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The electrochemical reduction of nitrate in low conductivity aqueous solutions

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THE ELECTROCHEMICAL REDUCTION OF NITRATE IN LOW CONDUCTIVITY
AQUEOUS SOLUTIONS

A Thesis

Presented to

The Faculty of the Department of Chemical and Materials Engineering

San Jose State University

In Partial Fulfillment of the Requirements for the Degree

Masters of Science

by

Khavin Sinha

August 2005

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ABSTRACT

THE ELECTROCHEMICAL REDUCTION OF NITRATE IN LOW CONDUCTIVITY AQUEOUS SOLUTIONS

By Xhavin Sinha

This thesis investigates the effects of the initial reaction pH, the initial nitrate concentration and the working electrode on the electrochemical reduction of nitrate in a low conductivity aqueous solution. The ranges tested were at an initial pH of 7 and 9, and an initial concentration of 100 ppm nitrate and 500 ppm nitrate. Experiments were conducted under conditions of constant current and constant voltage. A full factorial analysis was done on the data. The most significant factor observed was found to be the copper electrode while very little reduction took place using a carbon-felt electrode. The reaction seemed to stop as the nitrate concentration increased. A possible reason being the reaction did not have an appropriate voltage and current levels. Additional experiments were conducted to test the effects of sulfate and chloride ions as possible electrolytes; however these ions did not appear to help the reaction.

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Table of Contents

CHAPTER 1 – INTRODUCTION	1
1.1 General Background.....	1
1.2 Electrochemistry.....	3
1.3 Competing Technologies.....	14
1.4 Advantages and Disadvantages.....	18
CHAPTER 2 – LITERATURE REVIEW	19
2.1 Theoretical Nitrate Reduction Reactions	19
2.2 Nitrate Reduction Kinetics on Noble Metal Cathodes	21
2.3 Nitrate Reduction Using Carbon Based Cathodes	25
2.4 Nitrate Reduction Using Copper Cathodes	28
2.5 Other Metal Electrodes and Materials.....	29
2.6 Other Effects	30
2.7 Summary	31
CHAPTER 3 – RESEARCH OBJECTIVES	32
3.1 Research Objectives	32
3.2 Justification	32
CHAPTER 4 – EXPERIMENTAL PROCEDURE AND ANALYSIS.....	33
4.1 Experimental Set-up and Procedure.....	33
4.2 Experimental Analysis	36
4.3 Data Analysis	36

CHAPTER 5 – EXPERIMENTAL DESIGN	38
CHAPTER 6 – RESULTS AND DISCUSSIONS	39
6.1 Degradation of Nitrate Ion on a Copper Cathode under Constant Current Conditions	39
6.2 Analysis of the Effects of Initial pH and Initial Concentration using a Copper Cathode under a Constant Current on Nitrite and Ammonia Formation in the Product	53
6.3 Error Analysis of Results using a Copper Cathode under Constant Current Conditions	56
6.4 Degradation of Nitrate Ion on a Copper Cathode under Constant Potential Conditions	58
6.5 Analysis of the Effects of Initial pH and Initial Concentration using a Copper Cathode under a Constant Potential on Nitrite and Ammonia Formation	71
6.6 Error Analysis of Results using a Copper Cathode Under Constant Potential Conditions	74
6.7 Comparison Between Constant Current and Constant Voltage Experimental Results	76
6.8 Additional Ion Concentration	77
6.9 Degradation Nitrate Ion on a Carbon-Felt Cathode under Constant Current Conditions	79
6.10 Degradation Nitrate Ion on a Carbon-Felt Cathode under Constant Potential Conditions	80
CHAPTER 7 – CONCLUSIONS AND FUTURE DIRCTIONS	81
NOMENCLATURE	84
REFERENCES	85

APPENDIX A – SAMPLE CALCULATIONS.....	88
APPENDIX B – RAW DATA FOR EXPERIMENTS CARRIED OUT UNDER CONSTANT CURRENT CONDITIONS.....	97
APPENDIX C – RAW DATA FOR EXPERIMENTS CARRIED OUT UNDER CONSTANT POTENTIAL CONDITIONS.....	145
APPENDIX D – RAW DATA FOR EXPERIMENTS CARRIED OUT UNDER VARIOUS CHLORIDE AND SULFATE CONCETRATIONS.....	193

List of Figures

Figure 1. A General Schematic of an Electrochemical Cell [6].....	4
Figure 2. Schematic for Reverse Osmosis Process for Nitrate Removal [4].	15
Figure 3. Schematic for Distillation Removal of Nitrate [4].....	15
Figure 4. Schematic for an Ion Exchange Nitrate Removal System [4].	16
Figure 5. The Nitrogen Cycle [1].	17
Figure 6. Possible Reaction Mechanisms for the Reduction of Nitrate [15].....	22
Figure 7. Schematic of the Experimental Set-Up.....	34
Figure 8. The Total Coulombic Efficiency versus Average Solution Conductivity under Constant Current Conditions.....	48
Figure 9. The Effects Plot for the Average Total Coulombic Efficiency under Constant Current Conditions using a Copper Cathode.	49
Figure 10. The Effects Plot for the Average Percent Nitrate Removal under Constant Current Conditions Using a Copper Cathode.	50
Figure 11. The Effects Plot for the Average Percent Product Formation (Average Percent NO_2 and NH_3) under Constant Current Conditions using a Copper Cathode.	51
Figure 12. The Effects Plot for the Average Percent of Product Formed that was Nitrite under Constant Current Conditions using a Copper Cathode.	54
Figure 13. The Effects Plot for the Average Percent of Product Formed that was Ammonia under Constant Current Conditions using a Copper Cathode.	55
Figure 14. The Coulombic Efficiency versus Average Solution Conductivity under Constant Potential Conditions.	66
Figure 15. Effects Plot for the Average Total Coulombic Efficiency under Constant Potential Conditions Using a Copper Cathode.	67

Figure 16. Effects Plot for the Average Percent Nitrate Removal under Constant Potential Conditions Using a Copper Cathode.	68
Figure 17. Effects Plot for the Average Percent Product Formation under Constant Potential Conditions Using a Copper Cathode.	72
Figure 18. Effects Plot for the Percentage of the Product Formed that is Ammonia Under Constant Potential Conditions using a Copper Cathode	73

List of Tables

Table 1. Some Common Electrode Materials [7].	8
Table 2. Comparison of Advantages and Disadvantages for Methods of Nitrate Removal.	18
Table 3. Summary of Activity and Selectivity for Different Metal Electrodes.	31
Table 4. Summary of Experimental Conditions.	35
Table 5. Experiment Runs.	38
Table 6. Experimental Results using a Copper Cathode under a 0.10 A Constant Current.	44
Table 7. Overall Results at a 0.100 A Constant Current using a Copper Cathode.	46
Table 8. Full Factorial DOE Analysis using a Copper Cathode under Constant Current Conditions.	47
Table 9. Statistical Data of the Results under Constant Current Conditions Using a Copper Cathode.	57
Table 10. Results Obtained using a Copper Cathode under a 10.0 V Constant Potential.	62
Table 11. Overall Results at a 10.0 V Constant Potential using a Copper Cathode.	64
Table 12. Full Factorial DOE Analysis using a Copper Cathode under Constant Potential Conditions.	65
Table 13. Minimum Current Densities for the Nitrate Reduction Reaction.	69
Table 14. Statistical Data of the Results on under Constant Potential Conditions Using a Copper Cathode.	75
Table 15. A Comparison between Constant Current and Constant Voltage Experiments.	76

Table 16. Overall Results at Constant Current with Various Chloride and Sulfate Concentrations.	77
Table 17. Problems with using Possible Electrolytes for Nitrate Reduction.	78
Table 18. Overall Results at a 0.100 A Constant Current using a Carbon-Felt Cathode.	79
Table 19. Overall Results at a 10.0 V Constant Potential using a Carbon-Felt Cathode.	80

CHAPTER 1

INTRODUCTION

1.1 General Background

Nitrate (NO_3^-) is a major inorganic pollutant in groundwater and industrial wastewaters throughout the world [1]. It is used mainly in farming as a fertilizer because plants can only use nitrate as a source of nitrogen for their biological processes [1]. The addition of fertilizers ensures that this important nutrient is in abundance for the plants. Though an increase in crop production results, the excess nitrate seeps into the groundwater as nitrate where it is very soluble presenting a major health problem [1].

According to the Environmental Protection Agency (EPA) an increased exposure to nitrate can cause serious health problems, and even death in some cases, for livestock and human infants [2]. Nitrate is reduced to nitrite (NO_2^-) in the stomach by certain bacteria [2]. Nitrite reacts with hemoglobin to form methemoglobin, which is a molecule incapable of carrying oxygen. As a result the infant or livestock gradually suffocate as the oxygen levels in the body decrease [1, 3]. This condition is known as methemoglobinemia and human infants exhibit a bluish color to their skin [1, 3]. As the infant reaches three months, the stomach begins to produce larger quantities of hydrochloric acid that kills off the nitrite-forming bacteria. Adult humans simply excrete the excess nitrates. Due to this hazard in infants, the EPA has set a limit of 10 mg/L of

nitrate-nitrogen (44.3 mg/L nitrate) and 1 mg/L of nitrite-nitrogen (3.3 mg/L nitrite) in drinking water [2]. Therefore nitrate must be effectively removed from wastewater.

There are several methods that are being used to remove nitrate from groundwater and industrial wastewater [4]. The major methods used include reverse osmosis, distillation, ion exchange, thermal reduction, biological reduction, chemical reduction, ion exchange and electrochemical reduction [4, 5]. Electrochemical reduction is a relatively recent technique and has the potential to achieve the goal of complete mineralization of nitrate, nitrite and other intermediates to produce nitrogen gas (N_2) and ammonia (NH_3). However, more research is required in order to be able to determine electrode materials and operating conditions.

1.2 Electrochemistry

Electrochemistry is a discipline of chemistry that involves the study of how electricity is used to either drive a chemical reaction (such as in a metal plating), or how a chemical reaction maybe used to produce electricity (such as in a battery) [6]. An electrochemical reaction is a heterogeneous chemical process that involves the transfer of a charge to or from an electrode [7]. When electrons are transferred to an electrode from a chemical species, the process is known as oxidation or loss of electrons from the chemical species, and the electrode is called the anode [8]. When electrons are transferred from an electrode to a chemical species, the process is known as reduction or a gain of electrons for the chemical species, and the electrode is called the cathode [8]. Every electrochemical cell has an anode and a cathode [9]. In order for electrons to flow between the anode and the cathode, a conducting bridge or solution known as an electrolyte is required [6]. The salt bridge or electrolyte contains charged ions that allow for a charge to be carried in the solution between the anode and the cathode and thus complete the circuit. If there is no salt bridge or if the electrolyte is non-conducting then no charge will flow and there will be an open circuit. A basic electrochemical cell schematic is shown in Figure 1 [6].

Figure 1. A General Schematic of an Electrochemical Cell [6].

If DC power is placed on the circuit it supplies electrons that cause a chemical reaction to occur. This is known as electrolysis [6]. If a load is placed on the circuit, then the cell is known as a galvanic cell, or a cell in which chemical energy is changed to electricity. The process of electrolysis can be used to force the chemical reaction for the decomposition of nitrate using a DC power supply. A salt bridge is not used for electrolysis. Instead, a conducting electrolyte is used between the electrodes.

There are several important factors to consider in carrying out an electrochemical reaction [7]. These factors include the reaction potential, the applied current, current density, concentration of the electroactive species in the solution, temperature, pH, the

applied potential, the electrode materials and structure, the spacing between the electrode materials, and the fluid dynamics of the electrolyte [6, 7].

Thermodynamics plays a vital role in an electrochemical reaction. For an electrochemical reaction to take place, the free energy of the reaction (ΔG) must be negative [6]. The mathematical definition of the potential (ϵ) is given in Equation 1 [6]. Using this definition, the free energy of the system may be defined as shown in Equation 2 [6].

$$\epsilon_{MAX} = \frac{\text{Max. work}}{\text{charge}} = \frac{-w_{MAX}}{q} = \frac{-\Delta G}{n \cdot F} \quad \text{Equation 1}$$

$$\Delta G^o = -n \cdot F \cdot \epsilon^o \quad \text{Equation 2}$$

The charge is defined as the number of moles of electrons (n) multiplied by the charge on one mole of electrons (F = Faraday's Constant = 96485 coulombs if charge per mole of electron). The standard potential ϵ^o (potential at 25°C and 101.325 kPa) for a reaction can be found on standard reduction tables [6].

The effect of current can be seen from Faraday's Law shown in Equation 3 [7]. The mass of product formed (m) is dependant on the amount of electrons supplied for the reaction (n), Faraday's Constant (F), the current (I) and the time (t). The molecular mass (M_w) of the species is a constant required to convert from moles to mass.

$$m = \frac{I \cdot t \cdot M_w}{n \cdot F} \quad \text{Equation 3}$$

From Faraday's Law the relationship between the current density (i) or current passing through the unit area of electrode surface area and the electrochemical rate can be seen as shown in Equation 4 [7].

$$i = \frac{I}{A} = \frac{n \cdot F \cdot m}{M_w \cdot t \cdot A} = n \cdot F \cdot v \quad \text{Equation 4}$$

The term v represents mass (m) per unit time (t) per unit area (A), which is the unit for a reaction rate. As a result the current density is also known as the electrochemical reaction rate [7].

The concentration of the electroactive species plays a role that can be seen mathematically from the Nernst Equation shown in Equation 5 [6]. The Nernst Equation is derived from the thermodynamic relationship between the standard free energy (ΔG^0) and the free energy of the system (ΔG) at a given temperature (T) and concentration.

$$\mathcal{E} = \mathcal{E}^o - \frac{R \cdot T}{n \cdot F} \cdot \ln(Q) \quad \text{Equation 5}$$

In Equation 5, R is the gas constant, T is the temperature of the solution, F is Faraday's constant and n is the number of moles of electrons. Q is the reaction quotient and is a ratio of the product species activity to the reactant species activity [10]. When $Q = K$ (the

equilibrium constant) the cell has reached equilibrium and $\Delta G = 0$ for the system. Also the resistive properties of the solution may change as the concentration of electroactive species changes [11].

The effect of temperature can be seen from Equation 5. As the temperature of the cell increases, the voltage required for the chemical reaction decreases. The resistance of the solution changes as the temperature changes. Ohm's Law, given in Equation 6, shows that a change in resistance (R) will affect the system's applied voltage (V) and current (I) ratio through the system.

$$V = I \cdot R \quad \text{Equation 6}$$

The pH of a solution is important as shown by Equation 5. Water is an electroactive species and the concentration of hydrogen ions (H^+) and the hydroxide ions (OH^-) in solution affects the potential of the system. A Pourbaix diagram shows the thermodynamic relationship that exists between the pH, the potential and the form of the chemical [10]. A change in the pH of the system can greatly affect the outcome of the reaction.

The electrode material is critical [10]. An electrode material should adsorb the reactant, transfer the electrons to the electroactive species and allow the product to desorb from the surface [7]. The choice of electrode material depends on the reaction [10] and usually requires trial-and-error research. However, Table 1 shows some common electrode materials [7].

Table 1. Some Common Electrode Materials [7].

Cathodes	Anodes
Hg, Pb, Cu, Ni	Pt, Pt/Ti, Ir/Ti, Pb in acid sulfate media
Graphite and other forms of C	Graphite and other forms of C, Magnetite Fe_3O_4
Steels	PbO_2 on Ti or Nb, Ni in alkaline media
Coatings of low H_2 overpotential materials on steel, e.g. Ni, Ni/Al, Ni/Zn	Dimensionally stable anodes (DSA), i.e. RuO_2 on TiO_2/Ti
Boron Doped Diamond Electrodes	Other oxide coatings, e.g. $\text{Cu}_x\text{Co}_{3-x}\text{O}_4$, IrO_2 , on Ti
	Boron Doped Diamond (BDD) Electrodes

The ability of the electrode material to resist corrosion in the electrochemical environment is important [10]. The electrode if not properly chosen may electrochemically dissolve in a solution instead of catalyzing a reaction. The electrode may react with the reactants, products or other electrochemical species in the solution thereby changing its electrochemical properties.

The electrode structure plays an important role. Usually, an electrochemical reaction takes place at the surface of the electrode [7]. Therefore, the electrode surface area is a factor. The surface area will affect the current density and the cell voltage [10].

The electric field formed between the anode and the cathode is influenced by the geometrical and molecular structure of the electrode. The structure usually is determined by the material and by the needs of the electrochemical reaction.

The choice of electrode materials also affects the overvoltage and overpotential [11]. The overpotential (η) is defined as the difference in the electrode potential ($\Delta\varepsilon$) operating in its equilibrium potential and its operating potential when current is applied to the electrode [9]. It is expressed mathematically in Equation 7 [11].

$$\eta = \Delta\varepsilon = \varepsilon_{actual} - \varepsilon_{Equilibrium} \quad \text{Equation 7}$$

The overpotential is the additional energy required for an electrode reaction to proceed. Therefore the operating potential of the anode is more positive compared to its equilibrium potential while that of the cathode is more negative than its equilibrium potential [11].

The current density increases as the overpotential increases. This relationship is expressed in the Butler-Volmer equation as shown in Equation 8 [11].

$$i = i_o \cdot \left[\exp\left(\frac{\alpha_A \cdot n \cdot F}{R \cdot T} \cdot \eta_A\right) - \exp\left(\frac{\alpha_C \cdot n \cdot F}{R \cdot T} \cdot \eta_C\right) \right] \quad \text{Equation 8}$$

In Equation 8 the equation variables are defined as follows:

i = the current density

i_o = the exchange current density or the current density when there is no net reaction

α_A = the anodic charge transfer co-efficient = $(1 - \alpha_C)$. Usually about 0.5

α_C = the cathodic charge transfer co-efficient = $(1 - \alpha_A)$. Usually about 0.5

η_A = anodic overpotential = $-\eta_C$ (see Equation 7)

η_C = cathodic overpotential = $-\eta_A$ (see Equation 7)

The value of the overpotential also depends on the reaction kinetics of the electrode. A slow reaction (a reaction with a small exchange current density) will need a higher overpotential for a given current density than a fast reaction (a reaction with a large exchange current density) [9].

There is also a concentration overpotential that exists in the solution [8]. The concentration variation right next to the electrode and the concentration in the solution leads to potential difference by the Nernst Equation (Equation 5). The concentration overpotential ($\eta_{conc.}$) can be calculated by Equation 9 [8].

$$\eta_{conc.} = \frac{R \cdot T}{n \cdot F} \cdot \ln \frac{C_b}{C_s} = \frac{R \cdot T}{n \cdot F} \cdot \ln \left(1 - \frac{i}{i_L} \right) \quad \text{Equation 9}$$

In Equation 9, the equation variables are defined as follows:

R = universal gas constant

T = temperature

n = moles of electrons transferred during the reaction

F = Faraday Constant

C_b is the concentration of the species in the bulk

C_s is the concentration of the species at the surface of the electrode

i_L is the limiting current density (calculated by Equation 10)

The limiting current (i_L) represents the current at which the rate of the electrochemical reaction is no longer dependant on the rate of the current but on the diffusion from the bulk to the electrode surface. Essentially the concentration at the electrode surface is zero and the electrochemical rate is limited to mass transfer and diffusion to the electrode surface. The limiting current density is calculated by Equation 10 [8].

$$i_L = n \cdot F \cdot D \cdot \frac{C_b}{\delta} \quad \text{Equation 10}$$

In Equation 10, the equation variables are defined as follows:

n = moles of electrons transferred during the reaction

F = Faraday Constant

C_b is the concentration of the species in the bulk

D = Diffusion co-efficient

δ = Diffusion layer thickness between electrode surface and bulk concentration

The applied cell voltage plays a critical role in electrolysis. Depending on the applied voltage, different reactions may take place [6]. Research indicates for nitrate reduction, different products are formed at different applied voltages [6]. Therefore the applied voltage must be carefully chosen and maintained in order to achieve the desired end products.

The spacing between the electrodes affects the voltage applied to the system. The larger the distance between the electrodes, the more work is required to force the charge from one electrode to the other [7].

The movement of fluid within the system is very important in terms of ion flow. In a dilute aqueous solution, mass transfer of the electroactive species to the electrode surface may be considered to be the limiting rate step for the entire process [8, 11]. The Nernst-Planck Equation, shown in Equation 11, describes the mass transfer of the electroactive species from the bulk phase to the reaction area [11]. The equation consists of the convective transport due to convection (forced and natural), the diffusive transport between the viscous layer and the electrical double layer (the layer of charged particles of opposite polarity near an electrode only a few nanometers thick) and the migration of the charged species due to the electrical field in the electrolyte.

$$N_i = [c_i \cdot u] + [-D_i \cdot (\nabla \cdot c_i)] + \left[-\frac{n_i \cdot F \cdot D_i}{R \cdot T} \cdot c_i \cdot (\nabla \cdot \varepsilon) \right] \quad \text{Equation 11}$$

In Equation 11, the equation variables are defined as follows:

N_i = flux of the i^{th} chemical species

u = velocity of fluid

n_i = moles of electrons transferred during the reaction involving the i^{th} chemical species

F = Faraday Constant

C_i = the concentration of the i^{th} species in the bulk

D_i = Diffusion co-efficient

R = universal gas constant

T = temperature

ε = cell potential

1.3 Competing Technologies

There are several major technologies that are used to remove nitrate from groundwater and industrial wastewaters. These include reverse osmosis, distillation, ion exchange, thermal reduction, biological reduction, chemical reduction and electrochemical reduction [4, 5]. Electrochemical reduction has been mentioned briefly in Section 1.2 and will be dealt with to a greater extent in Section 2.0. A brief introduction to the other technologies will be discussed with each technology's advantages and disadvantages.

Reverse osmosis involves using pressure to force water through a permeable selective membrane [4]. Water passes through the membrane; however, nitrate does not, and concentrates on one side of the membrane. The amount of water that passes through the membrane is dependant upon the applied pressure, but the amount of water cleaned may be anywhere from one-third to two-thirds of the original volume. Figure 2 shows a schematic for reverse osmosis [4].

Figure 2. Schematic for Reverse Osmosis Process for Nitrate Removal [4].

Distillation is another process to remove nitrate. Distillation involves evaporating water while nitrates and other minerals remain behind [4]. The water is then condensed. Figure 3 shows a schematic of this process.

Figure 3. Schematic for Distillation Removal of Nitrate [4].

For ion exchange, the untreated water is passed through a resin bed charged with an anion similar to nitrate that will “exchange” places with the nitrate ion [4]. Usually the other anion that is substituted for nitrate is the chloride ion [4]. Figure 4 shows a schematic for an ion exchange removal system for nitrate.

Figure 4. Schematic for an Ion Exchange Nitrate Removal System [4].

Biological Removal involves the use of microorganisms or bacteria that reduce nitrate to nitrogen gas, nitrous oxide or nitric oxide. The genera of bacteria include *Pseudomonas*, *Micrococcus*, *Archromobacter*, *Thiobacillus* and *Bacillus* [5].

Chemical Removal includes Super Critical Water Oxidation (SCWO) and Wet-Air Oxidation (WAO). Both techniques involve using oxygen, or another oxidizing agent (such as nitrate) to oxidize organics or other contaminants. These processes take place under high temperature and pressure [5].

Electrochemical methods may produce ammonia [5]. Ammonia is not a desirable contaminant in wastewater because bacteria use it to make nitrate as part of the Nitrogen Cycle [4, 5]. The Nitrogen Cycle is shown in Figure 5 [1]. However, ammonia can be removed from wastewaters using an air stripper that allows for ammonia to escape as a gas from the wastewater [12]. Therefore, based on the potential advantages, electrochemical reduction presents huge potential and must be explored for nitrate reduction in wastewaters.

Figure 5. The Nitrogen Cycle [1].

1.4 Advantages and Disadvantages

The advantage and disadvantages are presented in Table 2 for the different methods of nitrate removal discussed above.

Table 2. Comparison of Advantages and Disadvantages for Methods of Nitrate Removal.

Method of Nitrate Removal	Advantages	Disadvantages
Biological Removal	1. Nitrate efficiently reduced to nitrogen gas 2. Able to handle large flows	1. Bacteria sensitive to other contaminants 2. Organic wastes produced
Distillation	1. All nitrate removed 2. Very pure water	1. Energy Intensive 2. Not suitable to treat large amounts of water 3. Nitrate still has to be dealt with.
Chemical Removal	1. Nitrate reduced to nitrogen gas efficiently and quickly. 2. Able to handle large flows	1. High temperature and pressure required. Energy intensive and not cost effective
Ion Exchange	1. Nitrate is removed from the water 2. Able to handle large flows quickly	1. Resin needs to be regenerated 2. Nitrate solution still has to be dealt with.
Reverse Osmosis	1. Nitrate is removed from water 2. Very pure water	1. High pressures required. Energy intensive and not cost effective 2. Nitrate solution still has to be dealt with. 3. In-efficient
Electrochemical Reduction	1. Nitrate reduced to nitrogen gas or ammonia. 2. Able to handle large flows 3. Cost effective (potential) 4. Carried out at low temperature and low pressure	1. Fouling of the electrode from other contaminants 2. Efficiency to reduce nitrate is a question 3. Ammonia selectivity

CHAPTER 2

LITERATURE REVIEW

There has been some research done in the area of nitrate electrochemical reduction. Most of the research has focused on determining suitable electrodes and conditions to carry out the electrochemical reduction. However it is important to understand the previous research to learn the kinetics and conditions under which nitrate reduction occurred.

2.1 Theoretical Nitrate Reduction Reactions

The electrochemical reduction of nitrate in an aqueous solution is a complex process. The overall reactions of interest occurring at the cathode that researchers generally agree upon for the reduction of nitrate to nitrogen gas, nitrite, hydroxylamine (NH_2OH), and ammonia are shown in Equations 12 to 17 (along with each reactions equilibrium reduction potential) [13, 14].

There are also other competing reactions occurring at the cathode in addition to the stated reactions. The important side reaction of note is the reduction of water to hydrogen. This is shown in Equation 18 [13].

This side reaction becomes prevalent depending on the conditions such as the voltage, nitrate concentration and the pH.

The main reaction occurring at the anode is the oxidation of water to oxygen. This is shown in Equation 19 [13].

The main overall reactions giving nitrogen gas and ammonia, assuming that sodium nitrate (NaNO_3) is the source of the nitrate, are given in Equations 20 and 21 respectively [13, 14].

