be added to aid in the drilling process, depending on the type of formation that is being drilled through. These additives and their potential uses are listed below:

- Bentonite clay (2:1 soil mineral), which is used for sealing the bore wall (prevent seepage of drilling fluid). Bentonite is also used for increasing fluid viscosity in order to make the fluid more “flowable” with regard to carrying the suspended cuttings back out of the hole to the surface. Bentonite is a clay mineral with a high cation exchange capacity, which can improve the ability of a soil to retain nutrients after land application.
- Polymers such as polyacrylamide (PAM) may be used as a substitute or in addition to bentonite for the same purpose. Polyacrylamide is a common soil conditioner used to stabilize soil on road construction sites and also to prevent erosion on furrow irrigated soils.
- Soda ash (sodium carbonate) used for pH control. Soda ash is used in the production of certain foods, toothpaste, and as a water softening agent in laundry and boilers.
- Surfactants (a.k.a. wetting agents), used to disperse clay particles during the drilling process. This prevents heavy clays from “balling up” at the drill bit, and allows clay particles to flow back toward the surface. Surfactants reduce the surface tension between two liquids or between a liquid and a solid. While some surfactants are synthesized naturally by living cells, manufactured surfactants are commonly used in shampoos, soaps, pharmaceuticals, and foods.

The regulations and bylaws for the proper disposal of the spent HDD mud vary by municipality as well as by state and are not federally regulated. In Oklahoma the OCC has the regulating

authority for disposal however, there are no regulations or guidelines currently available. HDD mud containment and disposal is a growing issue as the number of directional drilling rigs is on the rise (Allouche et al. 2000) paired with the stigma of its association to spent oil and gas drilling mud. Disposal can substantially increase the cost of the project, particularly when large diameter products are installed (Allouche et al. 2000). Current disposal methods include hauling to a landfill, leaving onsite, and land application. The majority of contractors in a survey conducted in conjunction with this study, stated that they had to haul their spent drilling fluid to a landfill, which is both costly and time consuming. Others stated that some landfills will not accept the mud due to unfamiliarity. When asked to rank how big of an issue mud disposal is, 47% of those surveyed considered it a major issue and 30% considered it a moderate concern.

Today there is growing speculation about HDD mud and the safety of its constituents or chemical makeup. As stated above, it is often mistaken for oil and gas drilling mud even though the two by-products are very different. There is a need to separate the public's perception of the spent drilling mud from the HDD industry from that of the oil and gas industry. To date, there is no published research on the chemical and physical makeup of spent HDD mud or its environmental impact as a residual for land application. This research is a first step to examine the safety of land application of HDD drilling residuals.

The overall objective of this project is to characterize the physical and chemical makeup of spent directional drilling fluid and to determine any possible environmental and agronomic impact of its use as a residual for land application.

Specific Objectives

1. Collect and characterize random HDD mud samples representing a large geographic area, for various chemical and physical parameters that could either inhibit land application through environmental degradation, or benefit growing plants.

2. Conduct a land application study of HDD mud representing potential agricultural and urban scenarios in order to assess potential impacts on plants.

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CHAPTER II

THE CHEMICAL AND PHYSICAL CHARACTERIZATION OF HORIZONTAL DIRECTIONAL DRILLING RESIDUALS

ABSTRACT

Horizontal directional drilling (HDD) is a trenchless technology used for the installation of gas lines, sewer lines, fiber optic cable, and many other common utilities. Spent HDD fluid is a by-product of the boring process. However, little is known of its constituency. Currently, the most common method of disposal is landfilling. Land application of spent HDD residuals would be a more economical and less time consuming method of disposal. A chemical and physical analysis of spent HDD residuals from a broad geographic range was conducted in order to determine if land application would be an environmentally safe option for disposal. Fifty eight samples were received from 26 states from throughout the United States. After separation of the liquid and solid portion, the materials were assessed for plant nutrients by Mehlich-3, soluble and total trace metals, potential “leachable metals” by the synthetic precipitation leaching procedure (SPLP), total dissolved solids and sodium absorption ratio (SAR). Calcium was the only nutrient in abundance. All trace metal concentration in the digested soil portion were well below the limits for biosolids set by the EPA 40 Part 503 rule for land application. Metal concentrations in the analytes of the SPLP were compared to drinking water standards, no sample exceeded these limits. Because there was nothing chemically limiting found in the samples, total solids appeared to be the most limiting factor for the land application of spent HDD residuals. However, when boring through a soil with suspected contamination, testing the residual prior to application is recommended.

INTRODUCTION

Humans have been land applying a wide variety of waste materials and by-products, in many forms, as soil amendments to supplement and improve the soil for over 2000 years (Lederer and Rechberger, 2010). Many by-products are applied for the purpose of waste disposal. Often the sources of these waste residuals are agricultural, municipal, and industrial wastes or by-products. These materials, in their broadest sense, vary widely in terms of their overall beneficial and environmental impacts. Information acquired over years of research suggests that sustainable land application systems can be established and maintained under a wide range of conditions (Bastian, 2005). Because of environmental concerns and economic restraints in regards to land-filling and incineration, public interest in land application of wastes is continuing to grow (O'Connor et al., 2005). As the interest in land application increases so does speculation of the public as to what potential contaminants could possibly be in the residuals being land applied. Probably the most important factor in regards to the land application of agricultural, municipal, and industrial by-products is earning the public's trust with regard to safety. Gaining the public's trust will require waste management in such a way that protects human and animal health, safeguards soil and water resources, and maintains a quality ecosystem for the long term (Larney et al., 2000).

The agricultural by-product most commonly land applied is manure. In fact, application of more than 100 million dry Mg annually makes it the most widely land applied residual. The benefits of land applying manure are numerous. Manure is an excellent source of nitrogen (N), phosphorus (P), potassium (K), and secondary nutrients (Risse et al., 2003). It is also a good source of organic matter. Organic matter acts as a buffering material, chelating agent, and can play a vital

role in soil physical properties. There is one major drawback that may occur with mis-application of manure: nutrient loss to surface waters that can lead to eutrophication. This is a major problem especially in concentrated animal feeding operations (CAFOs).

Another common residual that is land applied is treated sewage sludge, also known as biosolids. Biosolids are a by-product of the municipal waste water or sewage treatment process. Like manure, biosolids can be an excellent source of nutrients as well as organic matter. Unfortunately, biosolids may contain heavy metals as well as pathogens. For this reason the land application of biosolids is heavily regulated by the EPA under Part 503. The EPA has set strict concentration limits for metals and pathogens for biosolids to be land applied.

An industrial by-product with a growing need for disposal, especially in Oklahoma, is the spent drilling fluid from the oil and natural gas industry. It is the chemical makeup and physical properties of drilling mud that influence its hazardous characteristics and its ability to impact the environment (Onwukwe and Nwakaudu, 2012). Drilling muds generally contain bentonite clays, synthetic organic polymers, production water, barite, formation cuttings, and salts (Bauder et al., 2005), but may have other constituents such as petroleum hydrocarbons, heavy metals and high salt or sodium concentrations. The land application of spent drilling fluid is regulated by the Oklahoma Corporation Commission (OCC).

The horizontal directional drilling (HDD) industry is certainly on the rise worldwide. In North America alone, the number of drilling units grew from 12 units in 1984 to more than 2,000 units in 1995 (Kirby et al., 1996) and this number has only continued to grow over the last twenty years. HDD is a trenchless technology and its popularity is likely a direct result of its

noninvasive nature and the reduced project cost that is a result thereof (Allouche et al., 2000). It is usually used for the installation of underground utilities for municipalities ranging from 50 mm utility conduits to 900 mm sewer lines (Allouche et al., 2000). Spent HDD residuals are an ‘industrial’ by-product resulting from the drilling or boring process, which utilizes drilling fluid. Drilling fluids are used in the boring process as a means to stabilize the bore hole, float cuttings to the surface, and keep the bit cool. Once the drilling fluid’s density reaches 9-10 lbs/gallon it is no longer viable and needs to be disposed of (Ariaratnam and Eng, 2001). At that point, the by-product is known as “mud” or “residual”. HDD fluid constituents are usually water, bentonite (montmorillinite) and/or polymers (Ariaratnam and Eng, 2001) as well as surfactants and soda ash when needed. These additives may or may not be added to aid in the drilling process, depending on the type of formation that is being drilled through.

The regulations and bylaws for the proper disposal of the spent HDD residuals vary by municipality as well as by state and are not federally regulated. In Oklahoma the OCC has the regulating authority for disposal. However, there are no regulations or guidelines currently available. HDD residual containment and disposal is a growing issue as the number of directional drilling rigs is on the rise (Allouche et al. 2000) paired with the stigma of its association to spent oil and gas drilling mud. Disposal can substantially increase the cost of the project, particularly when large diameter products are installed (Allouche et al. 2000). Current disposal methods include hauling to a landfill, leaving onsite, and land application. The majority of contractors in a survey conducted in conjunction with this study, stated that they had to haul their spent drilling residuals to a landfill, which is both costly and time consuming. Others stated that some landfills will not accept the residuals due to unfamiliarity. When asked to rank how big of an issue residual disposal is, 47% of those surveyed considered it a major issue and 30% considered it a moderate concern.

Today there is a growing speculation about HDD residuals and the safety of their constituents or chemical makeup. As stated above, it is often mistaken for oil and gas drilling mud even though the two by-products are very different. There is a need to separate the public's perception of the spent HDD drilling residuals from that of the oil and gas industry. To date, there is no published research on the chemical and physical makeup of spent HDD residuals or their environmental impact as a media for land application. Thus, the objective of this research was to collect and characterize many residuals from throughout the United States in order to make an overall assessment of their physical and chemical makeup in terms of their suitability for land application.

MATERIALS AND METHODS

In order to obtain a broad overall characterization of HDD residuals it was necessary to obtain samples from throughout the United States. To do this, packages were sent to contractors throughout the country. These packages contained sample bottles, instructions on how to obtain a representative sample and a written survey (Appendix A). A total of 58 samples were received representing 26 different states. Chemical analyses of all residuals were evaluated in terms of their safety regarding land application.

Solids content (gravimetric) was determined by shaking samples for 20 minutes followed by subsampling and placement in an oven at 65°C for 24 hours. In order to conduct several analyses, the solids portion was separated from the liquid by centrifugation at 5000 rpm for 10 minutes followed by filtration with a Whatman No 41 paper.

For the solids portion, samples were extracted via total soil digestion using EPA 3050 method (EPA 1996) using two grams of solid residual, trace metal grade nitric acid (HNO₃) and trace metal grade hydrochloric acid (HCl), and a 30% hydrogen peroxide solution, in conjunction with heat. Following digestion, analytes were filtered using 41 Whatman paper and brought to volume with deionized (DI) water in 50 mL volumetric flasks. Samples were then analyzed for various elements including aluminum (Al), sodium (Na), sulfur as sulfate (SO₄-S), calcium (Ca), magnesium (Mg), P, boron (B), barium (Ba), K, chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), arsenic (As), and lead (Pb). Analytes were quantified using Inductively Coupled Plasma Atomic Emission Spectroscopy

(ICP). The results were compared to the EPA Regional Residential Screening Levels (EPA 2013) as well as EPA 40 CFR 503 Criteria for Exceptional Quality Biosolids (EPA 2007).

A modified version of the synthetic precipitation leaching procedure (SPLP) was also performed on the solids portion in order to simulate potential leachability to groundwater (Hageman et al., 2000). Extraction fluid was made using DI water acidified to a pH of 4.2 using a 60/40 ratio solution of trace metal grade nitric acid (HNO₃) to reagent grade sulfuric acid (H₂SO₄). Solids were extracted at a 1:20 solid:solution ratio for 18 hours followed by centrifugation at 5000 RPM for 10 minutes and filtration through .45 µm glass fiber filters using vacuum filtration. Extracts were analyzed for Mo, Al, B, Na, SO₄-S, Ca, Mg, P, K, Cr, Mn, Fe, Co, Cu, Zn, Cd, As, and Pb by ICP.

The water portion was separated using a centrifuge at 5000 RPM's for a minimum of 10 minutes. The separated water portion was then filtered using 42 Whatman paper. Samples were then analyzed for Mo, Al, B, Na, SO₄-S, Ca, Mg, P, K, Cr, Mn, Fe, Co, Cu, Zn, Cd, As, and Pb by ICP. This will be referred to as "water soluble". Results were compared to EPA Aquatic Life Criteria Table (EPA 2013). Sodium adsorption ratio was calculated for each sample:

$$SAR = \frac{Na}{\sqrt{\frac{(Ca + Mg)}{2}}}$$

Where Na, Ca, and Mg are in units of meq L⁻¹. Electrical Conductivity (EC) and pH were tested on both the solid residuals (prior to solid/liquid separation) and their separated water portion using standard EC and pH meters. Plant available nutrients that were analyzed included P, K, Mg, Ca, Mn, Na, Fe, SO₄-S, Zn, Cu, and B. Of those, Mehlich-3 extraction was used for K, P, Mg, Ca, Mn, and Fe. Mehlich 3 nutrients were extracted at a ratio of 1:10 solids:solution for five

minutes, followed by filtration with Whatman #42 filter paper and analysis by ICP (Mehlich, 1984). Total N and C were quantified using LECO TruSpec Analyzer (Leco Corporation, St. Joseph, MI).

Statistical Analysis

Descriptive statistical analysis was performed using Microsoft Excel (2013). Mean, median, minimum, maximum, and standard deviation were calculated for all elements of interest from the extraction methods listed above. The same parameters were also calculated for pH, EC, and total C and N.

RESULTS AND DISCUSSION

Chemical Properties of HDD Residuals

Overall, the residuals were low in total carbon, as percent C range was 0.09 to 6.61 with a mean of 1.42 and median of 0.51. Percent N ranged from 0.01 to 0.26 with a mean and median of 0.04 (Table 1). Low percentages of C and N were expected as the majority of the residual is smectitic clay and subsurface (deeper than 1 meter) soil and cuttings. The lack of C and N in the residual makes it of little value as a potential C or N source. Solids content had a broad range of 0.04 – 0.72 grams solid grams slurry⁻¹ with mean and median values of 0.35 and 0.34 grams solid grams slurry⁻¹, respectively (Table 2). This wide range is likely due to the variability of the original drilling fluid used and the various formations or sub-soils being bored through; for example, sometimes only water is used without bentonite clay. Values for EC of the residuals ranged from a low of 118 $\mu\text{S cm}^{-1}$ to a high of 3950 $\mu\text{S cm}^{-1}$ with mean and median values of 1198 and 1124 $\mu\text{S cm}^{-1}$, respectively (Table 2). All samples were under the threshold of 4000 $\mu\text{S cm}^{-1}$ for saline sensitive plants (Pierzynski et al., 2005). EC and density were used to calculate loading rates for all samples. Using an application rate of 112 metric tons ha⁻¹, the largest loading rate was 3723 kg ha⁻¹. This is well below the limit of 6724 kg total dissolved solids ha⁻¹ set by the OCC for the land application of water-base drilling mud from oil and gas exploration. The rules for WBM application are stated in the Oklahoma administrative code and register, Title 165:10-7-19. The majority of samples had pH values similar to that of a typical subsoil ranging from 4.7-9.9 with a mean of 7.5 and median of 7.6 (Table 2). The high pH samples are likely indicative of free carbonates, which could occur naturally in the subsoils, or from bicarbonate additives in the original drilling fluid

Separated Water Portion

Sodium absorption ratio (SAR) was measured on the separated water portion of every sample. The mean SAR was 13 with a median of 12.5. SAR ranged from 0.5-90.2 (Table 3). A soil with an SAR ≥ 13 is considered sodic at a pH > 8.5 and an EC $< 4000 \mu\text{S cm}^{-1}$. High concentrations of Na can negatively impact soil structure, and compete with the Ca and Mg for plant uptake (Davis et al., 2003). Thus, depending on the residual, land application could cause some problems if the residual was surface applied with no incorporation. If a residual with high SAR and potential for dispersion was surface applied with no incorporation, it could possibly seal the surface causing it to be physically dispersed. However, depending on land application rates, the incorporation of such a residual would prevent this problem from occurring.

Elemental concentrations were also measured on the separated water portion (Table 3). Sample concentrations were compared to the EPA Surface Water Quality Criteria for Aquatic Life. Eleven samples exceeded this criteria. Eight samples exceeded the criteria for Zn and three for Cd. It is important to note that the maximum concentration for Cd in this criteria is $2 \mu\text{g L}^{-1}$ which is less than the detection limit of the ICP used for quantification. Based on the results of the metals measured in the liquid portion of the residuals, there is no risk of causing aquatic toxicity if the material was accidentally disposed directly into a surface water body, except for Zn and Cd in a few cases. However, such a toxicity is unlikely or negligible when the residual is used properly as a soil amendment.

Solids Analysis: Nutrients

Mehlich-3 extractions were performed on the solids portion of all the residuals in order to determine concentrations of K, P, Mg, and Ca potentially available for plant uptake (Table 4). When compared to the agronomic optimum (Zhang and Raun, 2006), mean concentrations for K, Mg, and Ca were sufficient, however P would be slightly limiting if the residual was solely used

as a medium for plant growth instead of as a soil amendment. A mean Ca level of 5660 mg kg⁻¹ suggests that a residual could be a source of that nutrient among soils that are limited in Ca.

Metals

The solids portion of all the residuals were digested using method EPA 3050 and analyzed for the trace metals Mn, Ba, Cu, Zn, Cr, Ni, Pb, Co, As and Cd (Table 5). Metal concentrations were compared to EPA Part 503 which regulates the concentrations and loading of metals for land application of biosolids. The strictest of standards set by EPA Part 503 are for 'Exceptional Quality' (EQ) biosolids (EPA 1994). Land application of EQ biosolids requires no permit and can be applied based on the N and P contents of the material only. Metals concentrations in every residual were considerably less than the ceiling for EQ biosolids (Table 5). However, it must be considered that the threshold metal EQ limits were determined based on expected bioavailability in the background matrix of organic matter. This is an important difference from the matrix of the residual samples, which are not rich in organic matter, and will therefore have an impact on the bioavailability of the metals. It is likely that the metal bioavailability of the residual samples will highly vary as a function of the mineralogy of the material. This difference should be taken into consideration when calculating loading rates for land applying HDD residuals. If all residuals examined in this study were land applied at 50 tons solid acre⁻¹ and compared to the EPA cumulative loading rates for biosolids, no samples would exceed the thresholds (Table 7). One sample would exceed the EPA annual loading rate for Cu.

Sample concentrations of metals were also compared to the background concentrations found in Oklahoma soils (Richards et al., 2012). Fifteen samples exceeded these background concentrations, twelve were for Cu. Sample concentrations were also compared to typical levels found in soils throughout the United States (McBride, 1994). Only seven samples were above these levels, of which five were for Cu (Table 5). When compared to the EPA Soil Screening

Levels for residential soils (Table 5), concentrations of two samples exceed the maximum level for As but are far lower than levels found in Oklahoma soils (Richards, et al., 2012).

The modified Synthetic Precipitation Leaching Procedure was also performed on the solids portion of all samples (Table 6). This procedure is used as a means to estimate the leachability of the constituents in a material. Sample concentrations were compared to Maximum Contaminant Levels (MCLs) set by the EPA for drinking water. No samples exceeded the MCLs.

RECOMMENDATIONS AND IMPLICATIONS

There are typically two criteria used to assess a residuals “suitability” for land application: (i) the by-product’s ability to enhance, or have no negative impact, on the productivity of the soil and plant growth, and (ii) a lack of negative environmental impacts (Whalen et al., 2000). Before land applying HDD residuals, one must consider the in situ soil or formation being bored through, from which the residuals originated. If boring through a site with known contamination, performing an analysis for contaminants, such as heavy metals, is suggested. This could potentially be a problem when boring through ground with a historic industrial use. For example, during the industrial revolution, industrial processes such as Cd plating and Cu smelting were commonly conducted outside of the major metropolitan areas. These areas are now referred to as ‘suburbs.’ This is especially true for areas along the east coast. If boring took place in an area where Cu smelting once occurred, drilling residuals would potentially contain high concentrations of Cu.

The EPA Part 503 criteria for metals with regard to land application of biosolids can be used as a guide for maximum allowable metals in a residual to be land applied. One key difference between biosolids and HDD residuals is the organic matter content. Because of the residuals low organic content, the bioavailability of metals is something to take into consideration. Because bioavailability differs with plant species (Kabata-Pendias, 2004), having an estimate of free metal concentration may be more useful. Separation by diffusion using the Donnan membrane technique is one method suggested to measure inorganic contaminants, or free metal concentrations, available for plant uptake (Harmsen, 2007). Other methods include using the

chelating agents diethylenetriaminepentaacetic acid (DTPA) or ethylenediamine tetraacetic acid (EDTA) to extract potentially bioavailable metals (Lindsay and Norvell, 1978; Wear and Evans, 1968). A study in China used the rhizosphere method, which uses low molecular weight organic acids to extract bioavailable metals, and compared results to the DTPA and EDTA methods. Their results showed the rhizosphere method to be the most robust in predicting the bioavailability of Cr, Cu, Zn, and Cd in soils to barely (Feng et al., 2005). However, this study showed that the metals contained in the residuals were poorly soluble in water, thus potentially indicating that bioavailability and mobility would also be minimal.

A residual with a high SAR and/or salt concentration could be a problem, especially if the receiving soil is borderline saline and/or sodic. Unlike oil and gas drilling mud, HDD residuals aren't going to contain geologic salts or return water from deep formations. However, this does not mean that HDD residuals don't have the potential to have a negative impact. Measuring the EC of a residual before it is land applied would be beneficial in determining whether or not it could have a negative impact on the soil that is receiving it. If the SAR of a residual is known to be above 13, incorporating the residual is recommended.

It is not recommended to use HDD residuals solely as a planting medium, although nothing was chemically limiting among the survey residual samples that would limit its use as a soil amendment. Total solids was found to be the most limiting factor. Because of the residuals high clay content, applying too much of a residual could smother the receiving soil and plants, resulting in poor plant growth and minimal water infiltration. If applied at a proper rate, the high clay content could improve a sandy soils ability to retain water and nutrients. In order to minimize environmental impact to the receiving soil and plant life, acceptable HDD residuals (i.e. low metals and salt concentrations) should be applied at a rate ≤ 112 metric tons solid ha^{-1} on ground with a slope $\leq 8\%$ in order to minimize erosion and loss to surface water.

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TABLES AND FIGURES

Table 1. Descriptive statistics for percent carbon (C) and nitrogen (N) and the carbon:nitrogen ratio of the solids portion of the residuals. n=57

	Mean	Median	Min	Max	Std Dev
Carbon	1.42	0.51	0.09	6.61	1.75
Nitrogen	0.04	0.04	0.01	0.26	0.04
C:N	42.89	14.04	2.97	276.39	62.62

Table 2. Descriptive statistics for the electrical conductivity (EC) in $\mu\text{S cm}^{-1}$ and pH for the separated water portion (water) and the “as is” sample (residual). Solids content expressed as gram solid gram total⁻¹. n=58

		Mean	Median	Min	Max	Std Dev
<u>Residual</u>	EC	1198	1124	118	3950	772
	pH	7.5	7.6	4.7	9.9	1.2
	Solids Content	0.35	0.34	0.04	0.72	0.20
<u>Water</u>	EC	1439	1334	241	4296	848
	pH	7.7	8.0	4.2	10.1	1.0

Table 3. Descriptive statistics for the elemental concentrations in mg L⁻¹ and sodium adsorption ratio (SAR) of the water portion of residual samples with ceiling concentrations for the EPA Aquatic Life Criteria. n=58

Element	Mean	Median	Min	Max	Std Dev	EPA Aquatic Life Criteria†
S	326.8	248.2	5.2	2708.3	400.8	NA
Na	285.5	272.7	18.0	856.2	191.2	NA
Ca	47.0	27.2	0.8	584.7	80.9	NA
Fe	16.9	0.4	BD	183.1	40.8	NA
Mg	16.4	10.9	BD	74.9	17.6	NA
K	8.4	5.2	0.7	78.2	11.4	NA
Mn	1.1	BD	BD	23.4	3.3	NA
P	0.6	0.1	BD	26.6	3.5	NA
B	0.2	0.1	BD	1.5	0.3	NA
Mo	0.1	BD	BD	0.6	0.1	NA
Zn	0.1	BD	BD	0.3	0.1	0.12
Cr	BD‡	BD	BD	0.5	0.1	0.57
Cu	BD	BD	BD	0.3	0.1	NA
Cd	BD	BD	BD	0.2	NA§	0.002
Co	BD	BD	BD	0.1	NA	NA
As	BD	BD	BD	BD	NA	0.34
Pb	BD	BD	BD	BD	NA	0.065
SAR	13.0	12.5	0.5	90.2	12.8	NA

†USEPA 2014. National Recommended Water Quality Criteria
<http://water.epa.gov/scitech/swguidance/standards/criteria/current/index.cfm#altable>

‡ Below Detection Limit

§ Not Applicable

Table 4. Descriptive statistics for Mehlich-3 extractable element concentrations in mg kg⁻¹ from the solids portion of residual samples. n=56

Element	Mean	Median	Minimum	Maximum	Std Dev
K	156.5	121.0	9.2	658.4	130.0
P	23.1	8.6	BD†	274.6	50.9
Mg	438.8	436.3	12.7	1318.4	310.7
Ca	5660.0	3569.4	181.8	39320.2	9060.8
Na	2198.3	844.0	41.7	17150.6	3496.1
Mn	142.0	71.0	2.2	1443.7	240.7
Fe	222.3	175.8	5.9	760.7	166.2
Al	512.3	341.1	10.1	1967.0	499.9

† Below Detection Limit

Table 5. Descriptive statistics for total elemental concentrations measured in solid residual samples, in mg kg⁻¹, as determined by EPA 3050 solids digestion. Values shown in comparison to typical levels found in all soils, Oklahoma soils, EPA 503 ceiling concentrations for exceptional quality (EQ) biosolids, and EPA Soil Screening Levels for Residential Soils. n=58

Element	Mean	Median	Min	Max	Std Dev	Typical Levels in Soil†	Typical Levels in Oklahoma Soils‡	EPA Ceiling Concentration for EQ biosolids§	EPA SSL for Residential Soils¶
Ca	23835.6	5751	192	223986	40309.9	NA	NA	NA	NA
Fe	16971.6	14361.5	570	51185	11299.1	NA	NA	NA	NA
Al	12814.1	10763.5	2454	42668	8960.3	NA	NA	NA	NA
Mg	5564.9	3641	102	34074	7333.5	NA	NA	NA	NA
Na	2157.7	829	51	16221	3313.7	NA	NA	NA	NA
K	1341.5	1087.5	85	6614	1209.1	NA	NA	NA	NA
S	971.8	549	19	6236	1186.5	NA	NA	NA	NA
P	460.6	287	30	5753	826.2	NA	NA	NA	NA
Mn	301	238	BD	1606	306	80-1300	27-2165	NA	NA
Ba	105.9	57.6	9.2	974.1	142.9	NA	NA	NA	5500
Cu	66.1	11	BD	1863	252.2	6-80	2-33	4300	NA
Zn	39.9	30.5	BD	341	46.4	17-125	15-142	7500	23000
B	37	31.2	BD	194.6	31.7	NA	NA	NA	NA
Cr	16.8	13.4	1.9	100.1	15.3	7-221	4-70	3000	230
Ni	11	8.5	BD	43.3	9.4	4-55	2-57	420	1600
Pb	2.4	BD	BD	58.4	8.3	10-84	3-32	840	NA
Co	2.3	BD	BD	15.6	4.1	1-22	NA	NA	NA
As	BD#	BD	BD	1.6	0.2	4-9	1-34	75	0.4
Cd	BD	BD	BD	BD	NA††	0.06-1.10	0.13-0.80	85	70

† McBride, 1994

‡ Richards, et al., 2012.

§ USEPA 1994. Land Application of Biosolids. USEPA.

http://water.epa.gov/scitech/wastetech/biosolids/upload/2002_06_28_mtb_biosolids_503pe_503pe_2.pdf

¶ USEPA 2002. GENERIC SSLs FOR THE RESIDENTIAL AND COMMERCIAL/INDUSTRIAL SCENARIOS

http://www.epa.gov/superfund/health/conmedia/soil/pdfs/ssg_appa-c.pdf

Below Detection Limit

†† Not Applicable

Table 6. Descriptive statistics for the elemental concentrations (mg L⁻¹) for analytes from the modified synthetic precipitation leaching procedure (SPLP) performed on the solids portion of residuals. Values compared to maximum levels allowed by EPA Drinking Water Criteria. n=61

Element	Mean	Median	Min	Max	Std Dev	EPA Drinking Water Criteria†
S	75.3	30.3	3.4	527.7	98.6	NA
Na	63.5	38.4	1.9	386.9	74.5	NA
Al	38.7	21.6	BD	204.1	51.0	NA
Fe	15.4	9.0	BD	105.0	20.6	NA
Ca	12.7	7.4	0.1	239.6	30.3	NA
Mg	6.3	4.1	BD	42.1	8.4	NA
K	4.1	3.6	BD	21.5	5.1	NA
B	0.6	0.7	0.1	1.6	0.4	NA
P	0.3	0.1	BD	5.5	0.8	NA
Mn	0.1	BD	BD	2.3	0.3	NA
Ba	0.1	0.1	BD	0.3	0.1	2.0
Cu	BD‡	BD	BD	2.1	0.3	1.3#
Zn	BD	BD	BD	0.2	0.1	NA
Cr	BD	BD	BD	0.1	NA¶	0.1
Ni	BD	BD	BD	0.2	NA	NA
Mo	BD	BD	BD	0.1	NA	NA
Co	BD	BD	BD	0.1	NA	NA
As	BD	BD	BD	BD	NA	0.01
Cd	BD	BD	BD	BD	NA	0.005
Pb	BD	BD	BD	BD	NA	BD

† USEPA 2014. Drinking Water Contaminants
<http://water.epa.gov/drink/contaminants/>

‡ Below Detection Limit

¶ Not Applicable

At Tap

Table 7. Calculated metal loading rates (kg ha^{-1}) for each residual sample tested in the study, assuming application rates of 112 metric tons solid ha^{-1} . Metal loading rates are compared to EPA Cumulative and Annual Pollutant loading rates for biosolids.

Sample	Zn	Cu	Ni	As	Cd	Cr	Pb
1	0.9	0.2	0.3	BD†	BD	3.1	BD
2	8.2	53.6	2.2	BD	BD	4.5	BD
3	3.7	1.1	1.1	BD	BD	1.5	BD
4	2.5	0.9	1.2	BD	BD	2.0	0.1
5	7.8	5.5	3.3	BD	BD	2.8	0.1
6	5.5	3.2	2.6	BD	BD	1.9	BD
7	2.0	1.3	0.9	BD	BD	1.0	BD
8	2.4	1.5	0.8	BD	BD	1.8	0.5
9	1.6	16.8	0.9	BD	BD	3.8	BD
10	0.7	11.5	0.4	BD	BD	2.9	BD
11	4.8	1.6	1.8	BD	BD	1.5	0.1
12	4.6	1.6	1.8	BD	BD	1.8	BD
13	1.8	2.3	0.7	BD	BD	0.7	BD
14	3.8	1.4	1.2	BD	BD	2.6	BD
15	7.1	2.6	3.2	BD	BD	2.7	BD
16	4.3	2.1	1.3	BD	BD	1.3	BD
17	1.3	0.1	0.3	BD	BD	1.0	0.9
18	1.4	0.9	0.3	BD	BD	0.9	0.4
19	2.3	0.9	1.3	BD	BD	1.2	BD
20	2.5	1.0	1.0	BD	BD	1.0	BD
21	8.4	49.7	1.8	BD	BD	1.8	2.1
22	7.9	230.2	1.7	BD	BD	1.9	2.1
23	8.0	4.2	0.3	BD	BD	0.9	BD
24	2.3	3.7	0.2	BD	BD	5.4	BD
25	3.6	2.7	1.3	BD	BD	1.4	BD
26	42.2	4.7	2.4	0.2	BD	2.2	7.2
27	3.8	BD	2.2	BD	BD	12.4	BD
28	BD	BD	0.1	BD	BD	0.7	0.3
29	4.0	4.0	1.6	BD	BD	2.3	BD
30	1.9	1.2	0.9	BD	BD	2.3	BD
EPA Cumulative Pollutant Loading Rate§	2800	1500	420	41	39	3000	300
EPA Annual Pollutant Loading Rate§	140	75	21	2	1.9	150	15

† Below Detection Limit

§ USEPA 1994. Land Application of Biosolids. USEPA.

http://water.epa.gov/scitech/wastetech/biosolids/upload/2002_06_28_mtb_biosolids_503pe_503pe_2.pdf

Table 7. Continued

Sample	Zn	Cu	Ni	As	Cd	Cr	Pb
31	1.4	0.2	0.5	BD†	BD	0.7	BD
32	8.6	1.2	0.8	BD	BD	1.1	BD
33	1.6	1.3	0.5	BD	BD	0.5	0.5
34	12.3	4.1	0.6	BD	BD	0.7	1.1
35	0.6	0.1	0.3	BD	BD	0.6	0.2
36	0.6	3.1	0.3	BD	BD	0.8	0.9
37	6.8	4.8	5.3	BD	BD	6.1	BD
38	5.7	1.4	1.3	BD	BD	2.0	BD
39	4.3	1.2	2.4	BD	BD	1.8	0.8
40	8.1	3.0	3.6	BD	BD	3.5	BD
41	10.6	6.9	4.7	BD	BD	4.6	BD
42	2.1	0.7	0.3	BD	BD	0.4	BD
43	5.9	1.4	2.2	BD	BD	2.4	BD
44	2.1	1.0	0.3	BD	BD	0.9	BD
45	6.7	0.8	1.8	BD	BD	0.8	BD
46	4.7	0.3	0.5	BD	BD	0.2	BD
47	2.9	1.3	0.5	BD	BD	0.5	BD
48	4.5	0.4	0.9	BD	BD	1.0	BD
49	3.1	0.1	2.8	BD	BD	3.6	BD
50	1.7	0.8	0.6	BD	BD	1.1	BD
51	2.5	1.0	0.4	BD	BD	3.0	BD
52	3.3	4.6	2.1	BD	BD	1.6	BD
53	3.6	1.4	1.7	BD	BD	1.3	BD
54	2.7	0.3	0.4	BD	BD	0.3	BD
55	4.9	2.0	2.2	BD	BD	2.3	BD
56	5.3	2.8	2.9	BD	BD	1.5	BD
57	7.1	9.9	BD	BD	BD	1.7	BD
58	11.4	6.8	BD	BD	BD	4.3	BD
EPA Cumulative Pollutant Loading Rate	2800	1500	420	41	39	3000	300
EPA Annual Pollutant Loading Rate	140	75	21	2	1.9	150	15

† Below Detection Limit

CHAPTER III

LAND APPLICATION OF HORIZONTAL DIRECTIONAL DRILLING RESIDUALS TO ESTABLISHED AND BARE SOILS

ABSTRACT

Horizontal directional drilling (HDD) is a non-invasive alternative to trenching used for the installation of many common utilities. The process involves drilling fluid used to stabilize the borehole, lubricate the bit, and float cuttings to the surface. Currently, landfilling is the most popular method of disposal for the spent drilling fluid. A more economical method of disposal to consider is land application. A recent study found total solids to be the most limiting factor for the land application of most HDD residuals. Two field studies were conducted in order to determine the optimum application rate of HDD residuals. Residuals were applied at six rates to both established grass and bare soils that received broadcast bermudagrass application prior to application of residuals. Soils were sampled 7, 30, and 90 days after application. Saturated paste and Mehlich-3 extractions were performed on all samples. No significant nutrients or trace metals were found in the treated soils. All samples fell in the range for a typical soil. However, TDS and sodium absorption ratio increased with application rate. Forage was harvested on the established grass plots 110 days after application. There was no significant difference in forage yield. Percent cover was measured on the bare soils that received bermudagrass seed at approximately 30 and 60 days after application. At day 60 plots that received the lowest rate had percent cover significantly higher than the control, and plots that received the highest rate had significantly less cover.

INTRODUCTION

In the last three decades there have been many scientific studies generating a great deal of information on the benefits and environmental impacts associated with the land application of many types of residuals (O'Connor et al., 2005). There are typically two criteria used to assess a residuals “suitability” for land application and whether or not the intended use will be beneficial: (i) the by-product’s ability to enhance, or have no negative impact, on the productivity of the soil and plant growth (ii) a lack of negative environmental impacts (Whalen et al., 2000). Because residuals differ so much in their physical, chemical, and biological properties (Whalen et al., 2000), each material should be examined independently for potential beneficial or negative impact

There are many by-products that are currently being land applied. With the recent boom in oil and gas activity in Oklahoma, the land application of spent oil and gas drilling mud has become a very popular method of disposal. Land application is the most attractive option to the industry due to the minimization of hauling distances to disposal sites (Clements et al., 2010). Adverse environmental impacts to both soil and plant growth after the land application of oil and gas drilling fluid may occur due to the high concentrations of soluble salts, which are found primarily in water based mud. Analysis of the water based drilling mud used in a study in Colorado indicated that sodium (Na), Cd and Mo were the only constituents found in higher concentrations than a typical soil. Ranges for Na, Cd and Mo were 9900-20000, 1.1-4.7 and 0.2-9.3 mg/kg respectively (Bauder et al., 2005). However, by adhering to the guidelines set by the Oklahoma Corporation Commission (OCC), land application can be a sustainable solution for the disposal of oil and natural gas drilling muds, with minimal long term impact to the receiving soil.

Biosolids are another by-product commonly land applied. Waste water treatment plants receive domestic sewage as well as industrial effluents and storm water runoff. Industrial effluents and storm water runoff can contain many toxic substances ranging from detergents to pesticides (Singh and Agrawal, 2008). However, biosolids contain both organic and inorganic plant available nutrients as well as soil conditioning properties, giving incentive for its use as a source of fertilizer (Singh and Agrawal, 2008). The range of N, phosphorus (P) and potassium (K) percentage in biosolids are 1.5-5.0, 0.5-2.8, and 0.0-1.0 respectively and median levels of Pb, Zn, Cu, Ni, Cd and Cr to be 500, 1740, 800, 80, 10 and 500 mg/kg respectively (Girovich, 1996). Because biosolids have the potential to be both beneficial and detrimental to the environment, there is need for regulation. The land application of biosolids is federally regulated by the EPA under 40 CFR Part 503 (EPA 2007). This law governs the use of biosolids for land application by putting limits on heavy metal concentration, pathogens present, and site and harvesting restrictions in order to minimize threats to public health and environmental degradation (Guo, 2012).

Another industrial by-product commonly land applied is fly ash. In the past fly ash was considered solely as a waste material but, thanks to numerous detailed studies conducted in the latter half of the 20th century, we now know its nature and composition. Due to the abundance of basic mineral elements resembling the Earth's crust, fly ash is an excellent replacement for natural materials (Jala and Goyal, 2006). The United States utilizes roughly 44% of its annual production of fly ash (American Coal Ash Association, 2012). It can be used as a partial substitute for Portland cement, road fills and bases, in asphalt, and for waste solidification and stabilization (Adriano and Weber, 2001). The application of fly ash to agricultural land cannot supply crops with the macronutrients nitrogen N or P, but it could supply crops with a sufficient amount of nutrients such as potassium (K), sulfur (S), Mo, calcium (Ca), boron (B), as well as others such as Zn (Sajwan et al., 2003). Land application of alkaline fly ash can reduce the

acidity of a soil, making certain toxic metals unavailable for plant uptake. Unfortunately, fly ash can also contain different amounts of trace metals such as Cu, Ni, Pb, and Cr which does put a limit on its potential use as a soil amendment. Analysis of fly ash from a coal based thermal power plant in the U.S. showed levels of As, Cr, Pb, Cu, and Zn to be 157, 154, 553, 214 and 3327 mg/kg respectively (Jegadeesan et al., 2008). Currently, individual states regulate the disposal of fly ash, but the EPA is proposing two federal regulatory options, Subtitle C and Subtitle D, under the nation's primary law for regulating solid waste, RCRA (EPA 2013). Each option will better protect the environment and allow both federal and state enforcement.

In contrast to animal manure, biosolids, and fly ash, there is much less known about the composition and potential impact of land application of urban horizontal directional drilling (HDD) residuals. Today there is growing speculation about HDD residuals and the safety of their constituents and chemical makeup. They are often confused with "drilling mud" from the oil and gas industry even though the two by-products are very different. There is a need to separate the public's perception of the spent HDD drilling residuals from that of the oil and gas industry. The cost of hauling spent residuals (sometimes long distances) to landfills costs a lot of money and takes a lot of time. In a survey conducted with the research mentioned above, some contractors reported the need to dispose of over 200,000 L of spent HDD residuals every week. To date, there is no published research on the use of spent HDD residuals or their environmental impact as a media for land application.

In a previous study, 58 different HDD residuals from 26 different states were characterized in terms of their physical and chemical makeup. Analysis included: solids content, EC and pH of the "as is" residual portion, total soluble salts and pH of the separated water portion, total metals in the separated solids portion, water soluble constituents of the separated water portion, sodium absorption ratio (SAR), Mehlich-3 extractable elements from the separated solids portion, Synthetic Precipitation Leaching Procedure (SPLP) of the separated solids portion, and total

carbon:total nitrogen of the separated solids portion. Results of all the analysis were compared to many different standards for soils, surface waters, and groundwater in order to determine if land application would be a safe method of disposal for the spent HDD residuals. That study found that there was low potential for the residuals to contain anything that would be chemically limiting to land application of residuals, although some samples had elevated levels of total dissolved solids and sodium. The sodium and total dissolved solids may have originated from some of the ingredients in the drilling fluid or the subsoil that was being drilled at the site. Total solids was found to potentially be the most limiting factor.

For land application to be a viable option for the disposal of spent HDD residuals, more research is needed in order to determine any impacts these residuals might have on the soil and vegetation receiving them. With solids potentially being the most limiting factor for land application, proper application rates need to be determined in order to minimize any potential negative environmental impact. There are two likely scenarios to consider when deciding where HDD residuals might be land applied. HDD is typically used in a municipal setting or at a construction site. In a municipal setting a contractor would likely find the nearest pasture or field to land apply his/her spent residual. In a situation where boring is done on a construction site the contractor will likely want to leave onsite because the soil is typically already bare. Thus, the objective of this research was to determine proper application rates at which HDD residuals may be applied with minimal environmental and agronomic impact to the soil and vegetation, for both established grass plots and bare soils.

MATERIALS AND METHODS

The field studies were conducted on pasture land approximately 5 km northwest of Stillwater, OK (USA). The soil was a Zaneis clay loam (fine-loamy, siliceous, active, thermic Udic Argiustolls), a well-drained soil found on slopes ranging from 0-8%. The dominant forage is bermudagrass (*Cynodon dactylon*). Mean annual precipitation is 86.36 cm. The field study included the establishment and monitoring of two field trials upon which HDD residuals were applied to both established grass and bare soils at six different application rates.

Residuals were obtained from a local contractor and transported to the site using a Ditch Witch MV800 vacuum excavator and then transferred into a 3785 L mixing tank. Residuals were mixed for approximately 20 minutes prior to acquiring a sub-sample for all analysis to be performed. Solids content (gravimetric) was determined by shaking samples for 20 minutes followed by subsampling and placement in an oven at 30°C for 24 hours. In order to conduct several analyses, the solids portion was separated from the liquid by centrifugation at 5000 rpm for 10 minutes followed by filtration with a 41 Whatman paper.

For the solids portion, samples were extracted via total soil digestion using EPA 3050 method (EPA 1996) using two grams of solid residual, trace metal grade nitric acid (HNO_3) and trace metal grade hydrochloric acid (HCl), and a 30% sodium hydroxide solution, in conjunction with heat. Following digestion, analytes were filtered using 41 Whatman paper and brought to volume with deionized (DI) water in 50 mL volumetric flasks. Samples were then analyzed for various metals including aluminum (Al), sodium (Na), sulfur as sulfate ($\text{SO}_4\text{-S}$), calcium (Ca), magnesium (Mg), phosphorus (P), boron (B), barium (Ba), potassium (K), chromium (Cr),

manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), arsenic (As), and lead (Pb). Analytes were quantified using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP).

A modified version of the synthetic precipitation leaching procedure (SPLP) was also performed on the solids portion in order to simulate potential leachability to groundwater (Hageman et al., 2000). Extraction fluid was made using DI water acidified to a pH of 4.2 using a 60/40 ratio solution of trace metal grade nitric acid (HNO₃) to reagent grade sulfuric acid (H₂SO₄). Solids were extracted at a 1:20 solid:solution ratio for 18 hours followed by centrifugation at 5000 RPM for 10 minutes and filtration through .45 µm glass fiber filters using vacuum filtration. Extracts were analyzed for Mo, Al, B, Na, SO₄-S, Ca, Mg, P, K, Cr, Mn, Fe, Co, Cu, Zn, Cd, As, and Pb by ICP.

The water portion was separated using a centrifuge at 5000 RPM's for a minimum of 10 minutes. The separated water portion was then filtered using 42 Whatman paper. Samples were then analyzed for Mo, Al, B, Na, SO₄-S, Ca, Mg, P, K, Cr, Mn, Fe, Co, Cu, Zn, Cd, As, and Pb by ICP. This will be referred to as "water soluble". Results were compared to EPA Aquatic Life Criteria Table (EPA 2013). Sodium adsorption ratio was calculated for each sample:

$$SAR = \frac{Na}{\sqrt{\frac{(Ca + Mg)}{2}}}$$

Where Na, Ca, and Mg are in units of meq L⁻¹. Electrical Conductivity (EC) and pH were tested on both the solid residuals (prior to solid/liquid separation) and their separated water portion using standard EC and pH meters. Plant available nutrients that were analyzed included P, K, Mg, Ca, Mn, Na, Fe, SO₄-S, Zn, Cu, and B. Of those, Mehlich-3 extraction was used for K, P,

Mg, Ca, Mn, and Fe (values from the saturated paste extractions were used for the nutrients Na, SO₄-S, Zn, Cu and B). Mehlich 3 nutrients were extracted at a ratio of 1:10 solids:solution for five minutes, followed by filtration with Whatman #42 filter paper and analysis by ICP (Mehlich, 1984). Total N and C were quantified using LECO TruSpec Analyzer (Leco Corporation, St. Joseph, MI).

Individual plot dimensions were 3.05 X 3.05 m with 4.6 m alleys. Each plot received residuals representing 0, 22, 45, 67, 90 or 112 metric tons of solid ha⁻¹. Each application rate was replicated three times resulting in 18 experimental units for each study. Residual application rates were achieved by applying the slurry, which had a known solids content, through a hose for various calculated time periods as a function of the calibrated and pre-determined flow rate.

For the established grass field study, plots were sampled 0, 7, 30 and 93 days after application.

For the bare soil field study, plots were sampled 0, 7, 29, and 96 day after application. Samples were separated by depth, 0-5cm and 5-15 cm. Samples were then dried in an oven for approximately 48 hours then ground using a Bico Soil Pulverizer.

Saturated paste extractions were performed on every sample using 150 grams of dried sample and deionized (DI) water. After mixing, samples sat for one hour prior to extraction. Samples were extracted using a Baroid pneumatic soil press. Analytes were measured for Na, Ca, Mg, K, S, B, P, Fe, Zn, Cu, Mn, Al, Ni, Mo, As, Ba, Cd, Co, Cr and Pb via inductively coupled plasma atomic spectroscopy (ICP). EC and pH were also measured on each extract using standard EC and pH meters.

Mehlich-3 extractions were used to measure plant available K, P, Mg, Ca, Mn, and Fe. Two grams of sample were used in conjunction with 20 mL of Mehlich 3. Samples were reacted for five minutes then filtered using 42 Whatman paper. Extracts were quantified using ICP.