2.2 Nitrate Reduction Kinetics on Noble Metal Cathodes

Many researchers have done work using noble metal electrodes to discern the kinetics of nitrate reduction [15, 16, 17, and 18]. The results from these papers, discussed below, are applicable over a wide range of metal electrodes as nitrate reduction kinetics are similar between noble metals (platinum, palladium) and metals (copper, nickel, zinc). While the reaction kinetics for nitrate reduction function in a similar manner over a metal electrode, the selectivity of the electrode to the reaction product and the activity (the rate of the nitrate reduction reaction on the electrode) are dependant upon the type of metal electrode and the electrochemical conditions of that metal.

de Groot and Koper [16] investigated the influence of nitrate concentration on the kinetics of nitrate reduction using a platinum cathode. In their paper they discussed that nitrate reduction can occur in either a direct mechanism (between 0.1 V-0.4 V vs. SCE) or an indirect mechanism (0.5 V-0.9 V vs. SCE). The direct mechanism of reduction is selective to ammonia formation while the indirect mechanism is selective to the reduction of nitrate to nitrite. In order for the indirect mechanism to occur, both nitrite and a low pH are required [16]. A low pH is not common in a dilute aqueous media for nitrate. The direct mechanism on platinum, which may also occur for other metals, is presented in Equations 22 to 26 [16].

For the reaction to form N_2O or N_2 , they postulate that NO^- must be in solution [19].

However they also postulate that NO^- bonds very strongly to platinum and cannot desorb from the electrode surface unless it reacts to NH_4^+ or NH_3OH^+ [16]. As a result NH_2OH and NH_3 are the only reaction products formed on a platinum cathode [16]. Figure 6 shows a schematic of the possible reaction mechanisms that can occur.

Figure 6. Possible Reaction Mechanisms for the Reduction of Nitrate [15].

de Groot and Koper's [16] research focused on the influence of nitrate concentration using a platinum cathode on the nitrate reduction kinetics. They concluded that at low nitrate concentrations the rate of nitrate reduction is determined by the amount of nitrate adsorbed on to the surface. Therefore, the electrochemical reduction of nitrate is directly related to the concentration of nitrate in the solution [16]. de Groot and Koper

also concluded that at low concentrations (<0.1 M) the rate can be hindered by other competing anions such as chlorine or sulfate. At high concentrations (> 0.1 M) the rate is not as dependant on the adsorption of nitrate, and other anions do not have as large of an effect on the rate because nitrate is more capable to compete for electrode sites because it is in a much higher concentration compared to the other competing anions [16].

de Groot and Koper discussed that the reduction of nitrate to nitrite, shown in Equation 23, does not occur very well on platinum [16]. As a result researchers have theorized that in order to increase the activity of platinum, a metal with a high activity for nitrate reduction should be allowed with platinum [17, 18, 19]. Kerkeni et al. [17] have postulated that the addition of copper to platinum can help to increase the activity of the electrode. They found that copper did help to increase the activity.

Palladium is another metal that has been tested for nitrate reduction as it has a high selectivity towards forming nitrogen gas [17]. However palladium also has a low activity [17]. De Vooy et al. [15] did work on a palladium/copper electrode at potentials of 0.1-0.9 V. Their thinking was that copper would enhance the activity of the reaction (by enhancing the nitrate to nitrite reduction reaction) where the palladium would control the selectivity of the reaction to produce nitrogen gas. They suggested that the activity and selectivity of the electrode is very dependant upon the surface structure of the electrode. De Vooy et al. also found that other anions in solution such as sulfate and chloride compete for sites on the electrode surface with nitrate. A surface that has a high coverage of nitrate tends to have a high selectivity to nitrogen gas and high activity, whereas a low surface coverage of nitrate gives ammonia [15]. In addition to the anions

in solution, the pH of the solution also affected the nitrate reduction reaction activity and selectivity [15, 20].

El-Deab [21] used the concept of self-assembling monolayers to coat gold cathodes with a thin layer of thiourea, urea, iodide ions and sulfide ions. He used gold and each of the gold-coated cathodes for the nitrate reduction. He had hoped that these compounds would enhance the electrocatalytic activity of gold towards nitrate reduction. El-Deab found that using a gold electrode in a solution of 0.5 M NaNO_3 at a pH of 12.5 would reduce nitrate to nitrite and ammonia via a similar mechanism presented in the work by de Groot and Koper using platinum [16]. The highest current efficiency was achieved using a gold electrode coated with thiourea at 85% followed by a bare gold electrode at 58% current efficiency. There was a higher conversion of nitrite to ammonia using the thiourea/gold electrode than on the gold electrode. However the activity was still low for these electrodes.

While noble metal electrodes may give control for selectivity, for wastewater destruction other electrodes that are more cost effective may suffice. These electrode materials include carbon and copper.

2.3 Nitrate Reduction Using Carbon Based Cathodes

Electrocatalytic activity is very important and material specific [10]. The electrocatalytic activity of a material can increase the exchange current density ten fold and as a result increase the reaction rate for a specific reaction, or maybe even decrease the exchange current density and hence the reaction rate for unwanted reactions [10]. There have been many researchers that have done work on various electrodes. Carbon is a popular electrode material that has been used in the battery industry and in aluminum manufacturing [7]. Researchers have also done some work using carbon-based materials for nitrate reduction.

Koparal and Ogutveren [13] did work using a carbon cloth electrode with a graphite rod anode. The experiment entailed testing nitrate reduction at various pH values, concentration values and applied potential values. Experiments were carried out at pH values of 5, 7 and 11, concentration values of 100 mg/L and 300 mg/L nitrate and applied potentials of 0.9 V, 1.5 V, 2.2 V and 2.9 V. However, it appears that only one variable was tested at a time while the others were held constant and a design of experiments matrix was not done.

The fastest reduction rate for nitrate was seen at 2.9 V at a pH of 7 and in a solution with an initial nitrate concentration of 100 mg/L. Koparal and Ogutveren indicated that an applied potential as high as 2.9 V could be applied without converting nitrate to ammonia [13]. However, only data showing nitrate reduction concentration

was presented and no product formation data was shown. It also appears that no experiments were done at any other pH value or at different concentrations.

Koparal and Ogutveren tested at pH values of 5, 7 and 11 for a concentration of 300 mg/L and an applied potential of 2.9 V. They concluded that pH is a factor that effects the conversion of nitrate to nitrogen gas. The highest removal rates were observed at the lower pH values. They found that they were able to reduce nitrate from concentrations of 100 mg/L and 300 mg/L to below around 50 mg/L at an applied potential of 2.9 V and a pH of 7. All experiments took about 3 to 4 hours to complete depending on the experimental conditions [13].

While this paper presents some interesting ideas and some useful information, it does not answer questions about using carbon to reduce nitrate. The biggest question is to accurately determine what the final products of the reaction were. The researchers assumed that nitrogen gas was the final product and this may not have been the case. There may have been nitrite and ammonia [14]. Though nitrate may have been reduced, nitrite, which is a more dangerous pollutant, may have remained [1]. An increase in pH is also an indication of ammonia formation [14].

It would have been useful if Koparal and Ogutveren had performed a Design of Experiments. In testing the different conditions in their presentation of the data they assumed that an applied voltage of 2.9 V and a pH of 7 would give the best results at various concentration levels. They ignored the possibility that there maybe interaction between the parameters so that at a pH of 11 there maybe a much lower energy consumption.

Peel et al. [22] did experiments using cathode materials composed of carbon cloth and carbon cloth coated on the surface with 30% rhodium ($1 \mu\text{g}/\text{cm}$) at an applied potential of -1.5 V relative to a standard calomel reference electrode (SCE or Ag/AgCl) under ambient conditions. While they found that the bare carbon cloth did not exhibit any nitrate reduction, the rhodium-coated carbon cloth showed electrocatalytic activity to reduce nitrate under the experimental conditions [22]. Their results showed that the rhodium-coated carbon cloth showed a 36% increase in the reduction current in the presence of nitrate as opposed to the carbon cloth in the presence of nitrate. The fact that Peel et al. did not see any nitrate reduction using carbon cloth is in contrast to the results presented by Koparal and Ogutveren [13].

Peel et al. also did experiments with pH values of 5.8, 7.0 and 8.0 and found that a higher reduction current draw was observed at a higher pH for the rhodium-carbon cloth electrodes [22]. Perchloric acid was used to modify the pH.

Peel et al. found that for an aqueous solution of 100 mg/L nitrate the reduction occurred within 40-60 minutes. Nitrate was found to reduce to nitrite, nitrogen gas, ammonia and hydroxylamine. The researchers concluded that the mechanism for the reduction was a direct electrochemical reduction. This is in contrast again to the paper by Koparal and Ogutveren who indicated that only nitrogen gas was formed as a product using a carbon cloth.

Given these papers' discrepancies between these research articles it may be interesting to study the reduction of nitrate on a carbon-based cathode. These studies also do not directly investigate the effect of current density on the reduction of nitrate (although applied potential is related to the current density).

2.4 Nitrate Reduction Using Copper Cathodes

Copper has very good electrical properties in that it is very conductive and has a very high electrocatalytic activity towards many electrochemical reactions. Copper also has the highest activity compared to silver and gold [20]. Research has been done using copper cathodes under non-aqueous conditions [23] however Paider et al. [14, 24] have done research using copper electrodes for nitrate reduction in wastewater.

Paider et al. [14] have studied the reduction of nitrate ions in waste solutions after regeneration of ion exchange columns. When using a copper cathode under ambient conditions they observed that they were able to reduce nitrate to ammonia with nitrite being an intermediate in the reaction process. The current density was varied over a range of 2.8-7.6 mA/cm² with the best current efficiency being obtained at 2.8 mA/cm². They also observed that the addition of copper ions at 6 mg/L to the solution enhances the removal efficiency of nitrate.

Paider et al. [24] did a paper on the influence of cell construction on the electrochemical reduction of nitrate. While this paper focused more on the engineering of a reactor it did use copper cathodes and investigated the reaction kinetics of nitrate

reduction. The reduction of nitrate took place within 500 minutes. Using plate electrodes they had a high current efficiency at low current densities (below 4 mA/cm²) which was in agreement with the work Paider et al. [14] as discussed above. They also did experiments using copper cathodes with a high surface area and found that these cathodes performed well as would be expected. However there has been no work found for the way concentration can affect an aqueous nitrate solution using copper cathodes.

2.5 Other Metal Electrodes and Materials

Other metals have also been tried for nitrate reduction. Li [5] used nickel, lead zinc and iron cathodes and found that nitrate was reduced to ammonia at values of -1.5 V to -2.0 V vs. SCE. For zinc and lead there was over 90% nitrate reduction after one hour.

A promising electrode that has been recently developed is the thin film boron doped diamond (BDD) electrode [25, 26, 27, and 28]. These electrodes have a very high overpotential for oxygen and as a result, like dimensionally stable anodes (DSA), are very stable anodes that do not corrode under very corrosive conditions. However these electrodes, like carbon, may also be used as a cathode and they have a very high overpotential for hydrogen evolution. Levy-Clement et. al. [25], Bouamrane et. al. [27] and Reuben et. al. [28], showed that nitrate could be efficiently reduced again to nitrite and then to ammonia. The amount of boron affects the activity of nitrate reduction [26]. However to manufacture boron doped diamond electrodes is extremely costly.

2.6 Other Effects

Researchers found that the addition of certain anions such as sulfate or chloride ions would compete with nitrate ions for sites on a metal electrode [16, 20]. At low nitrate concentrations this becomes a problem. There are also chemical effects with the addition of formic acid or copper ions to the solution [5, 24, 29]. Researchers have found that with high temperature and pressure, formic acid will reduce nitrate to nitrogen gas [5]. Copper ions also in solution act as a catalyst for nitrate reduction [24, 29].

2.7 Summary

Table 3 summarizes some of the work done using different cathodes to reduce nitrate.

Table 3. Summary of Activity and Selectivity for Different Metal Electrodes.

Cathode *[Ref.]	Activity	Selectivity	Notes
Carbon [13, 22]	Not known	High selectivity to Ammonia	The results presented in this table are general characteristics. Concentration, pH, current density and voltage affect the outcome of the reaction products and the rate of reaction. The data presented in this table is under given conditions which should be checked from each reference
Copper [14, 24]	High Activity	High selectivity to Ammonia	
Platinum [16]	Low Activity	High selectivity to Ammonia	
Gold [21]	Low Activity	High selectivity to Ammonia	
Palladium [15, 17]	Low Activity	High selectivity to nitrogen gas	
Lead [5]	High Activity	High selectivity to Ammonia	
Zinc [5]	High Activity	High selectivity to Ammonia	
Iron [5]	High Activity	High selectivity to Ammonia	
Nickel [5]	High Activity	High selectivity to Ammonia	
Boron Doped Diamond [25, 26, 27, 29]	High Activity	High selectivity to Ammonia	

CHAPTER 3

RESEARCH OBJECTIVES

3.1 Research Objectives

The purpose is to study the effect of initial nitrate concentration and initial solution pH on the electrochemical reduction of nitrate in a dilute low conductivity aqueous system using a graphite-felt (carbon) cathode and copper cathode.

3.2 Justification

From the literature it is expected that there will be a greater selectivity towards ammonia forming at both the graphite felt electrode and the copper electrode given the conditions planned for this experiment. Copper will be a better electrode material than carbon under the experimental conditions as indicated by the literature [24]. The literature also indicates that a higher initial nitrate concentration and higher initial aqueous pH should give better conditions for nitrate reduction with ammonia forming as the main product [16, 22].

CHAPTER 4

EXPERIMENTAL PROCEDURE AND ANALYSIS

4.1 Experimental Set-up and Procedure

The experiments were performed in a small batch reactor using parallel flat plate electrodes. A schematic is shown in Figure 7. The cathode used was either graphite felt or copper. The anode used was constructed from titanium metal. Titanium oxidizes to titanium oxide but will not degrade significantly into the solution under the proposed experimental conditions. Each electrode had dimensions of 100 mm wide by 100 mm in length. Power was supplied using a DC power supply. The electrodes were spaced to control the current to 100 mA.

The batch reactor held 750 mL of solution. The batch reactor was a plastic beaker open to the atmosphere. The plastic did not appear to react electrochemically or interfere with the experiment. The solution was made up of sodium nitrate (NaNO_3) dissolved in water. The water used to make-up the dilute nitrate solution was put through a water softener to remove any calcium ions that might plate out on the cathode and interfere with the experiment. This setup was done to simulate an ideal condition for wastewater being treated electrochemically to remove nitrate. The concentrations of nitrate tested were 100 mg/L nitrate and 500 mg/L nitrate. The initial pH was controlled using a small amount of sodium hydroxide (NaOH).

Figure 7. Schematic of the Experimental Set-Up.

Prior to each experiment the copper electrode was sanded and washed to ensure that the surface was clean. A new graphite felt electrode was used for each experiment. A magnetic mixer was used to ensure that there was good mixing of the solution. The connection to the copper cathode was made directly. For the graphite felt cathode the connection to the power supply was made using a copper header bar. A header bar was used to ensure proper current distribution throughout the electrode. Table 4 summarizes the important experiment parameters, values, conditions and reasons for the conditions.

Table 4. Summary of Experimental Conditions.

Experimental Parameter	Condition/Status	Reason
Cathode	Graphite Felt Copper	Carbon behavior is not known. Copper has a very high activity and can be compared to carbon results
Anode	Titanium	Titanium oxidizes to TiO_2 but should not affect reaction or degrade under experiment conditions
Solution	750 mL Soft Water using NaNO_3	Soft water has no calcium that will plate out. This simulates an ideal electrochemical treatment.
Initial Solution pH	pH 7 and pH 9	Simulate incoming wastewater treatment influent. Controlled using NaOH .
Applied Voltage	10.0 V	The voltage will be controlled closely to this value, however as literature has indicated, voltage plays a role in selectivity
Current	100 mA	Controlled to give the appropriate current density
Current Density	0.8 mA/cm^2	Literature has indicated this value is acceptable for nitrate reduction
Reaction pH	Not controlled	Cl^- ions and SO_4^{2-} ions inhibit nitrate reduction reaction. Addition of nitric acid adds more reactant. Addition of perchloric acid becomes a problem because ClO_4^- is regulated by the EPA
Initial Nitrate Concentration	100 ppm, 500 ppm	Simulate a low conc. and high $[\text{NO}_3^-]$ wastewater
Temperature	$\sim 20^\circ\text{C}$	Simulation of wastewater treatment where temperature will not ideally be changed
Additives (i.e. buffers etc.)	None	May affect results
Spacing	34 mm	Used to control the applied voltage, current and current density
Mixing	High mixing	To ensure that process is not limited by mass transfer but by the kinetics
Run Time	hours	The theoretical time is calculated using Faraday's Law to determine how long it will take to decompose the entire nitrate to ammonia at the calculated concentration

4.2 Experimental Analysis

Samples were taken at the beginning and at the end of each experiment. Each sample was analyzed for chloride, sulfate, nitrate, nitrite and ammonia by ion chromatography at ENTECH Analytical Laboratories. The temperature of the solution was monitored along with the salt content as NaCl%, the total dissolved solids (TDS) and the conductivity of the soft water used to make the initial and the final solution. As solution is important for electrochemical reactions monitoring the conductivity, TDS and NaCl% helped to ensure that the solution had similar properties for each experiment.

4.3 Data Analysis

From the literature review it seems that gaseous nitrogen products are not produced in any significant quantity for copper or carbon cathodes under the experimental conditions. Therefore, a mass balance on the nitrogen in the system using nitrate, nitrite and ammonia values was done in accordance to Equation 27. The unaccounted nitrogen was assumed to be in another intermediate in solution, adsorbed onto the cathode, or have left the system as a gaseous product.

The response variables investigated were the coulombic efficiency, the amount of nitrate removed, the amount of product formed and the composition of the product.

The coulombic efficiency measured the efficiency of the electrochemical reaction. It was calculated using Equations 28.

The amount of nitrate removed was calculated using Equation 29. This response variable was important as it was used to determine how effective the method of electrochemical reduction was in reducing nitrate in wastewater. The calculation of the amount of product formed in Equation 30 is also an indication of this and it also confirms that nitrate was indeed electrochemically reduced. The split of the product as calculated in Equations 31 and 32 give an insight into the reaction mechanism.

$$0 = \text{Nitrogen}_{\text{initial nitrate}} - \text{Nitrogen}_{\text{final nitrate}} - \text{Nitrogen}_{\text{ammonia}} - \text{Nitrogen}_{\text{nitrite}} \quad \text{Equation 27}$$

$$\text{Coulombic Efficiency} = \frac{\text{Coulombs calculated to form desired product}}{\text{Total Coulombs}} \quad \text{Equation 28}$$

$$\text{Nitrate Removal \%} = \frac{\text{Nitrate in Influent} - \text{Nitrate in Effluent}}{\text{Nitrate in Influent}} \quad \text{Equation 29}$$

$$\text{Amount of Product} = \frac{\text{Moles of Ammonia and Nitrite Formed}}{\text{Total Moles of Product Formed}} \quad \text{Equation 30}$$

$$\% \text{ Split of Nitrite} = \frac{\text{Moles of Nitrite}}{\text{Total Moles of Ammonia and Nitrite Formed}} \quad \text{Equation 31}$$

$$\% \text{ Split of Ammonia} = \frac{\text{Moles of Ammonia}}{\text{Total Moles of Ammonia and Nitrite Formed}} \quad \text{Equation 32}$$

CHAPTER 5

EXPERIMENTAL DESIGN

The experimental design was based on the Design of Experiments (DOE) Methodology [30]. Three factors were varied, each having two levels. The three factors varied were the type of cathode (copper and graphite felt), the initial pH (7 and 9) and the initial nitrate concentration (100 mg/L and 500 mg/L). However, in order to check for curvature, the experiments will be designed as two, two-by-two experiments. This is known as a two level factorial design experimental design. Center point experiments were carried out for each two-by-two experimental set. The experiments are shown in Table 5. The term +/- indicates the upper and the lower limits of the DOE.

Table 5. Experiment Runs.

Basic Experiments		
Samples: Initial and final solution samples of 250 mL each for each experiential run. Experimental Conditions: Same conditions for constant current and constant potential experiments (see Table 4).		
Type of Cathode (+/-)	Control Factors	
	Initial pH (+/-)	Initial [NO ₃] Concentration (+/-) (mg/L)
Graphite Felt (+)	7 (-)	100 (-)
	7 (-)	500 (+)
	9 (+)	100 (-)
	9 (+)	500 (+)
	8 (o)	300 (o)
Copper (-)	7 (-)	100 (-)
	7 (-)	500 (+)
	9 (+)	100 (-)
	9 (+)	500 (+)
	8 (o)	300 (o)
Repeatability Experiments – Repeat experiments above at least once.		

CHAPTER 6

RESULTS AND DISCUSSIONS

The electrolytic reduction of nitrate on a copper and a carbon-working electrode was performed using three reaction conditions, constant current, constant voltage and constant current with various chloride and sulfate ion concentrations. In the sections that follow, the results obtained with the copper cathode are presented and discussed first and separately from those with the carbon electrode.

6.1 Degradation of Nitrate Ion on a Copper Cathode under Constant Current Conditions

Five batch experiments were performed using 750 mL solutions that were stirred at a constant rate with a magnetic stirrer and held at 22°C ($\pm 1.5^\circ\text{C}$). A flat plate copper electrode with an immersed area of 120 cm² area was used with a flat plate titanium counter electrode of equal area placed 34 mm apart. The compositions of the initial solutions included three pH conditions, 7, 8 and 9, and three nitrate ion concentrations, 100 ppm, 300 ppm and 500 pm. These conditions are tabulated in Table 5. The random error in the pH readings was 0.0 to 0.08 pH units for the initial solutions and 0.21 to 0.33 for the final solutions.

The electrolysis reactions were performed using 0.100 A constant current supplied by a DC power supply. The current was monitored before and after each experiment and found to remain constant within the average error of ± 0.001 A. The

electrolysis times were 2:36 hours, 7:46 hours and 13 hours. These times were selected based on the amount of time required to fully react the moles of nitrate in the solution with the applied current and using n equal to 8 in Faraday's law (See Equation 3). There was a slight volume change due to evaporation for the experiments carried out at longer electrolysis times. At a reaction time of 7:46 hours the final volume was 740 mL, while at 13 hours the final volume was 730 mL. Tiny bubbles formed sporadically at both electrodes. At the end of each experiment the solution was slightly yellow and had a weakly detectable ammonia like odor.

The pH of the solution initially at pH 7 and 8 increased slightly during the electrolysis reaction, while the pH of 9 solution had no pH change within the experimental error. The solutions were weakly conductive and had initial conductivities between 1.06 to 2.15 mS with an average reproducibility of 0.033 mS, depending on the nitrate ion concentration. The conductivity did not vary during the electrolysis within the experimental error. The initial voltages were between 11 and 7.5 V and the voltage dropped circa 0.5 to 1.0 V during the electrolysis. The results are summarized in Table 6 and the values represent the average of three runs.

Aliquots of the solution before and after the electrolysis were analyzed for nitrate plus the reaction products, nitrite and ammonia at the same time. The results are summarized both in parts per million (ppm) of nitrogen (original data) and in parts per million of each species (calculated data). The error was quite high in some cases. The error analysis is presented in Section 6.3

The percent of the unreacted nitrate and the nitrite and ammonia products in the electrolyzed solution were calculated using the data in Table 6 and Equations 27-32 and are summarized in Table 7. As can be seen in Table 7, very little nitrate reduction took place. The % NO_3^- remaining (third column) and the corresponding % NO_3^- reacted provided a good mass balance. The nitrogen balances on the experiments closed within experimental error. Hence, no additional products were formed besides nitrite and ammonia in contrast with the report by Paider et al. [24]. Thus, these are the major products in the reaction. These values were calculated using Equations 29 and 30, the initial and final nitrate concentrations.

The column labeled “% NO_2^- and NH_3 Formed” is the sum of NO_2^- and NH_3 formed based on the total moles of NO_3^- , NO_2^- and NH_3 in the final solution, and was calculated using Equation 30. The ($\text{NO}_2^- / \text{NH}_3$) product ratio (Equations 31 and 32) shown in the next two columns indicates that more ammonia was produced as the initial [NO_3^-] increased. The coulombic efficiency (the last column in the table) was calculated using Equation 28. The column is split into two sub columns with one sub column a coulombic efficiency of the experiment to only ammonia and another reflecting a coulombic efficiency to ammonia and nitrite (denoted as the total coulombic efficiency). As the differences between these two efficiencies are small and follow the same trend, an analysis of the total coulombic efficiency will represent the analysis for the coulombic efficiency to ammonia.

Table 8 shows the DOE results and analysis for the investigation of the initial solution pH and the initial nitrate concentration using a copper cathode under constant current conditions. The results were analyzed as a Full Factorial DOE set-up analyzing the data as two factors with two variables. The factors investigated for all DOE were the initial solution pH and the initial nitrate concentration. The variables that were monitored were the coulombic efficiency, the amount of NO_3^- removed, the amount of product formed (total amount of NO_2^- and NH_3 formed) and the breakdown of the product in terms of percentage NO_2^- and NH_3 . The DOE values calculated indicated that the larger the value of the effect, the larger the magnitude of the effect value. The sign of the value also shows which variable had the greater effect.

The low coulombic efficiencies, the low removal of nitrate reacted and the low amount of product formed during the experiments can be attributed to several factors. These include the reaction of the side reaction becoming more prominent; the improper voltage and current set-up; the reversible reaction of nitrate [NO_3^-] reducing to nitrite [NO_2^-] and then the nitrite oxidizing back to nitrate; and the adsorption/desorption properties of the electrode material under the solution conditions.

As can be seen in Table 7, both the extent of NO_3^- degradation and the coulombic efficiency are fairly low for the long electrolysis times. Thus, the side reactions, presumably the electrolysis of water to produce H_2 is much more efficient under these conditions. Of all the solution parameters tested, the $[\text{NO}_3^-]$ had the biggest effect on the coulombic efficiency of the reaction, which decreased almost linearly with the initial $[\text{NO}_3^-]$, and with the increase in the solution conductivity. Because of the ionic nature of the NO_3^- it had an effect on the conductivity of the weakly conducting solutions. This is seen graphically in Figure 8. This correlation suggests that the increase in the solution conductivity preferentially promotes the side reaction over the reduction of NO_3^- . Somehow, the concentration of the negatively charged NO_3^- is reduced at the cathode in the solutions with higher ionic strength and higher conductivity. Clearly a big issue is the strong competition by the electrolysis of water reaction. The majority of the electrons that were targeted for nitrate reduction went to the competing reactions for water electrolysis shown in Equation 18. This maybe attributed to improper voltage and current levels.

The results from Tables 7 and 8 showed that the coulombic efficiency, the amount of nitrate removed and the amount of product formed were all not greatly affected by the initial solution pH but were significantly affected by the initial nitrate concentration. The Effect plots in Figures 9 thru 11 show this visually that essentially there is no interaction with in experimental error between the initial solution pH and the measured variables but there is a large interaction for the initial nitrate concentrations between concentrations at 100 ppm and 500 ppm.