Established Grass Plots

In order to simulate a typical hay pasture, plots were only mowed prior to residuals application. A Carter harvester was used to harvest forage in order to determine biomass approximately 120 days after residual application. Forage was then dried in an oven for approximately 48 hours then ground using a Fritsch Pulverisette 19. Forage was then digested using concentrated HNO₃ and a heating block. Digests were analyzed for Ca, P, Na, Mg, K, S, Mn, Cu, Fe and Zn, quantified using ICP.

Bare Soil Plots

For the bare soil field study, the vegetation and first layer of top soil were removed with a skid steer loader in order to simulate a typical construction site that has exposed subsoil. Prior to residual application, every plot was seeded with Sta-Green bermudagrass seed (Spectrum Brands Middleton, WI). A rotary spreader was used to spread the seed at the application rate of 1.15 kg of pure live seed per 93 m². Percent cover was calculated at day 29 and 63 using a digital camera and SamplePoint software. Three pictures were randomly taken within each plot. In order to standardize the photos, pictures were taken using a pole with a camera mount approximately 100 cm up from the ground. One hundred evenly spaced pixels on every picture were determined to either have cover or no cover. Values for all three pictures were then averaged to get an overall percent cover for the entire plot.

Statistics

Soil chemical data, forage biomass, and percent cover were analyzed using SAS (SAS Institute, 2002). An ANOVA model of the response variables we created using PROC GLM routine. The ANOVA model used application rate, time, depth, and replications as treatment effects. Type III least square means from the PROC GLM routine were used for mean separation tests using the PDIFF option. Mean separation for all treatments were considered significant at the $P < 0.05$ level.

RESULTS AND DISCUSSION

Background Soil Properties

Background soil tests for the established grass plots indicated P was 99 to 75% sufficient for bermudagrass with concentrations ranging from 8 to 30 mg kg⁻¹ at depths of 0-5 and 5-15 cm. Soil tests for K indicated 100 to 92% sufficient for bermudagrass with concentrations of 96 to 186 mg kg⁻¹ at the two depths. Soil tests for the bare soil plots indicated P was 99 to 100% sufficient for bermudagrass with concentrations ranging from 31 to 38 mg kg⁻¹ at the two depths. Soil tests for K indicated 83 to 90% sufficiency for bermudagrass with concentrations 73 to 91 mg kg⁻¹ for the two depths.

The residuals applied to the established grass plots had a solids content of 39%, and an EC, pH, and SAR of 1049 $\mu\text{S cm}^{-1}$, 8.2, and 16 respectively. In the separated water portion, Na was the most abundant constituent at 334.2 mg L⁻¹. In separated solids portion, Mn was found to have the highest concentration at 1444 mg kg⁻¹. Mehlich 3 extractable Ca was very high at 28526 mg kg⁻¹ (Mehlich 1984).

Residuals applied to the bare soil plots had a solids content of 28% and an EC, pH, and SAR of 5417 $\mu\text{S cm}^{-1}$, 8.0, and 19 respectively. Like the residual applied to the established grass plots, Na was the most abundant constituent in the separated water portion at 1224.6 mg L⁻¹. In the separated solids portion, Mn was found to have the highest concentration at 92 mg kg⁻¹. Of the Mehlich 3 extractable elements Ca was the highest, however much lower than in the residuals applied to the established grass plot, with a concentration of 3766 mg kg⁻¹ (Mehlich 1984). All chemical and physical properties of the residuals applied to the established grass and bare soil plots can be seen in Tables 1 and 2 respectively.

Established Grass Plots

Considering all sample times, mean pH ranged from 6.3-8.0 in the top 5 cm of soil and 6.7-7.9 at the 5-15 cm depth (Table 3). Application rate, time, and depth were all significant with an interaction between depth and time at $P=0.05$. The residuals applied to each plot had a pH of 8.2 so an increase in pH in the 0-5 cm soil with an increase in application rate was plausible, but was only statistically significant at day 93.

As expected, the addition of residuals to the established grass increased the EC of the receiving soil as application rate increased, with a mean EC range of 463-748 $\mu\text{S cm}^{-1}$ averaged over depth and time. Application rate, time, and depth were all statistically significant. The application rates of 67, 90, and 112 metric tons solid ha^{-1} averaged across depth and sample time, were significantly higher than the control (Table 4). Soil EC was also significantly different between depths with a mean EC of 887 $\mu\text{S cm}^{-1}$ in the 0-5 cm depth and 420 $\mu\text{S cm}^{-1}$ at the 5-15 cm depth (Table 5). The EC of the residual applied was 1049 $\mu\text{S cm}^{-1}$ so an increase in EC in the 0-5 cm layer with an increase in application rate was expected. Over the 93 day sampling period 23.6 cm of precipitation was received, however no significant leaching of salts was observed. This could be a result of the extreme heat coupled with transpiration of soil moisture between rain events, wicking salts back up in the profile.

Copper, Zn, and Ba were the only trace metals detected in the saturated paste extracts, with no values exceeding 1.1 mg L^{-1} . This suggests that the trace metals applied with the residuals were not water soluble and therefore relatively low bioavailability. There was no statistical difference between application rate, depth, and time for trace metals. Soil SAR was also not statistically different between application rate, depth or time, with a maximum value of 3.1. Loading rate for total dissolved solids at the maximum application rate of 112 metric tons solid ha^{-1} was 145 kg ha^{-1} . This is well below the limit of 6724 $\text{kg total dissolved solids ha}^{-1}$ set by the OCC for the land application of water-base drilling mud from oil and gas exploration. The rules for water-base

drilling mud application are stated in the Oklahoma administrative code and register, Title 165:10-7-19. It is highly unlikely that 145 kg salts ha⁻¹ would have a negative impact on vegetation. If the loading rate were considerably higher, it could possibly have a negative impact on some of the more salt sensitive species depending on the concentration of salts already present in the receiving soil.

The residual applied to the plots contained appreciable concentration of Mehlich-3 extractable Ca at 28,526 mg kg⁻¹ (Table 1). Application rate and depth were both significant for Ca with an interaction between application rate and depth (Table 6). In the top 0-5 cm of soil, concentrations of Mehlich-3 Ca increased with application rate with mean concentrations ranging from 1459 mg kg⁻¹ for the control and 7672 mg kg⁻¹ at the highest application rate of 112 metric tons solid ha⁻¹. This particular residual would be a good soil amendment if applied to a Ca deficient soil, however Ca deficient soils are uncommon in Oklahoma (Zhang and Raun, 2006).

At 110 days after HDD residuals application, there was no significant difference in forage yield, by weight, for all application rates of residuals applied when compared to the control. Forage yield had a range of 7.4 – 12.8 metric tons ha⁻¹. Forage was also digested and analyzed for the trace metals Cu, Zn, Ni, As, Cd, Cr, Ba, Pb, and Co. The control had higher concentrations of metals compared to forage samples from treated plots, except for Ni and Cr. Results were highly variable with no statistical significance among application rates.

Bare Soil Plots

Mean pH ranged from 7.8 to 7.9 in the top 0-5 cm and from 7.4 to 7.8 at the 5-15 cm depth (Table 7). Time of sampling and depth were statistically significant with an interaction between application rate and depth at P=0.05. With pH values for both residuals applied to the field plots ≥ 8.0 , their application to an acidic soil could be beneficial. For both plot studies (established

grass and bare soil), the pH for the majority of treated-plots in the top 0-5 cm was slightly higher than the 5-15 depth for both the established grass and bare soil plots (Tables 3 and 7).

The applied residuals increased the EC of the receiving soil as a function of application rate. Application rate and depth were statistically significant with a two way interaction between application rate and depth and a two way interaction between depth and sampling time. There was also a three way interaction between application rate, sampling time, and depth at $P=0.05$. Table 8 shows that soil EC increased with residuals application rate, at each sampling time. This was expected since the residuals applied (Table 2) up to 1214 kg TDS ha⁻¹, at the highest residual application rate. The highest EC value, 3545 $\mu\text{S cm}^{-1}$, was measured at day 7 for the highest application rate. An EC of this magnitude is approaching the 4000 $\mu\text{S cm}^{-1}$ limit for a saline soil, and could possibly adversely affect salt sensitive plants. Because of this high EC value incorporation would be recommended if applying a similar residual at the 112 metric tons ha⁻¹ application rate. Table 9 illustrates the leaching of the applied salts, as evident by a general trend of decreasing EC values with time in the surface 0-5 cm depth, concurrent with an increasing trend at the subsurface 5-15 cm depth. This was expected because of the ample amount of precipitation received; 10.14 cm rainfall within a 22 day time period combined with the lack of transpiration since the plant was just beginning to become established.. This trend was not as obvious between days 29 and 96. EC actually increased in the top 0-5 cm layer in the control and at the application rates of 22 and 112 metric tons solid ha⁻¹, although not significantly (Table 9). During this 67 day time period, only 16.03 cm was received with an average temperature well into the 90's, so this is likely a result of wicking salts up through the soil profile as soil moisture was evaporated and transpired as the plant developed.

There was no statistical difference between application rates, depth, or sample time, with regard to trace metals measured in soil saturated paste extracts. Concentrations for Ba were all less than 1.0 mg L⁻¹ and all other metal concentrations were ≤ 0.05 mg L⁻¹. Maximum mean value for SAR

was 7.8 at the 5-15 cm depth for the control. Application rate and depth were both significant for SAR with an interaction between application rate and depth (Table 10). As expected, increasing application rate resulted in a greater SAR at the 0-5 cm depth. Notice that the SAR was greater for bare soil plots that received residuals compared to the established grass plots; this was due to the higher SAR content in the residuals applied to the bare soil plots (Tables 1 and 2). SAR values observed in this field study are of little concern with a loading rate of only 429 kg ha⁻¹ Na when applied at the highest application rate of 112 metric tons solid ha⁻¹. However, applying the same residual with a SAR of 19, to a soil that already has a high SAR would likely have negative implications. A soil is considered 'sodic' when the SAR ≥ 13, the EC < 4000 μS cm⁻¹, and the pH > 8.5. A 'saline-sodic' soil has a SAR ≥ 13, an EC > 4000 μS cm⁻¹, and a pH < 8.5. Sodic soils typically have poor structure and have a tendency to crust, restricting water infiltration (Davis et al., 2003). High concentrations of Na compete with plant available Ca and Mg which can limit plant growth. Application of a high SAR residual to a bare soil with no incorporation could create a thin dispersed layer at the surface, which could possibly inhibit germination of seed and also inhibit the infiltration of water.

When applied at the maximum application rate of 112 metric tons solid ha⁻¹, loading rates for TDS was 1214 kg ha⁻¹. This is much higher than the residuals applied to the established grass plots, but still well below the OCC threshold of 6724 kg ha⁻¹ for the land application of water-base oil and gas drilling mud. This loading rate could potentially have a negative impact on species of plants that are salt sensitive. This loading rate would be of even more concern if the receiving soil had high concentrations of salt prior to application. If the receiving soil had high salt concentrations and a pH ≤ 8.5 prior to application, this loading rate could have the potential to make the receiving soil 'saline', if EC levels were driven above 4000 μS cm⁻¹.

Compared to the residual applied to the established grass plots, there was less Mehlich-3 extractable Ca in the residual applied to the bare soil plots. Due to the lack of base cations applied, there was no significant difference between application rates, depth, or sampling time.

Twenty-nine days after residuals application to bare soil plots seeded with bermudagrass, percent cover was not significantly different from the un-amended control compared to any treatment. At day 63, mean percent cover for plots receiving the lowest application rate of residual applied, 22 metric tons solid ha⁻¹, was 75% and was significantly higher than the control at 57% (Figure 1). Mean percent cover for plots receiving the highest application rate, 112 metric tons solid ha⁻¹, was 44% and was significantly lower than the control. Growth in all plots was much faster between days 30 and 63 than days 0 and 30 (Figure 2). The measurement of cover at day 63 suggests two things. First, at a lower application rate of residuals at 22 metric tons solid ha⁻¹, might aid in the germination of bermudagrass seed. Second, at the highest application rate of 112 metric tons solid ha⁻¹, applied residuals possibly hinder germination. All other application rates were not significantly different from the control. Cover consisted of nearly 100 % bermudagrass. One explanation for the increase in germination at the lowest application rate is that the residuals applied some cover to the applied seed under it. By applying a small amount of residual to exposed surface seed, the seed is essentially being buried or incorporated, unlike the control. The seed is also receiving a source of water with residual application, unlike the control. It is possible that low application rates of the residual may have improved water retention in the thin layer where the seed was located. Low germination at the highest application rate could possibly be a result of the seed being excessively buried at too great a depth. After drying, a layer of clay from the residual application, could be difficult for a germinating seed to penetrate if it is excessively deep.

RECOMMENDATIONS AND IMPLICATIONS

The land application of any by-product needs to be conducted in a manner that is sustainable and not harmful to the environment or human health. In order to do this properly the physical or chemical limiting factor should be known, which may be specific to each individual HDD residual material. For the two HDD residuals used in these field studies the most limiting factor was total solids, which supports the results of the survey-characterization study conducted on residual samples from around the U.S. (Chapter 2). However, the limiting factor for land application of HDD residuals could vary depending on where the boring is taking place. If boring through a site with historic heavy metal contamination, metal concentrations would likely be the most limiting factor for land application. For this reason, testing the residual when boring through a known contaminated site is highly recommended.

An increase in soil pH in the top 0-5 cm was observed in both field studies. Soda ash (sodium carbonate) is a common additive used to increase the pH of HDD residuals when necessary. However, not all residuals are alkaline as observed in the survey study. In the survey study, pH ranged from 4.7 to 10.0. Thus, any potential to raise pH in the top 0-5 cm of soil with the application of HDD residuals would vary depending on the residual being applied, and any long term effect would depend on the receiving soil's buffering capacity and organic matter content.

In both field studies EC increased in the top 0-5 cm as application rate increased. Therefore a residuals EC could potentially be a limiting factor. This is especially true if the receiving soil's EC is already high. Application rates exceeding the rate of 112 metric tons solid ha⁻¹ could

potentially be harmful to salt sensitive plants depending on the residual's initial EC as well as the EC of the soil receiving the amendment.

If a residual with a high concentration of water soluble metals, in the context of aquatic life or drinking water criteria, were applied, it does not necessarily mean that they would remain water soluble after application to the soil. Depending on the mineralogy, organic matter content, and pH of the receiving soil, the constituents would be retained by the soil through several processes such as ligand exchange/chemisorption, precipitation, or ion exchange (Essington, 2004), thus making them mostly unavailable for plant uptake. Results from the digestion of forage from the established grass plots implies nothing added was bioavailable because concentrations of almost all metals were higher in the control than any other sample.

As observed for the Mehlich-3 extracts from the residuals applied to the established grass plots, HDD residuals have the potential to be a source of secondary nutrients. This will vary depending on the source of the residual. If boring through a soil or formation that is rich in nutrients, the residual will likely contain a higher concentration of nutrients. Possible nutrients include the macro nutrients P and K, and almost all the other secondary and micronutrients with the exception of Cl and B. The addition of any N is highly unlikely.

For this study, application rates up to 112 metric tons solid ha⁻¹ had no significant impact on forage yield from the established grass plots. Applying at rates higher than 112 metric tons solid ha⁻¹ to plants that have low height could possibly retard forage and turf yield as a result of smothering the plant. This was not observed for the established grass plots in this study since the grass was already at a height of around 15 cm. Another possible problem with over application of solids is that a thick clay layer could seal off the soil causing poor water infiltration and crusting, both of which will hinder plant growth. For the scenario of a bare receiving soil where seed germination is desired, the solids application rates becomes more critical since a germinating seed is much more sensitive than an established plant. For this study, application of solids at 112

metric tons solid ha⁻¹ significantly reduced percent germination compared to the un-amended control. As a result, exceeding application rates of 112 metric tons solid ha⁻¹ is not recommended on bare soils where seed germination is desired. However, based on the results of this study, a low application rate of solids (22 metric tons solid ha⁻¹) can be beneficial to germination of bermudagrass seed, thus making the HDD residuals valuable for such purposes. Recommended application rates, based only on solids, should be ≤ 90 metric tons solid ha⁻¹ for bare soils receiving HDD residuals. Similarly, the SAR of the HDD residual applied is also an important consideration along with the type of cover, and this may dictate management and application rates. For example, a residual with an excessive SAR is not likely to cause a problem when applied to an established forage. However, a high SAR residual could cause problems for bare soils as previously described, even at low application rates; this problematic situation could be prevented by incorporating the residual. For this study, application of a residual with a SAR of 19 to bare soil was not a problem for seed germination until application rates reached 112 metric tons solid ha⁻¹.

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TABLES AND FIGURES

Table 1. Chemical and physical characterization of “as is”, separated water portion, and separated solids portion of HDD residuals applied to established grass plots.

	<u>Value</u>	<u>Unit</u>
<u>“As is” sample</u>		
EC	1049	$\mu\text{S cm}^{-1}$
pH	8.2	
Solids Content	39	%
<u>Separated Water Portion</u>		
EC	1474	$\mu\text{S cm}^{-1}$
pH	8.4	
SAR	16	
Na	334.2	mg L^{-1}
Ca	22.0	mg L^{-1}
Mg	6.1	mg L^{-1}
Cu	BD†	mg L^{-1}
Zn	BD	mg L^{-1}
Mo	0.06	mg L^{-1}
As	0.03	mg L^{-1}
Cd	BD	mg L^{-1}
Co	BD	mg L^{-1}
Cr	0.02	mg L^{-1}
Pb	BD	mg L^{-1}
<u>Separated Solids Portion – total digestion</u>		
Cu	8	mg kg^{-1}
Zn	13	mg kg^{-1}
Mn	1444	mg kg^{-1}
Ni	6	mg kg^{-1}
As	BD	mg kg^{-1}
Cd	BD	mg kg^{-1}
Cr	7	mg kg^{-1}
Ba	34	mg kg^{-1}
Pb	BD	mg kg^{-1}
Co	2	mg kg^{-1}

Table 1. Continued

<u>Separated Solids Portion – Mehlich-3</u>		
K	81	mg kg ⁻¹
P	BD	mg kg ⁻¹
Mg	367	mg kg ⁻¹
Ca	28526	mg kg ⁻¹
Mn	221	mg kg ⁻¹

† Below detection limit

Table 2. Chemical and physical characterization of “as is”, separated water portion, and separated solids portion of HDD residuals applied to bare soil plots.

	<u>Value</u>	<u>Unit</u>
<u>“As is” Sample</u>		
EC	5417	$\mu\text{S cm}^{-1}$
pH	8.0	
Solids Content	28	%
<u>Separated Water Portion</u>		
EC	6775	$\mu\text{S cm}^{-1}$
pH	8.0	
SAR	19	
Na	1224.6	mg L^{-1}
Ca	193.5	mg L^{-1}
Mg	6.1	mg L^{-1}
Cu	BD†	mg L^{-1}
Zn	BD	mg L^{-1}
Mo	0.04	mg L^{-1}
As	BD	mg L^{-1}
Cd	BD	mg L^{-1}
Co	BD	mg L^{-1}
Cr	0.01	mg L^{-1}
Pb	BD	mg L^{-1}
<u>Separated Solids Portion – total digestion</u>		
Cu	7	mg kg^{-1}
Zn	18	mg kg^{-1}
Mn	92	mg kg^{-1}
Ni	5	mg kg^{-1}
As	BD	mg kg^{-1}
Cd	BD	mg kg^{-1}
Cr	7	mg kg^{-1}
Ba	84	mg kg^{-1}
Pb	BD	mg kg^{-1}
Co	3	mg kg^{-1}
<u>Separated Solids Portion – Mehlich-3</u>		
K	149	mg kg^{-1}
P	4	mg kg^{-1}
Mg	599	mg kg^{-1}
Ca	3766	mg kg^{-1}
Mn	71	mg kg^{-1}

† Below detection limit

Table 3. Mean pH at 7, 30, and 93 days after application of HDD residuals (Table 1) to established grass plots as a function of application rate, soil depth, and sampling time. Comparison between application rates of 0, 22, 45, 67, 90, and 112 metric tons solid ha⁻¹.and also between sampling time. Uppercase letters represent mean separation between application rates within depth. Lower case letters represent mean separation between depths within application rate. P=0.05

Depth 0-5 cm			
<u>Application rate</u>	<u>Day 7</u>	<u>Day 30</u>	<u>Day 93</u>
0	7.5 Aa	6.3 Ab	7.0 Ba
22	7.8 Aa	6.4 Aa	7.5 Aa
45	7.6 Aa	6.6 Ab	7.5 Aa
67	7.7 Aa	6.4 Ab	7.4 Aa
90	7.9 Aa	6.5 Ab	7.5 Aa
112	8.0 Aa	6.9 Aa	7.6 Aa

Depth 5-15 cm			
<u>Application rate</u>	<u>Day 7</u>	<u>Day 30</u>	<u>Day 93</u>
0	7.1 Aa	7.1 Ca	6.7 Ca
22	7.9 Aa	7.2 BCa	7.6 Aa
45	7.7 Aa	7.1 Ca	7.3 ABa
67	7.9 Aa	7.3 BC	7.2 Ba
90	7.8 Aa	7.8 Aa	7.2 Ba
112	7.7 Aa	7.6 ABa	7.7 Aa

Table 4. Mean soil electrical conductivity (EC; $\mu\text{S cm}^{-1}$) after application of HDD residuals (Table 1) to established grass at HDD residual application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha^{-1} . Soil EC averaged across depth and sampling time, uppercase letters represent mean separation between application rates. $P=0.05$

<u>Application Rate of HDD Residuals (metric tons solid ha^{-1})</u>					
<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
463 B	594 BA	625 BA	721 A	748 A	745 A

Table 5. From saturated paste extraction: Electrical conductivity (EC) in $\mu\text{S cm}^{-1}$ by depth, at HDD residual application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha^{-1} to established grass. Sampled 7, 30, and 93 days after application. Values are averaged across time and application rates. $P=0.05$

Depth	EC ($\mu\text{S cm}^{-1}$)
0-5 cm	887 A
5-15 cm	420 B

Table 6. Mehlich 3 extractable Ca in mg kg⁻¹ for established grass plots at depths 0-5 cm and 5-15 cm for each HDD residual application rate in metric tons solid ha⁻¹, averaged over time. Uppercase letters represent mean separation between application rates. Lowercase letters represent mean separation between depths. P=0.05

<u>Depth</u>	<u>Application Rate of HDD Residuals (metric tons solid ha⁻¹)</u>					
	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	1459 Ca	4181 Ba	4303 Ba	5141 Ba	6677 BAa	7672 Aa
5-15	1012 Bb	2421 Ab	1216 Bb	1175 Bb	1301 Bb	1341 Bb

Table 7. From saturated paste extraction: Mean pH at HDD residual application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha⁻¹ on bare soil at depths 0-5 cm and 5-15 cm averaged across sampling. Uppercase letters represent mean separation between application rates within depth. Lowercase letters represent mean separation between depths within application rate. P=0.05

<u>Depth (cm)</u>	<u>Application Rate of HDD Residuals (metric tons solid ha⁻¹)</u>					
	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	7.8 Aa	7.9 Aa	7.8 Aa	7.8 Aa	7.8 Aa	7.9 Aa
5-15	7.8 Aa	7.6 BAb	7.6 BAa	7.6 BAa	7.4 Bb	7.6 BAa

Table 8. From soil saturated paste extraction: Electrical conductivity (EC; $\mu\text{S cm}^{-1}$) as a function of soil depth (0-5 cm and 5-15 cm), residual application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha^{-1} , and time after days after application of residuals (7, 29, and 96) to bare soil. Uppercase letters represent mean separation between application rates within depth. Lowercase letters represent mean separation between depths within application rate. $P=0.05$

<u>Day 7</u>						
<u>Application Rate of HDD Residuals (metric tons solid ha^{-1})</u>						
<u>Depth (cm)</u>	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	599 Ca	1295 CBa	1810 Ba	3064 Aa	2940 Aa	3545 Aa
5-15	658 Aa	326 Ab	431 Ab	766 Ab	602 Ab	475 Ab

<u>Day 29</u>						
<u>Application Rate of HDD Residuals (metric tons solid ha^{-1})</u>						
<u>Depth (cm)</u>	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	513 Ca	573 Ca	1190 Ba	1899 Aa	2008 Aa	1242 Ba
5-15	581 Ba	489 Ba	1041 BAa	1553 Aa	1602 Aa	1130 BAa