A high initial concentration nitrate at 500 ppm inhibits the electrochemical reaction as indicated by the low coulombic efficiencies, nitrate reduction and product formation shown in Figures 9-11. Table 8 also shows that the initial concentration of nitrate in the solution has the highest effect on the coulombic efficiency with a value of -2.58, the amount of nitrate removed from the reaction at -2.30 and the amount of product formed at -2.37 meaning that the highest coulombic efficiencies, nitrate removal and product formation are obtained at the lower initial nitrate concentration of 100 ppm. The results are contrary to the literature results [16], which indicated that a higher concentration of nitrate in solution enhances the reaction, product formed and coulombic efficiencies since the concentration of nitrate ions adsorbed at the copper cathode increases.

Table 6. Experimental Results using a Copper Cathode under a 0.10 A Constant Current.

	pH	[NO ₃] (as N)	Reaction Time (hrs:min)	Voltage (V)	Cond. (mS)	[NO ₂] (as N)	[NH ₃] (as N)
		[NO ₃] (ppm)				[NO ₂] (ppm)	[NH ₃] (ppm)
Initial	7.27	25.67	0:00	11.05	1.051	0.00	0.00
		113.66				0.00	0.00
Std. Dev.		4.62		0.09		0.00	0.00
		20.45				0.00	0.00
Final	8.34	23.67	2:36	10.39	1.065	1.21	1.16
		104.80				3.99	1.41
Std. Dev.		3.79		0.67		0.34	0.50
		16.77				1.11	0.61

Table 6 Continued. Experimental Results using a Copper Cathode under a 0.10 A Constant Current.

	pH	[NO ₃] (as N)	Reaction Time (hrs:min)	Voltage (V)	Cond. (mS)	[NO ₂] (as N)	[NH ₃] (as N)
		[NO ₃] (ppm)				[NO ₂] (ppm)	[NH ₃] (ppm)
Initial	9.05	24.00	0:00	9.75	1.286	0.00	0.00
		106.28				0.00	0.00
Std. Dev.		0.00		0.78		0.00	0.00
		0.00				0.00	0.00
Final	9.08	22.33	2:36	8.81	1.297	0.69	150
		98.90				2.27	1.82
Std. Dev.		0.58		0.44		0.60	0.17
		2.56				1.98	0.21
Initial	8.22	71.00	0:00	9.10	1.575	0.00	0.00
		314.41				0.00	0.00
Std. Dev.		2.65		0.43		0.00	0.00
		11.72				0.00	0.00
Final	8.29	68.33	7:46	8.90	1.603	0.48	2.93
		302.60				1.58	3.57
Std. Dev.		2.31		0.85		0.42	2.24
		10.23				1.37	2.72
Initial	7.18	110.00	0:00	7.89	1.987	0.00	0.00
		487.11				0.00	0.00
Std. Dev.		0.00		0.16		0.00	0.00
		0.00				0.00	0.00
Final	8.75	110.00	12:57	8.14	2.037	0.22	2.63
		487.11				0.72	3.20
Std. Dev.		0.00		0.31		0.20	3.00
		0.00				0.67	3.65
Initial	9.03	110.00	0:00	7.88	2.097	0.02	0.00
		487.11				0.08	0.00
Std. Dev.		0.00		1.04		0.00	0.00
		0.00				0.00	0.00
Final	8.67	110.00	12:57	8.54	2.160	0.35	2.35
		487.11				1.13	2.86
Std. Dev.		0.00		0.11		0.05	0.35
		0.00				0.16	0.43

Table 7. Overall Results at a 0.100 A Constant Current using a Copper Cathode.

Initial pH	Initial [NO ₃] (ppm)	% NO ₃ Remaining	%NO ₃ Reacted	% Break-Down of Product				Coulombic Efficiency (%)	
				%NO ₃ Unreacted	% NO ₂ and NH ₃ Formed	% NO ₂ of prd.	% NH ₃ of prd.	NH ₃ Rxn.	Total Rxn
7	100	92.43	7.57	90.94	9.06	52.30	47.70	5.01	6.32
9	100	93.06	6.94	91.08	8.92	26.74	73.26	6.58	7.34
8	300	95.00	5.00	95.27	4.73	12.97	87.03	4.23	4.40
7	500	97.33	2.67	97.51	2.49	37.17	62.83	2.52	2.57
9	500	97.33	2.67	97.61	2.39	12.99	87.01	2.14	2.21

Table 8. Full Factorial DOE Analysis using a Copper Cathode under Constant Current Conditions.

Conditions (Initial pH, Initial [NO ₃])	Initial pH	[NO ₃] _o		CE	CE	NO ₃	Product	Product Split	
	P	C	PC	Total %	NH ₃ %	Removed %	%	NO ₂ %	NH ₃ %
(7,100)	-	-	+	6.32	5.01	7.57	9.06	52.30	47.70
(7,500)	-	+	-	2.57	2.52	2.67	2.49	37.17	62.83
(9,100)	+	-	-	7.34	6.58	6.94	8.92	26.74	73.26
(9,500)	+	+	+	2.21	2.14	2.67	2.39	12.99	87.01
(8,300)	0	0	0	4.40	4.23	5.00	4.73	12.97	87.03
Total Coulombic Eff.									
Effect	0.166	-2.219	-0.343						
Curvature	0.20								
Coulombic Eff. NH ₃									
Effect	0.298	-1.733	-0.488						
Curvature	-0.17								
NO ₃ Removed									
Effect	-0.157	-2.296	0.157						
Curvature	-0.04								
Product									
Effect	-0.0594	-3.273	0.011						
Curvature	0.98								
NO ₂ Split									
Effect	-12.435	-7.220	0.345						
Curvature	19.32								
NH ₃ Split									
Effect	12.435	7.220	-0.345						
Curvature	-19.32

Figure 8. The Total Coulombic Efficiency versus Average Solution Conductivity under Constant Current Conditions.

Figure 9. The Effects Plot for the Average Total Coulombic Efficiency under Constant Current Conditions using a Copper Cathode.

Figure 10. The Effects Plot for the Average Percent Nitrate Removal under Constant Current Conditions Using a Copper Cathode.

Figure 11. The Effects Plot for the Average Percent Product Formation (Average Percent NO_2 and NH_3) under Constant Current Conditions using a Copper Cathode.

de Groot and Koper [16] indicated that for platinum there are problems of nitrate adsorbing onto the surface in addition to the products desorbing from the surface of the electrode. These same adsorption/desorption issues may be the problem with the copper electrode. Nitrite may not easily desorb from the surface of the electrode leading to sites being “poisoned” or not being able to be used for nitrate reduction. Equation 6 shows a direct relationship between the voltage and the current. At a constant current, as the reaction proceeds, the ion concentration of the solution increased, along with joule heating of the solution. This lead to the resistivity of the solution to decrease and as a result, the applied voltage decreased in order to maintain the constant current. As discussed in Section 1.2, the voltage is a critical parameter to an electrochemical reaction. The voltage level for solutions at an initial nitrate concentration of 100 ppm was circa 9.0-10.0 V while the voltage levels required to drive 100 mA through the solution was circa 7.0-8.0 V. This slight difference may have been another reason for the discrepancy. Assuming that the voltage is held constant, the final coulombic efficiencies, nitrate removed and product formed may be slightly better under all conditions.

Another possible reason that the reaction had a low efficiency was that it took place in a single compartment electrochemical cell. As a result nitrate reduced to nitrite and released into solution at the cathode may have made its way to be adsorbed on the anode and reduced back to nitrate as indicated in Equation 12. This argument is difficult to track, as theoretically the amount of nitrite in solution should correspond to a lower reaction coulombic efficiency. It was observed however, that, most of the nitrite in

solution was at the 100 ppm initial nitrate concentrations. It was these low initial nitrate concentrations that had the higher coulombic efficiencies.

Ideally there should be 100% nitrate removal, however the minimum amount of nitrate that should be removed is approximately 42% at 100 ppm initial nitrate level to meet the discharge levels set by the EPA. This value becomes about 80% at an initial concentration of 300 ppm and approximately 88% at an initial concentration of 500 ppm.

6.2 Analysis of the Effects of Initial pH and Initial Concentration using a Copper Cathode under a Constant Current on Nitrite and Ammonia Formation in the Product

The product was analyzed for nitrite and ammonia since these were the reaction products described in the literature [14, 16, 24]. The results are given in Table 7 and Figures 12 and 13 show the effect plots for these breakdown products. The sum of the ammonia and nitrite content were made equal to 100 %. It may be noted that more nitrite formed at a pH of 7 while more ammonia formed at an initial pH of 9. The figures show that there is no interaction between the initial pH and the initial nitrate concentration. They are independent variables. Table 8 only indicates that the pH affects this conversion more than the initial nitrate concentration.

The higher conversion to nitrite only at pH 7 may reflect the effective detachment of the nitrite from the electrode that would eliminate the possibility of further reaction to produce ammonia. Also the voltage may have dropped which may have lead to more nitrite not being converted to ammonia.

At a solution pH of 7 the amount of ammonia formed may have been low also due to a pH buffer close to a pH of 9. Once the pH 9 was reached any nitrite in solution may have required a higher voltage to be reduced to ammonia.

Figure 12. The Effects Plot for the Average Percent of Product Formed that was Nitrite under Constant Current Conditions using a Copper Cathode.

Figure 13. The Effects Plot for the Average Percent of Product Formed that was Ammonia under Constant Current Conditions using a Copper Cathode.

6.3 Error Analysis of Results using a Copper Cathode under Constant Current Conditions

While the results show definite trends, there is a large variance in the data shown in Table 9. This table shows the statistical analysis of the calculated results giving the average in the third column, the range of the (difference between the maximum value and the minimum value) in the fourth column and the standard deviation under the test conditions in the last column.

There are several reasons for this variance. The first is the stability of the power supply to maintain a constant current. The power supplies used were designed to maintain a constant voltage. To maintain a constant current, a potentiometer may be required. Secondly, an analysis more accurate than ion chromatography may be required for these concentrations. As an example, in the calculation of the coulombic efficiency, an error of 1 ppm nitrate in the final solution amounts to 20% error. This variance limits the reliability of the conclusions that may be drawn from the results.

Table 9. Statistical Data of the Results under Constant Current Conditions Using a Copper Cathode.

Initial pH	Initial [NO ₃] ppm	Average % (see Factor)	Range of Data Between Max, Min. Values	Standard Deviation for 3 Experimental Runs
Factor: The Total Coulombic Efficiency				
7	100	6.32	4.23	2.41
7	500	2.57	5.63	2.87
9	100	7.34	2.46	1.24
9	500	2.21	0.67	0.33
8	300	4.40	6.20	3.33
Factor: The Coulombic Efficiency to NH ₃				
7	100	5.01	3.61	2.08
7	500	2.52	5.72	2.92
9	100	6.58	1.31	0.74
9	500	2.14	0.68	0.34
8	300	4.23	5.94	3.24
Factor: The Nitrate Removed				
7	100	7.58	5.33	2.84
7	500	2.67	2.67	1.34
9	100	6.94	4.16	2.40
9	500	2.67	0.00	0.00
8	300	5.00	5.41	2.83
Factor: The Product Formed				
7	100	9.06	5.29	2.70
7	500	2.49	4.73	2.40
9	100	8.92	5.55	2.86
9	500	2.39	0.38	0.19
8	300	4.73	6.58	3.40

6.4 Degradation of Nitrate Ion on a Copper Cathode under Constant Potential Conditions

Five batch experiments were carried out similar to those experiments described in the Section 6.1 except under constant potential conditions. A flat plate copper electrode (120 cm^2 area) and a flat plate titanium counter electrode (120 cm^2 area) were placed 34 mm apart in a 750 mL solution and stirred at 21°C ($\pm 1.5^\circ\text{C}$). The compositions of the initial solutions included pH 7, 8 and 9, and three nitrate ion concentrations, 100 ppm, 300 ppm and 500 ppm. These conditions are tabulated in Table 10.

The electrolysis reactions were performed using 10.0 V constant potential supplied by a DC power supply. The electrolysis times were 2:36 hours, 7:46 hours and 12 hours. There was a slight volume change due to evaporation for experiments carried out at longer electrolysis times. At a reaction time of 7:46 hours the final volume was 740 mL, while at 13 hours the final volume was 730 mL. Tiny bubbles formed at both electrodes. The solutions changed from a transparent, colorless solution to a slightly yellow, colorless solution. A very weak ammonia odor was noticed.

The pH of the solution initially at pH 7 and 8 increased slightly during the electrolysis reaction, while the pH 9 solution had no pH change within the experimental error. The solutions were weakly conductive and had initial conductivities between 1.09 to 2.00 mS. The conductivities increased monotonically with the increase of nitrate ion concentration, and were not changed with the electrolysis. The initial currents were between 80 mA and 140 mA depending on the solution composition.

The initial and final solutions were analyzed for the concentrations of nitrate, nitrite and ammonia. The results are summarized in Table 10 and are the average of two runs. The results are reported in parts per million (ppm) of nitrogen (original data) and in parts per million of each species (calculated data). The average deviations were quite high in some cases, compared to the results in the constant current experiments.

The percent nitrate, nitrite and ammonia in the electrolyzed solution were calculated using the data in Table 10 and Equations 27 - 32 and are summarized in Table 11. The % NO_3 remaining (third column) and the % NO_3 that decomposed provide an indication of the mass balance in the process. If they are equal, as was found with the constant current experiments, then there were no other products. A difference was found with these reactions indicating that ammonia may have escaped as a gaseous product, other products may have been produced, or simply, a large error is present in the analysis. These values were calculated using Equations 29, 30 and the initial and final nitrate concentrations. The calculations indicate that very little nitrate reduction occurred.

The column labeled “% NO_2 and NH_3 Formed” is the total amount of NO_2 and NH_3 formed and was calculated on the basis of the total moles of NO_3 , NO_2 and NH_3 in the final solution. Unlike the constant current conditions these conditions produced ammonia as the sole reaction product. The last column gives the coulombic efficiency calculated using Equation. 28. The column is split into two sub columns with one sub column a coulombic efficiency of the experiment to only ammonia and another reflecting a coulombic efficiency to ammonia and nitrite (denoted as the total coulombic efficiency). As the differences between these two efficiencies are small and follow the

same trend, an analysis of the total coulombic efficiency will represent the analysis for the coulombic efficiency to ammonia.

The results were analyzed as a Full Factorial DOE analyzing the data as two factors with two variables. The factors investigated were the initial solution pH and the initial nitrate concentration. The variables that were monitored were the coulombic efficiency, the amount of NO_3 removed, the amount of product formed (total amount of NO_2 and NH_3 formed) and the percent of NO_2 and NH_3 in the product. Table 12 presents the DOE results for the analysis of the effect of initial pH and the initial nitrate concentration using a copper cathode under a constant voltage on the electrochemical reduction of nitrate.

The trends in Tables 11 and 12 are similar to those in Tables 7 and 8 under constant current conditions. The initial nitrate concentration has a larger effect on the coulombic efficiency and nitrate removed than the initial solution pH. Figures 15 and 16 show a similar effect plot to Figure 9 and 10. At an initial nitrate concentration of 100 ppm the coulombic efficiencies and amount of nitrate removed are almost the same. While these efficiencies and removal percentages are higher than those done under the conditions of a sustained current, the efficiencies and removal percentages are still not 100%. This enforces the idea that the electrochemical nitrate reduction reaction had problems with the competing side reaction of water splitting; the ability of nitrate to be adsorbed or desorbed by the working electrode; the reversible reaction of nitrate [NO_3^-] reducing to nitrite [NO_2^-] and then the nitrite oxidizing back to nitrate of the solution; and the proper voltage/current selection. All of these are similar problems faced during

experiments run under a constant current. The discussions in Section 6.2 about the adsorption/desorption ability of nitrate and the reversible reaction of nitrate $[\text{NO}_3^-]$ reducing to nitrite $[\text{NO}_2^-]$ and then the nitrite oxidizing back to nitrate of the solution are applicable to this situation as well.

The extent of nitrate degradation and the coulombic efficiency are fairly low for the long electrolysis times (~2%). Thus, the side reactions, presumably the electrolysis of water to produce hydrogen is much more efficient under these conditions. The nitrate ion concentration had the biggest effect on the extent of reaction and the coulombic efficiency. The extent and the coulombic efficiency of the reaction decreased with an increase in the initial concentration of nitrate ion and subsequently the solution conductivity. This is seen in Figure 14. This correlation indicates that increasing the solution conductivity preferentially promotes the side reaction over the nitrate reduction. Somehow, the concentration of the negative nitrate ion at the cathode may be reduced with this increase in conductivity. Figure 14 compared to Figure 8 has more curvature. This is due in part to the larger coulombic efficiency at the constant potential at initial nitrate levels of 100 ppm as opposed to the sustained current level under the same conditions. It appears that at the low conductivity (approx. 1mS), a constant potential of 10.0V and a low initial nitrate concentration of 100 ppm allows for there to be both a high nitrate electrochemical reduction along with less of a reaction of the water splitting. However as the conductivity of the solution increases, the side reaction increases. This also means that the nitrate reaction decreases as more electrons go into the water splitting

reaction. The coulombic efficiency drops quickly as a result and the nitrate decomposition reaction essentially falls to almost nothing at the high concentrations.

Table 10. Results Obtained using a Copper Cathode under a 10.0 V Constant Potential.

	PH	[NO ₃] (as N)	Reaction Time (hrs:min)	Current (A)	Cond. (mS)	[NO ₂] (as N)	[NH ₃] (as N)
		[NO ₃] (ppm)				[NO ₂] (ppm)	[NH ₃] (ppm)
Initial	7.63	27.00	0:00	0.08390	1.090	0.00	0.00
		119.56				0.00	0.00
Std. Dev.		0.00		0.00198		0.00	0.00
		0.00				0.00	0.00
Final	8.61	23.50	2:36	0.09025	1.108	0.00	1.01
		104.06				0.00	1.23
Std. Dev.		0.71		0.00318		0.00	0.41
		3.13				0.00	0.50
Initial	9.00	24.00	0:00	0.08800	1.155	0.00	0.20
		106.28				0.00	0.24
Std. Dev.		0.00		0.00283		0.00	0.00
		0.00				0.00	0.00
Final	9.03	20.50	2:36	0.09040	1.153	0.00	1.45
		90.78				0.00	1.76
Std. Dev.		0.71		0.00000		0.00	0.21
		3.13				0.00	0.26

Table 10 Continued. Results Obtained using a Copper Cathode under a 10.0 V Constant Potential.

	PH	[NO ₃] (as N)	Reaction Time (hrs:min)	Current (A)	Cond. (mS)	[NO ₂] (as N)	[NH ₃] (as N)
		[NO ₃] (ppm)				[NO ₂] (ppm)	[NH ₃] (ppm)
Initial	8.91	69.00	0:00	0.11330	1.571	0.00	0.00
		305.55				0.00	0.00
Std. Dev.		0.00		0.01230		0.00	0.00
		0.00				0.00	0.00
Final	9.03	66.00	7:17	0.10815	1.600	1.20	1.62
		292.27				3.94	1.96
Std. Dev.		1.41		0.00375		0.57	1.82
		6.26				1.86	2.21
Initial	7.69	110.00	0:00	0.13530	1.973	0.02	0.00
		487.11				0.07	0.00
Std. Dev.		0.00		0.01315		0.00	0.00
		0.00				0.00	0.00
Final	9.15	110.00	12:25	0.11145	2.007	0.31	2.25
		487.11				1.02	2.74
Std. Dev.		0.00		0.00049		0.03	0.35
		0.00				0.09	0.43
Initial	9.02	110.00	0:00	0.14035	1.996	0.00	0.00
		487.11				0.00	0.00
Std. Dev.		0.00		0.01110		0.00	0.00
		0.00				0.00	0.00
Final	9.38	110.00	11:47	0.11935	2.055	0.00	2.65
		487.11				0.00	3.22
Std. Dev.		0.00		0.00559		0.00	0.07
		0.00				0.00	0.09

Table 11. Overall Results at a 10.0 V Constant Potential using a Copper Cathode.

Initial pH	Initial [NO ₃] (ppm)	% NO ₃ Remaining	%NO ₃ Reacted	% Break-Down of Product				Coulombic Efficiency (%)	
				%NO ₃ Unreacted	% NO ₂ and NH ₃ Formed	% NO ₂ of prd.	% NH ₃ of prd.	NH ₃ Rxn.	Total Rxn
7	100	87.04	12.96	95.87	4.13	0.00	100.00	17.75	17.75
9	100	85.42	14.58	93.41	6.59	0.00	100.00	17.35	17.35
8	300	95.34	4.66	95.91	4.09	52.26	47.74	2.81	3.20
7	500	97.33	2.67	97.73	2.27	12.16	87.84	1.91	1.96
9	500	97.33	2.67	97.65	2.35	0.00	100.00	2.00	2.00

Table 12. Full Factorial DOE Analysis using a Copper Cathode under Constant Potential Conditions.

Conditions (Initial pH, Initial [NO ₃])	Initial pH	[NO ₃] ₀		CE	CE	NO ₃	Product	Product Split	
	P	C	PC	Total %	NH ₃ %	Removed %	%	NO ₂ %	NH ₃ %
(7,100)	-	-	+	17.75	17.75	12.96	4.13	0.00	100.00
(7,500)	-	+	-	1.96	1.91	2.67	2.27	12.16	87.84
(9,100)	+	-	-	17.35	17.35	14.58	6.59	0.00	100.00
(9,500)	+	+	+	2.00	2.00	2.67	2.35	0.00	100.00
(8,300)	0	0	0	3.20	2.81	4.66	4.09	67.81	47.74
Total Coulombic Eff.									
Effect	-0.089	-7.785	0.113						
Curvature	6.56								
Coulombic Eff. NH ₃									
Effect	-0.077	-7.443	0.125						
Curvature	6.94								
NO ₃ Removed									
Effect	-0.157	-2.296	0.157						
Curvature	3.56								
Product									
Effect	0.635	-1.524	-0.595						
Curvature	-0.26								
NO ₂ Split									
Effect	-3.041	3.041	-3.041						
Curvature	-64.77								
NH ₃ Split									
Effect	3.041	-3.041	3.041						
Curvature	49.22

Figure 14. The Coulombic Efficiency versus Average Solution Conductivity under Constant Potential Conditions.

Figure 15. Effects Plot for the Average Total Coulombic Efficiency under Constant Potential Conditions Using a Copper Cathode.

Figure 16. Effects Plot for the Average Percent Nitrate Removal under Constant Potential Conditions Using a Copper Cathode.

In addition to the water splitting reaction, another problem was the voltage and current selection. The current should be selected in order to supply the electrochemical reaction with the proper current per unit area or current density. Equation 10 was used to calculate the limiting current density. For the electrochemical reduction of nitrate to nitrite by Equation 23, there are two electrons required. Assuming that the diffusion coefficient of nitrate in water calculated using the Wilke-Chang method is $1.027 \times 10^{-9} \text{ m}^2/\text{s}$ [31] and the diffusion layer thickness for the diffusion layer thickness between the electrode surface and the bulk concentration is 0.01 mm [32], then, Table 13 shows the limiting current density for various initial bulk nitrate concentrations.

Table 13. Minimum Current Densities for the Nitrate Reduction Reaction.

Initial [NO ₃ ⁻] (ppm)	Calculated Limiting Current Density (mA/cm ²)	Experimental Limiting Current Density (mA/cm ²)	Calculated Limiting Current Density / Experimental Limiting Current Density
100	1.60	0.83	1.92
300	4.79	0.83	5.77
500	7.99	0.83	9.63

The calculated value of the limiting current density at a diffusion layer thickness between the electrode surface and the bulk concentration of 0.01 mm is similar to the experimental value that Pader et al. [14] determined when using 2.8 mA/cm^2 . It can be seen that the minimum current density is 1.92 times higher than the experimental current density with 100 ppm nitrate to a minimum of 9.6 times higher than the experimental current density at 500 ppm nitrate. Especially at the high initial nitrate concentrations, the experimental current density is an appreciable fraction below the calculated limiting current. At an initial nitrate concentration of 500 ppm, the coulombic efficiencies and nitrate removal were on the same order of the constant current experiments. At an initial nitrate concentration of 100 ppm, the coulombic efficiencies and nitrate removal percentages were lower in the experiments done under a constant current. With currents that are below the limiting current density, considerations of the concentration variations near the working electrode, the surface overpotential associated with the electrode reaction and the ohmic potential drop in the bulk solution have to be taken into account [11].

6.5 Analysis of the Effects of Initial pH and Initial Concentration using a Copper Cathode under a Constant Potential on Nitrite and Ammonia Formation

Table 17 shows that the largest effect for the product formation is due to the initial nitrate concentration. Figure 17 supports this statement. Discussion on the effect of concentration on the coulombic efficiency and the nitrate removal discussed in Section 6.4 also apply to the amount of product formed. However Figure 13 shows that while the coulombic efficiency and the nitrate removal under low initial nitrate concentrations were essentially the same, the amount of product formed for each is significantly different as indicated in Figure 17. It was observed that the mass balances for the experiments with pH 7 and an initial nitrate concentration of 100 ppm NO_3 as well as the mass balances for the experiments with pH 7 and an initial nitrate concentration of 100 ppm NO_3 did not close as shown in Figure 17 under the same experimental conditions. A possible explanation for this discrepancy may be that at an initial pH of 7 and a higher voltage more ammonia is released as a gaseous product. Another possibility is another product is formed at a higher voltage in addition to ammonia and nitrite. The possible products are nitrous oxide, which may not have reacted, or gaseous N_2O , which may have formed. If a product such as NO_2 was produced, it could have adhered strongly to the working electrode and would not have been released into the solution. Additional experiments should capture the gaseous products and analyze the solution for additional products.

Figure 17. Effects Plot for the Average Percent Product Formation under Constant Potential Conditions Using a Copper Cathode.

Another interesting note is shown in Figure 18. The conversion from nitrite to ammonia was almost 100% in all cases. This is probably due to the higher voltage that was applied and sustained during the experiment. Although the rate-limiting step is the conversion to nitrite shown in Equation 23, there needs to be a high enough voltage so that there is an electrochemical reaction to the other products such as the reaction shown in Equation 24 in Section 2.2.

Figure 18. Effects Plot for the Percentage of the Product Formed that is Ammonia Under Constant Potential Conditions using a Copper Cathode

6.6 Error Analysis of Results using a Copper Cathode Under Constant Potential Conditions

Table 14 shows the statistical analysis of the calculated results giving the average in the third column, the range of the data (difference between the maximum value and the minimum value) in the fourth column and the standard deviation under the test conditions in the last column. The results show a slight variation, not as large as those in Table 9. However an additional run should be repeated to give more credibility to the data.

As discussed in Section 6.3, there are several reasons for this variance. The major sources of error involve the analytical techniques. An analysis more accurate than ion chromatography may be required for these low nitrate, nitrate and ammonia concentrations. As an example, in the calculation of the coulombic efficiency, an error of 1 ppm nitrate in the final solution amounts to a 20% error. This variance limits the reliability of the conclusions that may be drawn from the results.

Table 14. Statistical Data of the Results on under Constant Potential Conditions Using a Copper Cathode.

Initial pH	Initial [NO ₃] ppm	Average % (see Factor)	Range of Data Between Max, Min. Values	Standard Deviation for 2 Experimental Runs
Factor: The Total Coulombic Efficiency				
7	100	17.75	4.34	3.069
7	500	1.96	0.16	0.113
9	100	17.35	4.58	3.239
9	500	2.00	0.19	0.1344
8	300	3.20	4.38	3.097
Factor: The Coulombic Efficiency to NH ₃				
7	100	17.75	4.34	3.069
7	500	1.91	0.17	0.120
9	100	17.35	4.58	3.239
9	500	2.00	0.19	0.1344
8	300	2.81	4.60	3.25
Factor: The Nitrate Removed				
7	100	12.96	3.7	2.616
7	500	2.67	0	0
9	100	14.58	4.17	2.949
9	500	2.67	0	0
8	300	4.665	3.53	2.496
Factor: The Product Formed				
7	100	4.13	2.44	1.725
7	500	2.275	0.47	0.332
9	100	6.59	0.98	0.693
9	500	2.355	0.09	0.064
8	300	4.095	2.59	1.831

6.7 Comparison Between Constant Current and Constant Voltage Experimental Results

Table 15 shows the similarities and differences between the experiments conducted under constant current and constant voltage conditions.

Table 15. A Comparison between Constant Current and Constant Voltage Experiments.

	Coulombic Efficiency	Nitrate Reduction	Product	Nitrite/Ammonia Split
Similarities	The trend is similar with the highest efficiencies being at the nitrate concentrations of 100 ppm. The initial pH has very little interaction.	The trend was similar to the coulombic efficiency.	At the high concentrations the product formation was low.	At an initial pH of 9, the entire product was ammonia.
Differences	The coulombic efficiencies for experiments under a constant voltage were higher at the initial nitrate concentrations of 100 ppm.	There was a higher reduction that took place under a lower initial nitrate concentration for constant potential conditions.	There is a possibility that there were other products that formed at a pH 7 and low initial nitrate concentration for constant potential conditions.	More nitrite was formed under the initial pH of 7 for a constant current.

While the higher voltage solved some problems such as increasing the coulombic efficiency at the lower concentrations and the formation of more ammonia in the product, it did not solve all of the problems. There were problems with the electrochemical reaction at the high initial nitrate concentrations. This was probably due to a number of

reasons; the major reason being that at there may not have been enough current supplied for the reaction, as there was competition for current with the water splitting reaction.

6.8 Additional Ion Concentration

Experiments conducted in which the solution sulfate concentration was spiked and the solution chloride concentration was spiked under constant current conditions at an initial pH of 7 and an initial nitrate concentration of 100 ppm gave results that were predicted by literature [16]. Table 16 summarizes these results.

Table 16. Overall Results at Constant Current with Various Chloride and Sulfate Concentrations.

Summary at Constant Current, Initial pH of 7, Initial $[\text{NO}_3] = 100$ ppm							
Chloride/ Sulfate Conc. (ppm)	% NO_3 Remaining	% NO_3 Reacted	% Break-Down of Product				Total Coulombic Efficiency %
			% NO_3 Unreacted	% NO_2 and NH_3 Formed	% NO_2 of prd.	% NH_3 of prd.	
22/44	92.43	7.57	90.94	9.06	52.30	47.70	6.32 $\pm$ 2.40
22/440	93.75	6.25	95.51	4.49	0.00	100.00	6.43 $\pm$ 3.05
200/44	95.83	4.17	97.54	2.46	0.00	100.00	4.35 $\pm$ 0.01

As with all the previous results, the data has a large standard deviation. The trends show that chloride negatively effects the reaction while sulfate does not inhibit the reaction as poorly as chloride and is essentially the same with-in experimental error as the experiments conducted with low ion concentrations. This would be a similar effect to sulfate that only takes up sites on the copper cathode while chloride may inhibit the catalytic activity of the copper cathode sites in some manner. These results are in-line with the work done by de Groot and Koper [16] for platinum.

de Groot and Koper [16] indicated that on platinum, sulfide ions and chloride ions in solution can have a negative effect on nitrate reduction. Sulfate ions compete for sites on the cathode while chloride ions, in addition to competing for sites, may also inhibit the catalytic effect of the electrode. This is especially the case for copper where Sonoyama et al. report that in the reduction of dichloromethane using a copper cathode, the reaction involves the chloride adsorbing onto the copper surface [33].

However, literature has not indicated the effect of sulfate ions specifically for copper cathodes. Possible electrolytes are difficult to determine for the electrochemical reduction of nitrate in the treatment of water for a number of reasons. Table 17 outlines some of the possible electrolytes and the problem they may present.

Table 17. Problems with using Possible Electrolytes for Nitrate Reduction.

Possible Electrolyte	Problem(s)
Hydrochloric Acid	Addition of Cl^- ions to the system decreases system pH, which can change nitrate reduction kinetics.
Perchloric Acid	Addition of ClO_4^- (EPA regulated compound), decreases system pH.
Sulfuric Acid	Addition of SO_4^{2-} ions to the system decreases system pH.
Sodium Chloride	Addition of Cl^- ions to the system.
Nitric Acid	Addition of Nitrate to the system decreases system pH.
Formic Acid	Expensive (possibly chemically reduced nitrate – cannot be used in a study unless specified), decreases system pH.
Ammonium Hydroxide	Increases ammonia in system and water pH.
Sodium Hydroxide	Increases system pH and water pH.

6.9 Degradation Nitrate Ion on a Carbon-Felt Cathode under Constant Current Conditions

Effectively no electrochemical reduction of nitrate occurred using a carbon (graphite)-felt cathode under constant current conditions. Experiments were carried out exactly as described in Section 6.1 with the exception that the working electrode was a flat piece of carbon-felt (120 cm²). The results are summarized in Table 18. They show that there was almost no nitrate reduction as compared with the results of the copper electrode (Table 7). Under a sustained current, almost no nitrate reduction occurred using a carbon-felt electrode. The coulombic efficiencies were ~1-2%. This result is in line with the work done by Peel et al. [22] and contrasts to the work presented by Koparal and Ogutveren [13].

Table 18. Overall Results at a 0.100 A Constant Current using a Carbon-Felt Cathode.

Initial pH	Initial [NO ₃] (ppm)	% NO ₃ Remaining	%NO ₃ Reacted	% Break-Down of Product				Coulombic Efficiency (%)	
				%NO ₃ Unreacted	% NO ₂ and NH ₃ Formed	% NO ₂ of prd.	% NH ₃ of prd.	NH ₃ Rxn.	Total Rxn
7	100	100.00	0.00	100.00	0.00	0.00	0.00	0.00	0.00
9	100	98.92	1.08	98.92	1.08	0.00	100.00	1.20	1.20
8	300	98.68	1.32	98.85	1.15	0.00	100.00	1.21	1.21
7	500	97.92	2.08	99.51	0.49	0.00	100.00	0.46	0.46
9	500	98.22	1.78	99.90	0.10	17.52	82.48	0.08	0.08

6.10 Degradation Nitrate Ion on a Carbon-Felt Cathode under Constant Potential Conditions

As with the constant current conditions, effectively no nitrate reduction was observed using a carbon-felt electrode, and the coulombic efficiencies were 1-2%. The results are shown in Table 19.

Table 19. Overall Results at a 10.0 V Constant Potential using a Carbon-Felt Cathode.

Initial pH	Initial [NO ₃] (ppm)	% NO ₃ Remaining	%NO ₃ Reacted	% Break-Down of Product				Coulombic Efficiency (%)	
				%NO ₃ Unreacted	% NO ₂ and NH ₃ Formed	% NO ₂ of prd.	% NH ₃ of prd.	NH ₃ Rxn.	Total Rxn
7	100	98.15	1.85	97.80	2.20	0.00	100.00	2.29	2.29
9	100	100.00	0.00	97.09	2.91	0.00	100.00	0.00	0.00
8	300	99.33	0.67	99.47	0.53	3.03	96.97	0.54	0.55
7	500	98.67	1.33	99.84	0.16	6.04	93.96	1.01	1.01
9	500	97.33	2.67	99.65	0.35	0.00	100.00	1.64	1.64

CHAPTER 7

CONCLUSIONS AND FUTURE DIRECTIONS

The electrochemical reduction of nitrate in low conductivity aqueous solutions was investigated using a copper and a carbon electrode. Tests were run varying the initial pH of the water between 7 and 9 and the initial nitrate concentration between 100 ppm and 500 ppm nitrate. It was found that nitrate could be reduced electrochemically using a copper cathode. Results using a carbon felt electrode indicated that the carbon felt was not very catalytically active to nitrate reduction and in all experiments very little reduction took place (around the order of 0-2% coulombic efficiencies). A copper cathode was much more catalytically active with 2-17% coulombic efficiencies. As a result the bulk of the investigation focused on how the initial pH and the initial nitrate concentration affected the reaction using a copper cathode. The parameters used to measure the electro-reduction reaction of nitrate included the coulombic efficiency, the percentage of nitrate reduced, the amount of product that was formed and the split of the product as either ammonia or nitrite.

The results were that the initial nitrate concentration had a larger effect on the reaction rather than the initial pH under all conditions tested. As the initial nitrate concentration increased, the amount of nitrate reduced was diminished and the electrochemical reduction reaction decreased.

Investigating the possible reasons for this trend leads to development for future work. A possible reason included that as the nitrate concentration increased, the conductivity of the solution increased, and the competing side reaction of water splitting increased as opposed to the nitrate reduction reaction. Other reasons included the ability of nitrate to be adsorbed or desorbed by the working electrode; the reversible reaction of nitrate $[\text{NO}_3^-]$ reducing to nitrite $[\text{NO}_2^-]$ and then the nitrite oxidizing back to nitrate of the solution; and the proper voltage/current selection.

Future work should include first establishing the repeatability of the experiments. Therefore proper equipment with appropriate tolerances should be used. This includes a proper power supply, an accurate pH meter, conductivity meter and determining an appropriate analytical method to detect low levels of nitrate, nitrite and ammonia. After the repeatability of the runs have been established, experiments should be conducted under a constant voltage of 10.0 V at an initial nitrate concentration of 100 ppm to determine if almost all of the nitrate can be reduced in the solution given a 17% current efficiency.

Additional experiments should be conducted varying the parameters such as the spacing between the electrodes and electrode area or geometry to supply a proper voltage and current for the reaction. The next step would be to find an appropriate electrolyte for this reaction so that supplying a proper voltage and current, and controlling the solution conductivity can be realized. It is difficult to find an electrolyte to increase the conductivity of the solution and not interfere with the nitrate reduction reaction. Using cyclic voltametry would be useful to define voltage and current relationships as well.

A way to test for the amount of nitrate reducing to nitrite at the cathode and then the nitrite oxidizing back to nitrate at the anode would be to conduct experiments in a divided cell where there is a separate anolyte and catholyte with a semi-permeable membrane. This membrane would not be permeable to nitrate, nitrite or other possible products. It will be easy to track the products formed and reduce the possibility of any nitrite in solution to reduce back to nitrate. Theoretically this should increase the coulombic efficiency.

Additional work should be done to ensure that all gaseous products are captured during the experiment. This would allow for proof that an increase in solution conductivity would lead to the water splitting reaction becoming more prominent. A mass balance on the system accounting for all of the current could then be completed.

NOMENCLATURE

Symbol	Units	Definition
ΔG	kJ/mol	Change in Gibbs free energy of a reaction
n	moles e^-	Moles of electrons
A	m^2	Area
F	Coulombs/mole	Faraday's Constant = 96485 C/mol
m	g	mass of product formed
I	A	Current
ε	V	Potential
t	s	Time measured in seconds
M_w	g/mol	Molar mass of a substance
i	A/m^2	Current density = I/A
O	V	Standard Potential at 25°C, 101.325kPa
ν	moles/ m^3s	Electrochemical reaction rate
Q	--	$[Activity\ of\ Product]/[Activity\ of\ Reactants]$
K	--	$[Activity\ of\ Product]/[Activity\ of\ Reactants]$ at Equilibrium
η	V	Overpotential
i_o	A/m^2	Exchange current density or the current density when there is no net reaction
α_A	--	Anodic charge transfer co-efficient = $(1 - \alpha_C)$. Usually about 0.5 (see Equation 8)
α_C	--	Cathodic charge transfer co-efficient = $(1 - \alpha_A)$. Usually about 0.5 (see Equation 8)
η_A	V	Anodic overpotential = $-\eta_C$
η_C	V	Cathodic overpotential = $-\eta_A$
i_L	A/m^2	Limiting Current Density
δ	nm	Diffusion layer thickness between electrode surface and bulk concentration
D	cm^2/s	Diffusion co-efficiente
c	M	Concentration
∇	--	Gradient
N_i	mol/ m^2/s	Flux of the i^{th} species
u	m/s	Velocity of fluid

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APPENDIX A
SAMPLE CALCULATIONS

1. CALCULATION FOR TIME

Equation 3 solved for time:

$$t = \frac{m \cdot n \cdot F}{I \cdot M_w} \quad \text{Equation 3}$$

Using the following raw data taken from an initial [NO₃] of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions:

$$m = 100 \text{ ppm} = 100 \text{ mg/L} = 0.1 \text{ g/L}$$

$$n = 8 \text{ electrons / mol}$$

$$F = 96485 \text{ C/mol}$$

$$M_w = 62.04 \text{ g/mol}$$

$$I = 0.1 \text{ A} = 0.1 \text{ C/s}$$

$$\text{Volume of Solution} = 0.75 \text{ L}$$

Therefore;

$$t = [0.1 \text{ g/L} \cdot 0.75 \text{ L} \cdot 8 \text{ e}^-/\text{mol} \cdot 96485 \text{ C/mol}] / [0.1 \text{ A} \cdot 62.04 \text{ g/mol}]$$

$$t = 9331 \text{ s} = 2.60 \text{ hours}$$

2. CALCULATION ON THE MASS BALANCE ON NITROGEN

Using Equation 27:

$$0 = \text{Nitrogen}_{\text{initial nitrate}} - \text{Nitrogen}_{\text{final nitrate}} - \text{Nitrogen}_{\text{ammonia}} - \text{Nitrogen}_{\text{nitrite}} \quad \text{Equation 27}$$

Using the following raw data taken from an initial $[\text{NO}_3]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

$\text{Nitrogen}_{\text{initial nitrate}} = 23.25 \text{ mg/L}$ (Nitrate reported as Nitrogen)

$\text{Nitrogen}_{\text{final nitrate}} = 21.00 \text{ mg/L}$ (Nitrate reported as Nitrogen)

$\text{Nitrogen}_{\text{final nitrite}} = 1.13 \text{ mg/L}$ (Nitrite reported as Nitrogen)

$\text{Nitrogen}_{\text{final ammonia}} = 1.13 \text{ mg/L}$ (Ammonia reported as Nitrogen)

$M_w = 14.01 \text{ g/mol} = 14\,010 \text{ mg/mol}$

Therefore;

$$0 = [(23.35 \text{ mg/L}) - (21.00 \text{ mg/L} + 1.13 \text{ mg/L} + 1.13 \text{ mg/L})] * 14\,010 \text{ mg/mol}$$

3. CALCULATION FOR THE COULOMBIC EFFICIENCY FOR AMMONIA

Using Equation 28:

$$\text{Coulombic Efficiency} = \frac{\text{Coulombs calculated to form desired product}}{\text{Total Coulombs}} \quad \text{Equation 28}$$

Using the following raw data taken from an initial $[\text{NO}_3]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

Coulombs required to form Ammonia

$n = 8 \text{ electrons/mol}$

$F = 96485 \text{ C/mol}$

Amt of NO_3 that reduced to $\text{NH}_3 = 8.03\text{E-}05 \text{ mol}$

Coulombs required for Ammonia = $8 \text{ e}^-/\text{mol} * 96485 \text{ C/mol} * 8.03\text{E-}05 \text{ mol} = 62.0 \text{ C}$

Total Coulombs passed through the experiment =

avg. experimental current * total time = $0.1 \text{ A} * 9331 \text{ s} = 997 \text{ C}$

Therefore:

Coulombic Efficiency to Ammonia = $(62.0 \text{ C} / 997 \text{ C}) * 100\% = 6.2\%$

4. CALCULATION FOR THE TOTAL COULOMBIC EFFICIENCY CALCULATION

Using Equation 28:

$$\text{Coulombic Efficiency} = \frac{\text{Coulombs calculated to form desired product}}{\text{Total Coulombs}} \quad \text{Equation 28}$$

Using the following raw data taken from an initial $[\text{NO}_3]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

Coulombs required for Ammonia and Coulombs required for Nitrite

$n = 8 \text{ e}^-/\text{mol}$ (for Ammonia)

$n = 2 \text{ e}^-/\text{mol}$ (for Nitrite)

$F = 96485 \text{ C/mol}$

Amt of NO_3 that reduced to $\text{NH}_3 = 8.03\text{E-}05 \text{ mol}$

Amt of NO_3 that reduced to $\text{NO}_2 = 8.03 \text{ E-}05 \text{ mol}$

Coulombs required to form Ammonia = $8 \text{ e}^-/\text{mol} * 96485 \text{ C/mol} * 8.03\text{E-}05 \text{ mol} = 62.0 \text{ C}$

Coulombs required to form Nitrite = $2 \text{ e}^-/\text{mol} * 96485 \text{ C/mol} * 8.03\text{E-}05 \text{ mol} = 15.4 \text{ C}$

Total Coulombs passed through the experiment =

avg. experimental current * total time = $0.1 \text{ A} * 9331 \text{ s} = 997 \text{ C}$

Therefore:

Total Coulombic Efficiency = $[(15.4 \text{ C} + 62.0 \text{ C}) / 997 \text{ C}] * 100\% = 7.8\%$

5. CALCULATION FOR THE NITRATE REMOVED

Using Equation 29:

$$\text{Nitrate Removal \%} = \frac{\text{Nitrate in Influent} - \text{Nitrate in Effluent}}{\text{Nitrate in Influent}} \quad \text{Equation 29}$$

Using the following raw data taken from an initial $[\text{NO}_3]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

Nitrate in Influent = 23.25 mg (reported as mg/L Nitrate-Nitrogen)

$$\begin{aligned} \text{Converting to Nitrate} &= 23.25 \text{ mg} * [(62.04 \text{ g/mol Nitrate}) / (14.01 \text{ g/mol Nitrogen})] \\ &= 103.0 \text{ mg} \end{aligned}$$

Nitrate in Effluent = 21.00 mg (reported as mg/L Nitrate-Nitrogen)

$$\begin{aligned} \text{Converting to Nitrate} &= 21.00 \text{ mg} * [(62.04 \text{ g/mol Nitrate}) / (14.01 \text{ g/mol Nitrogen})] \\ &= 93.0 \text{ mg} \end{aligned}$$

Nitrate Removal % =

$$\begin{aligned} &[(103.0 \text{ mg} - 93.0 \text{ mg}) * 62\,040 \text{ mg/mol}] / [103.0 \text{ mg} * 62\,040 \text{ mg/mol}] * 100\% \\ &= 9.7\% \end{aligned}$$

6. CALCULATION FOR THE AMOUNT OF PRODUCT FORMED

Using Equation 30:

$$\text{Amount of Product} = \frac{\text{Moles of Ammonia and Nitrite Formed}}{\text{Total Moles of Product Formed}} \quad \text{Equation 30}$$

Using the following raw data taken from an initial $[\text{NO}_3^-]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

Moles of Ammonia formed:

$$\begin{aligned} & (1.13 \text{ mg} * [(17.04 \text{ g/mol Ammonia})/(14.01 \text{ g/mol Nitrogen})]) / 17.04 \text{ mg/mmol} \\ & = 0.08 \text{ mmol ammonia} \end{aligned}$$

Moles of Nitrite formed:

$$\begin{aligned} & (1.13 \text{ mg} * [(46.05 \text{ g/mol Nitrate})/(14.01 \text{ g/mol Nitrogen})]) / 46.05 \text{ mg/mmol} \\ & = 0.08 \text{ mmol nitrite} \end{aligned}$$

Total moles of product formed = moles of ammonia + moles of nitrite

$$= 0.08 \text{ mmol} + 0.08 \text{ mmol} = 0.161 \text{ mmol}$$

Moles of unreacted nitrate (i.e. nitrate in effluent)

$$= 93.0 \text{ mg} / 62.04 \text{ mg/mmol}$$

$$= 1.5 \text{ mmol}$$

Total moles in final product = unreacted nitrate + moles of ammonia formed + moles of

$$\text{nitrite formed} = 0.161 \text{ mmol} + 1.5 \text{ mmol} = 1.66 \text{ mmol}$$

$$\text{Amount of Product} = (0.161 \text{ mmol} / 1.66 \text{ mmol}) * 100\% = 9.7 \%$$

7. CALCULATION OF PERCENT SPLIT OF REACTION PRODUCT

Using Equations 31 and 32:

$$\% \text{ Split of Nitrite} = \frac{\text{Moles of Nitrite}}{\text{Total Moles of Ammonia and Nitrite Formed}} \quad \text{Equation 31}$$

$$\% \text{ Split of Ammonia} = \frac{\text{Moles of Ammonia}}{\text{Total Moles of Ammonia and Nitrite Formed}} \quad \text{Equation 32}$$

Using the following raw data taken from an initial $[\text{NO}_3^-]$ of 100 ppm and an initial solution pH of 7 using a copper electrode under constant current conditions (third run):

Moles of nitrite in final product = 0.08 mmol

Moles of ammonia in final product = 0.08 mmol

Total moles of ammonia and nitrite = 0.08 mol + 0.08 mmol = 0.160 mmol

Therefore;

$\% \text{ Split of nitrite} = [0.08 \text{ mmol} / 0.160 \text{ mmol}] * 100\% = 50\%$ of product is nitrite

$\% \text{ Split of ammonia} = [0.08 \text{ mmol} / 0.160 \text{ mmol}] * 100\% = 50\%$ of product is ammonia

8. CALCULATION OF THE LIMITING CURRENT

Using Equation 10:

$$i_L = n \cdot F \cdot D \cdot \frac{C_b}{\delta} \quad \text{Equation 10}$$

Using the following raw data taken from an initial [NO₃] of 100 ppm:

Coulombs required.