<u>Day 96</u>						
<u>Application Rate of HDD Residuals (metric tons solid ha^{-1})</u>						
<u>Depth (cm)</u>	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	914 Ba	870 Ba	836 Ba	1649 Aa	1469 Aa	1346 Aa
5-15	892 Ba	656 Bb	900 Ba	2059 Aa	1237 BAa	1260 BAa

Table 9. From soil saturated pasted extraction: Comparison of electrical conductivity (EC; $\mu\text{S cm}^{-1}$) between 7, 29, and 96 days after HDD residual application to bare soil at application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha^{-1} , for soil depths 0-5 cm and 5-15 cm. Uppercase letters represent mean separation between days within application rate and depth. $P=0.05$

<u>Depth 0-5 cm</u>			
<u>Application rate</u>	<u>Day 7</u>	<u>Day 29</u>	<u>Day 96</u>
0	599 B	513 B	914 B
22	1295 A	573 B	870 BA
45	1810 A	1190 B	836 C
67	3064 A	1899 B	1649 B
90	2940 A	2008 B	1469 B
112	3545 A	1242 B	1346 B
<u>Depth 5-15 cm</u>			
<u>Application rate</u>	<u>Day 7</u>	<u>Day 29</u>	<u>Day 96</u>
0	658 A	581 A	892 A
22	326 B	489 BA	656 A
45	431 A	1041 A	900 A
67	766 B	1553 BA	2059 A
90	602 A	1602 A	1237 A
112	475 B	1130 A	1260 A

Table 10. Mean sodium absorption ratio (SAR, by saturated pasted extract) values on soils treated with HDD residuals at application rates equivalent to 0, 22, 45, 67, 90, and 112 metric tons solid ha⁻¹. Soil SAR values averaged across sampling time. Uppercase letters represent mean separation between application rates and lowercase letters represent mean separation between depths. P=0.05

<u>Application Rate of HDD Residuals (metric tons solid ha⁻¹)</u>						
<u>Depth (cm)</u>	<u>0</u>	<u>22</u>	<u>45</u>	<u>67</u>	<u>90</u>	<u>112</u>
0-5	3.8 CBb	2.0 Ca	4.2 Ba	7.3 Aa	7.7 Aa	6.3 Aa
5-15	7.8 Aa	0.8 Cb	2.1 BCb	6.1 BAa	5.0 BACa	1.4 Cb

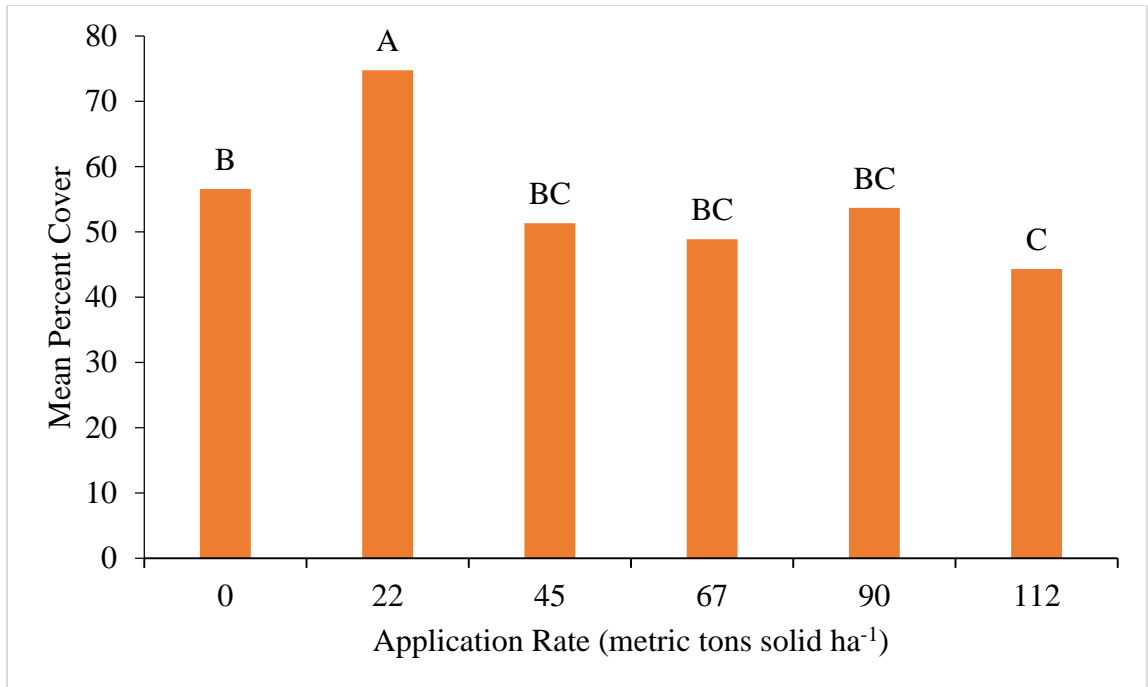


Figure 1. Mean percent cover of bare soil plots 63 days after residual application. Upper case letters represent mean separation between application rates. Bare soil was seeded with bermudagrass seed prior to residual application.

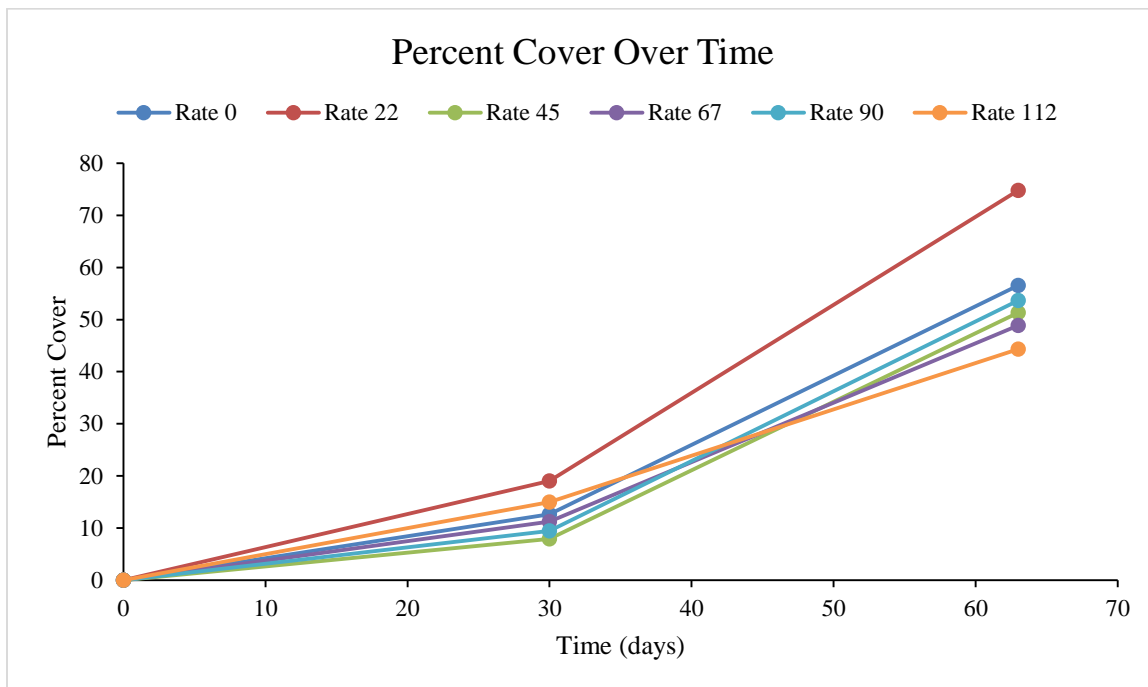


Figure 2. Mean percent cover for HDD residual application rates of 0, 22, 45, 67, 90, and 112 metric tons solid ha⁻¹ to bare soil plots. Measured at days 30 and 63.

Appendix A

Instructions for Mud Survey

1. **Box and Contents:** Keep box and packing contents in good condition as it will be re-used to return samples. Contents of each box include: 1 pen; 1 return mailing label; 2 sample bottles, 1 roll duct tape; 1 sample ladle; 2 gallon zip-lock bag, packing materials, these instructions and written survey.
2. **Written Survey:** Please complete the included written survey, attached as page 3 and 4. If samples are obtained from jobsites for different HDD companies; complete one survey for each company. Include a note on the survey indicating company name and which sample (1 or 2) goes with which survey.
3. **Two Locations:** If possible, please plan to obtain two different mud samples (from different locations) for us to analyze as part of this nation-wide survey.
4. **Label: before the bottle is filled with mud,** complete the label attached with permanent marker. The more information that you provide, the more useful it will be to us. Note, we understand that some drillers may be reluctant, specific information regarding the drilling company is optional and will not be maintained long term. Only the basic address (nearest cross streets) where sample is obtained will be kept to allow us to correlate the information with known soils type maps for each location. Also, label the sample by circling either sample number (1 or 2), and circle the corresponding number on the survey.
5. **Sample: Completely** fill each bottle with mud. The goal of the sampling is to obtain a representative sample of the mud for that entire job. There are two possible methods to do this using the 32 oz. plastic bottle provided. Please choose one of the methods below. Note: depending on your time available to take the sample, a mixture of methods A & B are fine to obtain the most representative sample.

Method A: Completely fill the sample bottle with mud over an extended period of time as it flows out of the borehole throughout the drilling job.

- Depending on the time required to complete the job, take several “grab samples” spread out evenly over the boring portion; or at least a 2-4 hour period during the boring process, enough to fill the bottle.
 - Obtain each of the grab samples with the ladle provided as the mud is actively flowing out of the borehole, near the exit point where mud is flowing. Danger! To prevent entanglement and injury, make sure not to get close to the rotating drill rod.

- For Example, if drilling is projected to require 4 hours, then take one grab sample of one ladle-full every 30 minutes to one hour and pour into the 32 oz. plastic bottle until it is full.

Method B: Take samples directly from the ditch or “pit” shortly after job is complete.

- After drilling is complete and all mud is in the pit or ditch.
 - Remove a grab sample from several different locations and depths within the pit or ditch and pour into the 32 oz. plastic bottle until the bottle is completely full.
6. **Packing and Shipping:** *After the plastic container is completely filled with mud:*
- Clean threads with clean paper towel or rag and secure the lid tightly. Then wipe or wash off the container with clean water and dry. Tape lid securely with the duct tape provided.
 - If possible, please keep samples refrigerated if they cannot be immediately returned.
 - Place both containers in the Zip-lock bag to contain accidental spillage during shipping.
 - Place the two samples in the original shipping container and cushion with packing material.
 - Place the written survey into the box. No need to return tape, ladle or pen.
 - Tape the box shut with duct tape. Attach the pre-paid shipping label to the outside of the box and ship as soon as possible.

Company Name: _____

Circle Sample # (1) or (2) corresponding with a number circled for specific sample bottle.

1. Which of the following best describes your position with your company? (Choose one)

- Purchasing General management Equipment user or job-site Supervisor Other_____

Primary State where HDD Company is Located: _____

2. Which market do you primarily work in?

- Municipal/ Govt Utility Co. Utility Contractor Oil & Gas Other_____

3. Approximately how many drills of the following sizes do you own? (at locations you are involved with)

0-50K	
50-100K	
Over 100K	

4. How long have you worked in the industry? _____

5. Please describe what your usual “fluid disposal” activity looks like: _____

6. Please rank how big of an issue mud disposal is for you on a scale of 1-10, with 10 being a major issue: (please circle)

1 2 3 4 5 6 7 8 9 10

Why? _____

8. Do you utilize a reclaimer? Yes No

8a. If no, do you anticipate needing a reclaimer in the future? Yes No

9. Has mud/fluid disposal changed for you over the past 10 years? Yes No

9a. If yes, in what way? _____

10. Thinking of mud disposal, how much mud did you dispose of per week? _____

11. About how many dollars would you estimate were spent on mud disposal in the past year?

(please mark which one or all that you know)

- Not Sure \$ _____ % of job costs _____
 % of revenue _____

12. Please mark what contributes to your top two costs:

- Additives Travel to dump \$ paid to farmers \$ paid to landfill
 Other _____ Not incurring cost today

13. Any other comments/needs regarding fluid/mud disposal: _____

APPENDIX B

Table B1. EC in $\mu\text{S cm}^{-1}$ and pH of the mud and the separated water portion with solids content and Total Dissolved Solids (TDS) in lbs. acre⁻¹ applied at 112 metric tons ha⁻¹.

Sample #	E.C. mud	pH mud	EC Water	pH Water	Solids Content
1	267	5.8	343	5.1	0.45
2	440	6.3	978	6.6	0.61
3	1625	7.6	1686	8.3	0.29
4	905	7.9	1008	8.3	0.23
5	1967	7.5	1857	7.8	0.16
6	846	7.6	1191	8.0	0.35
7	571	7.6	655	7.9	0.57
8	587	7.6	533	7.6	0.11
9	372	6.1	441	6.2	0.31
10	305	6.0	331	6.1	0.34
11	1271	7.4	1491	8.2	0.28
12	1235	7.4	1532	8.1	0.29
13	1409	8.1	1650	8.6	0.17
14	185	5.9	300	6.8	0.36
15	885	7.5	1312	8.0	0.53
16	720	7.7	996	8.1	0.42
17	1385	6.8	1630	7.8	0.34
18	926	7.8	1356	8.2	0.49
19	2064	7.9	3172	8.6	0.50
20	2928	9.9	3526	10.1	0.27
21	1684	9.1	1763	8.8	0.04
22	1698	9.1	1942	9.0	0.05
23	943	6.6	1055	6.8	0.24
24	143	5.8	259	6.0	0.57
25	1438	7.3	1714	7.8	0.32
26	848	7.3	1270	7.5	0.54
27	416	4.7	592	4.2	0.36
28	473	5.9	611	6.0	0.67
29	354	6.1	572	6.8	0.49
30	118	5.2	241	5.4	0.53
31	1168	10.0	2017	6.9	0.46
32	1277	9.4	1661	8.8	0.13
33	2075	8.1	2252	8.4	0.13
34	2826	8.3	3093	8.6	0.14
35	818	7.3	1688	8.0	0.62

Table B1. Continued

Sample #	E.C. mud	pH mud	EC Water	pH Water	Solids Content
36	1775	6.9	2042	7.7	0.40
37	3950	8.1	4296	8.1	0.41
38	2460	8.6	3251	8.2	0.41
39	720	7.7	980	7.3	0.72
40	1041	7.2	1037	7.4	0.10
41	1088	7.0	962	7.2	0.06
42	1962	8.6	1913	8.3	0.32
43	703	7.7	780	7.9	0.47
44	558	7.7	1180	7.9	0.56
45	2009	8.3	1918	8.9	0.04
46	1317	8.6	1696	8.8	0.30
47	1492	8.6	1424	8.1	0.09
48	1279	9.5	1044	8.2	0.08
49	715	7.1	1149	8.2	0.72
50	230	6.5	406	7.5	0.68
51	1160	6.7	1179	8.0	0.69
52	1781	9.5	1734	8.9	0.13
53	756	8.0	1287	8.2	0.52
54	2036	8.4	2183	8.7	0.12
55	1195	7.0	1784	8.0	0.51
56	1708	7.7	1656	8.4	0.14

Table B2. Primary constituent concentrations in mg kg⁻¹ of digested separated solids portion of mud. EPA 3050

Sample	Na	Ca	Mg	K	S	P	Fe	Zn	Cu	Mn	Al
1	193	477	332	240	268	110	33150	7	2	BD	30806
2	90	1511	4294	1357	131	338	39501	66	434	296	26910
3	1588	5624	2855	1552	265	114	9704	30	9	75	10679
4	1247	1569	2028	1821	198	43	13627	20	7	204	12840
5	2624	4856	2662	1103	780	294	26289	63	45	1606	14779
6	759	59347	31588	2027	587	285	21453	45	26	386	10621
7	168	32247	2734	1236	298	161	7765	16	10	102	7939
8	465	3504	1469	672	156	30	11475	19	12	BD†	18752
9	534	393	365	240	229	102	34368	13	136	BD	37080
10	329	349	194	113	216	80	25730	6	93	BD	21460
11	928	5789	3693	1032	1689	582	20280	39	13	273	7434
12	892	5713	3876	1538	1629	587	21234	37	13	269	9403
13	2361	93706	8222	1526	6236	3061	9488	14	19	688	5749
14	111	610	478	943	78	212	29049	31	11	710	8621
15	482	52321	14246	4423	870	503	28851	58	21	391	19236
16	401	29668	7379	1141	431	442	12756	35	17	242	7584
17	1088	1522	556	444	217	57	3400	10	1	78	11899
18	434	4317	600	282	446	85	4312	11	7	41	3022
19	1352	75311	25027	1703	1590	199	9325	18	7	150	6888
20	4119	41873	13718	708	678	157	7299	20	8	137	4254
21	13455	7042	3736	1449	1521	283	13703	68	402	463	14041
22	12927	11518	3589	1348	1604	334	13106	64	1863	417	13383
23	677	1950	5743	6614	373	281	24660	65	34	393	23209

† Below Detection Limit

Table B2. Continued

Sample	Na	Ca	Mg	K	S	P	Fe	Zn	Cu	Mn	Al
24	51	445	310	408	243	290	51185	19	30	190	33104
25	1266	18562	7180	849	2012	289	11392	30	22	334	7255
26	406	30789	3447	1797	838	700	21096	341	38	493	12039
27	327	5171	2206	1524	141	5753	36491	31	BD†	BD	42668
28	138	339	102	88	174	544	570	BD	BD	BD	4275
29	263	1282	1690	1028	83	196	22247	33	32	234	19487
30	196	192	707	503	46	73	17417	15	10	13	20938
31	363	104773	4766	951	1102	80	5096	11	1	44	5118
32	3392	8736	6041	3800	685	819	25613	69	10	452	12951
33	3443	5257	1408	823	1340	183	7623	13	10	455	4153
34	5488	3719	1796	812	1509	195	5362	100	34	95	5311
35	244	603	230	85	86	702	1016	5	1	BD	3852
36	766	1666	334	135	245	224	1833	5	25	80	5654
37	1394	9648	7347	2127	1692	444	22616	55	39	231	17704
38	1866	19420	7430	4519	792	791	21073	46	11	259	17876
39	177	36006	5592	665	547	351	14893	35	9	359	6714
40	2641	8085	6953	1072	551	548	25445	65	24	513	20389
41	4402	8705	8445	1483	1232	666	31464	85	56	690	24119
42	2107	1831	984	347	525	88	3025	17	5	48	2505
43	220	3031	3742	1199	43	489	20305	47	11	657	13789
44	331	618	193	153	44	54	4888	17	8	16	5066
45	16221	9703	5572	1833	2021	584	14411	54	7	475	15530
46	2003	1357	989	922	181	110	5934	38	3	232	4092

† Below Detection Limit

Table B2. Continued

Sample	Na	Ca	Mg	K	S	P	Fe	Zn	Cu	Mn	Al
47	3488	122638	3883	1690	2289	356	9224	23	11	468	7491
48	7085	6106	3424	2346	1107	311	13139	36	4	562	10848
49	289	7892	4113	1679	132	554	34320	25	1	558	12095
50	109	472	462	310	19	100	4961	14	6	BD†	11069
51	2390	1451	631	1031	461	171	27171	20	8	22	8109
52	6455	2526	1087	743	1067	105	8539	27	37	41	4832
53	436	100412	34074	764	3253	174	4861	29	11	185	2454
54	4646	223986	11582	671	4262	111	3940	22	2	72	3838
55	352	25137	8481	1165	274	318	14312	39	17	438	8578
56	3849	62623	25512	1185	1068	401	18769	43	23	1236	7541
57	959	36583	6199	1659	1380	547	23383	57	80	445	11989
58	159	71482	6470	3927	4433	1051	30213	92	55	641	23196

† Below Detection Limit

Table B3. Secondary constituent concentrations in mg kg⁻¹ of digested separated solids portion of mud. EPA 3050

Sample	Ni	B	As	Cd	Cr	Ba	Pb	Co
1	2.3	56.9	BD†	BD	25.1	15.5	BD	BD
2	17.7	64.4	BD	BD	36.0	47.5	BD	BD
3	8.6	21.6	BD	BD	11.9	54.2	BD	BD
4	9.9	29.7	BD	BD	15.9	82.4	0.5	BD
5	27.0	49.5	BD	BD	22.7	218.7	0.6	5.5
6	21.2	40.7	BD	BD	15.4	62.7	BD	BD
7	7.5	17.4	BD	BD	8.1	51.6	BD	BD
8	6.8	22.7	BD	BD	14.6	69.8	4.4	BD
9	7.3	58.8	BD	BD	30.6	20.2	BD	BD
10	3.4	43.7	BD	BD	23.5	14.1	BD	BD
11	14.5	36.4	BD	BD	12.5	27.2	1.1	BD
12	14.8	40.2	BD	BD	14.8	31.1	BD	BD
13	5.7	23.4	BD	BD	5.8	44.4	BD	BD
14	9.6	49.8	BD	BD	21.1	37.9	BD	3.9
15	25.8	60.8	BD	BD	22.0	113.8	BD	BD
16	10.2	26.5	BD	BD	10.4	37.0	BD	BD
17	2.3	8.0	BD	BD	8.1	46.7	7.4	BD
18	2.6	10.1	BD	BD	7.3	20.9	2.9	BD
19	10.7	25.2	BD	BD	9.6	41.9	BD	BD
20	8.3	17.7	BD	BD	7.9	27.8	BD	BD
21	14.6	32.2	BD	BD	14.5	125.2	16.9	BD
22	13.5	31.4	BD	BD	15.6	112.5	16.9	BD
23	2.6	41.7	BD	BD	7.5	117.9	BD	BD
24	1.8	89.6	BD	BD	43.4	28.6	BD	BD
25	10.3	24.2	BD	BD	11.3	86.4	BD	BD
26	19.7	44.0	1.6	BD	18.2	178.1	58.4	BD
27	18.2	63.3	BD	BD	100.1	230.0	BD	BD
28	0.6	BD	BD	BD	5.6	9.2	2.1	BD
29	12.7	41.3	BD	BD	18.7	65.9	BD	BD
30	7.5	32.3	BD	BD	18.2	46.8	BD	BD
31	3.9	14.4	BD	BD	5.3	392.8	BD	BD
32	6.2	46.7	BD	BD	8.9	108.7	BD	BD
33	3.8	17.7	BD	BD	4.2	71.3	3.7	BD
34	5.1	16.2	BD	BD	5.7	60.9	9.0	BD
35	2.5	3.7	BD	BD	4.7	32.2	1.4	BD
36	2.0	5.4	BD	BD	6.8	41.9	7.7	BD
37	43.3	49.3	BD	BD	49.3	230.4	BD	BD

† Below Detection Limit

Table B3. Continued

Sample	Ni	B	As	Cd	Cr	Ba	Pb	Co
38	10.3	52.7	BD†	BD	15.9	128.0	BD	BD
39	19.3	27.0	BD	BD	14.9	25.3	6.8	BD
40	28.8	42.8	BD	BD	28.2	138.9	BD	10.3
41	38.3	53.5	BD	BD	37.2	163.3	BD	14.8
42	2.2	10.0	0.1	BD	3.5	19.0	BD	1.2
43	17.8	36.5	BD	BD	19.6	211.9	BD	7.6
44	2.4	9.6	BD	BD	7.5	21.7	BD	1.4
45	14.3	31.0	BD	BD	6.8	157.6	BD	6.9
46	3.8	13.1	BD	BD	1.9	51.5	BD	1.6
47	3.8	20.2	BD	BD	3.8	974.1	BD	4.3
48	7.1	27.4	BD	BD	8.2	146.4	BD	4.2
49	22.9	57.5	BD	BD	28.8	114.3	BD	15.6
50	5.0	10.4	BD	BD	9.1	35.5	BD	2.0
51	3.6	46.9	BD	BD	24.6	49.9	BD	3.6
52	16.7	21.4	BD	BD	12.9	34.9	BD	4.0
53	14.0	11.9	0.2	BD	10.2	35.2	BD	4.8
54	3.3	11.9	0.1	BD	2.2	24.8	BD	1.2
55	17.4	30.7	0.1	BD	18.6	107.1	BD	5.9
56	23.7	36.4	0.1	BD	12.5	107.0	BD	10.3
57	BD	145.2	BD	BD	13.9	397.8	BD	9.7
58	BD	194.6	BD	BD	35.1	195.9	BD	15.3

† Below Detection Limit

Table B4. Concentration in mg L⁻¹ of the major constituents in the separated water portion of mud and the sodium adsorption ratio.