$n = 2 \text{ e}^-/\text{mol}$ (for reaction limiting step electron transfer)

$F = 96485 \text{ C/mol}$

$C_b = 100 \text{ ppm}$

$\delta = 0.01 \text{ mm}$

D = calculated using the empirical correlation of Wilke and Change [12]

$$D_{AB}^o = \frac{(117.3 \times 10^{-18}) \cdot (\varphi \cdot M_B)^{0.5} \cdot T}{\mu \cdot \nu_A^{0.6}}$$

where

D_{AB}^o = diffusivity of A in very dilute solution in solvent B, m²/s

M_b = molecular weight of solvent, kg/kmol = 18.02 kg/kmol (water)

T = temperature, K = 293 K

μ = solution viscosity, kg/m.s = 0.001005 kg/m.s (water)

ν_A = solute molal volume at normal boiling point, m³/kmol = 0.0756 for nitrate as solute

φ = associate factor for solvent = 2.26 for water as solute

Therefore;

$D = 1.0276 \times 10^{-9} \text{ m}^2/\text{s}$ and

$i_L = 1.60 \text{ mA/cm}^2$

9. DOE CALCULATIONS

DOE Calculations were completed using a computer program. DOE Pack 2000 and MiniTab

APPENDIX B

RAW DATA FOR EXPERIMENTS CARRIED OUT UNDER CONSTANT CURRENT CONDITIONS

Summary at Constant Current							
Experiment	% Break-Down of Product			%NO ₃ Removed	Minimum % NO ₃ Removal	Coulombic Efficiency NH ₃	Coulombic Efficiency Total
	% NO ₂ and NH ₃ Formed	% NO ₂	% NH ₃				
DOEN-C71	9.06%	52.30%	47.70%	7.57%	47.02%	5.01%	6.32%
DOEN-C75	2.49%	37.17%	62.83%	2.67%	87.88%	2.52%	2.57%
DOEN-C91	8.92%	26.74%	73.26%	6.94%	44.44%	6.58%	7.34%
DOEN-C95	2.39%	12.99%	87.01%	2.67%	87.88%	2.14%	2.21%
DOEN-C83	4.73%	12.97%	87.03%	5.00%	81.20%	4.23%	4.40%
DOEN-GF83	1.15%	0.00%	100.00%	1.32%	81.21%	1.21%	1.21%
DOEN-GF71	0.00%	0.00%	0.00%	0.00%	47.02%	0.00%	0.00%
DOEN-GF75	0.49%	0.00%	100.00%	2.08%	87.88%	0.46%	0.46%
DOEN-GF91	1.08%	0.00%	100.00%	1.08%	51.48%	1.20%	1.20%
DOEN-GF95	0.10%	17.52%	82.48%	1.78%	58.59%	0.08%	0.08%

	Target Initial pH	Target Initial [NO ₃] (ppm)	Reaction Time (hrs:min)	Current (A)	Voltage (V)	pH	Vol. (L)	Temp. (°C)	TDS (ppm)	NaCl % (%)	Cond. (µS)	[NO ₃] (as N) (ppm)	[NO ₂] (as N) (ppm)	[NH ₃] (as N) (ppm)	[SO ₄] (ppm)	[Cl] (ppm)
Initial	7	100	02:36	0.10027	11.05	7.27	0.75	22	526	2.03	1051	25.67	0.00	0.00	44	23
Std. Dev.				0.00046	0.09	0.08	0.00	1.73	16.74	0.06	32	113.66	0.00	0.00	1	1
Final				0.10430	10.39	8.34	0.75	22	533	2.03	1065	4.62	0.00	0.00		
Std. Dev.				0.00745	0.67	0.24	0.00	1.73	22.94	0.06	45	20.45	0.00	0.00		
Initial	9	100	02:36	0.10077	9.75	9.05	0.75	21	643	2.47	1286	23.67	1.21	1.16		
Std. Dev.				0.00025	0.78	0.01	0.00	1.15	9.81	0.06	20	104.80	3.99	1.41		
Final				0.10107	8.81	9.08	0.75	22	649	2.47	1297	3.79	0.34	0.50		
Std. Dev.				0.00075	0.44	0.22	0.00	1.73	12.86	0.06	25	16.77	1.11	0.61		
Initial	8	300	07:46	0.10100	9.10	8.22	0.75	22	787	3.00	1575	24.00	0.00	0.00	46	34
Std. Dev.				0.00056	0.43	0.00	0.00	1.73	5.20	0.00	9	106.28	0.00	0.00	2	8
Final				0.10130	8.90	8.29	0.74	22	801	3.10	1603	0.00	0.00	0.00		
Std. Dev.				0.00115	0.85	0.30	0.00	1.73	1.73	0.00	5	22.33	0.69	1.50		
Initial				0.10100	9.10	8.22	0.75	22	787	3.00	1575	98.90	2.27	1.82		
Std. Dev.				0.00056	0.43	0.00	0.00	1.73	5.20	0.00	9	0.58	0.60	0.17		
Final				0.10130	8.90	8.29	0.74	22	801	3.10	1603	2.56	0.00	0.21		
Std. Dev.				0.00115	0.85	0.30	0.00	1.73	1.73	0.00	5	71.00	0.00	0.00	42	30
Initial	7	500	12:57	0.10023	7.89	7.18	0.75	21	994	3.87	1987	314.41	0.00	0.00	45	37
Std. Dev.				0.00025	0.16	0.08	0.00	1.15	22.52	0.12	42	487.11	0.00	0.00	3	7
Final				0.08810	8.14	8.75	0.73	21	1018	3.97	2037	0.00	0.00	0.00		
Std. Dev.				0.01341	0.31	0.21	0.01	1.15	38.73	0.12	76	110.00	0.22	2.65		
Initial	9	500	12:57	0.10040	7.88	9.03	0.75	22	1047	4.10	2097	487.11	0.00	0.00	49	42
Std. Dev.				0.00057	1.04	0.00	0.00	0.00	0.00	0.00	0	0.00	0.00	0.00	0	0
Final				0.09060	8.54	8.67	0.73	22	1080	4.20	2160	0.00	0.00	0.00		
Std. Dev.				0.01499	0.11	0.08	0.00	0.00	4.95	0.00	8	110.00	0.35	2.35		
												487.11	1.13	2.86		
												0.00	0.05	0.35		
												0.00	0.16	0.43		

DATA - CONSTANT CURRENT

Target Initial pH	Target Initial [NO ₃]	Avg. Soln. Conductivity μS	Coulombic Efficiency %
7	100	1058	6.32%
9	100	1292	7.34%
8	300	1589	4.40%
7	500	2012	2.57%
9	500	2129	2.21%

Coulombic Efficiency versus Solution Conductivity Under Constant Current Conditions

DOE Numbers - Copper Constant Current

Run	Treatments	INO3C	NO3Removed	CE Total	CE NH4	Product	Run1	Run2	Run3	Run1	Run2	Run3	Run1	Run2	Run3
1	7	100	4.35%	8.70%	9.60%	3.54%	7.64%	7.77%	2.61%	6.22%	6.10%	11.30%	58.71%	41.20%	51.85%
2	7	500	1.33%	2.67%	4.00%	0.09%	1.90%	5.72%	0.00%	5.72%	0.36%	2.03%	100.00%	100.00%	100.00%
3	9	100	8.33%	8.33%	4.17%	7.19%	8.64%	6.16%	1.03%	6.13%	7.84%	11.59%	59.07%	62.71%	100.00%
4	9	500	2.67%	2.67%	2.67%	1.66%	2.55%	2.21%	1.00%	2.14%	2.20%	2.58%	40.93%	84.66%	89.35%
C1	8	300	2.76%	8.19%	4.04%	2.99%	8.21%	2.01%	2.74%	7.95%	3.75%	8.51%	15.32%	72.80%	86.26%

DOE Numbers - Graphite Felt Constant Current

Run	Treatments	INO3C	NO3Removed	CE Total	CE NH4	Product	Run1	Run2	Run3	Run1	Run2	Run3	Run1	Run2	Run3
5	7	100	-4.35%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
6	7	500	2.67%	1.11%	0.90%	0.00%	2.77%	0.09%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
7	9	100	2.67%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
8	9	500	2.67%	1.78%	1.78%	0.11%	0.13%	0.13%	0.13%	0.00%	0.14%	0.17%	16.13%	83.87%	81.08%
C2	8	300	-0.10%	2.72%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%

Factors Initial pH Concentration Electrode
 P C WE
 + 7.0 100 Copper
 + 9.0 500 Graphite Felt +

DOE - Design of Experiments for Electrochemical Reduction of Nitrate

Trial	Mean	P	C	PC	PWE	CWE	PCWE	CE	CE NH4	NO3Removed	Product	NO2	NH3
1	+	+	-	+	+	+	+	6.32	5.01	7.57	9.06	52.30	47.70
2	+	+	+	-	+	+	+	7.34	6.94	6.94	8.92	26.74	73.26
3	+	+	+	+	-	+	+	2.57	2.52	2.67	2.49	37.17	62.83
4	+	+	+	+	+	-	+	2.21	2.14	2.67	2.39	12.99	87.01
5	+	+	+	+	+	+	+	0.20	0.20	1.08	1.08	0.00	99.92
6	+	+	+	+	+	+	+	0.46	0.46	2.08	0.43	0.00	100.00
7	+	+	+	+	+	+	+	0.08	0.08	2.37	0.10	11.68	54.96
CE Total	20.169	10.020	5.316	8.609	10.166	14.190	9.983						
Sum +	9.341	14.854	11.560	10.432	10.003	5.980	10.186						
Sum -	20.169	20.169	20.169	20.169	20.169	20.169	20.169						
Overall Sum	1.488	-9.538	-2.951	-16.694	0.163	9.210	-0.203						
Difference	2.571	0.372	-2.385	-4.173	0.041	2.052	-0.051						
Effect													

CE NH4	Sum +	17.985	10.001	5.194	7.220	7.737	8.809	12.126	9.119
Sum -	17.985	17.985	17.985	17.985	17.985	17.985	17.985	17.985	17.985
Overall Sum	17.985	2.016	-7.597	-3.528	-14.511	-0.368	6.267	0.373	0.093
Difference	2.244	-1.839	-0.882	-3.628	-0.092	1.567	0.093		
Effect									

NO3Removed	Sum +	13.060	9.782	12.612	12.612	13.689	18.957	11.985
Sum -	13.060	13.060	13.060	13.060	13.060	13.060	13.060	13.060
Overall Sum	25.377	25.377	25.377	25.377	25.377	25.377	25.377	25.377
Difference	25.377	0.743	-5.813	-0.153	-14.326	2.001	12.556	-1.411
Effect	3.172	0.166	-1.463	-0.038	-3.582	0.500	3.139	-0.353

Product	Sum +	24.521	12.489	5.468	11.552	1.665	12.726	18.564	11.509
	Sum -	..	12.032	19.052	12.969	22.855	11.794	5.957	13.011
	Overall Sum	24.521	24.521	24.521	24.521	24.521	24.521	24.521	24.521
	Difference	24.521	0.457	-13.564	-1.417	-1.190	0.932	12.607	-1.502
	Effect	3.865	0.114	-3.396	-0.354	-5.297	0.233	3.152	-0.375

NO2	Sum +	140.873	51.408	61.839	76.967	11.683	101.147	90.717	75.589
	Sum -	..	89.465	79.034	63.906	129.190	39.726	50.156	65.284
	Overall Sum	140.873	140.873	140.873	140.873	140.873	140.873	140.873	140.873
	Difference	140.873	-38.056	-17.196	13.061	11.717	61.422	40.561	10.305
	Effect	17.609	-9.514	-4.299	3.285	-29.177	15.305	10.140	2.576

NH3	Sum +	492.460	281.925	304.820	189.700	271.651	232.186	275.950	191.078
	Sum -	..	210.535	187.632	302.761	270.810	260.274	216.511	301.382
	Overall Sum	492.460	492.460	492.460	492.460	492.460	492.460	492.460	492.460
	Difference	492.460	71.390	117.196	-113.061	49.159	-28.088	59.439	-110.305
	Effect	61.558	17.847	29.229	-28.265	-12.250	-7.022	14.860	-27.976

DOE Numbers - Copper Constant Current

Run	Treatments		NO ₃ Removed			CE Total			CE NH ₃			Product			NH ₃		
	IPH	IND3C	Run1	Run2	Run3	Run1	Run2	Run3	Run1	Run2	Run3	Run1	Run2	Run3	Run1	Run2	Run3
1	7	100	4.35%	8.70%	9.68%	3.54%	7.64%	7.77%	2.61%	6.20%	6.22%	6.10%	11.39%	9.68%	50.00%	51.85%	50.00%
2	7	500	1.33%	2.67%	4.00%	0.06%	1.80%	5.72%	0.00%	1.84%	5.72%	0.39%	2.74%	5.09%	100.00%	86.50%	100.00%
3	9	100	8.33%	8.33%	4.17%	7.19%	8.64%	6.10%	6.13%	7.44%	6.10%	11.39%	11.29%	5.74%	56.07%	60.71%	100.00%
4	9	500	2.67%	2.67%	2.67%	1.86%	2.55%	2.21%	1.80%	2.46%	2.14%	2.20%	2.58%	15.32%	84.68%	86.35%	87.01%
5	8	300	2.76%	8.19%	4.04%	2.99%	8.21%	2.01%	2.74%	7.95%	2.01%	3.75%	8.51%	1.93%	72.80%	88.28%	100.00%

Factors

Initial pH

Concentration

C

7.0 100

9.0 500

DOEN - Design of Experiments for Electrochemical Reduction of Nitrate

Trial	Mean	P	C	PC	CE			NO ₃ Removed			Product			NH ₃		
					Total	CE	NH ₃	Total	CE	NH ₃	Total	CE	NH ₃	Total	CE	NH ₃
1	+	-	-	+	6.32	5.01	7.67	6.32	5.01	7.67	9.08	52.39	47.70	62.93	51.85	50.00%
2	+	-	+	-	2.57	2.52	2.49	2.57	2.52	2.49	2.74	3.75	62.93	62.93	86.50%	100.00%
3	+	+	-	-	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	56.07%	60.71%
4	+	-	+	-	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	2.34	84.68%	86.35%
5	0	0	0	0	4.40	4.23	5.00	4.40	4.23	5.00	4.73	12.97	87.03	87.03	88.28%	100.00%
CE Total	18.43	9.55	4.76	8.53												
Sum +	18.43	9.55	4.76	8.53												
Sum -	18.43	9.55	4.76	8.53												
Overall Sum	18.43	9.55	4.76	8.53												
Difference	18.43	9.55	4.76	8.53												
Effect	18.43	9.55	4.76	8.53												
Center Point Average	4.40	0.165578	-2.218524	-0.343												
Curvature	0.20															

CE NH ₃																	
Sum +	16.25	8.72	4.66	7.15													
Sum -	16.25	8.72	4.66	7.15													
Overall Sum	16.25	8.72	4.66	7.15													
Difference	16.25	8.72	4.66	7.15													
Effect	16.25	8.72	4.66	7.15													
Center Point Average	4.062	0.29799	-1.733096	-0.488													
Curvature	4.23																

NO ₃ Removed																	
Sum +	10.85	9.61	5.33	10.24													
Sum -	10.85	9.61	5.33	10.24													
Overall Sum	10.85	9.61	5.33	10.24													
Difference	10.85	9.61	5.33	10.24													
Effect	10.85	9.61	5.33	10.24													
Center Point Average	5.00	-0.157297	2.296186	0.157													
Curvature	-0.04																

Product																	
Sum +	22.86	11.31	4.98	11.45													
Sum -	22.86	11.31	4.98	11.45													
Overall Sum	22.86	11.31	4.98	11.45													
Difference	22.86	11.31	4.98	11.45													
Effect	22.86	11.31	4.98	11.45													
Center Point Average	4.73	-0.059412	3.273912	0.011													
Curvature	0.98																

NO ₂																	
Sum +	129.19	39.73	50.16	65.26													
Sum -	129.19	39.73	50.16	65.26													
Overall Sum	129.19	39.73	50.16	65.26													
Difference	129.19	39.73	50.16	65.26													
Effect	129.19	39.73	50.16	65.26													
Center Point Average	12.97	-12.43472	7.219643	0.345													
Curvature	19.32																

NH ₃																	
Sum +	270.81	160.27	140.94	134.72													
Sum -	270.81	160.27	140.94	134.72													
Overall Sum	270.81	160.27	140.94	134.72													
Difference	270.81	160.27	140.94	134.72													
Effect	270.81	160.27	140.94	134.72													
Center Point Average	87.03	12.45472	7.219643	-0.345													
Curvature	-19.32																

DOE Numbers - Graphite Fell Constant Current

Run	Treatments		NO3Removed		CE Total		CE NH3		Product		NO2		NH3	
	IPH	INOC	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	-4.35%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%
2	7	500	2.67%	2.67%	0.17%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	100.00%	100.00%
3	9	100	0.77%	2.46%	0.00%	0.00%	0.89%	0.00%	1.17%	0.19%	0.00%	0.00%	100.00%	0.00%
4	9	500	2.67%	2.67%	1.78%	0.00%	0.11%	0.13%	0.00%	0.17%	16.13%	18.92%	83.87%	0.00%
5	8	300	-0.10%	2.72%	1.33%	0.00%	0.00%	0.48%	0.00%	3.31%	0.00%	0.00%	100.00%	100.00%

Factors

Initial pH

Concentration

C

7.0 100

9.0 500

DOEN - Design of Experiments for Electrochemical Reduction of Nitrate											
Trial	Mean	P	C	PC	CE Total	CE NH3	NO3Removed	Product	NO2	NH3	
1	+	-	-	-	0.00	0.00	-2.90	0.00	0.00	0.00	
2	+	+	+	+	0.46	0.46	2.08	0.49	0.00	100.00	
3	+	+	+	+	1.20	1.20	1.08	1.08	0.00	66.67	
4	+	+	+	+	0.06	0.06	2.37	0.10	11.68	54.98	
5	0	0	0	0	1.21	1.21	1.32	1.15	0.00	66.67	
CE Total	1.74	1.28	0.54	0.08							
Sum +	1.74	1.28	0.54	0.08							
Sum -	-	-	-	-							
Overall Sum	1.74	1.28	0.54	0.08							
Difference	1.74	1.28	0.54	0.08							
Effect	0.434	0.2063	-0.166042	-0.394							
Centre Point Average	1.21										
Curvature	-0.77										

NO3 Removed											
Sum +	2.63	3.45	4.45	0.53							
Sum -	-	-	-	-							
Overall Sum	2.63	3.45	4.45	0.53							
Difference	2.63	3.45	4.45	0.53							
Effect	0.657	1.067606	1.567528	-0.920							
Centre Point Average	1.32										
Curvature	-0.65										

Product											
Sum +	1.67	1.16	0.59	0.10							
Sum -	-	-	-	-							
Overall Sum	1.67	1.16	0.59	0.10							
Difference	1.67	1.16	0.59	0.10							
Effect	0.416	0.173558	-0.122123	-0.365							
Centre Point Average	1.15										
Curvature	-0.75										

NO2											
Sum +	11.68	11.68	11.68	11.68							
Sum -	-	-	-	-							
Overall Sum	11.68	11.68	11.68	11.68							
Difference	11.68	11.68	11.68	11.68							
Effect	2.921	2.920663	2.920663	2.921							
Centre Point Average	0.00										
Curvature	2.92										

NH3											
Sum +	221.65	121.65	134.98	54.98							
Sum -	-	-	-	-							
Overall Sum	221.65	121.65	134.98	54.98							
Difference	221.65	121.65	134.98	54.98							
Effect	55.413	5.412671	22.07934	-27.921							
Centre Point Average	86.67										
Curvature	-11.25										

Experiment Name and Purpose	
Experiment Name:	DOEN-CL7/DOEN-CL7R/DOEN-C71R2
Date:	10/19/2004 -- 10/19/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed in Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	15:30	0.75	7.22	23	1032	516	2.0
Final	18:06	0.75	8.11	23	1034	517	2.0
Repeat							
Initial	15:40	0.75	7.22	23	1032	516	2.0
Final	18:16	0.75	8.33	23	1044	522	2.0
Repeat							
Initial	14:30	0.75	7.36	20	1088	545	2.1
Final	17:06	0.75	8.58	20	1117	559	2.1

Raw Data							
Elpsd. Time hrs:min	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	11.00	0.1000	23.00	0.00	0.00	44.00	23.00
02:36	10.00	0.1000	22.00	0.84	0.59	45.00	23.00
Repeat							
00:00	11.00	0.1000	23.00	0.00	0.00	44.00	23.00
02:36	10.00	0.1000	21.00	1.30	1.40	44.00	23.00
Repeat							
00:00	11.16	0.1008	31.00	0.00	0.00	43.00	24.00
02:36	11.16	0.1129	28.00	1.50	1.50	45.00	24.00

Mass Balance on Nitrogen			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	16.50 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.63 mg
NH ₃	0.00 mg	NH ₃	0.44 mg
Total Nitrogen Sources Initial	17.25 mg	Total Nitrogen Sources Final	17.57 mg
Mass Balance Final-Initial:	0.32 mg		
Repeat			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	15.75 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.98 mg
NH ₃	0.00 mg	NH ₃	1.05 mg
Total Nitrogen Sources Initial	17.25 mg	Total Nitrogen Sources Final	17.78 mg
Mass Balance Final-Initial:	0.53 mg		
Repeat 2			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	23.25 mg	NO ₃ ⁻	21.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	1.13 mg
NH ₃	0.00 mg	NH ₃	1.13 mg
Total Nitrogen Sources Initial	23.25 mg	Total Nitrogen Sources Final	23.25 mg
Mass Balance Final-Initial:	0.00 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	73.07 mg
% NO ₃ Removed:	4.35%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat</i>	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	69.75 mg
% NO ₃ Removed:	8.70%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	102.96 mg
Amount of NO ₃ Final:	92.99 mg
% NO ₃ Removed:	9.68%
Min. % NO ₃ Removal Req.:	56.99%
Average %NO ₃ Removal:	7.57%
Minimum % NO ₃ Removal:	47.02%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	2.61 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.077 mmol
% Product NO ₂ and NH ₃ :	3.45% by wt.
% Product NO ₂ and NH ₃ :	6.10% by mol.
% Split of NO ₂ :	79.37% by wt.
% Split of NH ₃ :	20.63% by wt.
% Split of NO ₂ :	58.74% by mol.
% Split of NH ₃ :	41.26% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	4.48 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.145 mmol
% Product NO ₂ and NH ₃ :	6.04% by wt.
% Product NO ₂ and NH ₃ :	11.39% by mol.
% Split of NO ₂ :	71.51% by wt.
% Split of NH ₃ :	28.49% by wt.
% Split of NO ₂ :	48.15% by mol.
% Split of NH ₃ :	51.85% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	5.07 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.161 mmol
% Product NO ₂ and NH ₃ :	5.17% by wt.
% Product NO ₂ and NH ₃ :	9.68% by mol.
% Split of NO ₂ :	72.99% by wt.
% Split of NH ₃ :	27.01% by wt.
% Split of NO ₂ :	50.00% by mol.
% Split of NH ₃ :	50.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	4.88% by wt.
% Product NO ₂ and NH ₃ :	9.06% by mol.
% Split of NO ₂ :	52.30% by mol.
% Split of NH ₃ :	47.70% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.1 A
Total Amt. of Coloumbs Passed:	933.12 C
Total Amt. of NO ₃ Decomposed:	3.32 mg
Total Moles of NO ₃ Decomposed:	5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	4.49679E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.158E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	8.677 C
Coulombs to NH ₃ :	24.380 C
Coulombic Efficiency to NH ₃ :	2.61% C
Coulombic Efficiency:	3.54%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1 A
Total Amt. of Coloumbs Passed:	933.12 C
Amt. of NO ₃ Decomposed:	6.64 mg
Moles of NO ₃ Decomposed:	1.071E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	6.95931E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	7.495E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	13.429 C
Coulombs to NH ₃ :	57.850 C
Coulombic Efficiency to NH ₃ :	6.20%
Coulombic Efficiency:	7.64%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.10685 A
Total Amt. of Coloumbs Passed:	997.04 C
Amt. of NO ₃ Decomposed:	9.96 mg
Moles of NO ₃ Decomposed:	1.606E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	8.02998E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	8.030E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	15.495 C
Coulombs to NH ₃ :	61.982 C
Coulombic Efficiency to NH ₃ :	6.22%
Coulombic Efficiency:	7.77%
Average Coulombic Efficiency to NH ₃ :	5.01%
Standard Deviation:	2.08%
Average Coulombic Efficiency Total:	6.32%
Standard Deviation:	2.40%

Experiment Name and Purpose	
Experiment Name:	DOEN-GFL7/DOEN-GFL7R/DOEN-GF71R2
Date:	10/19/2004 -- 10/19/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions								
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl%	
Initial	15:30	0.75	7.22	23	1032	516	2.0	
Final	18:06	0.75	7.57	24	1032	515	2.0	
Repeat								
Initial	15:30	0.75	7.22	23	1032	516	2.0	
Final	18:06	0.75	7.75	24	1038	519	2.0	
Repeat								
Initial	14:24	0.75	7.36	20	1088	545	2.1	
Final	17:00	0.75	8.58	20	1102	550	2.1	

Raw Data								
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm	
00:00	10.00	0.1000	23.00	0.00	0.00	44.00	23.00	
02:36	9.00	0.1000	24.00	0.00	0.00	45.00	23.00	
Repeat								
00:00	10.20	0.1000	23.00	0.00	0.00	44.00	23.00	
02:36	9.20	0.1000	24.00	0.00	0.00	45.00	23.00	
Repeat								
00:00	11.98	0.1004	31.00	0.00	0.00	43.00	24.00	
02:36	10.81	0.1022	31.00	0.00	0.00	45.00	23.00	

Mass Balance on Nitrogen			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrate Sources Initial	17.25 mg	Total Nitrate Sources Final	18.00 mg
Mass Balance Final-Initial:	0.75 mg		
Repeat			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrate Sources Initial	17.25 mg	Total Nitrate Sources Final	18.00 mg
Mass Balance Final-Initial:	0.75 mg		
Repeat 2			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	23.25 mg	NO ₃ ⁻	23.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrate Sources initial	23.25 mg	Total Nitrate Sources Final	23.25 mg
Mass Balance Final-Initial:	0.00 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	-4.35%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat</i>	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	-4.35%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	102.96 mg
Amount of NO ₃ Final:	102.96 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	56.99%
Average %NO ₃ Removal:	-2.90%
Minimum % NO3 Removal:	47.02%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.00 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.00 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.00 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.1 A
Total Amt. of Coloumbs Passed:	933.12 C
Total Amt. of NO ₃ Decomposed:	-3.32 mg
Total Moles of NO ₃ Decomposed:	-5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	0.00 C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1 A
Total Amt. of Coloumbs Passed:	933.12 C
Amt. of NO ₃ Decomposed:	-3.32 mg
Moles of NO ₃ Decomposed:	-5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	0.00 C
Coulombic Efficiency to NH ₃ :	0.00%
Coulombic Efficiency:	0.00%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1013 A
Total Amt. of Coloumbs Passed:	945.25 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	0.00 C
Coulombic Efficiency to NH ₃ :	0.00%
Coulombic Efficiency:	0.00%
Average Coulombic Efficiency to NH ₃ :	0.00%
Standard Deviation:	0.00%
Average Coulombic Efficiency Total:	0.00%
Standard Deviation:	0.00%

Experiment Name and Purpose	
Experiment Name:	DOEN-CL91/DOEN-CL91R/DOEN-C91R2
Date:	10/24/2004 -- 10/24/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper Plate		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	16:45	0.75	9.05	22	1297	649	2.5
Final	19:21	0.75	9.01	23	1314	658	2.5
Repeat							
Initial	16:35	0.75	9.05	22	1297	649	2.5
Final	19:11	0.75	8.9	23	1310	654	2.5
Repeat							
Initial	14:40	0.75	9.06	20	1263	632	2.4
Final	17:16	0.75	9.33	20	1268	634	2.4

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cr ppm
00:00	8.85	0.1008	24.00	0.00	0.00	47.00	40.00
02:36	8.52	0.1015	22.00	0.97	1.40	48.00	39.00
Repeat							
00:00	10.10	0.1010	24.00	0.00	0.00	47.00	40.00
02:36	8.60	0.1015	22.00	1.10	1.70	47.00	39.00
Repeat							
00:00	10.29	0.1005	24.00	0.00	0.00	43.00	24.00
02:36	9.32	0.1002	23.00	0.00	1.40	45.00	24.00

Mass Balance on Nitrogen			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	16.50 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.73 mg
NH ₃	0.00 mg	NH ₃	1.05 mg
Total Nitrogen Sources Initial	18.00 mg	Total Nitrogen Sources Final	18.28 mg
Mass Balance Final-Initial:		0.28 mg	
Repeat			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	16.50 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.83 mg
NH ₃	0.00 mg	NH ₃	1.28 mg
Total Nitrogen Sources Initial	18.00 mg	Total Nitrogen Sources Final	18.60 mg
Mass Balance Final-Initial:		0.60 mg	
Repeat 2			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.05 mg
Total Nitrogen Sources Initial	18.00 mg	Total Nitrogen Sources Final	18.30 mg
Mass Balance Final-Initial:		0.30 mg	