Sample	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
1	62.3	2.8	0.5	3.2	99.3	BD†	0.1	BD	BD	BD	0.1	BD	9
2	29.5	201.4	15.3	3.8	160.6	BD	0.1	BD	BD	0.1	23.4	BD	1
3	396.1	15.0	5.4	1.8	340.0	0.1	0.1	0.1	BD	BD	BD	0.1	22
4	234.7	25.3	50.7	78.2	166.5	0.8	BD	125.6	0.3	BD	BD	273.8	6
5	403.8	41.9	18.0	3.5	411.9	0.1	0.1	0.3	BD	BD	7.3	0.5	13
6	230.3	33.1	15.4	3.1	147.7	0.1	0.1	0.1	0.1	0.1	0.4	0.1	8
7	77.3	53.9	11.3	1.5	53.9	0.1	BD	BD	BD	BD	BD	BD	2
8	52.2	48.8	10.9	2.6	63.6	0.1	BD	BD	BD	BD	BD	BD	2
9	98.8	1.2	0.1	2.3	129.5	BD	BD	0.1	BD	BD	BD	BD	23
10	67.4	0.8	BD	1.1	103.8	BD	0.1	BD	BD	BD	BD	BD	21
11	315.6	30.3	8.0	10.7	203.6	0.1	0.1	1.4	BD	BD	0.4	4.5	13
12	308.8	31.0	8.2	10.1	199.8	0.1	0.1	1.3	BD	BD	0.4	4.1	13
13	374.6	26.9	11.3	13.3	484.1	0.2	0.3	8.7	BD	BD	BD	31.7	15
14	37.8	18.3	2.0	3.5	69.3	BD	0.1	BD	0.1	BD	1.7	BD	2
15	191.6	78.6	35.5	4.6	387.5	0.4	BD	BD	BD	BD	0.2	BD	5
16	172.4	49.5	9.9	4.7	198.2	0.1	0.1	0.1	BD	BD	0.2	BD	6
17	383.7	44.1	5.9	6.6	38.3	BD	0.1	0.4	BD	BD	2.9	0.4	14
18	277.9	38.5	7.1	4.5	193.9	0.2	0.1	1.3	BD	BD	0.2	3.1	11
19	804.8	44.9	47.0	40.4	447.4	0.5	0.4	133.3	0.2	BD	BD	326.0	20
20	856.2	1.4	3.3	6.5	355.7	0.1	0.2	0.8	BD	BD	BD	2.8	90
21	435.1	10.9	16.3	5.0	386.0	0.3	0.1	31.2	0.1	0.3	BD	105.0	20
22	462.2	9.4	5.8	4.3	434.0	0.3	0.1	8.7	BD	0.3	BD	33.9	29
23	165.8	44.0	16.2	2.1	469.4	BD	BD	BD	BD	BD	0.5	BD	5

† Below Detection Limit

Table B4. Continued

Sample	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
24	18.0	25.0	3.4	1.4	77.6	BD†	BD	BD	BD	BD	2.0	BD	1
25	381.1	49.5	12.4	6.6	327.8	BD	0.2	1.2	BD	BD	1.0	2.0	13
26	163.2	131.9	10.7	18.6	183.1	0.3	0.4	0.2	BD	BD	3.5	0.2	4
27	67.8	25.0	1.1	0.6	92.1	BD	0.1	0.3	BD	BD	BD	0.4	4
28	115.2	41.0	31.3	6.5	162.3	0.5	26.6	80.6	0.2	BD	BD	472.0	3
29	88.9	16.0	4.3	1.2	65.3	0.1	BD	0.1	BD	BD	2.1	BD	5
30	45.6	0.8	0.4	1.3	5.2	BD	BD	0.2	BD	BD	0.1	0.3	11
31	219.0	138.5	18.4	10.3	561.5	0.3	BD	BD	BD	BD	BD	BD	5
32	309.0	4.9	2.6	5.2	405.9	0.1	0.2	1.9	BD	BD	BD	9.0	28
33	385.6	40.3	16.3	10.6	468.8	0.1	0.2	22.2	BD	BD	BD	94.0	13
34	379.2	15.3	11.7	9.2	417.1	0.1	0.1	7.9	0.2	BD	BD	43.4	18
35	290.6	20.2	4.2	5.6	468.1	0.1	0.4	0.2	BD	BD	BD	0.8	15
36	389.1	54.8	11.1	5.7	350.4	0.1	0.1	4.4	BD	BD	2.6	0.3	13
37	642.0	584.7	65.1	12.7	2708.3	0.8	BD	BD	BD	BD	0.9	BD	7
38	667.9	71.0	14.6	3.7	1395.5	1.5	BD	BD	BD	BD	BD	BD	19
39	157.4	37.5	3.2	4.0	161.2	BD	0.1	0.2	BD	BD	BD	0.4	7
40	268.4	24.1	5.1	4.8	24.7	0.1	0.1	4.9	BD	BD	1.2	11.0	13
41	272.6	35.7	41.2	10.7	107.4	0.4	0.4	75.0	0.2	0.1	2.3	732.0	7
42	577.2	27.2	19.8	17.6	860.5	0.2	0.3	17.3	0.1	BD	BD	130.3	21
43	81.8	64.2	35.1	1.2	15.8	BD	BD	BD	BD	BD	2.7	BD	2
44	262.6	12.5	0.4	2.6	75.4	0.1	BD	0.2	BD	BD	BD	0.5	20
45	525.4	19.6	19.0	9.2	274.2	0.2	0.6	37.7	0.2	BD	0.8	254.9	20
46	419.5	21.0	4.5	13.8	255.4	0.2	2.4	3.3	BD	BD	BD	13.0	22

† Below Detection Limit

Table B4. Continued

Sample	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
47	320.7	19.0	4.6	6.0	464.3	BD†	BD	0.7	BD	BD	BD	2.5	17
48	286.3	4.2	5.3	6.5	342.5	0.1	0.1	11.1	BD	BD	BD	61.5	22
49	193.2	63.9	22.3	13.2	248.2	0.1	0.1	BD	BD	BD	BD	BD	5
50	72.2	17.8	2.3	3.9	26.6	0.1	0.1	0.6	BD	BD	0.2	1.5	4
51	315.3	25.9	52.2	14.4	328.0	0.6	0.4	161.8	0.2	BD	BD	1185.9	8
52	235.8	3.1	9.8	4.3	214.3	0.5	0.2	17.1	0.1	0.1	BD	102.5	15
53	247.9	39.2	43.8	19.9	463.0	0.2	BD	BD	BD	BD	0.2	0.1	6
54	537.1	21.0	16.4	10.1	802.3	0.1	BD	14.6	BD	BD	BD	107.6	21
55	154.3	95.5	54.3	6.1	68.4	0.1	BD	BD	BD	BD	1.1	BD	3
56	407.6	57.1	74.9	9.9	270.9	0.7	0.3	183.0	0.3	0.2	5.9	1030.9	8

† Below Detection Limit

Table B5. Concentration in mg L⁻¹ of the minor constituents in the separated water portion of the mud. † Below Detection Limit

Sample #	Mo	As	Cd	Co	Cr	Pb
1	BD†	BD	0.01	BD	0.02	BD
2	0.01	BD	0.01	0.07	0.02	0.02
3	0.12	BD	0.01	BD	0.02	BD
4	BD	BD	0.12	0.08	0.40	BD
5	0.03	BD	0.01	0.03	0.02	BD
6	0.03	BD	0.02	0.10	0.02	0.04
7	0.01	BD	0.01	BD	0.02	BD
8	0.02	BD	BD	BD	0.02	0.02
9	BD	BD	0.01	BD	0.02	BD
10	BD	BD	0.01	0.01	0.02	BD
11	0.15	BD	0.01	0.01	0.02	BD
12	0.14	BD	0.01	0.01	0.02	BD
13	0.10	BD	0.01	0.01	0.02	BD
14	BD	BD	0.01	0.02	0.02	BD
15	0.02	BD	0.01	BD	0.02	BD
16	0.06	BD	0.01	BD	0.01	0.02
17	0.01	BD	0.01	BD	0.02	BD
18	0.09	BD	0.01	BD	0.02	BD
19	0.06	BD	0.01	BD	0.12	BD
20	0.11	BD	0.01	0.01	0.02	BD
21	0.10	BD	0.01	BD	0.02	BD
22	0.13	BD	0.01	BD	0.02	BD
23	BD	BD	0.01	BD	0.02	0.02
24	BD	BD	0.01	0.01	0.02	BD
25	0.05	BD	0.01	BD	0.02	BD
26	0.18	BD	BD	0.01	0.02	0.02
27	BD	BD	0.02	BD	0.02	BD
28	BD	BD	0.11	0.10	0.51	BD
29	BD	BD	0.01	0.01	0.02	BD
30	BD	BD	0.16	0.01	0.02	BD
31	0.06	BD	0.02	0.01	0.02	BD
32	0.10	BD	0.01	0.01	0.02	BD
33	0.05	BD	BD	BD	0.03	BD
34	0.04	BD	BD	BD	0.02	BD
35	0.06	BD	BD	BD	BD	BD
36	0.11	BD	BD	BD	0.02	BD
37	0.63	BD	BD	BD	BD	BD

Table B5. Continued

Sample #	Mo	As	Cd	Co	Cr	Pb
38	0.37	BD†	BD	BD	0.03	BD
39	0.03	BD	BD	BD	0.02	BD
40	0.03	BD	BD	BD	0.02	BD
41	BD	BD	BD	0.01	0.04	BD
42	0.12	BD	BD	BD	0.01	BD
43	0.02	BD	BD	0.02	0.01	0.03
44	0.07	BD	BD	BD	0.02	BD
45	0.02	BD	BD	0.08	0.02	BD
46	0.04	BD	BD	BD	0.02	BD
47	0.06	BD	BD	BD	0.02	BD
48	0.07	BD	BD	BD	0.02	BD
49	BD	BD	BD	BD	0.02	BD
50	BD	BD	BD	BD	BD	BD
51	BD	BD	BD	0.02	0.13	BD
52	0.04	BD	BD	BD	0.02	BD
53	0.38	BD	BD	BD	0.01	0.02
54	0.06	BD	BD	BD	BD	BD
55	0.06	BD	BD	BD	0.01	BD
56	BD	BD	BD	0.07	0.06	BD

† Below Detection Limit

Table B6. Mehlich 3 extractable elemental concentrations in mg kg⁻¹ of the separated solids portion of mud.

Sample	K	P	Mg	Ca	Na	Mn	Fe	Al
1	58	2	92	504	185	12	35	908
2	60	9	59	740	53	84	303	736
3	141	7	496	4486	1590	41	65	112
4	148	2	509	1575	1225	102	76	616
5	134	12	954	3955	2665	1444	223	263
6	117	2	632	5231	682	172	113	163
7	70	2	282	8630	117	36	37	93
8	119	6	621	3479	458	14	110	856
9	77	2	127	411	568	8	87	867
10	36	2	94	370	350	13	68	855
11	110	3	302	2216	959	58	761	330
12	106	5	300	2286	882	57	727	333
13	170	1	362	37320	2200	130	92	42
14	61	2	75	689	99	191	152	598
15	158	3	521	12080	317	126	272	138
16	60	10	163	5350	306	117	245	885
17	113	8	162	1588	1110	80	278	855
18	44	8	133	2670	438	22	297	227
19	139	7	293	6600	1435	58	275	95
20	123	3	460	5860	4128	58	232	87
21	469	9	1001	3246	15689	98	287	118
22	401	22	856	3801	14125	73	210	83
23	73	10	380	1444	672	61	171	505
24	40	1	45	390	49	33	33	1061
25	123	17	435	4250	1219	149	484	91
26	291	102	226	5790	326	242	282	209
27	48	210	135	3660	317	5	479	1967
28	16	275	13	196	136	2	13	1894
29	90	4	249	1269	236	149	166	995
30	55	3	223	182	185	39	99	899
31	81	1	594	36690	277	2	6	47
32	291	16	696	4476	3588	90	252	86
33	195	14	483	3913	3456	131	305	61
34	281	12	796	2971	5353	50	146	298
35	31	217	76	418	321	6	176	903
36	63	52	207	1590	806	69	433	507
37	245	34	481	6180	1167	85	279	111

Table B6. Continued

Sample	K	P	Mg	Ca	Na	Mn	Fe	Al
38	152	51	438	5280	1480	73	68	79
39	27	17	67	4233	165	62	102	20
40	208	19	549	4119	2818	513	491	1574
41	340	20	855	4590	4259	653	383	1506
42	117	20	448	1577	2034	16	156	295
43	57	10	862	2110	183	305	67	604
44	24	5	47	629	350	14	127	350
39	9	4	31	2097	42	16	50	13
40	223	21	554	4110	2708	258	478	1621
41	386	21	857	4660	4189	319	397	1536
42	148	21	539	1802	2304	19	176	325
43	87	10	864	2078	190	185	82	674
44	24	5	48	654	363	14	118	359
45	658	43	1318	3977	17151	303	436	345
46	384	14	215	978	1579	54	81	361
47	276	BD†	822	37889	3707	84	107	24
48	462	5	638	3853	6679	134	266	442
49	197	10	319	4364	251	218	177	439
50	51	20	59	449	91	11	159	1117
51	146	3	320	1540	2567	23	392	571
52	261	9	644	2263	6336	20	432	337
53	106	2	885	19926	334	76	114	10
54	198	BD	822	39320	4886	14	20	22
55	175	6	616	5795	302	240	319	190
56	151	4	854	6121	3640	1075	315	60

† Below Detection Limit

Table B7. Percent C and N and C:N ratio for the separated solids portion of mud.

Sample	TC %	TN %	C/N Ratio
1	0.43	0.04	11.72
2	1.25	0.04	28.13
3	0.26	0.03	7.62
4	0.14	0.02	5.91
5	0.76	0.06	11.79
6	3.65	0.05	72.48
7	1.01	0.02	54.35
8	0.17	0.03	6.11
9	0.39	0.02	16.25
10	0.31	0.02	20.13
11	0.53	0.04	13.55
12	0.53	0.04	12.45
13	5.97	0.02	276.39
14	0.24	0.04	5.81
15	2.26	0.05	43.42
16	1.49	0.02	83.24
17	0.32	0.04	7.24
18	0.23	0.03	8.05
19	3.59	0.02	213.87
20	2.13	0.01	212.80
21	1.48	0.03	45.02
22	4.75	0.03	154.85
23	0.21	0.03	7.97
24	0.35	0.04	9.70
25	1.54	0.04	35.90
26	6.61	0.21	31.35
27	0.27	0.05	5.45
28	1.27	0.02	66.25
29	0.43	0.04	11.34
30	0.30	0.04	8.01
31	2.27	0.03	82.77
32	0.23	0.02	10.83
33	0.50	0.03	14.53
34	0.57	0.04	14.92
35	0.11	0.01	9.48
36	0.50	0.04	13.43
37	0.36	0.04	9.75

Table B7. Continued

Sample	TC %	TN %	C/N Ratio
38	0.52	0.04	14.79
39	1.76	0.02	89.39
40	2.11	0.10	20.24
41	4.35	0.26	16.95
42	0.20	0.02	12.54
43	0.18	0.05	3.92
44	0.18	0.03	6.47
45	1.09	0.05	20.55
46	0.09	0.03	2.97
47	4.39	0.06	69.41
48	0.19	0.04	5.17
49	0.25	0.06	4.21
50	0.18	0.03	5.90
51	0.33	0.10	3.36
52	0.37	0.04	9.42
53	5.17	0.04	125.77
54	6.06	0.03	225.13
55	1.52	0.06	26.08
56	3.40	0.04	96.62

Table B8. Primary elemental concentrations in mg L⁻¹ of leachate from the Synthetic Precipitation Leaching Procedure performed on the separated solids portion of mud.

Sample	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
1	8.8	0.8	BD†	BD	13.5	0.2	BD	BD	BD	BD	BD	BD
2	3.1	7.5	0.4	BD	10.2	0.2	BD	BD	BD	BD	0.6	0.1
3	61.6	4.0	5.2	3.9	37.6	0.3	BD	14.8	BD	BD	BD	29.1
4	41.5	6.7	14.9	21.5	27.5	0.7	0.1	54.7	0.1	BD	BD	110.8
5	105.8	5.4	1.4	BD	109.6	0.2	0.2	0.8	BD	BD	0.1	1.5
6	31.7	7.2	5.9	10.0	15.8	0.3	0.1	18.8	BD	BD	BD	25.3
7	6.8	10.4	3.6	BD	5.4	0.2	BD	8.8	BD	BD	BD	14.5
8	20.8	14.7	3.1	BD	22.1	0.2	BD	4.4	BD	BD	BD	12.0
9	15.4	0.2	BD	BD	19.1	0.5	BD	BD	BD	BD	BD	BD
10	12.6	0.3	BD	BD	16.7	0.5	BD	BD	BD	BD	BD	BD
11	45.8	5.5	4.9	15.7	34.2	0.7	0.1	18.2	BD	BD	BD	41.0
12	43.9	6.5	4.7	16.0	32.5	0.6	0.1	18.2	BD	BD	BD	39.3
13	105.0	21.8	14.6	14.3	160.8	0.7	1.3	26.4	BD	BD	BD	79.3
14	5.4	1.6	BD	BD	8.1	0.2	BD	0.1	BD	BD	0.2	0.2
15	15.7	12.3	3.1	4.1	25.0	0.2	BD	2.0	BD	BD	BD	5.5
16	16.8	12.8	1.9	3.9	16.8	0.7	0.1	7.3	BD	BD	0.1	9.7
17	39.0	9.0	9.5	8.2	18.0	0.5	0.2	43.6	0.1	BD	0.1	179.7
18	20.3	7.3	1.6	BD	15.3	0.6	0.2	8.9	BD	BD	BD	15.9
19	62.2	24.7	17.4	20.4	30.5	0.7	0.5	39.1	BD	BD	0.1	74.2
20	154.0	5.2	6.1	4.3	52.4	0.2	0.2	13.2	BD	BD	BD	36.0
21	266.6	12.2	32.4	5.5	262.2	1.5	BD	72.2	0.2	0.5	BD	192.3
22	265.9	13.6	36.4	6.3	247.0	1.4	0.2	83.0	0.2	2.1	BD	204.1
23	29.5	4.3	2.0	BD	63.8	0.1	BD	4.9	BD	BD	0.1	13.3

† Below Detection Limit

Table B8. Continued

Sample	Na mg/l	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
24	3.5	2.4	BD†	BD	7.9	0.5	BD	BD	BD	BD	0.2	BD
25	52.2	11.4	9.0	3.8	42.7	0.7	0.4	27.9	BD	BD	0.2	56.0
26	16.0	17.0	2.0	6.7	15.7	0.7	0.7	7.8	0.1	BD	0.2	10.8
27	11.4	2.1	0.2	BD	12.2	0.6	1.3	3.0	BD	BD	BD	7.5
28	6.9	0.6	BD	BD	4.8	0.3	0.5	0.9	BD	BD	BD	5.2
29	9.7	2.1	2.5	3.4	7.7	0.8	0.2	26.2	BD	BD	0.1	41.8
30	6.8	0.1	BD	BD	3.8	0.7	BD	0.9	BD	BD	BD	2.2
31	16.5	15.9	2.5	BD	39.0	0.7	BD	BD	BD	BD	BD	BD
32	107.1	13.1	22.4	4.8	91.5	1.1	0.2	43.1	0.1	BD	BD	123.4
33	126.0	10.8	8.1	4.2	165.5	0.8	0.3	17.8	BD	BD	BD	44.1
34	108.8	5.0	6.4	4.3	212.9	1.0	0.1	10.7	0.1	BD	BD	40.1
35	15.1	5.2	4.0	BD	15.3	0.7	5.5	12.3	BD	BD	BD	51.2
36	34.9	11.6	6.0	BD	27.1	0.7	1.8	23.8	BD	BD	0.1	117.4
37	52.1	46.7	4.0	BD	189.9	0.6	BD	0.1	BD	BD	BD	0.3
38	62.2	7.8	6.0	BD	84.7	0.9	0.4	12.4	BD	BD	BD	24.5
39	8.5	9.6	0.5	BD	7.6	0.6	BD	1.7	BD	BD	0.1	2.5
40	73.3	9.5	5.7	BD	36.7	0.8	0.4	13.4	BD	BD	0.3	35.0
41	119.8	6.5	1.6	3.5	158.9	0.8	0.4	2.1	BD	BD	0.3	6.2
42	68.0	3.6	4.2	BD	77.7	0.7	0.4	7.3	BD	BD	BD	28.0
43	9.2	3.9	2.4	BD	3.6	0.5	BD	4.4	BD	BD	BD	5.4
44	17.8	5.5	1.8	BD	5.9	0.7	0.1	21.1	0.1	BD	BD	43.0
45	386.9	5.9	1.0	6.5	334.5	1.4	1.7	1.5	BD	BD	0.1	4.5
46	52.7	2.8	2.4	5.4	28.7	0.7	0.4	5.6	BD	BD	BD	18.6

† Below Detection Limit

Table B8. Continued

Sample	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
47	150.7	7.4	3.0	6.2	223.2	0.8	BD†	4.0	BD	BD	BD	13.4
48	143.9	3.9	6.9	4.0	162.2	1.6	0.1	13.6	BD	BD	BD	46.7
49	12.2	8.9	1.9	7.7	11.9	0.5	BD	4.7	BD	BD	BD	10.0
50	5.5	2.5	1.8	BD	3.4	0.6	0.5	24.5	BD	BD	BD	64.6
51	58.5	1.7	2.3	BD	63.5	0.7	BD	11.2	BD	BD	BD	26.9
52	154.0	3.9	6.8	3.3	156.4	1.3	0.3	13.5	BD	BD	BD	47.1
53	15.6	9.8	5.3	3.7	27.4	0.4	BD	1.1	BD	BD	BD	3.3
54	164.5	12.2	19.8	7.6	236.5	0.9	0.1	32.1	0.1	BD	BD	112.3
55	13.8	13.3	4.2	BD	6.4	0.4	0.1	9.0	BD	BD	0.1	10.0
56	124.5	23.2	42.1	5.7	84.5	1.0	0.3	105.0	0.2	0.1	2.3	192.0
57	40.1	14.7	7.4	4.5	62.2	0.2	BD	1.0	BD	BD	0.1	1.8
58	6.3	239.6	4.5	9.8	527.7	0.2	BD	BD	BD	BD	0.3	BD

† Below Detection Limit

Table B9. Secondary elemental concentrations in mg L⁻¹ of leachate from the Synthetic Precipitation Leaching Procedure performed on the separated solids portion of the mud.

Sample	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
1	BD†	BD	BD	BD	BD	BD	0.01	BD
2	BD	BD	BD	0.02	BD	BD	0.01	BD
3	BD	0.02	BD	0.06	BD	BD	0.02	BD
4	BD	BD	BD	0.34	BD	0.01	0.09	BD
5	BD	0.05	BD	0.04	BD	BD	0.01	BD
6	BD	0.03	BD	0.09	BD	0.02	0.03	BD
7	BD	0.01	BD	0.06	BD	BD	0.02	BD
8	BD	BD	BD	0.08	BD	0.01	0.01	BD
9	BD	BD	BD	BD	BD	BD	BD	BD
10	BD	BD	BD	BD	BD	BD	0.01	BD
11	BD	0.02	BD	0.10	BD	0.01	0.04	BD
12	BD	0.02	BD	0.09	BD	0.01	0.03	BD
13	BD	0.03	BD	0.13	BD	0.02	0.02	BD
14	BD	BD	BD	BD	BD	BD	0.01	BD
15	BD	0.02	BD	0.05	BD	BD	0.01	BD
16	BD	BD	BD	0.06	BD	BD	0.02	BD
17	0.03	BD	BD	0.24	BD	0.02	0.12	BD
18	BD	BD	BD	0.05	BD	BD	0.02	BD
19	BD	BD	BD	0.33	BD	0.02	0.06	BD
20	BD	0.02	BD	0.05	BD	BD	0.01	BD
21	BD	0.05	BD	0.12	BD	BD	BD	BD
22	BD	0.05	BD	0.14	BD	BD	BD	BD
23	BD	BD	BD	0.04	BD	BD	0.01	BD
24	BD	BD	BD	0.01	BD	BD	0.01	BD
25	BD	0.03	BD	0.13	BD	0.01	0.03	BD
26	BD	0.02	BD	0.07	BD	0.01	0.02	BD
27	BD	BD	BD	0.06	BD	BD	0.02	BD
28	BD	BD	BD	BD	BD	BD	0.01	BD
29	BD	BD	BD	0.08	BD	0.01	0.03	BD
30	BD	BD	BD	BD	BD	BD	BD	BD
31	BD	BD	BD	0.07	BD	BD	BD	BD
32	BD	0.02	BD	0.12	BD	BD	BD	BD
33	BD	0.03	BD	0.04	BD	BD	0.01	BD
34	BD	0.02	BD	0.04	BD	BD	BD	BD
35	BD	BD	BD	0.20	BD	BD	0.04	BD
36	BD	0.02	BD	0.24	BD	BD	0.10	BD
37	BD	0.04	BD	0.04	BD	BD	0.01	BD

† Below Detection Limit

Table B9. Continued

Sample	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
39	BD†	BD	BD	0.02	BD	0.01	0.01	BD
40	BD	0.02	BD	0.06	BD	BD	0.01	BD
41	0.04	0.05	BD	0.03	BD	BD	0.01	BD
42	BD	0.03	BD	0.02	BD	0.01	BD	BD
43	BD	BD	BD	0.04	BD	BD	0.01	BD
44	BD	0.01	BD	0.05	BD	BD	0.03	BD
45	0.16	0.05	BD	0.04	BD	0.06	0.01	BD
46	BD	BD	BD	0.04	BD	BD	BD	BD
47	BD	0.04	BD	0.05	BD	BD	BD	BD
48	BD	0.04	BD	0.03	BD	BD	BD	BD
49	BD	BD	BD	0.06	BD	BD	0.02	BD
50	BD	BD	BD	0.13	BD	0.01	0.05	BD
51	BD	BD	BD	0.07	BD	BD	0.03	BD
52	BD	0.03	BD	0.03	BD	0.01	0.01	BD
53	BD	0.02	BD	0.02	BD	BD	0.01	BD
54	BD	0.02	BD	0.05	BD	BD	BD	BD
55	BD	0.03	BD	0.06	BD	BD	0.02	BD
56	0.08	0.01	BD	0.23	BD	0.05	0.03	BD
57	BD	BD	BD	0.09	BD	BD	0.01	BD
58	BD	BD	BD	0.06	BD	BD	0.01	BD

† Below Detection Limit

Table B10. Loading rates in kg ha⁻¹ of trace metals in the solids portion and Total Dissolved Solids in kg ha⁻¹ of mud if applied at 112 metric tons solid ha⁻¹.

Sample	Ni	B	As	Cd	Cr	Ba	Pb	Co	TDS
1	0.3	7.0	BD†	BD	3.1	1.9	BD	BD	31
2	2.2	8.0	BD	BD	4.5	5.9	BD	BD	32
3	1.1	2.7	BD	BD	1.5	6.7	BD	BD	324
4	1.2	3.7	BD	BD	2.0	10.2	0.1	BD	236
5	3.3	6.1	BD	BD	2.8	27.0	0.1	0.7	782
6	2.6	5.0	BD	BD	1.9	7.7	BD	BD	136
7	0.9	2.1	BD	BD	1.0	6.4	BD	BD	47
8	0.8	2.8	BD	BD	1.8	8.6	0.5	BD	355
9	0.9	7.3	BD	BD	3.8	2.5	BD	BD	69
10	0.4	5.4	BD	BD	2.9	1.7	BD	BD	51
11	1.8	4.5	BD	BD	1.5	3.4	0.1	BD	273
12	1.8	5.0	BD	BD	1.8	3.8	BD	BD	254
13	0.7	2.9	BD	BD	0.7	5.5	BD	BD	547
14	1.2	6.1	BD	BD	2.6	4.7	BD	0.5	28
15	3.2	7.5	BD	BD	2.7	14.1	BD	BD	80
16	1.3	3.3	BD	BD	1.3	4.6	BD	BD	90
17	0.3	1.0	BD	BD	1.0	5.8	0.9	BD	231
18	0.3	1.3	BD	BD	0.9	2.6	0.4	BD	93
19	1.3	3.1	BD	BD	1.2	5.2	BD	BD	202
20	1.0	2.2	BD	BD	1.0	3.4	BD	BD	646
21	1.8	4.0	BD	BD	1.8	15.5	2.1	BD	2614
22	1.7	3.9	BD	BD	1.9	13.9	2.1	BD	2582
23	0.3	5.2	BD	BD	0.9	14.6	BD	BD	241
24	0.2	11.1	BD	BD	5.4	3.5	BD	BD	12
25	1.3	3.0	BD	BD	1.4	10.7	BD	BD	258
26	2.4	5.4	0.2	BD	2.2	22.0	7.2	BD	74
27	2.2	7.8	BD	BD	12.4	28.4	BD	BD	64
28	0.1	BD	BD	BD	0.7	1.1	0.3	BD	30
29	1.6	5.1	BD	BD	2.3	8.1	BD	BD	35
30	0.9	4.0	BD	BD	2.3	5.8	BD	BD	11
31	0.5	1.8	BD	BD	0.7	48.5	BD	BD	129
32	0.8	5.8	BD	BD	1.1	13.4	BD	BD	660
33	0.5	2.2	BD	BD	0.5	8.8	0.5	BD	1065
34	0.6	2.0	BD	BD	0.7	7.5	1.1	BD	1357
35	0.3	0.5	BD	BD	0.6	4.0	0.2	BD	58
36	0.3	0.7	BD	BD	0.8	5.2	0.9	BD	243
37	5.3	6.1	BD	BD	6.1	28.5	BD	BD	515

† Below Detection Limit

Table B10. Continued

Sample	Ni	B	As	Cd	Cr	Ba	Pb	Co	TDS
38	1.3	6.5	BD†	BD	2.0	15.8	BD	BD	322
39	2.4	3.3	BD	BD	1.8	3.1	0.8	BD	40
40	3.6	5.3	BD	BD	3.5	17.2	BD	1.3	733
41	4.7	6.6	BD	BD	4.6	20.2	BD	1.8	1288
42	0.3	1.2	BD	BD	0.4	2.3	BD	0.1	351
43	2.2	4.5	BD	BD	2.4	26.2	BD	0.9	77
44	0.3	1.2	BD	BD	0.9	2.7	BD	0.2	46
45	1.8	3.8	BD	BD	0.8	19.5	BD	0.9	3723
46	0.5	1.6	BD	BD	0.2	6.4	BD	0.2	256
47	0.5	2.5	BD	BD	0.5	120.3	BD	0.5	1101
48	0.9	3.4	BD	BD	1.0	18.1	BD	0.5	1066
49	2.8	7.1	BD	BD	3.6	14.1	BD	1.9	39
50	0.6	1.3	BD	BD	1.1	4.4	BD	0.2	14
51	0.4	5.8	BD	BD	3.0	6.2	BD	0.4	69
52	2.1	2.6	BD	BD	1.6	4.3	BD	0.5	919
53	1.7	1.5	BD	BD	1.3	4.3	BD	0.6	71
54	0.4	1.5	BD	BD	0.3	3.1	BD	0.1	1091
55	2.2	3.8	BD	BD	2.3	13.2	BD	0.7	114
56	2.9	4.5	BD	BD	1.5	13.2	BD	1.3	791
57	BD	17.9	BD	BD	1.7	49.1	BD	1.2	
58	BD	24.0	BD	BD	4.3	24.2	BD	1.9	-

† Below Detection Limit

APPENDIX C

Table C3. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm. † Below Detection Limit

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C1	40	0-5	93.1	92.6	22.4	26.4	71.4	BD†	0.9	0.1	BD	0.1	0.2	0.1	2.3
C1	40	5-10	18.9	87.5	17.0	8.1	46.9	BD	0.3	0.5	0.1	0.1	0.3	0.7	0.5
C1	0	0-5	11.7	81.5	24.5	35.3	40.2	BD	1.3	0.2	0.1	0.1	0.4	0.2	0.3
C1	0	5-10	9.5	30.0	6.5	4.7	23.1	0.1	0.5	0.2	BD	0.6	0.1	0.2	0.4
C1	30	0-5	64.4	119.8	30.6	30.2	77.4	BD	1.2	0.2	0.1	0.2	0.3	0.1	1.4
C1	30	5-10	16.1	72.5	14.8	5.8	34.4	BD	0.3	BD	BD	0.1	0.2	0.1	0.5
C1	40	0-5	65.6	120.0	29.2	41.5	91.6	BD	1.3	0.2	0.1	0.2	0.3	0.1	1.4
C1	40	5-10	17.5	96.3	18.3	12.4	56.3	BD	0.4	0.1	BD	0.1	0.2	0.1	0.4
C1	20	0-5	48.1	110.8	25.4	31.8	72.5	BD	0.9	0.4	0.1	0.1	0.4	0.4	1.1
C1	20	5-10	13.2	78.8	15.2	8.1	41.7	BD	0.5	0.1	BD	0.1	0.2	0.1	0.4
C1	50	0-5	137.5	108.2	24.7	31.9	151.7	BD	1.1	0.5	0.3	0.2	0.3	0.2	3.1
C1	50	5-10	16.2	89.0	16.8	7.2	73.6	BD	0.4	BD	BD	BD	0.3	0.1	0.4
C2	30	0-5	67.8	111.1	29.9	45.8	75.2	BD	1.3	0.4	0.1	0.1	0.4	0.5	1.5
C2	30	5-10	18.2	63.8	13.7	12.4	46.6	BD	0.4	0.3	0.2	0.1	0.3	0.4	0.5
C2	40	0-5	94.7	95.7	25.2	31.8	95.1	BD	1.1	0.5	0.1	0.1	0.2	0.2	2.2
C2	40	5-10	18.4	84.3	15.4	10.3	57.2	BD	0.5	0.4	0.2	BD	0.3	0.1	0.5
C2	50	0-5	126.9	103.9	27.2	45.6	107.1	BD	1.2	0.2	0.1	0.1	0.2	0.1	2.9
C2	50	5-10	20.8	76.6	13.8	9.5	55.6	BD	0.4	0.4	0.1	BD	0.2	0.5	0.6

Table C1. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C2	40	0-5	112.9	87.6	22.5	37.4	87.0	BD†	1.0	0.1	BD	0.1	0.2	0.1	2.8
C2	40	5-10	21.3	92.1	16.2	10.8	57.4	BD	0.3	0.1	BD	BD	0.2	0.1	0.5
C2	0	0-5	10.4	106.5	31.8	39.2	43.6	BD	1.1	0.2	0.1	0.2	0.6	0.2	0.2
C2	0	5-10	8.7	32.4	7.0	5.3	22.5	BD	0.5	0.1	BD	BD	0.2	0.2	0.4
C2	10	0-5	32.6	124.3	32.6	36.2	58.6	BD	1.1	0.4	0.1	0.1	0.4	0.1	0.7
C2	10	5-10	12.9	84.2	15.1	4.8	30.0	BD	0.3	BD	BD	BD	0.2	0.1	0.3
C3	0	0-5	8.1	95.4	25.1	31.7	50.2	BD	0.9	0.7	0.1	0.1	0.4	1.0	0.2
C3	0	5-10	5.8	31.2	6.5	4.9	26.8	BD	0.5	0.5	0.1	BD	0.2	0.7	0.2
C3	30	0-5	58.1	106.9	24.6	31.6	71.6	BD	1.0	0.1	0.1	0.2	0.3	0.1	1.3
C3	30	5-10	18.4	83.1	15.0	8.4	44.5	BD	0.4	0.1	BD	0.1	0.2	0.1	0.5
C3	20	0-5	38.7	127.5	29.3	39.3	72.3	BD	1.1	0.1	0.1	0.8	0.3	0.1	0.8
C3	20	5-10	11.5	75.9	13.7	8.0	34.7	BD	0.4	0.3	0.1	BD	0.2	0.4	0.3
C3	20	0-5	53.3	117.5	28.9	32.9	76.3	BD	1.1	0.2	0.1	0.1	0.3	0.1	1.1
C3	20	5-10	12.3	80.2	14.8	6.1	42.0	BD	0.4	BD	BD	BD	0.1	BD	0.3
C3	10	0-5	27.0	126.9	28.3	20.0	57.3	BD	0.9	0.1	0.1	0.3	0.2	0.1	0.6
C3	10	5-10	12.1	71.3	10.7	3.5	35.7	0.1	0.5	0.3	0.1	0.1	0.1	0.5	0.4
C3	10	0-5	15.8	119.2	24.6	24.8	46.4	0.1	1.0	0.4	0.1	0.1	0.1	0.2	0.3
C3	10	5-10	10.1	68.9	9.5	3.3	32.4	0.1	0.4	0.2	0.1	BD	0.1	0.3	0.3
C3	50	0-5	103.3	102.1	22.2	22.5	86.0	BD	1.2	0.3	0.1	0.1	0.2	0.1	2.4
C3	50	5-10	20.8	74.1	13.3	4.9	53.1	BD	0.5	0.2	BD	0.1	0.1	0.1	0.6

† Below Detection Limit

Table C4. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 30 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C1	40	0-5	62.8	110.3	27.6	47.4	61.6	BD†	0.6	0.6	0.1	0.1	0.2	0.3	1.4
C1	40	5-10	31.7	77.0	15.4	9.4	25.7	BD	0.4	0.2	BD	0.1	0.1	BD	0.9
C1	0	0-5	9.4	63.4	21.6	44.0	49.3	0.1	2.4	0.7	0.1	BD	0.4	0.3	0.3
C1	0	5-10	6.7	18.9	4.7	5.1	16.0	0.1	0.2	0.2	0.1	BD	0.1	0.1	0.4
C1	30	0-5	43.7	137.2	37.0	50.7	87.0	BD	2.4	0.6	0.1	0.1	0.3	0.2	0.9
C1	30	5-10	18.3	33.5	8.1	5.3	24.3	BD	0.3	0.2	BD	0.1	0.2	0.2	0.7
C1	40	0-5	42.5	105.7	26.3	42.9	75.8	BD	1.8	0.5	0.1	0.1	0.2	0.1	1.0
C1	40	5-10	17.4	46.0	9.7	9.8	30.8	BD	0.4	0.2	0.1	0.1	0.1	0.1	0.6
C1	20	0-5	19.6	124.3	33.2	61.1	71.7	BD	2.0	0.8	0.2	0.1	0.3	0.2	0.4
C1	20	5-10	11.0	43.1	9.5	11.3	25.6	BD	0.4	0.2	0.1	0.1	0.2	0.1	0.4
C1	50	0-5	61.5	107.8	23.8	28.3	77.2	BD	0.8	0.2	BD	0.1	0.1	0.2	1.4
C1	50	5-10	18.6	40.2	8.4	6.4	31.0	BD	0.4	0.1	BD	BD	0.1	0.1	0.7
C2	30	0-5	48.8	117.7	33.8	52.5	56.4	BD	1.5	0.3	BD	0.1	0.2	0.2	1.0
C2	30	5-10	24.1	76.5	16.5	11.2	27.2	BD	0.3	0.1	BD	BD	0.2	BD	0.7
C2	40	0-5	47.3	106.6	29.1	34.0	53.7	BD	2.0	0.5	0.1	0.1	0.2	0.2	1.0
C2	40	5-10	16.2	57.4	11.2	8.3	23.2	BD	0.2	0.1	BD	BD	0.1	BD	0.5
C2	50	0-5	74.1	101.9	26.0	46.4	60.9	BD	1.0	0.4	0.1	0.1	0.2	0.2	1.7
C2	50	5-10	29.5	76.0	14.1	12.0	44.0	BD	0.4	0.2	0.1	0.1	0.2	0.1	0.8
C2	40	0-5	53.6	105.3	29.4	46.4	60.9	BD	0.8	0.4	0.1	BD	0.2	0.2	1.2

† Below Detection Limit

Table C2. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C2	40	5-10	26.6	70.0	14.0	15.8	31.9	BD†	0.4	0.1	0.1	0.1	0.2	0.1	0.8
C2	0	0-5	7.8	115.5	36.3	49.3	48.7	BD	1.6	0.7	0.1	0.1	0.7	0.3	0.2
C2	0	5-10	6.1	20.2	4.5	3.5	14.5	BD	0.5	0.2	BD	BD	0.1	0.1	0.3
C2	10	0-5	15.8	133.6	36.2	45.7	75.2	BD	1.6	0.5	0.1	BD	0.7	0.2	0.3
C2	10	5-10	9.9	28.4	5.9	3.4	20.1	BD	0.3	0.1	BD	BD	0.1	0.1	0.4
C3	0	0-5	5.7	84.8	24.5	50.1	57.4	BD	1.5	0.7	0.1	BD	0.4	0.4	0.1
C3	0	5-10	5.3	48.8	9.7	13.9	22.8	BD	0.6	0.2	BD	BD	0.2	0.1	0.2
C3	30	0-5	50.0	94.8	23.0	37.8	54.9	BD	1.3	0.2	BD	BD	0.2	0.2	1.2
C3	30	5-10	19.9	44.4	9.0	9.3	23.4	BD	0.5	0.3	BD	0.1	0.1	0.2	0.7
C3	20	0-5	24.5	98.3	24.3	26.2	51.6	BD	1.1	0.2	0.1	0.1	0.1	0.1	0.6
C3	20	5-10	10.5	35.5	7.3	5.9	19.0	BD	0.3	0.4	BD	BD	0.1	0.3	0.4
C3	20	0-5	29.3	121.7	29.2	33.6	68.9	BD	1.3	0.6	0.1	0.1	0.2	0.2	0.6
C3	20	5-10	12.4	38.5	7.8	4.0	18.2	BD	0.3	0.1	BD	BD	0.1	BD	0.5
C3	10	0-5	20.1	94.7	20.0	19.2	45.2	0.1	0.9	0.2	0.2	0.2	0.2	0.1	0.5
C3	10	5-10	13.8	65.2	9.8	2.7	34.5	0.1	0.2	BD	BD	BD	BD	BD	0.4
C3	10	0-5	17.5	126.9	28.3	27.0	44.5	0.1	1.4	0.3	0.1	0.1	0.1	0.3	0.4
C3	10	5-10	11.2	53.7	8.5	3.3	24.3	0.1	0.3	0.1	BD	0.1	BD	0.2	0.4
C3	50	0-5	64.6	90.1	20.0	21.6	64.9	BD	1.1	0.2	0.1	0.2	0.1	0.1	1.6
C3	50	5-10	30.1	46.7	9.1	5.0	30.2	BD	0.3	0.3	BD	BD	0.1	0.1	1.1

† Below Detection Limit

Table C5. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 93 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C1	40	0-5	24.0	128.3	29.5	30.5	55.7	BD†	2.6	0.2	BD	0.1	0.2	0.1	0.5
C1	40	5-10	31.4	66.5	14.0	7.5	29.9	BD	1.1	0.1	BD	BD	0.3	BD	0.9
C1	0	0-5	7.4	71.9	23.5	32.9	47.7	BD	4.2	0.3	BD	1.1	0.4	0.1	0.2
C1	0	5-10	13.4	41.8	9.6	9.2	25.1	BD	0.9	0.2	BD	0.1	0.4	0.2	0.5
C1	30	0-5	29.9	135.1	37.1	29.9	56.4	BD	3.4	0.2	BD	0.4	0.4	0.2	0.6
C1	30	5-10	23.6	55.2	13.2	8.7	29.7	BD	1.3	0.4	BD	0.1	0.5	0.6	0.7
C1	40	0-5	27.0	192.4	45.8	83.6	124.7	BD	7.4	0.3	BD	0.2	0.5	0.2	0.5
C1	40	5-10	19.0	51.5	11.5	12.4	28.3	BD	1.2	0.1	BD	BD	0.3	BD	0.6
C1	20	0-5	15.6	138.1	33.5	180.6	67.9	BD	3.8	0.2	BD	0.1	0.3	0.1	0.3
C1	20	5-10	18.3	46.8	10.1	7.2	24.8	BD	1.2	0.3	BD	BD	0.3	0.5	0.6
C1	50	0-5	26.2	152.3	33.2	48.3	81.2	BD	5.1	0.2	BD	0.1	0.2	0.2	0.5
C1	50	5-10	42.7	60.6	12.4	6.9	31.9	BD	1.3	0.1	BD	BD	0.3	BD	1.3
C2	30	0-5	20.9	167.3	43.9	46.0	73.3	BD	4.8	0.4	BD	0.2	0.4	0.3	0.4
C2	30	5-10	27.1	81.9	17.4	11.1	28.7	BD	0.9	0.1	BD	BD	0.3	0.1	0.7
C2	40	0-5	31.4	103.0	23.4	21.7	38.0	BD	1.3	0.1	BD	0.1	0.2	0.1	0.7
C2	40	5-10	26.9	55.2	10.8	12.2	31.5	BD	1.1	0.1	BD	BD	0.3	0.2	0.9
C2	50	0-5	27.5	148.8	37.3	39.6	67.3	BD	2.9	0.2	BD	0.1	0.3	0.2	0.5
C2	50	5-10	40.7	70.5	13.6	11.7	30.9	BD	1.4	0.1	BD	BD	0.2	0.1	1.2
C2	40	0-5	56.9	118.1	28.1	29.0	62.7	BD	2.4	0.2	BD	0.1	0.3	BD	1.2

† Below Detection Limit

Table C3. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
C2	40	5-10	61.1	78.5	16.0	13.8	52.2	BD†	1.6	0.1	BD	0.1	0.4	0.2	1.6
C2	0	0-5	6.6	70.7	24.0	28.8	45.9	BD	3.4	0.2	BD	0.1	0.6	0.4	0.2
C2	0	5-10	7.9	40.3	8.7	6.8	23.7	BD	1.4	0.1	BD	BD	0.4	0.1	0.3
C2	10	0-5	12.1	114.8	31.4	20.1	55.2	BD	2.8	0.2	BD	0.1	0.5	0.2	0.3
C2	10	5-10	14.2	46.8	9.4	5.2	24.6	BD	1.3	0.1	BD	BD	0.3	0.1	0.5
C3	0	0-5	5.5	88.7	27.8	33.2	64.9	BD	3.0	0.4	BD	0.1	0.5	0.5	0.1
C3	0	5-10	8.0	64.0	14.0	10.7	38.1	BD	1.3	0.1	BD	0.1	0.6	0.1	0.2
C3	30	0-5	18.3	135.4	32.0	30.4	64.1	BD	3.1	0.2	BD	0.1	0.2	0.2	0.4
C3	30	5-10	23.9	81.3	15.4	10.9	32.8	BD	0.9	0.1	BD	BD	0.4	0.1	0.6
C3	20	0-5	13.7	116.8	26.5	22.7	57.3	BD	2.4	0.1	BD	0.1	0.2	BD	0.3
C3	20	5-10	15.5	51.8	9.8	9.0	26.5	BD	1.0	0.1	BD	0.1	0.3	0.2	0.5
C3	20	0-5	17.7	126.0	32.5	25.5	62.5	BD	2.8	0.2	BD	0.1	0.2	0.3	0.4
C3	20	5-10	18.5	52.4	10.8	7.6	25.5	BD	1.1	0.2	BD	0.1	0.3	0.3	0.6
C3	10	0-5	9.2	116.5	27.7	15.6	54.5	BD	2.5	0.3	BD	0.6	0.2	0.2	0.2
C3	10	5-10	16.2	72.3	13.0	5.6	34.6	BD	1.1	BD	BD	0.1	0.1	BD	0.5
C3	10	0-5	7.5	112.6	24.6	13.7	38.6	BD	2.5	0.1	BD	0.1	0.1	BD	0.2
C3	10	5-10	12.4	79.5	11.8	4.4	31.3	BD	1.2	BD	BD	BD	BD	BD	0.3
C3	50	0-5	27.8	136.0	28.0	24.6	70.1	BD	3.5	0.1	BD	0.3	0.1	0.2	0.6
C3	50	5-10	32.9	62.2	10.9	15.7	28.2	BD	1.2	0.1	BD	BD	0.1	BD	1.0