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	73.07 mg
% NO ₃ Removed:	8.33%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	73.07 mg
% NO ₃ Removed:	8.33%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	76.39 mg
% NO ₃ Removed:	4.17%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	6.94%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	3.67 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.127 mmol
% Product NO ₂ and NH ₃ :	4.78% by wt.
% Product NO ₂ and NH ₃ :	9.73% by mol.
% Split of NO ₂ :	65.19% by wt.
% Split of NH ₃ :	34.81% by wt.
% Split of NO ₂ :	40.93% by mol.
% Split of NH ₃ :	59.07% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	4.26 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.150 mmol
% Product NO ₂ and NH ₃ :	5.51% by wt.
% Product NO ₂ and NH ₃ :	11.29% by mol.
% Split of NO ₂ :	63.62% by wt.
% Split of NH ₃ :	36.38% by wt.
% Split of NO ₂ :	39.29% by mol.
% Split of NH ₃ :	60.71% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.28 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.075 mmol
% Product NO ₂ and NH ₃ :	1.64% by wt.
% Product NO ₂ and NH ₃ :	5.74% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	3.98% by wt.
% Product NO ₂ and NH ₃ :	8.92% by mol.
% Split of NO ₂ :	26.74% by mol.
% Split of NH ₃ :	73.26% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.10115 A
Total Amt. of Coloumbs Passed:	943.85 C
Total Amt. of NO ₃ Decomposed:	6.64 mg
Total Moles of NO ₃ Decomposed:	1.071E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	5.19272E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	7.495E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	10.020 C
Coulombs to NH ₃ :	57.850 C
Coulombic Efficiency to NH ₃ :	6.13% C
Coulombic Efficiency:	7.19%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.10125 A
Total Amt. of Coloumbs Passed:	944.79 C
Amt. of NO ₃ Decomposed:	6.64 mg
Moles of NO ₃ Decomposed:	1.071E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	5.88865E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	9.101E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	11.363 C
Coulombs to NH ₃ :	70.246 C
Coulombic Efficiency to NH ₃ :	7.44%
Coulombic Efficiency:	8.64%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.10035 A
Total Amt. of Coloumbs Passed:	936.39 C
Amt. of NO ₃ Decomposed:	3.32 mg
Moles of NO ₃ Decomposed:	5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	7.495E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	57.850 C
Coulombic Efficiency to NH ₃ :	6.18%
Coulombic Efficiency:	6.18%
Average Coulombic Efficiency to NH ₃ :	6.58%
Standard Deviation:	0.74%
Average Coulombic Efficiency Total:	7.34%
Standard Deviation:	1.24%

Experiment Name and Purpose	
Experiment Name:	DOEN-GF91/DOEN-GF91R/DOEN-GF91R2
Date:	10/24/2004 -- 10/24/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds 2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	16:50	0.75	9.05	22	1314	649	2.5
Final	19:26	0.75	8.74	23	1290	645	2.5
<i>Repeat</i>							
Initial	16:55	0.75	9.05	22	1314	649	2.5
Final	19:31	0.75	8.78	23	1310	655	2.5
<i>Repeat</i>							
Initial	14:48	0.75	9.06	20	1263	632	2.4
Final	17:24	0.75	9.16	20	1238	619	2.4

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.55	0.1000	26.00	0.00	0.00	47.00	40.00
02:36	7.08	0.1000	26.00	0.00	0.20	49.00	40.00
<i>Repeat</i>							
00:00	8.60	0.1006	26.00	0.00	0.00	47.00	40.00
02:36	8.63	0.1080	26.00	0.00	0.64	51.00	41.00
<i>Repeat</i>							
00:00	10.22	0.1123	31.00	0.00	0.00	43.00	24.00
02:36	9.35	0.1107	31.00	0.00	0.00	45.00	24.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	19.50 mg	NO ₃ ⁻	19.35 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.15 mg
Total Nitrogen Souces Initia	19.50 mg	Total Nitrogen Souces Final	19.50 mg
Mass Balance Final-Initial:		0.00 mg	

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	19.50 mg	NO ₃ ⁻	19.02 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.48 mg
Total Nitrogen Souces Initia	19.50 mg	Total Nitrogen Souces Final	19.50 mg
Mass Balance Final-Initial:		0.00 mg	

<i>Repeat 2</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	23.25 mg	NO ₃ ⁻	23.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Souces Initia	23.25 mg	Total Nitrogen Souces Final	23.25 mg
Mass Balance Final-Initial:		0.00 mg	

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	86.35 mg
Amount of NO ₃ Final:	85.69 mg
% NO ₃ Removed:	0.77%
Min. % NO ₃ Removal Req.:	48.72%
<i>Repeat</i>	
Amount of NO ₃ Initial:	86.35 mg
Amount of NO ₃ Final:	84.23 mg
% NO ₃ Removed:	2.46%
Min. % NO ₃ Removal Req.:	48.72%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	102.96 mg
Amount of NO ₃ Final:	102.96 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	56.99%
Average %NO ₃ Removal:	1.08%
Minimum % NO ₃ Removal:	51.48%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.18 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.011 mmol
% Product NO ₂ and NH ₃ :	0.21% by wt.
% Product NO ₂ and NH ₃ :	0.77% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.58 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.034 mmol
% Product NO ₂ and NH ₃ :	0.69% by wt.
% Product NO ₂ and NH ₃ :	2.46% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	- mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.30% by wt.
% Product NO ₂ and NH ₃ :	1.08% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.1 A
Total Amt. of Coloumbs Passed:	933.12 C
Total Amt. of NO ₃ Decomposed:	0.66 mg
Total Moles of NO ₃ Decomposed:	1.071E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.071E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	8.264 C
Coulombic Efficiency to NH ₃ :	0.89% C
Coulombic Efficiency:	0.89%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1043 A
Total Amt. of Coloumbs Passed:	973.25 C
Amt. of NO ₃ Decomposed:	2.13 mg
Moles of NO ₃ Decomposed:	3.426E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.426E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	26.446 C
Coulombic Efficiency to NH ₃ :	2.72%
Coulombic Efficiency:	2.72%
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1115 A
Total Amt. of Coloumbs Passed:	1040.43 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00%
Coulombic Efficiency:	0.00%
Average Coulombic Efficiency to NH ₃ :	1.20%
Standard Deviation:	1.39%
Average Coulombic Efficiency Total:	1.20%
Standard Deviation:	1.39%

Experiment Name and Purpose	
Experiment Name:	DOEN-C83/DOEN-C83R/DOEN-C38R2
Date:	10/20/2004 -- 10/20/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 8 - Constant Current - 300ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper Plate		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	467	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	300 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	67.75 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	27994 seconds
	7.8 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	14:35	0.75	8.22	23	1580	790	3.0
Final	22:21	0.74	8.50	23	1605	802	3.1
Repeat							
Initial	15:20	0.75	8.22	23	1580	790	3.0
Final	23:06	0.74	8.07	23	1606	802	3.1
Repeat							
Initial	02:30	0.75	8.22	20	1564	781	3.0
Final	10:16	0.74	8.82	20	1597	799	3.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	8.86	0.1005	68.00	0.00	0.00	41.00	34.00
07:46	8.37	0.1017	67.00	0.71	1.90	44.00	34.00
Repeat							
00:00	8.85	0.1016	72.00	0.00	0.00	41.00	34.00
07:46	8.46	0.1000	67.00	0.73	5.50	58.00	35.00
Repeat							
00:00	9.6	0.1009	73.00	0.00	0.00	42.00	27.00
07:46	9.88	0.1022	71.00	0.00	1.40	23.00	15.00

Mass Balance on Nitrogen			
Nitrogen Sources Initial (as mg Nitrogen)		Nitrogen Sources Final (as mg Nitrogen)	
NO ₃ ⁻	51.00 mg	NO ₃ ⁻	49.58 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.53 mg
NH ₃	0.00 mg	NH ₃	1.41 mg
Total Nitrogen Sources Initial	51.00 mg	Total Nitrogen Sources Final	51.51 mg
Mass Balance Final-Initial:	0.51 mg		
Repeat			
Nitrogen Sources Initial (as mg Nitrogen)		Nitrogen Sources Final (as mg Nitrogen)	
NO ₃ ⁻	54.00 mg	NO ₃ ⁻	49.58 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.54 mg
NH ₃	0.00 mg	NH ₃	4.07 mg
Total Nitrogen Sources Initial	54.00 mg	Total Nitrogen Sources Final	54.19 mg
Mass Balance Final-Initial:	0.19 mg		
Repeat 2			
Nitrogen Sources Initial (as mg Nitrogen)		Nitrogen Sources Final (as mg Nitrogen)	
NO ₃ ⁻	54.75 mg	NO ₃ ⁻	52.54 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.04 mg
Total Nitrogen Sources Initial	54.75 mg	Total Nitrogen Sources Final	53.58 mg
Mass Balance Final-Initial:	-1.17 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	225.84 mg
Amount of NO ₃ Final:	219.55 mg
% NO ₃ Removed:	2.78%
Min. % NO ₃ Removal Req.:	80.39%
<i>Repeat</i>	
Amount of NO ₃ Initial:	239.13 mg
Amount of NO ₃ Final:	219.55 mg
% NO ₃ Removed:	8.19%
Min. % NO ₃ Removal Req.:	81.48%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	242.45 mg
Amount of NO ₃ Final:	232.66 mg
% NO ₃ Removed:	4.04%
Min. % NO ₃ Removal Req.:	81.74%
Average %NO ₃ Removal:	5.00%
Minimum % NO ₃ Removal:	81.20%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	3.44 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.138 mmol
% Product NO ₂ and NH ₃ :	1.54% by wt.
% Product NO ₂ and NH ₃ :	3.75% by mol.
% Split of NO ₂ :	50.25% by wt.
% Split of NH ₃ :	49.75% by wt.
% Split of NO ₂ :	27.20% by mol.
% Split of NH ₃ :	72.80% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	6.73 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.329 mmol
% Product NO ₂ and NH ₃ :	2.97% by wt.
% Product NO ₂ and NH ₃ :	8.51% by mol.
% Split of NO ₂ :	26.40% by wt.
% Split of NH ₃ :	73.60% by wt.
% Split of NO ₂ :	11.72% by mol.
% Split of NH ₃ :	88.28% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.26 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.074 mmol
% Product NO ₂ and NH ₃ :	0.54% by wt.
% Product NO ₂ and NH ₃ :	1.93% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.68% by wt.
% Product NO ₂ and NH ₃ :	4.73% by mol.
% Split of NO ₂ :	12.97% by mol.
% Split of NH ₃ :	87.03% by mol.

Coulombic Efficiency	
Time for Experiment:	27994 s
Current Passed:	0.1011 A
Total Amt. of Coloumbs Passed:	2830.16 C
Total Amt. of NO ₃ Decomposed:	6.29 mg
Total Moles of NO ₃ Decomposed:	1.014E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	3.75018E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.004E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	7.237 C
Coulombs to NH ₃ :	77.463 C
Coulombic Efficiency to NH ₃ :	2.74% C
Coulombic Efficiency:	2.99%
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.1008 A
Total Amt. of Coloumbs Passed:	2821.77 C
Amt. of NO ₃ Decomposed:	19.57 mg
Moles of NO ₃ Decomposed:	3.155E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	3.85582E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.905E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	7.441 C
Coulombs to NH ₃ :	224.236 C
Coulombic Efficiency to NH ₃ :	7.95%
Coulombic Efficiency:	8.21%
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.10155 A
Total Amt. of Coloumbs Passed:	2842.76 C
Amt. of NO ₃ Decomposed:	9.79 mg
Moles of NO ₃ Decomposed:	1.577E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	7.395E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	57.078 C
Coulombic Efficiency to NH ₃ :	2.01%
Coulombic Efficiency:	2.01%
Average Coulombic Efficiency to NH ₃ :	4.23%
Standard Deviation:	3.24%
Average Coulombic Efficiency Total:	4.40%
Standard Deviation:	3.33%

Experiment Name and Purpose	
Experiment Name:	DOEN-GF83/DOEN-GF83R/DOEN-GF38R2
Date:	10/20/2004 -- 10/20/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 8 - Constant Current - 300ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	467	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	300 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	67.75 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	27994 seconds
	7.8 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	14:55	0.75	8.22	23	1580	790	3.0
Final	22:41	0.74	7.84	23	1590	795	3.1
<i>Repeat</i>							
Initial	15:10	0.75	8.22	23	1580	790	3.0
Final	22:56	0.74	8.05	23	1625	812	3.1
<i>Repeat</i>							
Initial	02:35	0.75	8.22	20	1564	781	3.0
Final	10:21	0.74	8.03	20	1575	787	3.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	8.70	0.1005	69.00	0.00	0.00	41.00	34.00
07:46	7.44	0.1019	70.00	0.00	0.00	45.00	36.00
<i>Repeat</i>							
00:00	8.52	0.1005	71.00	0.00	0.00	41.00	34.00
07:46	8.35	0.1006	70.00	0.00	2.40	52.00	36.00
<i>Repeat</i>							
00:00	9.75	0.102	73.00	0.00	0.00	42.00	27.00
07:46	9.39	0.1034	73.00	0.00	0.10	44.00	27.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	51.75 mg	NO ₃ ⁻	51.80 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Souces Initial	51.75 mg	Total Nitrogen Souces Final	51.80 mg
Mass Balance Final-Initial:	0.05 mg		
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	53.25 mg	NO ₃ ⁻	51.80 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.78 mg
Total Nitrogen Souces Initial	53.25 mg	Total Nitrogen Souces Final	53.58 mg
Mass Balance Final-Initial:	0.33 mg		
<i>Repeat 2</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	54.75 mg	NO ₃ ⁻	54.02 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.07 mg
Total Nitrogen Souces Initial	54.75 mg	Total Nitrogen Souces Final	54.09 mg
Mass Balance Final-Initial:	-0.66 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	229.16 mg
Amount of NO ₃ Final:	229.38 mg
% NO ₃ Removed:	-0.10%
Min. % NO ₃ Removal Req.:	80.68%
<i>Repeat</i>	
Amount of NO ₃ Initial:	235.81 mg
Amount of NO ₃ Final:	229.38 mg
% NO ₃ Removed:	2.72%
Min. % NO ₃ Removal Req.:	81.22%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	242.45 mg
Amount of NO ₃ Final:	239.21 mg
% NO ₃ Removed:	1.33%
Min. % NO ₃ Removal Req.:	81.74%
Average %NO ₃ Removal:	1.32%
Minimum % NO3 Removal:	81.21%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	- mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	2.16 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.127 mmol
% Product NO ₂ and NH ₃ :	0.93% by wt.
% Product NO ₂ and NH ₃ :	3.31% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.09 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.005 mmol
% Product NO ₂ and NH ₃ :	0.04% by wt.
% Product NO ₂ and NH ₃ :	0.14% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.32% by wt.
% Product NO ₂ and NH ₃ :	1.15% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	27994 s
Current Passed:	0.1012 A
Total Amt. of Coloumbs Passed:	2832.96 C
Total Amt. of NO ₃ Decomposed:	-0.22 mg
Total Moles of NO ₃ Decomposed:	-3.569E-06 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.10055 A
Total Amt. of Coloumbs Passed:	2814.77 C
Amt. of NO ₃ Decomposed:	6.42 mg
Moles of NO ₃ Decomposed:	1.035E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.268E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	97.849 C
Coulombic Efficiency to NH ₃ :	3.48%
Coulombic Efficiency:	3.48%
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.1027 A
Total Amt. of Coloumbs Passed:	2874.95 C
Amt. of NO ₃ Decomposed:	3.23 mg
Moles of NO ₃ Decomposed:	5.211E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.282E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	4.077 C
Coulombic Efficiency to NH ₃ :	0.14%
Coulombic Efficiency:	0.14%
Average Coulombic Efficiency to NH ₃ :	1.21%
Standard Deviation:	1.97%
Average Coulombic Efficiency Total:	1.21%
Standard Deviation:	1.97%

Experiment Name and Purpose	
Experiment Name:	DOEN-C75/DOEN-C75R/DOEN-C57R2
Date:	10/21/2004 -- 10/21/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper Plate		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	10/21/2004 00:37	0.75	7.13	22	1963	981	3.8
Final	10/21/2004 13:34	0.74	8.62	22	1998	998	3.9
Repeat							
Initial	10/21/2004 01:12	0.75	7.13	22	1963	981	3.8
Final	10/21/2004 14:09	0.73	8.64	22	1988	994	3.9
Repeat							
Initial	10/25/2004 21:28	0.75	7.27	20	2035	1020	4.0
Final	10/26/2004 10:25	0.72	9.00	20	2125	1063	4.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.82	0.1002	110.00	0.00	0.00	43.00	32.00
12:57	8.18	0.1018	110.00	0.40	0.00	45.00	33.00
Repeat							
00:00	8.07	0.1000	110.00	0.00	0.00	43.00	32.00
12:57	8.42	0.0875	110.00	0.26	2.00	44.00	33.00
Repeat							
00:00	7.77	0.1005	110.00	0.00	0.00	47.00	43.00
12:57	7.81	0.0750	110.00	0.00	5.90	50.00	47.00

Mass Balance on Nitrogen			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	81.40 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.30 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Sources Initial	82.50 mg	Total Nitrogen Sources Final	81.70 mg
Mass Balance Final-Initial:	-0.80 mg		
Repeat			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.19 mg
NH ₃	0.00 mg	NH ₃	1.46 mg
Total Nitrogen Sources Initial	82.50 mg	Total Nitrogen Sources Final	81.95 mg
Mass Balance Final-Initial:	-0.55 mg		
Repeat 2			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	79.20 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	4.25 mg
Total Nitrogen Sources Initial	82.50 mg	Total Nitrogen Sources Final	83.45 mg
Mass Balance Final-Initial:	0.95 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	360.46 mg
% NO ₃ Removed:	1.33%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	350.72 mg
% NO ₃ Removed:	4.00%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.97 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.021 mmol
% Product NO ₂ and NH ₃ :	0.27% by wt.
% Product NO ₂ and NH ₃ :	0.36% by mol.
% Split of NO ₂ :	100.00% by wt.
% Split of NH ₃ :	0.00% by wt.
% Split of NO ₂ :	100.00% by mol.
% Split of NH ₃ :	0.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	2.40 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.118 mmol
% Product NO ₂ and NH ₃ :	0.67% by wt.
% Product NO ₂ and NH ₃ :	2.01% by mol.
% Split of NO ₂ :	26.00% by wt.
% Split of NH ₃ :	74.00% by wt.
% Split of NO ₂ :	11.50% by mol.
% Split of NH ₃ :	88.50% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	5.17 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.303 mmol
% Product NO ₂ and NH ₃ :	1.45% by wt.
% Product NO ₂ and NH ₃ :	5.09% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.80% by wt.
% Product NO ₂ and NH ₃ :	2.49% by mol.
% Split of NO ₂ :	37.17% by mol.
% Split of NH ₃ :	62.83% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.101 A
Total Amt. of Coloumbs Passed:	4712.28 C
Total Amt. of NO ₃ Decomposed:	4.87 mg
Total Moles of NO ₃ Decomposed:	7.852E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	2.11278E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	4.077 C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.09%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.09375 A
Total Amt. of Coloumbs Passed:	4374.02 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.35475E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.042E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	2.614 C
Coulombs to NH ₃ :	80.439 C
Coulombic Efficiency to NH ₃ :	1.84%
Coulombic Efficiency:	1.90%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.08775 A
Total Amt. of Coloumbs Passed:	4094.08 C
Amt. of NO ₃ Decomposed:	14.61 mg
Moles of NO ₃ Decomposed:	2.355E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.032E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	234.043 C
Coulombic Efficiency to NH ₃ :	5.72%
Coulombic Efficiency:	5.72%
Average Coulombic Efficiency to NH ₃ :	2.52%
Standard Deviation:	2.92%
Average Coulombic Efficiency Total:	2.57%
Standard Deviation:	2.87%

Experiment Name and Purpose	
Experiment Name:	DOEN-GF75/DOEN-GF75R/DOEN-GF57R2
Date:	10/21/2004 -- 10/21/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	6000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	6000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	10/21/2004 01:00	0.75	7.13	22	1963	981	3.8
Final	10/21/2004 13:57	0.73	8.03	22	2005	1002	3.9
<i>Repeat</i>							
Initial	10/21/2004 00:37	0.75	7.13	22	1963	981	3.8
Final	10/21/2004 13:34	0.73	8.00	23	1936	979	3.8
<i>Repeat</i>							
Initial	10/25/2004 21:20	0.75	7.27	20	2035	1020	4.0
Final	10/26/2004 10:17	0.73	8.00	20	2069	1035	4.0

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.87	0.1005	110.00	0.00	0.00	43.00	32.00
12:57	6.46	0.1023	110.00	0.00	1.30	48.00	33.00
<i>Repeat</i>							
00:00	7.42	0.1007	110.00	0.00	0.00	43.00	32.00
12:57	7.42	0.1123	110.00	0.00	0.21	45.00	34.00
<i>Repeat</i>							
00:00	7.23	0.1006	110.00	0.00	0.00	47.00	43.00
12:57	6.49	0.1018	112.00	0.00	0.11	49.00	44.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.95 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	81.25 mg
Mass Balance Final-Initial:	-1.25 mg		
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.15 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	80.45 mg
Mass Balance Final-Initial:	-2.05 mg		
<i>Repeat 2</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	81.76 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.08 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	81.84 mg
Mass Balance Final-Initial:	-0.66 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	362.05 mg
% NO ₃ Removed:	0.90%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	2.08%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	1.15 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.068 mmol
% Product NO ₂ and NH ₃ :	0.32% by wt.
% Product NO ₂ and NH ₃ :	1.17% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.19 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.011 mmol
% Product NO ₂ and NH ₃ :	0.05% by wt.
% Product NO ₂ and NH ₃ :	0.19% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.10 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.006 mmol
% Product NO ₂ and NH ₃ :	0.03% by wt.
% Product NO ₂ and NH ₃ :	0.10% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.13% by wt.
% Product NO ₂ and NH ₃ :	0.49% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.1014 A
Total Amt. of Coloumbs Passed:	4730.94 C
Total Amt. of NO ₃ Decomposed:	9.74 mg
Total Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	6.774E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	52.285 C
Coulombic Efficiency to NH ₃ :	1.11% C
Coulombic Efficiency:	1.11%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.1065 A
Total Amt. of Coloumbs Passed:	4968.88 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.094E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	8.446 C
Coulombic Efficiency to NH ₃ :	0.17%
Coulombic Efficiency:	0.17%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.1012 A
Total Amt. of Coloumbs Passed:	4721.61 C
Amt. of NO ₃ Decomposed:	3.28 mg
Moles of NO ₃ Decomposed:	5.282E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.732E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	4.424 C
Coulombic Efficiency to NH ₃ :	0.09%
Coulombic Efficiency:	0.09%
Average Coulombic Efficiency to NH ₃ :	0.46%
Standard Deviation:	0.56%
Average Coulombic Efficiency Total:	0.46%
Standard Deviation:	0.56%

Experiment Name and Purpose	
Experiment Name:	DOEN-C95/DOEN-C95R/DOEN-C59R2
Date:	10/24/2004 -- 10/24/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper Plate		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	10/24/2004 21:30	0.75	9.03	22	2097	1047	4.1
Final	10/25/2004 10:27	0.73	8.73	22	2166	1083	4.2
Repeat							
Initial	10/24/2004 21:55	0.75	9.03	22	2097	1047	4.1
Final	10/25/2004 10:52	0.73	8.61	22	2154	1076	4.2
Repeat							
Initial	10/25/2004 21:38	0.75	9.00	20	2176	1088	4.2
Final	10/26/2004 10:35	0.73	9.24	20	2217	1109	4.3

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.14	0.1000	110.00	0.02	0.00	49.00	42.00
12:57	8.46	0.1012	110.00	0.38	2.10	49.00	42.00
Repeat							
00:00	8.61	0.1008	110.00	0.02	0.00	49.00	42.00
12:57	8.61	0.0800	110.00	0.31	2.60	49.00	42.00
Repeat							
00:00	6.75	0.1003	110.00	0.00	0.00	47.00	43.00
12:57	7.68	0.1011	100.00	0.00	7.40	53.00	45.00

Mass Balance on Nitrogen			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.28 mg
NH ₃	0.00 mg	NH ₃	1.53 mg
Total Nitrogen Sources Initial	82.52 mg	Total Nitrogen Sources Final	82.11 mg
Mass Balance Final-Initial:	-0.41 mg		
Repeat			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.23 mg
NH ₃	0.00 mg	NH ₃	1.90 mg
Total Nitrogen Sources Initial	82.52 mg	Total Nitrogen Sources Final	82.42 mg
Mass Balance Final-Initial:	-0.09 mg		
Repeat 2			
<u>Nitrogen Sources Initial (as mg Nitrogen)</u>		<u>Nitrogen Sources Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	73.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	5.40 mg
Total Nitrogen Sources Initial	82.50 mg	Total Nitrogen Sources Final	78.40 mg
Mass Balance Final-Initial:	-4.10 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	323.26 mg
% NO ₃ Removed:	11.52%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	2.78 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.129 mmol
% Product NO ₂ and NH ₃ :	0.77% by wt.
% Product NO ₂ and NH ₃ :	2.20% by mol.
% Split of NO ₂ :	32.84% by wt.
% Split of NH ₃ :	67.16% by wt.
% Split of NO ₂ :	15.32% by mol.
% Split of NH ₃ :	84.68% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	3.05 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.152 mmol
% Product NO ₂ and NH ₃ :	0.85% by wt.
% Product NO ₂ and NH ₃ :	2.58% by mol.
% Split of NO ₂ :	24.37% by wt.
% Split of NH ₃ :	75.63% by wt.
% Split of NO ₂ :	10.65% by mol.
% Split of NH ₃ :	89.35% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	6.57 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.386 mmol
% Product NO ₂ and NH ₃ :	1.99% by wt.
% Product NO ₂ and NH ₃ :	6.89% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.81% by wt.
% Product NO ₂ and NH ₃ :	2.39% by mol.
% Split of NO ₂ :	12.99% by mol.
% Split of NH ₃ :	87.01% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.1006 A
Total Amt. of Coloumbs Passed:	4693.61 C
Total Amt. of NO ₃ Decomposed:	9.74 mg
Total Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.85153E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.094E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	3.573 C
Coulombs to NH ₃ :	84.461 C
Coulombic Efficiency to NH ₃ :	1.80% C
Coulombic Efficiency:	1.88%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.0904 A
Total Amt. of Coloumbs Passed:	4217.72 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.4868E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.355E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	2.869 C
Coulombs to NH ₃ :	104.570 C
Coulombic Efficiency to NH ₃ :	2.48%
Coulombic Efficiency:	2.55%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.1007 A
Total Amt. of Coloumbs Passed:	4698.28 C
Amt. of NO ₃ Decomposed:	42.07 mg
Moles of NO ₃ Decomposed:	6.781E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.856E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	297.623 C
Coulombic Efficiency to NH ₃ :	6.33%
Coulombic Efficiency:	6.33%
Average Coulombic Efficiency to NH ₃ :	2.14%
Standard Deviation:	0.48%
Average Coulombic Efficiency Total:	2.21%
Standard Deviation:	0.47%

Experiment Name and Purpose	
Experiment Name:	DOEN-GF91/DOEN-GF91R/DOEN-GF91R2
Date:	10/24/2004 -- 10/24/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	10/24/2004 21:36	0.75	9.03	22	2097	1047	4.1
Final	10/25/2004 10:33	0.73	7.98	22	2148	1072	4.1
<i>Repeat</i>							
Initial	10/24/2004 21:45	0.75	9.03	22	2097	1047	4.1
Final	10/25/2004 10:42	0.73	8.07	22	2119	1059	4.1
<i>Repeat</i>							
Initial	10/25/2004 21:50	0.75	9.00	20	2176	1088	4.2
Final	10/26/2004 10:47	0.73	8.26	20	2159	1080	4.2