† Below Detection Limit

Table C6. Secondary elemental concentrations in mg L⁻¹ of saturated paste extractions at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C1	40	0-5	0.02	0.01	0.03	0.29	BD†	BD	0.01	BD
C1	40	5-10	0.02	BD	0.01	0.38	0.01	BD	0.02	BD
C1	0	0-5	0.02	BD	BD	0.35	BD	BD	0.01	BD
C1	0	5-10	0.05	BD	BD	0.14	BD	BD	0.01	BD
C1	30	0-5	0.03	BD	BD	0.39	BD	BD	0.01	BD
C1	30	5-10	0.03	BD	BD	0.32	BD	BD	0.01	BD
C1	40	0-5	0.03	BD	BD	0.44	BD	BD	0.02	BD
C1	40	5-10	0.01	BD	BD	0.46	BD	BD	0.02	BD
C1	20	0-5	0.01	BD	BD	0.42	BD	BD	0.01	BD
C1	20	5-10	0.03	BD	BD	0.37	BD	BD	0.01	BD
C1	50	0-5	0.04	BD	BD	0.35	BD	0.01	0.01	BD
C1	50	5-10	0.03	BD	0.02	0.39	BD	BD	0.01	BD
C2	30	0-5	0.05	0.01	0.01	0.34	BD	0.01	0.02	BD
C2	30	5-10	0.04	BD	BD	0.32	BD	BD	0.02	BD
C2	40	0-5	0.03	0.01	BD	0.29	BD	0.01	0.01	BD
C2	40	5-10	0.04	BD	BD	0.35	BD	BD	0.01	BD
C2	50	0-5	0.04	0.01	BD	0.36	BD	0.01	0.01	BD
C2	50	5-10	0.02	BD	BD	0.35	BD	BD	0.01	BD
C2	40	0-5	0.02	0.02	0.03	0.33	BD	BD	0.01	BD

† Below Detection Limit

Table C4. Continued

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C2	40	5-10	0.03	BD†	BD	0.40	BD	BD	0.01	BD
C2	0	0-5	0.03	BD	0.01	0.55	BD	BD	0.01	BD
C2	0	5-10	0.02	BD	BD	0.18	BD	BD	0.01	BD
C2	10	0-5	0.03	BD	BD	0.51	BD	BD	0.02	BD
C2	10	5-10	0.02	BD	0.01	0.36	BD	BD	0.01	BD
C3	0	0-5	0.02	BD	BD	0.44	BD	BD	0.02	BD
C3	0	5-10	0.02	BD	BD	0.18	BD	BD	0.01	BD
C3	30	0-5	0.06	BD	0.02	0.37	BD	BD	0.01	BD
C3	30	5-10	0.15	BD	BD	0.34	BD	BD	0.01	BD
C3	20	0-5	0.04	0.01	0.01	0.49	BD	BD	0.01	BD
C3	20	5-10	0.02	BD	BD	0.35	BD	BD	0.01	BD
C3	20	0-5	0.02	0.01	BD	0.46	BD	0.01	0.02	BD
C3	20	5-10	0.01	BD	BD	0.34	BD	BD	0.01	BD
C3	10	0-5	0.04	0.02	BD	0.83	BD	BD	0.02	BD
C3	10	5-10	0.02	BD	BD	0.40	BD	BD	0.02	BD
C3	10	0-5	0.02	0.03	BD	0.86	BD	BD	0.01	BD
C3	10	5-10	0.02	BD	BD	0.40	BD	BD	0.01	BD
C3	50	0-5	0.03	0.03	BD	0.48	BD	BD	0.02	BD
C3	50	5-10	0.02	BD	BD	0.33	BD	BD	0.01	BD

† Below Detection Limit

Table C7. Secondary elemental concentrations in mg L⁻¹ of saturated paste extractions at day 30 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C1	40	0-5	BD†	0.01	0.04	0.35	BD	BD	0.02	BD
C1	40	5-10	BD	BD	0.02	0.32	BD	BD	0.01	BD
C1	0	0-5	BD	BD	BD	0.29	BD	BD	0.02	BD
C1	0	5-10	BD	BD	BD	0.11	BD	BD	0.01	BD
C1	30	0-5	BD	0.02	0.02	0.47	BD	BD	0.02	BD
C1	30	5-10	BD	BD	BD	0.19	BD	BD	0.01	BD
C1	40	0-5	BD	BD	BD	0.41	BD	BD	0.02	BD
C1	40	5-10	BD	BD	BD	0.29	BD	BD	0.01	BD
C1	20	0-5	BD	BD	BD	0.44	BD	BD	0.02	BD
C1	20	5-10	BD	BD	BD	0.25	BD	BD	0.02	BD
C1	50	0-5	BD	BD	0.02	0.33	BD	BD	0.01	BD
C1	50	5-10	BD	BD	BD	0.21	BD	BD	0.01	BD
C2	30	0-5	BD	BD	BD	0.39	BD	BD	0.02	BD
C2	30	5-10	BD	BD	BD	0.38	BD	BD	0.01	BD
C2	40	0-5	BD	0.02	BD	0.32	BD	BD	0.02	BD
C2	40	5-10	BD	BD	BD	0.26	BD	BD	0.01	BD
C2	50	0-5	BD	0.02	BD	0.36	BD	BD	0.01	BD
C2	50	5-10	BD	BD	BD	0.37	BD	BD	0.01	BD
C2	40	0-5	BD	0.02	BD	0.41	BD	BD	0.02	BD

† Below Detection Limit

Table C5. Continued

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C2	40	5-10	BD†	BD	BD	0.38	BD	BD	0.01	BD
C2	0	0-5	BD	BD	BD	0.58	BD	BD	0.01	BD
C2	0	5-10	BD	BD	BD	0.11	BD	BD	0.01	BD
C2	10	0-5	BD	BD	BD	0.63	BD	BD	0.02	BD
C2	10	5-10	BD	BD	0.02	0.15	BD	BD	0.01	BD
C3	0	0-5	BD	BD	0.02	0.42	BD	BD	0.02	BD
C3	0	5-10	BD	BD	BD	0.27	BD	BD	0.01	BD
C3	30	0-5	BD	BD	BD	0.34	BD	BD	0.01	BD
C3	30	5-10	BD	BD	BD	0.23	BD	BD	0.02	BD
C3	20	0-5	BD	0.02	BD	0.35	BD	BD	0.01	BD
C3	20	5-10	BD	BD	BD	0.18	BD	BD	0.01	BD
C3	20	0-5	BD	0.02	0.01	0.49	BD	BD	0.02	BD
C3	20	5-10	BD	BD	BD	0.19	BD	BD	0.01	BD
C3	10	0-5	BD	0.02	BD	0.65	BD	BD	0.01	BD
C3	10	5-10	BD	BD	BD	0.41	BD	BD	0.01	BD
C3	10	0-5	BD	0.04	0.03	0.96	BD	BD	0.02	BD
C3	10	5-10	BD	0.01	BD	0.31	BD	BD	0.02	BD
C3	50	0-5	BD	0.05	BD	0.37	BD	BD	0.01	BD
C3	50	5-10	BD	BD	BD	0.25	BD	BD	0.02	BD

† Below Detection Limit

Table C8. Secondary elemental concentrations in mg L⁻¹ of saturated paste extractions at day 93 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on pastureland at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C1	40	0-5	BD†	BD	BD	0.33	BD	BD	BD	BD
C1	40	5-10	BD	BD	BD	0.27	BD	BD	BD	BD
C1	0	0-5	0.13	BD	BD	0.27	BD	BD	BD	BD
C1	0	5-10	BD	BD	BD	0.22	BD	BD	BD	BD
C1	30	0-5	BD	BD	BD	0.39	BD	BD	BD	BD
C1	30	5-10	BD	BD	BD	0.27	BD	BD	BD	BD
C1	40	0-5	BD	BD	BD	0.58	BD	BD	BD	BD
C1	40	5-10	BD	BD	BD	0.26	BD	BD	BD	BD
C1	20	0-5	BD	0.02	BD	0.39	BD	BD	BD	BD
C1	20	5-10	BD	BD	BD	0.21	BD	BD	BD	BD
C1	50	0-5	BD	BD	BD	0.39	BD	BD	BD	BD
C1	50	5-10	BD	BD	BD	0.24	BD	BD	BD	BD
C2	30	0-5	BD	0.02	BD	0.47	BD	BD	BD	BD
C2	30	5-10	BD	0.01	BD	0.32	BD	BD	BD	BD
C2	40	0-5	BD	0.01	BD	0.35	BD	BD	BD	BD
C2	40	5-10	BD	0.01	BD	0.24	BD	BD	BD	BD
C2	50	0-5	BD	0.01	BD	0.40	BD	BD	BD	BD
C2	50	5-10	BD	0.02	BD	0.28	BD	BD	BD	BD
C2	40	0-5	BD	0.02	BD	0.33	BD	BD	BD	BD

† Below Detection Limit

Table C6. Continued

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
C2	40	5-10	BD†	0.02	BD	0.34	BD	BD	BD	0.01
C2	0	0-5	BD	0.02	BD	0.33	BD	BD	BD	0.02
C2	0	5-10	BD	0.02	BD	0.20	BD	BD	BD	BD
C2	10	0-5	BD	0.02	BD	0.42	BD	BD	BD	BD
C2	10	5-10	BD	0.02	BD	0.23	BD	BD	BD	BD
C3	0	0-5	BD	0.02	BD	0.36	BD	BD	BD	BD
C3	0	5-10	BD	0.03	BD	0.32	BD	BD	BD	BD
C3	30	0-5	BD	0.03	BD	0.37	BD	BD	BD	BD
C3	30	5-10	BD	0.03	BD	0.32	BD	BD	BD	BD
C3	20	0-5	BD	0.03	BD	0.34	BD	BD	BD	BD
C3	20	5-10	BD	0.03	BD	0.22	BD	BD	BD	BD
C3	20	0-5	BD	0.03	BD	0.45	BD	BD	BD	BD
C3	20	5-10	BD	0.03	BD	0.20	BD	BD	BD	BD
C3	10	0-5	BD	0.03	BD	0.66	BD	BD	BD	BD
C3	10	5-10	BD	0.04	BD	0.38	BD	BD	BD	BD
C3	10	0-5	BD	0.04	BD	0.63	BD	BD	BD	BD
C3	10	5-10	BD	0.04	BD	0.41	BD	BD	BD	BD
C3	50	0-5	BD	0.04	BD	0.62	BD	BD	BD	BD
C3	50	5-10	BD	0.04	BD	0.30	BD	BD	BD	BD

† Below Detection Limit

Table C9. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on bare soil at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B1	40	0-5	429.8	234.9	49.4	13.5	588.3	0.08	0.29	0.05	0.03	0.03	BD†	0.09	6.7
B1	40	5-15	22.7	90.2	17.4	5.5	74.5	0.11	0.15	0.02	0.06	0.02	0.04	0.01	0.6
B1	20	0-5	211.0	177.1	30.0	9.7	253.9	0.11	0.21	0.04	0.06	0.07	0.07	0.05	3.9
B1	20	5-15	21.9	73.5	12.9	3.7	65.7	0.09	0.17	0.01	0.03	0.02	0.04	0.04	0.6
B1	30	0-5	379.4	288.8	34.2	11.6	353.1	0.36	0.21	0.02	0.03	0.08	0.02	0.07	5.6
B1	30	5-15	14.7	64.6	7.7	3.8	43.9	0.21	0.20	0.03	0.01	0.01	0.01	0.04	0.5
B1	10	0-5	124.6	168.6	21.3	5.5	118.9	0.18	0.23	0.04	0.01	0.06	BD	0.10	2.4
B1	10	5-15	16.3	53.7	7.5	2.9	33.4	0.26	0.13	0.02	0.01	0.03	BD	0.07	0.6
B1	50	0-5	568.5	307.4	48.2	21.1	518.6	0.13	0.22	0.03	0.02	0.03	BD	0.07	8.0
B1	50	5-15	25.5	94.8	15.5	8.9	61.0	0.16	0.15	0.01	0.01	0.04	BD	0.04	0.6
B1	0	0-5	23.9	89.3	13.5	5.9	51.8	0.19	0.16	0.01	0.01	0.05	BD	0.04	0.6
B1	0	5-15	28.6	61.3	11.4	3.3	41.6	0.23	0.13	BD	0.01	0.03	BD	0.07	0.9
B2	10	0-5	106.7	165.6	27.1	4.1	122.0	0.17	0.21	0.04	0.01	0.02	BD	0.05	2.0
B2	10	5-15	19.8	50.3	8.0	3.8	30.3	0.32	0.21	0.02	0.02	0.03	BD	0.07	0.7
B2	20	0-5	289.2	200.9	45.6	9.8	377.0	0.04	0.22	0.02	0.01	0.03	0.01	0.03	4.8
B2	20	5-15	18.3	57.0	12.9	3.6	47.2	0.08	0.25	0.03	0.04	0.04	0.04	0.05	0.6
B2	50	0-5	580.1	271.4	75.1	18.6	724.2	0.06	0.24	0.05	0.02	0.02	0.30	0.11	8.0
B2	50	5-15	31.8	49.3	18.1	5.3	50.4	0.12	0.21	0.06	0.07	0.02	0.03	0.08	1.0
B2	30	0-5	643.7	251.2	63.7	19.7	680.6	0.26	0.31	0.02	0.07	0.41	BD	0.03	9.4

† Below Detection Limit

Table C7. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B2	30	5-15	406.0	31.2	11.7	4.6	445.2	1.25	0.48	0.12	0.02	0.09	BD†	0.13	15.7
B2	40	0-5	637.3	195.1	54.6	15.4	634.6	0.22	0.24	0.01	BD	0.05	BD	0.05	10.4
B2	40	5-15	195.2	19.4	6.1	4.4	153.3	0.75	0.33	0.24	0.03	0.37	BD	0.07	9.9
B2	0	0-5	215.1	22.1	7.4	6.2	137.8	0.63	0.33	0.50	0.10	0.10	BD	0.28	10.1
B2	0	5-15	358.3	14.5	5.2	3.3	432.0	1.97	0.35	0.46	0.05	0.09	BD	0.33	20.5
B3	50	0-5	356.0	257.2	48.8	9.3	465.0	0.11	0.14	BD	0.04	0.04	BD	0.02	5.3
B3	50	5-15	22.3	52.3	14.1	4.8	42.3	0.15	0.10	BD	0.02	0.03	BD	0.04	0.7
B3	30	0-5	422.1	227.6	54.7	10.3	489.6	0.08	0.15	BD	0.01	0.02	BD	0.05	6.5
B3	30	5-15	56.0	40.9	12.8	6.1	41.8	0.15	0.22	0.03	0.06	0.02	BD	0.04	2.0
B3	0	0-5	51.5	41.0	12.4	3.2	52.6	0.57	0.16	0.01	0.02	0.03	BD	0.04	1.8
B3	0	5-15	109.6	13.2	6.1	2.3	34.9	1.01	0.22	0.03	0.01	0.06	BD	0.11	6.2
B3	20	0-5	266.4	197.1	38.7	9.0	262.4	0.19	0.30	0.02	0.03	0.06	BD	0.05	4.5
B3	20	5-15	109.5	21.2	5.4	2.7	49.5	0.31	0.30	0.15	0.03	0.04	0.01	0.11	5.5
B3	40	0-5	521.1	241.0	43.2	8.6	625.2	0.27	0.15	BD	0.01	0.02	0.01	0.05	8.1
B3	40	5-15	38.0	53.0	10.8	3.4	45.6	0.31	0.18	0.03	0.01	0.03	0.02	0.06	1.2
B3	10	0-5	131.6	151.6	24.5	8.5	149.7	0.33	0.15	0.01	BD	0.02	BD	0.09	2.6
B3	10	5-15	21.9	51.0	10.1	3.8	33.4	0.29	0.10	0.04	0.07	0.04	0.02	0.06	0.7

† Below Detection Limit

Table C10. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 29 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on bare soil at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B1	40	0-5	354.6	132.4	28.9	13.2	429.1	0.04	0.44	0.04	0.01	0.06	BD†	0.05	7.3
B1	40	5-15	56.2	184.2	36.6	5.9	114.4	0.05	0.17	BD	0.01	0.02	0.32	BD	1.0
B1	20	0-5	183.9	102.6	13.0	7.2	172.8	0.13	0.22	0.01	0.02	0.07	BD	0.10	4.5
B1	20	5-15	49.1	221.4	27.7	4.6	100.4	0.14	BD	BD	BD	0.03	0.01	0.03	0.8
B1	30	0-5	303.3	159.5	25.4	11.6	398.5	0.27	0.38	0.02	BD	0.06	BD	0.08	5.9
B1	30	5-15	48.7	225.9	33.7	6.4	93.8	0.11	0.13	BD	0.01	0.01	0.10	0.02	0.8
B1	10	0-5	59.5	61.1	8.0	2.4	72.7	0.20	0.20	0.04	0.01	0.06	BD	0.10	1.9
B1	10	5-15	27.1	94.9	11.4	2.1	58.6	0.22	0.10	BD	BD	0.03	BD	0.04	0.7
B1	50	0-5	252.0	79.9	13.5	7.3	265.2	0.12	0.27	0.04	0.01	0.11	BD	0.14	6.9
B1	50	5-15	98.5	184.6	22.7	3.3	134.3	0.21	0.13	0.02	0.01	0.02	0.04	0.23	1.8
B1	0	0-5	26.2	81.1	8.6	4.4	102.6	0.40	0.11	0.01	BD	0.05	BD	0.10	0.7
B1	0	5-15	20.1	49.3	6.9	1.8	29.6	0.34	0.09	BD	BD	0.04	BD	0.11	0.7
B2	10	0-5	79.7	84.9	11.0	2.8	95.4	0.17	0.29	0.05	0.01	0.22	BD	0.07	2.2
B2	10	5-15	20.1	96.0	12.8	2.5	55.6	0.18	0.09	BD	0.01	0.04	0.02	0.06	0.5
B2	20	0-5	195.7	89.4	20.0	5.7	235.9	0.05	0.21	0.03	0.04	0.07	0.21	0.06	4.9
B2	20	5-15	37.4	118.8	28.2	4.1	95.8	0.06	BD	BD	0.02	0.01	0.34	0.03	0.8
B2	50	0-5	227.3	81.0	21.7	9.2	245.6	0.07	0.35	0.03	0.01	0.07	BD	0.05	5.8
B2	50	5-15	57.7	116.1	42.2	6.6	84.7	0.11	0.22	BD	0.01	0.03	0.24	0.05	1.2
B2	30	0-5	576.7	112.5	34.2	11.8	567.8	0.27	0.20	0.01	BD	0.07	BD	0.10	12.2

† Below Detection Limit

Table C8. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B2	30	5-15	358.3	24.3	10.0	4.4	247.8	0.80	0.30	0.19	0.01	0.06	BD†	0.14	15.5
B2	40	0-5	572.8	81.0	25.3	9.7	563.9	0.45	0.30	0.03	0.01	0.12	0.07	0.09	14.2
B2	40	5-15	556.1	37.4	16.3	4.6	476.9	1.50	0.60	0.07	0.01	0.41	0.01	0.16	19.1
B2	0	0-5	172.1	18.0	5.0	3.7	88.4	0.51	0.32	0.24	0.02	0.08	BD	0.14	9.2
B2	0	5-15	271.4	11.3	3.4	1.8	223.8	1.54	0.90	1.07	0.03	0.12	BD	0.29	18.2
B3	50	0-5	269.2	88.6	13.8	5.5	301.6	0.17	0.17	0.01	BD	0.12	BD	0.07	7.0
B3	50	5-15	60.0	181.3	42.2	4.7	143.7	0.20	BD	BD	BD	0.03	0.01	0.02	1.0
B3	30	0-5	271.5	85.4	19.0	6.2	314.0	0.06	0.24	0.02	BD	0.14	0.03	0.12	6.9
B3	30	5-15	136.7	216.2	64.5	6.4	269.2	0.14	BD	BD	0.03	0.01	0.01	BD	2.1
B3	0	0-5	39.2	42.4	9.6	2.5	34.2	0.49	0.11	BD	BD	0.04	BD	0.09	1.4
B3	0	5-15	76.2	29.2	10.4	1.9	38.9	0.57	0.13	0.02	0.01	0.03	BD	0.07	3.1
B3	20	0-5	266.2	87.6	18.0	4.4	300.4	0.15	0.13	0.01	BD	0.09	BD	0.08	6.8
B3	20	5-15	185.8	65.8	17.3	2.9	132.4	0.32	0.08	BD	BD	0.03	BD	0.02	5.3
B3	40	0-5	288.1	83.6	13.8	4.6	318.1	0.28	0.11	BD	BD	0.04	BD	0.11	7.7
B3	40	5-15	90.3	113.2	27.5	3.2	98.0	0.38	0.07	BD	BD	0.03	BD	0.04	2.0
B3	10	0-5	76.8	51.1	7.1	3.1	61.2	0.30	0.24	0.06	0.02	0.10	BD	0.11	2.7
B3	10	5-15	35.3	44.4	7.6	2.0	38.7	0.24	0.15	0.03	0.02	0.03	BD	0.02	1.3

† Below Detection Limit

Table C9. Primary elemental concentrations in mg L⁻¹ and SAR of saturated paste extractions at day 96 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on bare soil at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B1	40	0-5	240.2	121.9	23.7	21.1	276.0	BD†	3.2	0.3	BD	0.1	0.2	0.2	5.2
B1	40	5-15	125.4	162.7	29.6	9.1	232.8	0.1	1.3	0.2	BD	BD	0.3	0.3	2.4
B1	20	0-5	83.9	90.7	17.3	12.6	120.5	BD	2.3	0.3	BD	0.1	0.2	0.4	2.1
B1	20	5-15	38.9	56.1	10.7	5.8	74.3	BD	1.3	0.3	BD	BD	0.1	0.4	1.2
B1	30	0-5	212.7	117.3	21.5	18.5	218.6	BD	3.7	0.2	BD	0.2	0.2	0.1	4.7
B1	30	5-15	115.8	147.8	26.5	11.0	199.3	BD	1.4	0.1	BD	BD	0.3	0.1	2.3
B1	10	0-5	69.7	115.7	10.3	6.1	106.7	0.2	2.5	0.1	BD	0.1	BD	0.1	1.7
B1	10	5-15	36.0	105.6	10.8	5.1	103.5	0.3	1.4	BD	BD	0.1	BD	0.1	0.9
B1	50	0-5	220.3	93.0	8.6	7.0	267.0	0.3	2.7	0.2	BD	0.2	BD	0.3	5.9
B1	50	5-15	90.5	131.9	12.7	4.6	151.4	0.3	0.9	0.3	BD	BD	BD	0.5	2.0
B1	0	0-5	34.9	177.6	16.2	8.7	279.0	0.6	1.6	0.1	BD	0.1	BD	0.3	0.7
B1	0	5-15	20.8	79.8	9.1	3.7	51.1	0.4	0.9	BD	BD	0.1	BD	0.1	0.6
B2	10	0-5	57.3	120.4	12.6	4.3	109.9	0.2	2.3	0.2	BD	0.1	BD	0.3	1.3
B2	10	5-15	34.9	97.2	11.6	4.5	71.9	0.2	1.6	0.1	BD	0.1	BD	0.1	0.9
B2	20	0-5	83.9	64.2	13.4	6.9	111.5	BD	1.9	0.4	BD	0.1	0.2	0.4	2.5
B2	20	5-15	55.6	118.4	24.3	6.4	125.5	BD	1.4	0.1	BD	BD	0.3	0.1	1.2
B2	50	0-5	189.5	92.1	22.1	113.3	220.1	0.1	2.3	0.3	BD	0.1	0.2	0.3	4.6
B2	50	5-15	93.6	103.9	32.1	7.9	180.0	0.1	1.2	0.1	BD	BD	0.2	0.1	2.1
B2	30	0-5	347.3	60.3	17.1	9.7	305.2	0.3	2.6	0.6	BD	0.1	0.1	0.7	10.2

† Below Detection Limit

Table C11. Continued

Rep	Rate	Depth	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al	SAR
B2	30	5-15	582.8	83.9	31.4	11.3	574.5	0.7	1.8	0.1	BD†	0.1	BD	0.1	13.8
B2	40	0-5	283.4	118.1	26.6	18.2	278.8	0.2	4.1	0.3	BD	0.2	0.2	0.2	6.1
B2	40	5-15	207.6	69.7	18.3	7.3	259.2	0.2	1.4	0.1	BD	BD	BD	0.1	5.7
B2	0	0-5	200.4	30.0	8.0	5.4	150.6	0.4	2.7	0.6	BD	0.1	BD	0.6	8.4
B2	0	5-15	343.3	22.1	7.5	3.4	350.3	1.1	3.2	0.7	BD	0.1	BD	0.5	16.1
B3	50	0-5	177.2	82.9	9.7	6.9	216.2	0.1	1.7	0.1	BD	0.1	BD	0.2	4.9
B3	50	5-15	99.8	147.3	24.0	6.8	187.7	0.1	0.7	BD	BD	BD	BD	BD	2.0
B3	30	0-5	162.8	72.2	14.7	7.3	162.3	0.1	1.6	0.2	BD	0.1	BD	0.3	4.6
B3	30	5-15	119.0	117.2	30.5	7.2	213.7	0.1	0.6	BD	BD	BD	BD	BD	2.5
B3	0	0-5	41.9	99.2	17.3	7.9	100.8	0.4	1.8	0.1	BD	0.1	BD	0.1	1.0
B3	0	5-15	86.5	31.3	9.6	3.7	45.4	0.5	1.3	0.2	BD	BD	BD	0.3	3.5
B3	20	0-5	117.1	68.4	9.2	7.3	116.5	0.2	2.0	0.5	BD	0.1	BD	0.9	3.5
B3	20	5-15	111.2	67.0	12.5	4.5	116.0	0.2	1.1	BD	BD	0.1	BD	0.1	3.3
B3	40	0-5	120.2	64.9	8.5	5.8	126.1	0.3	2.1	0.1	BD	0.1	BD	0.1	3.7
B3	40	5-15	83.5	50.4	10.2	4.2	89.7	0.3	1.1	0.1	BD	BD	BD	0.1	2.8
B3	10	0-5	58.3	125.0	14.9	8.4	161.0	0.5	1.6	0.1	BD	0.1	BD	0.2	1.3
B3	10	5-15	41.1	73.3	11.8	4.2	56.0	0.3	1.1	0.2	BD	BD	BD	0.3	1.2

† Below Detection Limit

Table C12. Secondary elemental concentrations in mg L⁻¹ of saturated paste extractions at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on bare soil at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
B1	40	0-5	BD†	BD	BD	0.32	BD	BD	0.02	BD
B1	40	5-15	BD	BD	BD	0.32	BD	BD	0.02	BD
B1	20	0-5	BD	BD	BD	0.60	BD	0.01	0.02	BD
B1	20	5-15	BD	BD	BD	0.32	BD	BD	0.02	BD
B1	30	0-5	BD	0.03	BD	0.76	BD	BD	0.02	BD
B1	30	5-15	BD	0.02	BD	0.33	BD	BD	0.02	BD
B1	10	0-5	BD	0.03	BD	0.70	BD	BD	0.01	BD
B1	10	5-15	BD	0.04	BD	0.29	BD	BD	0.02	BD
B1	50	0-5	BD	0.02	BD	0.59	BD	BD	0.01	BD
B1	50	5-15	BD	BD	BD	0.56	BD	BD	0.02	BD
B1	0	0-5	BD	0.01	BD	0.61	BD	BD	0.02	BD
B1	0	5-15	BD	BD	BD	0.33	BD	BD	0.02	BD
B2	10	0-5	BD	0.01	BD	0.67	BD	BD	0.02	BD
B2	10	5-15	BD	0.02	BD	0.21	BD	BD	0.02	BD
B2	20	0-5	0.02	BD	BD	0.33	BD	BD	0.02	BD
B2	20	5-15	BD	BD	BD	0.25	BD	BD	0.02	BD
B2	50	0-5	BD	BD	BD	0.24	BD	BD	0.02	BD
B2	50	5-15	BD	BD	BD	0.27	BD	BD	0.02	BD
B2	30	0-5	0.03	0.01	BD	0.45	BD	BD	0.02	BD

† Below Detection Limit

Table C10. Continued

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
B2	30	5-15	BD†	0.02	BD	0.11	BD	BD	0.02	BD
B2	40	0-5	BD	0.01	BD	0.33	BD	BD	0.02	BD
B2	40	5-15	0.03	BD	BD	0.07	BD	BD	0.02	BD
B2	0	0-5	0.03	BD	BD	0.11	BD	BD	0.02	BD
B2	0	5-15	0.02	0.01	BD	0.07	BD	BD	0.02	BD
B3	50	0-5	0.03	BD	BD	0.29	BD	BD	0.02	BD
B3	50	5-15	BD	BD	BD	0.21	BD	BD	0.02	BD
B3	30	0-5	BD	BD	BD	0.25	BD	BD	0.02	BD
B3	30	5-15	BD	BD	BD	0.19	BD	BD	0.01	BD
B3	0	0-5	BD	0.04	BD	0.18	BD	BD	0.02	BD
B3	0	5-15	BD	0.10	BD	0.06	BD	BD	0.02	BD
B3	20	0-5	BD	BD	BD	0.43	BD	BD	0.01	BD
B3	20	5-15	BD	0.01	BD	0.10	BD	BD	0.02	BD
B3	40	0-5	BD	0.02	BD	0.29	BD	BD	0.02	BD
B3	40	5-15	BD	0.01	BD	0.18	BD	BD	0.02	BD
B3	10	0-5	BD	0.04	BD	0.58	BD	BD	0.02	BD
B3	10	5-15	BD	0.02	BD	0.20	BD	0.01	0.02	BD

† Below Detection Limit

Table C13. Secondary elemental concentrations in mg L⁻¹ of saturated paste extractions at day 29 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid on bare soil at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
B1	40	0-5	BD†	BD	BD	0.20	BD	BD	BD	BD
B1	40	5-15	BD	BD	BD	0.56	BD	BD	BD	BD
B1	20	0-5	BD	BD	BD	0.34	BD	BD	BD	BD
B1	20	5-15	BD	BD	BD	0.88	BD	BD	BD	BD
B1	30	0-5	BD	BD	BD	0.37	BD	BD	BD	BD
B1	30	5-15	BD	BD	BD	0.98	BD	BD	BD	BD
B1	10	0-5	BD	0.05	BD	0.32	BD	BD	BD	BD
B1	10	5-15	BD	BD	BD	0.49	BD	BD	BD	0.02
B1	50	0-5	BD	BD	BD	0.24	BD	BD	BD	BD
B1	50	5-15	BD	BD	BD	0.67	BD	BD	BD	BD
B1	0	0-5	BD	BD	BD	0.59	BD	BD	BD	0.01
B1	0	5-15	BD	0.03	BD	0.29	BD	BD	BD	BD
B2	10	0-5	BD	0.03	BD	0.39	BD	BD	BD	BD
B2	10	5-15	BD	BD	BD	0.42	BD	BD	BD	BD
B2	20	0-5	BD	BD	BD	0.17	BD	BD	BD	BD
B2	20	5-15	BD	BD	BD	0.42	BD	BD	BD	BD
B2	50	0-5	BD	BD	BD	0.17	BD	BD	BD	BD
B2	50	5-15	BD	BD	BD	0.45	BD	BD	BD	BD
B2	30	0-5	BD	BD	BD	0.18	BD	BD	BD	BD

† Below Detection Limit

Table C11. Continued

Rep	Rate	Depth	Ni	Mo	As	Ba	Cd	Co	Cr	Pb
B2	30	5-15	BD†	BD	BD	0.09	BD	BD	BD	BD
B2	40	0-5	BD	BD	BD	0.13	BD	BD	BD	BD
B2	40	5-15	BD	BD	BD	0.10	BD	BD	BD	BD
B2	0	0-5	BD	BD	BD	0.09	BD	BD	BD	BD
B2	0	5-15	BD	BD	BD	0.05	BD	BD	BD	BD
B3	50	0-5	BD	BD	BD	0.18	BD	BD	BD	BD
B3	50	5-15	BD	BD	BD	0.56	BD	BD	BD	BD
B3	30	0-5	BD	BD	BD	0.19	BD	BD	BD	BD
B3	30	5-15	BD	BD	BD	0.46	BD	BD	BD	BD
B3	0	0-5	BD	0.05	0.03	0.19	BD	BD	BD	0.03
B3	0	5-15	BD	0.04	BD	0.10	BD	BD	BD	BD
B3	20	0-5	BD	BD	BD	0.17	BD	BD	BD	BD
B3	20	5-15	BD	BD	BD	0.22	BD	BD	BD	BD
B3	40	0-5	BD	0.03	BD	0.18	BD	BD	BD	BD
B3	40	5-15	BD	BD	0.03	0.29	BD	BD	BD	BD
B3	10	0-5	BD	0.04	BD	0.26	BD	BD	BD	BD
B3	10	5-15	BD	BD	BD	0.16	BD	BD	BD	BD

† Below Detection Limit

Table C14. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to pastureland, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
C1	40	0-5	193	18	268	5973	98	122	178	7
C1	40	5-15	107	7	159	1419	30	124	168	530
C1	0	0-5	176	44	194	1111	21	74	222	385
C1	0	5-15	86	10	141	971	25	128	158	519
C1	30	0-5	143	26	232	4761	62	95	194	22
C1	30	5-15	77	9	154	1244	26	133	169	522
C1	40	0-5	215	21	249	5061	65	113	184	20
C1	40	5-15	121	9	160	1434	25	137	169	560
C1	20	0-5	161	16	210	4138	48	106	169	45
C1	20	5-15	100	7	152	1339	21	143	149	537
C1	50	0-5	150	16	235	5686	116	118	176	19
C1	50	5-15	92	5	155	1358	22	146	150	553
C2	30	0-5	211	15	276	8217	69	120	176	21
C2	30	5-15	113	8	142	1016	21	116	162	525
C2	40	0-5	195	11	300	10346	107	127	175	BD†
C2	40	5-15	98	7	146	1329	23	130	164	529
C2	50	0-5	202	4	296	14942	112	142	166	BD
C2	50	5-15	101	6	136	1182	25	131	148	506
C2	40	0-5	216	6	279	12702	111	142	151	0
C2	40	5-15	115	8	143	1259	30	148	155	553
C2	0	0-5	214	34	257	1624	18	133	187	458
C2	0	5-15	80	9	148	1002	19	139	130	537
C2	10	0-5	187	23	254	4021	35	129	134	83
C2	10	5-15	77	6	158	1457	23	141	115	615
C3	0	0-5	195	20	222	1653	19	109	133	398
C3	0	5-15	98	6	150	1060	16	118	109	583
C3	30	0-5	167	14	225	4982	55	121	131	11
C3	30	5-15	99	5	144	1324	23	122	113	522
C3	20	0-5	183	18	231	4297	39	117	133	37
C3	20	5-15	102	6	147	1412	21	130	122	498
C3	20	0-5	162	23	250	4117	47	116	130	37
C3	20	5-15	85	6	154	1347	20	142	115	495
C3	10	0-5	172	59	362	5207	42	110	233	916
C3	10	5-15	66	37	253	2494	27	108	235	937
C3	10	0-5	205	75	387	4487	36	117	254	1128
C3	10	5-15	70	45	297	3149	32	108	266	1201
C3	50	0-5	142	20	262	5423	95	138	133	25
C3	50	5-15	72	8	170	1380	25	173	118	530

† Below Detection Limit

Table C15. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 30 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to pastureland, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
C1	40	0-5	202	11	287	11283	95	132	169	BD†
C1	40	5-15	103	9	154	1512	51	108	158	450
C1	0	0-5	186	47	238	1311	31	70	225	365
C1	0	5-15	88	11	128	836	27	103	144	433
C1	30	0-5	205	31	270	5435	59	110	185	15
C1	30	5-15	90	12	157	1069	40	109	172	475
C1	40	0-5	206	23	295	6433	72	119	178	23
C1	40	5-15	137	19	206	1441	44	128	217	706
C1	20	0-5	235	26	281	4557	38	106	175	92
C1	20	5-15	132	7	152	1112	30	126	148	485
C1	50	0-5	163	20	268	5596	83	109	179	15
C1	50	5-15	98	6	153	1206	38	129	150	510
C2	30	0-5	181	23	273	5314	65	99	174	11
C2	30	5-15	107	9	150	1267	39	101	158	448
C2	40	0-5	164	22	291	5429	67	111	170	17
C2	40	5-15	103	7	152	1357	35	129	148	448
C2	50	0-5	182	3	315	18611	91	157	167	BD
C2	50	5-15	112	7	139	1254	43	123	148	468
C2	40	0-5	200	19	295	5283	74	120	136	12
C2	40	5-15	133	8	144	1257	39	119	136	440
C2	0	0-5	198	28	261	1629	28	109	151	378
C2	0	5-15	109	16	163	1069	28	131	123	478
C2	10	0-5	150	22	246	3331	37	116	132	365
C2	10	5-15	71	6	153	1115	33	134	116	507
C3	0	0-5	198	24	248	1564	30	108	120	349
C3	0	5-15	103	5	138	1136	24	107	100	467
C3	30	0-5	186	14	272	4855	71	103	112	14
C3	30	5-15	112	6	139	1109	36	108	104	441
C3	20	0-5	139	17	255	4514	46	101	115	30
C3	20	5-15	97	6	142	1099	30	113	108	442
C3	20	0-5	156	16	252	4634	45	108	103	29
C3	20	5-15	73	5	145	1160	36	125	99	418
C3	10	0-5	174	66	388	5162	44	101	217	881
C3	10	5-15	66	54	450	5671	56	45	312	1419
C3	10	0-5	181	78	490	5827	47	84	260	1191
C3	10	5-15	81	51	321	2776	44	98	256	1185
C3	50	0-5	147	18	284	5512	87	121	104	15
C3	50	5-15	77	8	164	1258	49	144	99	456

† Below Detection Limit

Table C16. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 93 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to pastureland, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
C1	40	0-5	144	22	239	4438	19	80	104	BD†
C1	40	5-15	88	7	149	1233	29	82	90	307
C1	0	0-5	163	38	222	1310	6	53	139	222
C1	0	5-15	83	12	135	940	9	69	93	293
C1	30	0-5	130	30	241	3688	22	64	118	20
C1	30	5-15	81	9	139	940	20	72	92	291
C1	40	0-5	234	28	284	4547	18	87	113	3
C1	40	5-15	113	9	152	1130	15	90	107	338
C1	20	0-5	167	24	272	4624	13	79	105	BD
C1	20	5-15	97	7	155	1199	22	91	89	331
C1	50	0-5	164	13	243	4460	24	82	84	BD
C1	50	5-15	91	6	153	1236	31	91	90	343
C2	30	0-5	168	28	267	4381	17	68	103	BD
C2	30	5-15	91	11	154	1258	18	70	97	302
C2	40	0-5	114	12	216	4058	26	80	90	BD
C2	40	5-15	98	9	137	1159	24	91	95	302
C2	50	0-5	160	11	274	4030	20	80	83	BD
C2	50	5-15	105	8	139	1361	30	78	84	248
C2	40	0-5	165	20	248	4573	46	88	90	BD
C2	40	5-15	95	10	124	1083	42	72	83	293
C2	0	0-5	182	26	255	1420	8	83	93	296
C2	0	5-15	75	7	143	1048	8	90	84	321
C2	10	0-5	126	19	260	2672	18	84	80	356
C2	10	5-15	69	8	150	1190	18	93	87	391
C3	0	0-5	182	19	254	1509	12	83	87	247
C3	0	5-15	101	7	148	1045	5	90	71	363
C3	30	0-5	156	16	254	4632	22	86	77	BD
C3	30	5-15	106	7	139	1350	19	83	77	327
C3	20	0-5	138	15	241	4150	13	87	82	BD
C3	20	5-15	92	6	132	1104	12	89	75	313
C3	20	0-5	164	20	277	3698	14	91	87	13
C3	20	5-15	70	6	149	1177	14	105	74	309
C3	10	0-5	138	50	313	3115	11	90	115	492
C3	10	5-15	72	20	203	1729	16	104	121	621
C3	10	0-5	147	59	335	3810	10	87	141	658
C3	10	5-15	76	27	223	2211	16	118	141	710
C3	50	0-5	162	37	306	4789	29	96	98	14
C3	50	5-15	75	24	198	1837	36	126	124	570

† Below Detection Limit

Table C17. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 7 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to bare soil, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
B1	40	0-5	11	3	30	219	32	14	22	74
B1	40	5-15	7	1	20	147	5	19	12	51
B1	20	0-5	10	5	34	321	21	10	29	118
B1	20	5-15	7	2	21	189	5	16	16	69
B1	30	0-5	12	3	59	1080	35	2	32	181
B1	30	5-15	7	5	32	381	6	11	29	123
B1	10	0-5	9	3	55	998	17	2	35	178
B1	10	5-15	6	5	48	672	7	3	35	162
B1	50	0-5	15	5	50	584	41	3	35	163
B1	50	5-15	9	5	46	494	7	3	37	154
B1	0	0-5	8	4	52	724	8	3	35	170
B1	0	5-15	6	5	39	347	7	9	32	111
B2	10	0-5	8	3	61	915	15	2	35	175
B2	10	5-15	8	3	33	313	7	13	28	97
B2	20	0-5	8	1	24	162	22	16	18	50
B2	20	5-15	7	1	26	167	5	20	15	53
B2	50	0-5	11	1	29	175	39	19	14	44
B2	50	5-15	9	1	35	142	7	24	16	52
B2	30	0-5	13	3	38	269	43	14	25	95
B2	30	5-15	9	1	42	146	56	28	14	53
B2	40	0-5	11	2	32	179	44	18	20	69
B2	40	5-15	7	BD†	33	118	28	27	12	47
B2	0	0-5	11	1	29	111	35	23	19	58
B2	0	5-15	9	1	34	87	66	26	11	42
B3	50	0-5	7	1	24	168	26	26	15	58
B3	50	5-15	8	BD	36	164	4	31	10	48
B3	30	0-5	8	1	27	152	29	29	13	48
B3	30	5-15	8	BD	33	139	9	57	12	51
B3	0	0-5	7	2	44	248	9	22	22	75
B3	0	5-15	11	BD	65	261	26	26	9	41
B3	20	0-5	9	3	35	247	21	18	26	99
B3	20	5-15	6	1	27	139	17	23	17	56
B3	40	0-5	8	3	39	379	35	16	29	116
B3	40	5-15	6	1	23	151	6	24	13	48
B3	10	0-5	11	3	69	931	17	4	37	189
B3	10	5-15	7	1	25	178	5	20	17	60

† Below Detection Limit

Table C18. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 29 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to bare soil, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
B1	40	0-5	11	2	27	203	29	10	19	59
B1	40	5-15	8	1	21	157	5	16	11	49
B1	20	0-5	11	5	46	713	20	3	31	151
B1	20	5-15	7	6	41	468	6	5	34	140
B1	30	0-5	13	5	42	446	26	5	31	124
B1	30	5-15	8	3	24	205	4	12	20	73
B1	10	0-5	7	5	49	730	10	2	32	162
B1	10	5-15	5	6	44	610	5	3	34	149
B1	50	0-5	11	5	45	476	27	5	32	134
B1	50	5-15	6	5	43	572	9	3	34	155
B1	0	0-5	10	3	51	1194	6	2	33	194
B1	0	5-15	4	5	47	788	5	3	35	161
B2	10	0-5	6	4	52	698	11	3	33	176
B2	10	5-15	6	4	30	296	3	10	27	87
B2	20	0-5	8	1	20	149	16	14	13	36
B2	20	5-15	6	BD†	23	149	3	15	11	39
B2	50	0-5	10	1	22	141	18	15	11	37
B2	50	5-15	7	1	32	132	4	19	12	38
B2	30	0-5	11	1	31	183	44	16	12	38
B2	30	5-15	7	BD	37	111	49	22	11	40
B2	40	0-5	11	1	28	146	50	16	14	51
B2	40	5-15	6	1	36	102	60	21	11	45
B2	0	0-5	9	1	28	108	24	19	14	49
B2	0	5-15	6	1	34	93	50	21	11	41
B3	50	0-5	8	4	44	383	25	9	32	112
B3	50	5-15	9	BD	33	180	5	24	10	43
B3	30	0-5	8	1	25	175	21	20	15	49
B3	30	5-15	7	BD	31	138	8	31	12	43
B3	0	0-5	7	4	45	357	7	13	29	87
B3	0	5-15	6	BD	39	144	9	23	10	39
B3	20	0-5	6	2	28	210	24	16	21	66
B3	20	5-15	6	1	26	122	17	19	12	43
B3	40	0-5	8	3	57	644	31	7	32	157
B3	40	5-15	6	1	28	165	7	20	11	40
B3	10	0-5	7	4	56	768	11	5	33	172
B3	10	5-15	6	3	28	228	5	16	23	81

† Below Detection Limit

Table C19. Mehlich 3 extractable element concentrations in mg kg⁻¹ at day 96 with mud application rates equivalent to 0, 20, 30, 40, and 50 tons acre⁻¹ solid applied to bare soil, sampled at depths 0-5 cm and 5-15 cm.

Rep	Rate	Depth	K	P	Mg	Ca	Na	Mn	Fe	Al
B1	40	0-5	10	2	20	156	23	4	11	46
B1	40	5-15	7	1	18	134	14	5	9	40
B1	20	0-5	11	2	21	160	12	4	13	49
B1	20	5-15	9	1	17	133	9	5	9	41
B1	30	0-5	12	3	25	204	25	3	19	65
B1	30	5-15	9	1	17	133	14	4	9	43
B1	10	0-5	7	6	37	665	13	1	30	147
B1	10	5-15	7	6	37	566	11	1	33	146
B1	50	0-5	10	5	44	1015	33	1	31	161
B1	50	5-15	5	4	44	982	16	BD†	34	168
B1	0	0-5	10	3	46	1435	11	1	29	166
B1	0	5-15	5	5	43	805	9	1	34	161
B2	10	0-5	7	4	46	749	13	1	32	159
B2	10	5-15	8	5	31	329	10	3	29	96
B2	20	0-5	7	1	17	117	13	5	9	32
B2	20	5-15	8	1	21	139	9	5	11	39
B2	50	0-5	10	1	21	119	20	5	9	34
B2	50	5-15	8	1	26	111	11	6	8	31
B2	30	0-5	10	1	25	113	38	6	10	36
B2	30	5-15	8	1	35	120	55	7	12	39
B2	40	0-5	11	3	26	170	26	4	17	64
B2	40	5-15	9	1	26	125	24	7	10	40
B2	0	0-5	9	1	25	98	30	6	10	35
B2	0	5-15	7	1	33	103	56	7	12	39
B3	50	0-5	8	4	29	295	23	5	24	91
B3	50	5-15	10	1	24	161	13	7	8	36
B3	30	0-5	9	1	21	156	19	6	9	35
B3	30	5-15	9	BD	29	138	15	19	11	40
B3	0	0-5	9	5	42	378	10	4	28	102
B3	0	5-15	9	1	40	164	17	7	13	47
B3	20	0-5	8	4	34	346	18	4	27	95
B3	20	5-15	7	1	25	162	16	6	12	48
B3	40	0-5	9	5	37	436	19	4	27	102
B3	40	5-15	7	1	23	147	14	6	10	37
B3	10	0-5	10	4	55	925	14	2	32	163
B3	10	5-15	8	5	41	388	11	4	32	110

† Below Detection Limit

VITA

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