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.48	0.1000	110.00	0.02	0.00	49.00	42.00
12:57	6.80	0.1009	110.00	0.03	0.13	49.00	43.00
<i>Repeat</i>							
00:00	7.82	0.1022	110.00	0.02	0.00	49.00	42.00
12:57	7.67	0.1003	110.00	0.04	0.15	50.00	42.00
<i>Repeat</i>							
00:00	7.80	0.1000	110.00	0.00	0.00	47.00	43.00
12:57	6.62	0.0806	111.00	0.00	0.00	99.00	86.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.02 mg
NH ₃	0.00 mg	NH ₃	0.09 mg
Total Nitrogen Souces Initial	82.52 mg	Total Nitrogen Souces Final	80.41 mg
Mass Balance Final-Initial:	-2.10 mg		
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.03 mg
NH ₃	0.00 mg	NH ₃	0.11 mg
Total Nitrogen Souces Initial	82.52 mg	Total Nitrogen Souces Final	80.44 mg
Mass Balance Final-Initial:	-2.08 mg		
<i>Repeat 2</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	81.03 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	81.03 mg
Mass Balance Final-Initial:	-1.47 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat 2</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	358.82 mg
% NO ₃ Removed:	1.78%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	1.78%
Minimum % NO ₃ Removal:	58.59%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.18 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.008 mmol
% Product NO ₂ and NH ₃ :	0.05% by wt.
% Product NO ₂ and NH ₃ :	0.14% by mol.
% Split of NO ₂ :	34.20% by wt.
% Split of NH ₃ :	65.80% by wt.
% Split of NO ₂ :	16.13% by mol.
% Split of NH ₃ :	83.87% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.22 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.010 mmol
% Product NO ₂ and NH ₃ :	0.06% by wt.
% Product NO ₂ and NH ₃ :	0.17% by mol.
% Split of NO ₂ :	38.67% by wt.
% Split of NH ₃ :	61.33% by wt.
% Split of NO ₂ :	18.92% by mol.
% Split of NH ₃ :	81.08% by mol.
<i>Repeat 2</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	- mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.04% by wt.
% Product NO ₂ and NH ₃ :	0.10% by mol.
% Split of NO ₂ :	17.52% by mol.
% Split of NH ₃ :	82.48% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.10045 A
Total Amt. of Coloumbs Passed:	4686.61 C
Total Amt. of NO ₃ Decomposed:	9.74 mg
Total Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.78444E-08 mol
Amt. Of NO ₃ Decomposed to NH ₃	6.774E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.003 C
Coulombs to NH ₃ :	5.229 C
Coulombic Efficiency to NH ₃ :	0.11% C
Coulombic Efficiency:	0.11%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.10125 A
Total Amt. of Coloumbs Passed:	4723.94 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	5.38901E-07 mol
Amt. Of NO ₃ Decomposed to NH ₃	7.816E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.104 C
Coulombs to NH ₃ :	6.033 C
Coulombic Efficiency to NH ₃ :	0.13%
Coulombic Efficiency:	0.13%
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.0903 A
Total Amt. of Coloumbs Passed:	4213.05 C
Amt. of NO ₃ Decomposed:	6.51 mg
Moles of NO ₃ Decomposed:	1.049E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	- C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00%
Coulombic Efficiency:	0.00%
Average Coulombic Efficiency to NH ₃ :	0.08%
Standard Deviation:	0.07%
Average Coulombic Efficiency Total:	0.08%
Standard Deviation:	0.07%

APPENDIX C

RAW DATA FOR EXPERIMENTS CARRIED OUT UNDER CONSTANT POTENTIAL CONDITIONS

Summary at Constant Potential							
Experiment	% Break-Down of Product			%NO ₃ Removed	Minimum % NO ₃ Removal	Coulombic Efficiency NH ₃	Coulombic Efficiency Total
	% NO ₂ and NH ₃ Formed	% NO ₂	% NH ₃				
DOEN-C71	4.13%	0.00%	100.00%	12.96%	50.62%	17.75%	17.75%
DOEN-C75	2.27%	12.16%	87.84%	2.67%	87.88%	1.91%	1.96%
DOEN-C91	6.59%	0.00%	100.00%	14.58%	44.44%	17.35%	17.35%
DOEN-C95	2.35%	0.00%	100.00%	2.67%	87.88%	2.00%	2.00%
DOEN-C83	4.09%	52.26%	47.74%	4.66%	80.68%	2.81%	3.20%
DOEN-GF83	0.53%	3.03%	96.97%	0.67%	80.68%	0.54%	0.55%
DOEN-GF71	2.20%	0.00%	100.00%	1.85%	50.62%	2.29%	2.29%
DOEN-GF75	0.16%	6.04%	93.96%	1.33%	88.89%	1.01%	1.00%
DOEN-GF91	2.91%	0.00%	100.00%	0.00%	44.44%	0.00%	0.00%
DOEN-GF95	0.35%	0.00%	100.00%	2.67%	87.88%	1.64%	1.64%

	Target Initial pH	Target Initial [NO ₃] (ppm)	Reaction Time (hrs:min)	Current (A)	Voltage (V)	pH	Vol. (L)	Temp. (°C)	TDS (ppm)	NaCl % (%)	Cond. (µS)	[NO ₃] (as N) [NO ₃] (ppm)	[NO ₂] (as N) [NO ₂] (ppm)	[NH ₃] (as N) [NH ₃] (ppm)	[SO ₄] (ppm)	[Cl] (ppm)
Initial	7	100	02:36	0.08390	10.00	7.63	0.75	20	545	2.10	1090	27.00 119.56	0.00 0.00	0.00 0.00	43	37
Std. Dev.				0.00198	0.00	0.00	0.00	0.00	0.00	0.00	0	0.00 0.00	0.00 0.00	0.00 0.00	1	1
Final				0.09025	10.00	8.61	0.75	20	553	2.10	1108	23.50 104.06	0.00 0.00	1.01 1.23		
Std. Dev.				0.00318	0.00	0.12	0.00	0.00	1.41	0.00	5	0.71 3.13	0.00 0.00	0.41 0.50		
Initial	9	100	02:36	0.08800	10.00	9.00	0.75	20	576	2.20	1155	24.00 106.28	0.00 0.00	0.20 0.24	42	36
Std. Dev.				0.00283	0.00	0.00	0.00	0.00	0.00	0.00	0	0.00 0.00	0.00 0.00	0.00 0.00	1	1
Final				0.09040	10.00	9.03	0.75	20	576	2.20	1153	20.50 90.78	0.00 0.00	1.45 1.76		
Std. Dev.				0.00000	0.00	0.00	0.00	0.00	2.83	0.00	6	0.71 3.13	0.00 0.00	0.21 0.26		
Initial	8	300	07:17	0.11330	10.00	8.91	0.75	20	785	3.00	1571	69.00 305.55	0.00 0.00	0.00 0.00	43	39
Std. Dev.				0.01230	0.00	0.00	0.00	0.00	0.00	0.00	0	0.00 0.00	0.00 0.00	0.00 0.00	1	1
Final				0.10815	10.00	9.03	0.748	20	800	3.10	1600	66.00 292.27	1.20 3.94	1.62 1.96		
Std. Dev.				0.00375	0.00	0.04	0.00	0.00	7.78	0.00	11	1.41 6.26	0.57 1.86	1.82 2.21		
Initial	7	500	12:25	0.13530	10.00	7.69	0.75	20	987	3.80	1973	110.00 487.11	0.02 0.07	0.00 0.00	44	42
Std. Dev.				0.01315	0.00	0.00	0.00	0.00	0.00	0.00	0	0.00 0.00	0.00 0.00	0.00 0.00	1	1
Final				0.11145	10.00	9.15	0.73	20	1004	3.85	2007	110.00 487.11	0.31 1.02	2.25 2.74		
Std. Dev.				0.00049	0.00	0.03	0.00	0.00	10.61	0.07	24	0.00 0.00	0.03 0.09	0.35 0.43		
Initial	9	500	11:47	0.14035	10.00	9.02	0.75	20	998	3.90	1996	110.00 487.11	0.00 0.00	0.00 0.00	43	39
Std. Dev.				0.01110	0.00	0.00	0.00	0	0.00	0.00	0	0.00 0.00	0.00 0.00	0.00 0.00	1	1
Final				0.11935	10.00	9.38	0.73	20	1029	4.00	2055	110.00 487.11	0.00 0.00	2.65 3.22		
Std. Dev.				0.00559	0.00	0.08	0.00	0.00	0.71	0.00	0	0.00 0.00	0.00 0.00	0.00 0.09		

DATA

Target Initial pH	Target Initial [NO ₃]	Avg. Soln. Conductivity μS	Coulombic Efficiency %
7	100	1099	17.75%
9	100	1154	17.35%
8	300	1586	3.20%
7	500	1990	1.96%
9	500	2026	2.00%

Coulombic Efficiency versus Average Solution Conductivity at Constant Potential

Run	IPH	Treatments	INOC	NO ₃ Removed		CE Total		CE NH ₄ ⁺		Products		NO ₂		NH ₄	
				Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100		14.81%	19.32%	15.58%	19.52%	2.81%	5.35%	0.00%		0.00%		100.00%	100.00%
2	7	500		2.67%	2.04%	1.95%	1.80%	2.64%	2.51%	12.66%	11.66%	0.00%		100.00%	100.00%
3	7	500		12.50%	19.64%	15.08%	16.14%	1.61%	1.61%	0.00%		0.00%		100.00%	100.00%
4	9	500		2.67%	2.04%	1.31%	2.10%	2.80%	2.31%	0.00%		0.00%		100.00%	100.00%
C1	8	300		2.90%	6.53%	0.51%	5.39%	5.11%	2.80%	82.90%	21.62%	1.70%	78.35%		

Run	In3C		NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	Treatments		Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
5	7	100	1.85%	2.00%	2.57%	2.00%	2.57%	2.43%	1.96%	0.00%	0.00%	100.00%	100.00%
6	7	500	1.33%	0.96%	1.05%	0.98%	1.05%	0.23%	0.09%	12.05%	0.00%	87.91%	100.00%
7	9	100	0.00%	0.00%	0.00%	0.00%	0.00%	4.39%	1.44%	0.00%	0.00%	100.00%	100.00%
8	9	500	2.67%	1.35%	1.71%	1.35%	1.71%	0.43%	0.26%	0.00%	0.00%	100.00%	100.00%
C2	300		0.67%	0.51%	0.58%	0.51%	0.58%	0.65%	0.36%	6.05%	0.00%	93.95%	100.00%

Factors	Initial pH P	Concentration C	Electrode WE
-	7.0	100	Copper
+	9.0	500	Graphite Felt

DOEN - Design of Experiments for Electrochemical Reduction of Nitrate

Trial	Mean	P	G	PC	WE	PWE	GWE	POWE	CE	CE	NO ₃ removed	Product	NO ₂	NH ₃
1	+	-	-	+	-	+	+	-	17.75	17.75	12.86	4.13	0.00	100.00
2	+	+	-	-	-	-	+	+	17.35	17.35	14.58	6.59	0.00	100.00
3	+	+	+	-	-	+	+	+	1.96	1.91	2.67	2.71	12.16	100.00
4	+	+	+	+	-	-	-	-	2.00	2.00	1.82	2.00	0.00	100.00
5	+	+	+	+	+	+	+	+	0.50	0.50	0.00	2.81	0.00	100.00
6	+	+	+	+	+	+	+	+	1.00	1.01	0.16	5.04	93.96	100.00
7	+	+	+	+	+	+	+	+	1.64	1.64	2.67	0.35	0.00	100.00

CE NH ₂	Sum *	20 990	6 554	23 680	4 927	21 298	37 746	23 719
Sum	43 943	37 369	20 264	38 017	22 646	6 198	43 943	20 765
Overall Sum	43 943	43 943	43 943	43 943	43 943	43 943	43 943	43 943
Difference	43 943	-1 953	-30 835	3 416	-34 090	-1 346	31 546	2 414
Final	5 492	-2 708	0 154	0 416	-8 522	-0 137	7 887	0 603

	Sum *	Sum -	Sum +	Sum -	Sum +	Sum -	Sum +
Overall Sum	36,731	19,917	9,333	20,148	6,652	18,296	31,546
Difference		18,815	29,398	18,983	32,860	20,435	16,963
		36,731	36,731	36,731	36,731	36,731	36,731
		1,102	1,565	-27,028	-2,139	24,361	4,806
	36,731	0,107	2,466	0,304	5,923	6,535	5,001

Product	Sum +	Sum -	Overall Sum	Difference
1	20,962	5,134	9,026	5,613
2	12,202	8,760	11,534	15,349
3	20,962	20,962	20,962	20,962
4	20,962	20,962	20,962	20,962
5	20,962	20,962	20,962	20,962
6	20,962	20,962	20,962	20,962
7	20,962	20,962	20,962	20,962
8	20,962	20,962	20,962	20,962
9	20,962	20,962	20,962	20,962
10	20,962	20,962	20,962	20,962
11	20,962	20,962	20,962	20,962
12	20,962	20,962	20,962	20,962
13	20,962	20,962	20,962	20,962
14	20,962	20,962	20,962	20,962
15	20,962	20,962	20,962	20,962
16	20,962	20,962	20,962	20,962
17	20,962	20,962	20,962	20,962
18	20,962	20,962	20,962	20,962
19	20,962	20,962	20,962	20,962
20	20,962	20,962	20,962	20,962
21	20,962	20,962	20,962	20,962
22	20,962	20,962	20,962	20,962
23	20,962	20,962	20,962	20,962
24	20,962	20,962	20,962	20,962
25	20,962	20,962	20,962	20,962
26	20,962	20,962	20,962	20,962
27	20,962	20,962	20,962	20,962
28	20,962	20,962	20,962	20,962
29	20,962	20,962	20,962	20,962
30	20,962	20,962	20,962	20,962
31	20,962	20,962	20,962	20,962
32	20,962	20,962	20,962	20,962
33	20,962	20,962	20,962	20,962
34	20,962	20,962	20,962	20,962
35	20,962	20,962	20,962	20,962
36	20,962	20,962	20,962	20,962
37	20,962	20,962	20,962	20,962
38	20,962	20,962	20,962	20,962
39	20,962	20,962	20,962	20,962
40	20,962	20,962	20,962	20,962
41	20,962	20,962	20,962	20,962
42	20,962	20,962	20,962	20,962
43	20,962	20,962	20,962	20,962
44	20,962	20,962	20,962	20,962
45	20,962	20,962	20,962	20,962
46	20,962	20,962	20,962	20,962
47	20,962	20,962	20,962	20,962
48	20,962	20,962	20,962	20,962
49	20,962	20,962	20,962	20,962
50	20,962	20,962	20,962	20,962
51	20,962	20,962	20,962	20,962
52	20,962	20,962	20,962	20,962
53	20,962	20,962	20,962	20,962
54	20,962	20,962	20,962	20,962
55	20,962	20,962	20,962	20,962
56	20,962	20,962	20,962	20,962
57	20,962	20,962	20,962	20,962
58	20,962	20,962	20,962	20,962
59	20,962	20,962	20,962	20,962
60	20,962	20,962	20,962	20,962
61	20,962	20,962	20,962	20,962
62	20,962	20,962	20,962	20,962
63	20,962	20,962	20,962	20,962
64	20,962	20,962	20,962	20,962
65	20,962	20,962	20,962	20,962
66	20,962	20,962	20,962	20,962
67	20,962	20,962	20,962	20,962
68	20,962	20,962	20,962	20,962
69	20,962	20,962	20,962	20,962
70	20,962	20,962	20,962	20,962
71	20,962	20,962	20,962	20,962
72	20,962	20,962	20,962	20,962
73	20,962	20,962	20,962	20,962
74	20,962	20,962	20,962	20,962
75	20,962	20,962	20,962	20,962
76	20,962	20,962	20,962	20,962
77	20,962	20,962	20,962	20,962
78	20,962	20,962	20,962	20,962
79	20,962	20,962	20,962	20,962
80	20,962	20,962	20,962	20,962
81	20,962	20,962	20,962	20,962
82	20,962	20,962	20,962	20,962
83	20,962	20,962	20,962	20,962
84	20,962	20,962	20,962	20,962
85	20,962	20,962	20,962	20,962
86	20,962	20,962	20,962	20,962
87	20,962	20,962	20,962	20,962
88	20,962	20,962	20,962	20,962
89	20,962	20,962	20,962	20,962
90	20,962	20,962	20,962	20,962
91	20,962	20,962	20,962	20,962
92	20,962	20,962	20,962	20,962
93	20,962	20,962	20,962	20,962
94	20,962	20,962	20,962	20,962
95	20,962	20,962	20,962	20,962
96	20,962	20,962	20,962	20,962
97	20,962	20,962	20,962	20,962
98	20,962	20,962	20,962	20,962
99	20,962	20,962	20,962	20,962
100	20,962	20,962	20,962	20,962

Sum +	12,262	6,044	12,162	6,044	12,162
Sum -	18,206	18,206	18,206	18,206	18,206
Overall Sum	18,206	18,206	18,206	18,206	18,206
Difference	18,206	-18,206	-6,118	-6,118	6,118

[illegible]

DOE Numbers - Copper Constant Potential

Run	Treatments		NO3Removed		CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	11.11%	14.81%	15.58%	19.92%	15.58%	19.92%	2.91%	5.35%	0.00%	0.00%	100.00%	100.00%
2	7	500	2.67%	2.67%	2.04%	1.88%	1.99%	1.82%	2.04%	2.51%	12.68%	11.66%	87.34%	88.34%
3	9	100	12.50%	16.67%	15.06%	19.64%	15.06%	19.64%	7.08%	6.10%	0.00%	0.00%	100.00%	100.00%
4	9	500	2.67%	2.67%	1.91%	2.10%	1.91%	2.10%	2.40%	2.31%	0.00%	0.00%	100.00%	100.00%
5	8	300	2.90%	6.43%	1.01%	5.39%	0.51%	5.11%	2.80%	5.39%	82.90%	21.62%	17.10%	78.38%

Factors

	Initial pH P	Concentration C
-	7.0	100
+	9.0	500

DOEN - Design of Experiments for Electrochemical Reduction of Nitrate

Trial		Mean	P	C	PC	CE Total	CE NH ₃	NO ₃ Removed	Product	NO ₂	NH ₃
1	+	-	-	+	-	17.75	17.75	12.95	4.13	0.00	100.00
2	+	-	-	+	-	1.96	1.91	2.67	2.27	12.16	87.84
3	+	+	-	-	-	17.35	17.35	14.58	6.59	0.00	100.00
4	+	+	+	+	+	2.00	2.00	2.67	2.35	0.00	100.00
5	0	0	0	0	0	3.20	2.81	4.66	4.09	52.26	47.74

CE Total											
Sum +		39.07	19.35	3.96	19.76						
Sum -		-	19.71	35.10	19.31						
Overall Sum		39.067	39.07	39.07	39.07						
Difference		39.067	-0.36	-31.14	0.45						
Effect		9.767	-0.089	-7.785	0.113						
Centre Point	Average	3.20									
Curvature		6.56									

CE NH ₃											
Sum +		39.02	19.35	5.33	19.76						
Sum -		-	19.66	35.10	19.26						
Overall Sum		39.017	39.02	40.44	39.02						
Difference		39.017	-0.31	-29.77	0.50						
Effect		9.754	-0.077	-7.443	0.125						
Centre Point	Average	2.81									
Curvature		6.94									

NO ₃ Removed											
Sum +		32.88	17.25	5.33	15.63						
Sum -		-	15.63	27.55	17.25						
Overall Sum		32.880	32.88	32.88	32.88						
Difference		32.880	1.62	-22.21	-1.62						
Effect		8.220	0.405	-5.553	-0.405						
Centre Point	Average	4.66									
Curvature		3.56									

Product											
Sum +		15.35	8.94	4.63	6.48						
Sum -		-	6.40	10.72	8.87						
Overall Sum		15.349	15.35	15.35	15.35						
Difference		15.349	2.54	-6.10	-2.36						
Effect		3.837	0.635	-1.524	-0.595						
Centre Point	Average	4.09									
Curvature		-0.26									

NO ₂											
Sum +		12.16	0.00	12.16	0.00						
Sum -		-	12.16	0.00	12.16						
Overall Sum		12.162	12.16	12.16	12.16						
Difference		12.162	-12.16	12.16	-12.16						
Effect		3.041	-3.041	3.041	-3.041						
Centre Point	Average	52.26									
Curvature		-49.22									

NH ₃											
Sum +		367.84	200.00	187.84	200.00						
Sum -		-	187.84	200.00	187.84						
Overall Sum		367.838	367.84	367.84	367.84						
Difference		367.838	12.16	-12.16	12.16						
Effect		96.959	3.041	-3.041	3.041						
Centre Point	Average	47.74									
Curvature		-49.22									

DOE Numbers - Graphite Felt Constant Potential

Run	Treatments		NO ₃ Removed		CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO ₃ C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	1.85%	1.85%	2.00%	2.57%	2.00%	2.57%	2.43%	1.96%	0.00%	0.00%	100.00%	100.00%
2	7	500	1.33%	1.33%	0.96%	1.05%	0.96%	1.05%	0.23%	0.08%	12.09%	0.00%	87.91%	100.00%
3	9	100	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	4.38%	1.44%	0.00%	0.00%	100.00%	100.00%
4	9	500	2.67%	2.67%	1.56%	1.71%	1.56%	1.71%	0.43%	0.26%	0.00%	0.00%	100.00%	100.00%
5	8	300	0.67%	0.67%	0.51%	0.56%	0.50%	0.56%	0.69%	0.36%	6.05%	0.00%	93.95%	100.00%

Factors

	Initial pH P	Concentration C
-	7.0	100
+	9.0	500

DOEN - Design of Experiments for Electrochemical Reduction of Nitrate												
Trial		Mean	P	C	PC	CE Total	CE NH ₃	NO ₃ Removed	Product	NO ₂	NH ₃	
1	+	-	-	+	2.29	2.29	1.85	2.20	0.00	100.00		
2	+	-	+	-	1.00	1.01	1.33	0.16	6.04	93.96		
3	+	+	-	-	0.00	0.00	0.00	2.91	0.00	100.00		
4	+	+	+	+	1.64	1.64	2.67	0.35	0.00	100.00		
5	0	0	0	0	0.55	0.54	0.67	0.53	3.03	96.97		

CE Total												
Sum +		4.93	1.64	2.64	3.92							
Sum -		--	3.29	2.29	1.00							
Overall Sum		4.926	4.93	4.93	4.93							
Difference		4.926	-1.66	0.36	2.92							
Effect		1.231	-0.413600608	0.0888901	0.729							
Centre Point Average		0.55										
Curvature		0.68										

CE NH ₃												
Sum +		4.93	1.64	2.64	3.92							
Sum -		--	3.29	2.29	1.01							
Overall Sum		4.927	4.93	4.93	4.93							
Difference		4.927	-1.66	0.36	2.91							
Effect		1.232	-0.413848677	0.08813817	0.729							
Centre Point Average		0.54										
Curvature		0.69										

NO ₃ Removed												
Sum +		5.85	2.67	4.00	4.52							
Sum -		--	3.19	1.85	1.33							
Overall Sum		5.852	5.85	5.85	5.85							
Difference		5.852	-0.52	2.15	3.19							
Effect		1.483	-0.12962963	0.53703704	0.766							
Centre Point Average		0.67										
Curvature		0.60										

Product												
Sum +		5.61	3.26	0.51	2.54							
Sum -		--	2.35	5.11	3.07							
Overall Sum		5.613	5.61	5.61	5.61							
Difference		5.613	0.90	-4.60	-0.53							
Effect		1.403	0.225982236	-1.1493365	-0.131							
Centre Point Average		0.53										
Curvature		0.88										

NO ₂												
Sum +		6.04	0.00	6.04	0.00							
Sum -		--	6.04	0.00	6.04							
Overall Sum		6.044	6.04	6.04	6.04							
Difference		6.044	-6.04	6.04	-6.04							
Effect		1.511	-1.510989011	1.51098901	-1.511							
Centre Point Average		3.03										
Curvature		-1.52										

NH ₃												
Sum +		393.96	200.00	193.96	200.00							
Sum -		--	193.96	200.00	193.96							
Overall Sum		393.956	393.96	393.96	393.96							
Difference		393.956	6.04	-6.04	6.04							
Effect		98.489	1.510989011	-1.510989	1.511							
Centre Point Average		96.97										
Curvature		1.52										

Experiment Name and Purpose	
Experiment Name:	DOEN-CVC71/DOEN-CVC71R
Date:	11/08/2004 -- 11/08/2004
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	11/08/2004 22:50	0.75	7.63	20	1090	545	2.1
Final	11/09/2004 01:26	0.75	8.69	20	1111	554	2.1
<i>Repeat</i>							
Initial	11/08/2004 22:45	0.75	7.63	20	1090	545	2.1
Final	11/09/2004 01:21	0.75	8.52	20	1104	552	2.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.0825	27.00	0.00	0.00	43.00	37.00
02:36	10.00	0.0880	24.00	0.00	0.72	43.00	36.00
<i>Repeat</i>							
00:00	10.00	0.0853	27.00	0.00	0.00	43.00	37.00
02:36	10.00	0.0925	23.00	0.00	1.30	42.00	36.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	20.25 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.54 mg
Total Nitrogen Souces Initial	20.25 mg	Total Nitrogen Souces Final	18.54 mg
Mass Balance Final-Initial:	-1.71 mg		
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	20.25 mg	NO ₃ ⁻	17.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.98 mg
Total Nitrogen Souces Initial	20.25 mg	Total Nitrogen Souces Final	18.23 mg
Mass Balance Final-Initial:	-2.03 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	89.67 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	11.11%
Min. % NO ₃ Removal Req.:	50.62%
<i>Repeat</i>	
Amount of NO ₃ Initial:	89.67 mg
Amount of NO ₃ Final:	76.39 mg
% NO ₃ Removed:	14.81%
Min. % NO ₃ Removal Req.:	50.62%
Average %NO ₃ Removal:	12.96%
Minimum % NO3 Removal:	50.62%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.66 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.039 mmol
% Product NO ₂ and NH ₃ :	0.82% by wt.
% Product NO ₂ and NH ₃ :	2.91% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.19 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.070 mmol
% Product NO ₂ and NH ₃ :	1.53% by wt.
% Product NO ₂ and NH ₃ :	5.35% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.17% by wt.
% Product NO ₂ and NH ₃ :	4.13% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.08525 A
Total Amt. of Coloumbs Passed:	795 C
Amt. of NO ₃ Decomposed:	9.96 mg
Moles of NO ₃ Decomposed:	1.606E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.606E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	123.96 C
Coulombic Efficiency to NH ₃ :	15.58% C
Coulombic Efficiency:	15.58%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.0889 A
Total Amt. of Coloumbs Passed:	829.55 C
Amt. of NO ₃ Decomposed:	13.28 mg
Moles of NO ₃ Decomposed:	2.141E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.141E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	165.28 C
Coulombic Efficiency to NH ₃ :	19.92% C
Coulombic Efficiency:	19.92%
Average Coulombic Efficiency to NH ₃ :	17.75%
Standard Deviation:	3.07%
Average Coulombic Efficiency:	17.75%
Standard Deviation:	3.07%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVGF71/DOEN-CVGF71R
Date:	11/08/2004 -- 11/08/2004
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 100ppm NO3

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO2	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	156	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/08/2004 22:55	0.75	7.63	20	1090	545	2.1
Final	11/09/2004 01:31	0.75	7.53	20	1082	540	2.1
<i>Repeat</i>							
Initial	11/08/2004 22:45	0.75	7.63	20	1090	545	2.1
Final	11/09/2004 01:21	0.75	7.78	20	1092	546	2.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.103	27.00	0.00	0.00	43.00	37.00
02:36	10.00	0.1189	26.50	0.00	0.66	43.00	37.00
<i>Repeat</i>							
00:00	10.00	0.0801	27.00	0.00	0.00	43.00	37.00
02:36	10.00	0.0919	26.50	0.00	0.53	42.00	36.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	20.25 mg	NO ₃ ⁻	19.88 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.50 mg
Total Nitrogen Souces Initial	20.25 mg	Total Nitrogen Souces Final	20.37 mg
Mass Balance Final-Initial:	0.12 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	20.25 mg	NO ₃ ⁻	19.88 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.40 mg
Total Nitrogen Souces Initial	20.25 mg	Total Nitrogen Souces Final	20.27 mg
Mass Balance Final-Initial:	0.02 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	89.67 mg
Amount of NO ₃ Final:	88.01 mg
% NO ₃ Removed:	1.85%
Min. % NO ₃ Removal Req.:	50.62%
<i>Repeat</i>	
Amount of NO ₃ Initial:	89.67 mg
Amount of NO ₃ Final:	88.01 mg
% NO ₃ Removed:	1.85%
Min. % NO ₃ Removal Req.:	50.62%
Average %NO ₃ Removal:	1.85%
Minimum % NO ₃ Removal:	50.62%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.60 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.035 mmol
% Product NO ₂ and NH ₃ :	0.68% by wt.
% Product NO ₂ and NH ₃ :	2.43% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.48 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.028 mmol
% Product NO ₂ and NH ₃ :	0.55% by wt.
% Product NO ₂ and NH ₃ :	1.96% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.61% by wt.
% Product NO ₂ and NH ₃ :	2.20% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.11095 A
Total Amt. of Coloumbs Passed:	1035 C
Amt. of NO ₃ Decomposed:	1.66 mg
Moles of NO ₃ Decomposed:	2.677E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.677E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	20.66 C
Coulombic Efficiency to NH ₃ :	2.00% C
Coulombic Efficiency:	2.00%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.086 A
Total Amt. of Coloumbs Passed:	802.49 C
Amt. of NO ₃ Decomposed:	1.66 mg
Moles of NO ₃ Decomposed:	2.677E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.677E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	20.66 C
Coulombic Efficiency to NH ₃ :	2.57% C
Coulombic Efficiency:	2.57%
Average Coulombic Efficiency to NH ₃ :	2.29%
Standard Deviation:	0.41%
Average Coulombic Efficiency:	2.29%
Standard Deviation:	0.41%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVC91/DOEN-CVC91R
Date:	11/08/2004 -- 11/08/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 100ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds 2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/08/2004 19:00	0.75	9.00	20	1155	576	2.2
Final	11/08/2004 21:36	0.75	9.03	20	1157	578	2.2
<i>Repeat</i>							
Initial	11/08/2004 22:45	0.75	9.00	20	1155	576	2.2
Final	11/09/2004 01:21	0.75	9.03	20	1148	574	2.2

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.0860	24.00	0.00	0.20	42.00	36.00
02:36	10.00	0.0904	21.00	0.00	1.60	42.00	36.00
<i>Repeat</i>							
00:00	10.00	0.0900	24.00	0.00	0.20	42.00	36.00
02:36	10.00	0.0904	20.00	0.00	1.30	43.00	37.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	15.75 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.15 mg	NH ₃	1.20 mg
Total Nitrogen Souces Initial	18.15 mg	Total Nitrogen Souces Final	16.95 mg
Mass Balance Final-Initial:	-1.20 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	15.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.15 mg	NH ₃	0.98 mg
Total Nitrogen Souces Initial	18.15 mg	Total Nitrogen Souces Final	15.98 mg
Mass Balance Final-Initial:	-2.18 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	69.75 mg
% NO ₃ Removed:	12.50%
Min. % NO ₃ Removal Req.:	44.44%
<hr/>	
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	66.42 mg
% NO ₃ Removed:	16.67%
Min. % NO ₃ Removal Req.:	44.44%
<hr/>	
Average %NO ₃ Removal:	14.58%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	1.46 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.086 mmol
% Product NO ₂ and NH ₃ :	2.05% by wt.
% Product NO ₂ and NH ₃ :	7.08% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.19 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.070 mmol
% Product NO ₂ and NH ₃ :	1.75% by wt.
% Product NO ₂ and NH ₃ :	6.10% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.90% by wt.
% Product NO ₂ and NH ₃ :	6.59% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.0882 A
Total Amt. of Coloumbs Passed:	823 C
Amt. of NO ₃ Decomposed:	9.96 mg
Moles of NO ₃ Decomposed:	1.606E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.606E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	123.96 C
Coulombic Efficiency to NH ₃ :	15.06% C
Coulombic Efficiency:	15.06%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.0902 A
Total Amt. of Coloumbs Passed:	841.68 C
Amt. of NO ₃ Decomposed:	13.28 mg
Moles of NO ₃ Decomposed:	2.141E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.141E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	165.28 C
Coulombic Efficiency to NH ₃ :	19.64% C
Coulombic Efficiency:	19.64%
Average Coulombic Efficiency to NH ₃ :	17.35%
Standard Deviation:	3.24%
Average Coulombic Efficiency:	17.35%
Standard Deviation:	3.24%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVGF91/DOEN-CVGF91R
Date:	11/08/2004 -- 11/08/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 100ppm NO3

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO2	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	156	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity μS	TDS ppm	NaCl% %
Initial	11/08/2004 19:10	0.75	9.00	20	1155	576	2.2
Final	11/08/2004 21:46	0.75	8.71	20	1161	580	2.2
<i>Repeat</i>							
Initial	11/08/2004 19:15	0.75	9.00	20	1155	576	2.2
Final	11/08/2004 21:51	0.75	8.73	20	1143	572	2.2

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.091	24.00	0.00	0.20	42.00	36.00
02:36	10.00	0.1035	24.00	0.00	1.10	43.00	37.00
<i>Repeat</i>							
00:00	10.00	0.0923	24.00	0.00	0.20	42.00	36.00
02:36	10.00	0.1040	24.00	0.00	0.35	43.00	36.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.15 mg	NH ₃	0.83 mg
Total Nitrogen Souces Initial	18.15 mg	Total Nitrogen Souces Final	18.83 mg
Mass Balance Final-Initial:	0.68 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.15 mg	NH ₃	0.26 mg
Total Nitrogen Souces Initial	18.15 mg	Total Nitrogen Souces Final	18.26 mg
Mass Balance Final-Initial:	0.11 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	44.44%
<hr/>	
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	44.44%
<hr/>	
Average %NO ₃ Removal:	0.00%
Minimum % NO3 Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	1.00 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.059 mmol
% Product NO ₂ and NH ₃ :	1.24% by wt.
% Product NO ₂ and NH ₃ :	4.38% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.32 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.019 mmol
% Product NO ₂ and NH ₃ :	0.40% by wt.
% Product NO ₂ and NH ₃ :	1.44% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.82% by wt.
% Product NO ₂ and NH ₃ :	2.91% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.09725 A
Total Amt. of Coloumbs Passed:	907 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.09815 A
Total Amt. of Coloumbs Passed:	915.86 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
Average Coulombic Efficiency to NH ₃ :	0.00%
Standard Deviation:	0.00%
Average Coulombic Efficiency:	0.00%
Standard Deviation:	0.00%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVC83/DOEN-CVC83R
Date:	11/09/2004 -- 11/09/2004
Test Type / Purpose:	Initial pH 8 - Constant Voltage - 300ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	467	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	300 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	67.75 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	27994 seconds 7.8 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 14:45	0.75	8.91	20	1571	785	3.0
Final	11/09/2004 22:00	0.75	9.05	20	1592	794	3.1
<i>Repeat</i>							
Initial	11/09/2004 14:40	0.75	8.91	20	1571	785	3.0
Final	11/09/2004 22:00	0.745	9.00	20	1608	805	3.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.1220	69.00	0.00	0.00	42.00	38.00
07:15	10.00	0.1108	67.00	1.60	0.33	43.00	39.00
<i>Repeat</i>							
00:00	10.00	0.1046	69.00	0.00	0.00	42.00	38.00
07:20	10.00	0.1055	65.00	0.80	2.90	43.00	39.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	51.75 mg	NO ₃ ⁻	50.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	1.20 mg
NH ₃	0.00 mg	NH ₃	0.25 mg
Total Nitrogen Souces Initial	51.75 mg	Total Nitrogen Souces Final	51.70 mg
Mass Balance Final-Initial:	-0.05 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	51.75 mg	NO ₃ ⁻	48.43 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.60 mg
NH ₃	0.00 mg	NH ₃	2.16 mg
Total Nitrogen Souces Initial	51.75 mg	Total Nitrogen Souces Final	51.18 mg
Mass Balance Final-Initial:	-0.57 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	229.16 mg
Amount of NO ₃ Final:	222.52 mg
% NO ₃ Removed:	2.90%
Min. % NO ₃ Removal Req.:	80.68%
<i>Repeat</i>	
Amount of NO ₃ Initial:	229.16 mg
Amount of NO ₃ Final:	214.44 mg
% NO ₃ Removed:	6.43%
Min. % NO ₃ Removal Req.:	80.68%
Average %NO ₃ Removal:	4.66%
Minimum % NO ₃ Removal:	80.68%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	4.25 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.103 mmol
% Product NO ₂ and NH ₃ :	1.87% by wt.
% Product NO ₂ and NH ₃ :	2.80% by mol.
% Split of NO ₂ :	92.91% by wt.
% Split of NH ₃ :	7.09% by wt.
% Split of NO ₂ :	82.90% by mol.
% Split of NH ₃ :	17.10% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	4.59 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.197 mmol
% Product NO ₂ and NH ₃ :	2.09% by wt.
% Product NO ₂ and NH ₃ :	5.39% by mol.
% Split of NO ₂ :	42.71% by wt.
% Split of NH ₃ :	57.29% by wt.
% Split of NO ₂ :	21.62% by mol.
% Split of NH ₃ :	78.38% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.98% by wt.
% Product NO ₂ and NH ₃ :	4.09% by mol.
% Split of NO ₂ :	52.26% by mol.
% Split of NH ₃ :	47.74% by mol.

Coulombic Efficiency	
Time for Experiment:	27994 s
Current Passed:	0.1164 A
Total Amt. of Coloumbs Passed:	3258 C
Amt. of NO ₃ Decomposed:	6.64 mg
Moles of NO ₃ Decomposed:	1.071E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	8.56531E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.141E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	16.53 C
Coulombs to NH ₃ :	16.53 C
Coulombic Efficiency to NH ₃ :	0.51% C
Coulombic Efficiency:	1.01%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.10505 A
Total Amt. of Coloumbs Passed:	2940.74 C
Amt. of NO ₃ Decomposed:	14.72 mg
Moles of NO ₃ Decomposed:	2.373E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	4.2541E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.948E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	8.21 C
Coulombs to NH ₃ :	150.35 C
Coulombic Efficiency to NH ₃ :	5.11% C
Coulombic Efficiency:	5.39%
Average Coulombic Efficiency to NH ₃ :	2.81%
Standard Deviation:	3.26%
Average Coulombic Efficiency:	3.20%
Standard Deviation:	3.10%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVGF83/DOEN-CVGF83R
Date:	11/08/2004 -- 11/08/2004
Test Type / Purpose:	Initial pH 8 - Constant Voltage - 300ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	467	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	300 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	67.75 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	27994 seconds 7.8 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 14:50	0.75	8.91	20	1571	785	3.0
Final	11/09/2004 22:00	0.745	8.49	20	1593	797	3.1
<i>Repeat</i>							
Initial	11/09/2004 15:00	0.75	8.91	20	1571	785	3.0
Final	11/09/2004 22:00	0.745	8.44	20	1592	795	3.1

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.126	69.00	0.00	0.00	42.00	38.00
07:10	10.00	0.128	69.00	0.03	0.45	43.00	39.00
<i>Repeat</i>							
00:00	10.00	0.1222	69.00	0.00	0.00	42.00	38.00
07:00	10.00	0.1108	69.00	0.00	0.25	43.00	39.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	51.75 mg	NO ₃ ⁻	51.41 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.02 mg
NH ₃	0.00 mg	NH ₃	0.34 mg
Total Nitrogen Souces Initial	51.75 mg	Total Nitrogen Souces Final	51.76 mg
Mass Balance Final-Initial:	0.01 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	51.75 mg	NO ₃ ⁻	51.41 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.19 mg
Total Nitrogen Souces Initial	51.75 mg	Total Nitrogen Souces Final	51.59 mg
Mass Balance Final-Initial:	-0.16 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	229.16 mg
Amount of NO ₃ Final:	227.63 mg
% NO ₃ Removed:	0.67%
Min. % NO ₃ Removal Req.:	80.68%
<i>Repeat</i>	
Amount of NO ₃ Initial:	229.16 mg
Amount of NO ₃ Final:	227.63 mg
% NO ₃ Removed:	0.67%
Min. % NO ₃ Removal Req.:	80.68%
Average %NO ₃ Removal:	0.67%
Minimum % NO ₃ Removal:	80.68%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.48 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.025 mmol
% Product NO ₂ and NH ₃ :	0.21% by wt.
% Product NO ₂ and NH ₃ :	0.69% by mol.
% Split of NO ₂ :	14.83% by wt.
% Split of NH ₃ :	85.17% by wt.
% Split of NO ₂ :	6.05% by mol.
% Split of NH ₃ :	93.95% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.23 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.013 mmol
% Product NO ₂ and NH ₃ :	0.10% by wt.
% Product NO ₂ and NH ₃ :	0.36% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.15% by wt.
% Product NO ₂ and NH ₃ :	0.53% by mol.
% Split of NO ₂ :	3.03% by mol.
% Split of NH ₃ :	96.97% by mol.

Coulombic Efficiency	
Time for Experiment:	27994 s
Current Passed:	0.12675 A
Total Amt. of Coloumbs Passed:	3548 C
Amt. of NO ₃ Decomposed:	1.53 mg
Moles of NO ₃ Decomposed:	2.463E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.542E-06 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.308E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.30 C
Coulombs to NH ₃ :	17.82 C
Coulombic Efficiency to NH ₃ :	0.50% C
Coulombic Efficiency:	0.51%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	27994 s
Current Passed:	0.1165 A
Total Amt. of Coloumbs Passed:	3261.27 C
Amt. of NO ₃ Decomposed:	1.53 mg
Moles of NO ₃ Decomposed:	2.463E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	2.463E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	19.01 C
Coulombic Efficiency to NH ₃ :	0.58% C
Coulombic Efficiency:	0.58%
Average Coulombic Efficiency to NH ₃ :	0.54%
Standard Deviation:	0.06%
Average Coulombic Efficiency:	0.55%
Standard Deviation:	0.05%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVC75/DOEN-CVC75R
Date:	11/9/2004 -- 11/9/2004
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed in Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds 12.96 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 22:45	0.75	7.69	20	1973	987	3.8
Final	11/10/2004 11:00	0.73	9.13	20	2024	1011	3.9
<i>Repeat</i>							
Initial	11/08/2004 22:45	0.75	7.69	20	1973	987	3.8
Final	11/09/2004 11:21	0.73	9.17	20	1990	996	3.8

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.1260	110.00	0.02	0.00	43.00	42.00
12:15	10.00	0.1118	110.00	0.29	2.00	44.00	42.00
<i>Repeat</i>							
00:00	10.00	0.1446	110.00	0.02	0.00	43.00	42.00
12:36	10.00	0.1111	110.00	0.33	2.50	44.00	43.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.21 mg
NH ₃	0.00 mg	NH ₃	1.46 mg
Total Nitrogen Souces Initial	82.52 mg	Total Nitrogen Souces Final	81.97 mg
Mass Balance Final-Initial:	-0.54 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.24 mg
NH ₃	0.00 mg	NH ₃	1.83 mg
Total Nitrogen Souces Initial	82.52 mg	Total Nitrogen Souces Final	82.37 mg
Mass Balance Final-Initial:	-0.15 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	2.47 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.119 mmol
% Product NO ₂ and NH ₃ :	0.69% by wt.
% Product NO ₂ and NH ₃ :	2.04% by mol.
% Split of NO ₂ :	28.15% by wt.
% Split of NH ₃ :	71.85% by wt.
% Split of NO ₂ :	12.66% by mol.
% Split of NH ₃ :	87.34% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	3.01 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.147 mmol
% Product NO ₂ and NH ₃ :	0.84% by wt.
% Product NO ₂ and NH ₃ :	2.51% by mol.
% Split of NO ₂ :	26.29% by wt.
% Split of NH ₃ :	73.71% by wt.
% Split of NO ₂ :	11.66% by mol.
% Split of NH ₃ :	88.34% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.77% by wt.
% Product NO ₂ and NH ₃ :	2.27% by mol.
% Split of NO ₂ :	12.16% by mol.
% Split of NH ₃ :	87.84% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.1189 A
Total Amt. of Coloumbs Passed:	5547 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.39329E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.431E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	2.69 C
Coulombs to NH ₃ :	110.45 C
Coulombic Efficiency to NH ₃ :	1.99% C
Coulombic Efficiency:	2.04%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.12785 A
Total Amt. of Coloumbs Passed:	5964.99 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	1.60171E-05 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.410E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	3.09 C
Coulombs to NH ₃ :	108.85 C
Coulombic Efficiency to NH ₃ :	1.82% C
Coulombic Efficiency:	1.88%
Average Coulombic Efficiency to NH ₃ :	1.91%
Standard Deviation:	0.12%
Average Coulombic Efficiency:	1.96%
Standard Deviation:	0.12%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVGF75/DOEN-CVGF75R
Date:	11/09/2004 -- 11/09/2004
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 500ppm NO ₃

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO ₂	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	778	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 22:45	0.75	7.69	20	1973	987	3.8
Final	11/10/2004 11:00	0.74	8.65	20	1971	983	3.8
<i>Repeat</i>							
Initial	11/09/2004 22:45	0.75	7.69	20	1973	987	3.8
Final	11/10/2004 11:00	0.74	8.56	20	1938	969	3.8

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.149	120.00	0.02	0.00	43.00	42.00
12:15	10.00	0.145	120.00	0.03	0.24	44.00	43.00
<i>Repeat</i>							
00:00	10.00	0.1375	120.00	0.02	0.00	43.00	42.00
12:15	10.00	0.1350	120.00	0.00	0.11	44.00	44.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	90.00 mg	NO ₃ ⁻	88.80 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.02 mg
NH ₃	0.00 mg	NH ₃	0.18 mg
Total Nitrogen Souces Initial	90.02 mg	Total Nitrogen Souces Final	89.00 mg
Mass Balance Final-Initial:	-1.01 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	90.00 mg	NO ₃ ⁻	88.80 mg
NO ₂ ⁻	0.02 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.08 mg
Total Nitrogen Souces Initial	90.02 mg	Total Nitrogen Souces Final	88.88 mg
Mass Balance Final-Initial:	-1.14 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	398.54 mg
Amount of NO ₃ Final:	393.23 mg
% NO ₃ Removed:	1.33%
Min. % NO ₃ Removal Req.:	88.89%
<i>Repeat</i>	
Amount of NO ₃ Initial:	398.54 mg
Amount of NO ₃ Final:	393.23 mg
% NO ₃ Removed:	1.33%
Min. % NO ₃ Removal Req.:	88.89%
Average %NO ₃ Removal:	1.33%
Minimum % NO ₃ Removal:	88.89%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.30 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.014 mmol
% Product NO ₂ and NH ₃ :	0.08% by wt.
% Product NO ₂ and NH ₃ :	0.23% by mol.
% Split of NO ₂ :	27.09% by wt.
% Split of NH ₃ :	72.91% by wt.
% Split of NO ₂ :	12.09% by mol.
% Split of NH ₃ :	87.91% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.10 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.006 mmol
% Product NO ₂ and NH ₃ :	0.03% by wt.
% Product NO ₂ and NH ₃ :	0.09% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.05% by wt.
% Product NO ₂ and NH ₃ :	0.16% by mol.
% Split of NO ₂ :	6.04% by mol.
% Split of NH ₃ :	93.96% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.147 A
Total Amt. of Coloumbs Passed:	6858 C
Amt. of NO ₃ Decomposed:	5.31 mg
Moles of NO ₃ Decomposed:	8.565E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	5.653E-07 mol
Amt. Of NO ₃ Decomposed to NH ₃	8.509E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.11 C
Coulombs to NH ₃ :	65.68 C
Coulombic Efficiency to NH ₃ :	0.96% C
Coulombic Efficiency:	0.96%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.13625 A
Total Amt. of Coloumbs Passed:	6356.91 C
Amt. of NO ₃ Decomposed:	5.31 mg
Moles of NO ₃ Decomposed:	8.565E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	-1.178E-06 mol
Amt. Of NO ₃ Decomposed to NH ₃	8.683E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	-0.23 C
Coulombs to NH ₃ :	67.02 C
Coulombic Efficiency to NH ₃ :	1.05% C
Coulombic Efficiency:	1.05%
Average Coulombic Efficiency to NH ₃ :	1.01%
Standard Deviation:	0.07%
Average Coulombic Efficiency:	1.00%
Standard Deviation:	0.06%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVC95/DOEN-CVC95R
Date:	11/9/2004 -- 11/9/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 500ppm NO ₃

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	12.96 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 02:00	0.75	9.02	20	1996	998	3.9
Final	11/09/2004 13:45	0.73	9.44	20	2055	1029	4.0
<i>Repeat</i>							
Initial	11/09/2004 01:55	0.75	9.02	20	1996	998	3.9
Final	11/09/2004 13:45	0.73	9.32	20	2055	1028	4.0

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.1482	110.00	0.00	0.00	42.00	38.00
11:45	10.00	0.1233	110.00	0.00	2.70	43.00	39.00
<i>Repeat</i>							
00:00	10.00	0.1325	110.00	0.00	0.00	42.00	38.00
11:50	10.00	0.1154	110.00	0.00	2.60	43.00	39.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.97 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	82.27 mg
Mass Balance Final-Initial:	-0.23 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.90 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	82.20 mg
Mass Balance Final-Initial:	-0.30 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	2.40 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.141 mmol
% Product NO ₂ and NH ₃ :	0.67% by wt.
% Product NO ₂ and NH ₃ :	2.40% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	2.31 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.135 mmol
% Product NO ₂ and NH ₃ :	0.65% by wt.
% Product NO ₂ and NH ₃ :	2.31% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.66% by wt.
% Product NO ₂ and NH ₃ :	2.35% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.13575 A
Total Amt. of Coloumbs Passed:	6334 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.570E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	121.21 C
Coulombic Efficiency to NH ₃ :	1.91% C
Coulombic Efficiency:	1.91%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.12395 A
Total Amt. of Coloumbs Passed:	5783.03 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.570E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	121.21 C
Coulombic Efficiency to NH ₃ :	2.10% C
Coulombic Efficiency:	2.10%
Average Coulombic Efficiency to NH ₃ :	2.00%
Standard Deviation:	0.13%
Average Coulombic Efficiency:	2.00%
Standard Deviation:	0.13%

Experiment Name and Purpose	
Experiment Name:	DOEN-CVGF95/DOEN-CVGF95R
Date:	11/09/2004 -- 11/09/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 500ppm NO3

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO2	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	778	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	500 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	112.91 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds 13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/09/2004 22:45	0.75	9.02	20	1996	998	3.9
Final	11/10/2004 11:00	0.73	9.14	20	2018	998	3.9
<i>Repeat</i>							
Initial	11/09/2004 22:45	0.75	9.02	20	1996	998	3.9
Final	11/10/2004 11:00	0.73	8.59	20	2026	1008	3.9

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.1758	110.00	0.00	0.00	42.00	38.00
12:15	10.00	0.1574	110.00	0.00	0.48	43.00	39.00
<i>Repeat</i>							
00:00	10.00	0.1525	110.00	0.00	0.00	42.00	38.00
12:15	10.00	0.1510	110.00	0.00	0.29	43.00	39.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.35 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	80.65 mg
Mass Balance Final-Initial:	-1.85 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	82.50 mg	NO ₃ ⁻	80.30 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.21 mg
Total Nitrogen Souces Initial	82.50 mg	Total Nitrogen Souces Final	80.51 mg
Mass Balance Final-Initial:	-1.99 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<hr/>	
<i>Repeat</i>	
Amount of NO ₃ Initial:	365.33 mg
Amount of NO ₃ Final:	355.59 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	87.88%
<hr/>	
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	87.88%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.43 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.025 mmol
% Product NO ₂ and NH ₃ :	0.12% by wt.
% Product NO ₂ and NH ₃ :	0.43% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.26 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.015 mmol
% Product NO ₂ and NH ₃ :	0.07% by wt.
% Product NO ₂ and NH ₃ :	0.26% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.10% by wt.
% Product NO ₂ and NH ₃ :	0.35% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.1666 A
Total Amt. of Coloumbs Passed:	7773 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.570E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	121.21 C
Coulombic Efficiency to NH ₃ :	1.56% C
Coulombic Efficiency:	1.56%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.15175 A
Total Amt. of Coloumbs Passed:	7080.08 C
Amt. of NO ₃ Decomposed:	9.74 mg
Moles of NO ₃ Decomposed:	1.570E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.570E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	121.21 C
Coulombic Efficiency to NH ₃ :	1.71% C
Coulombic Efficiency:	1.71%
Average Coulombic Efficiency to NH ₃ :	1.64%
Standard Deviation:	0.11%
Average Coulombic Efficiency:	1.64%
Standard Deviation:	0.11%

APPENDIX D

DATA FOR EXPERIMENTS CARRIED OUT UNDER VARIOUS CHLORIDE AND SULFATE CONCENTRATIONS

Summary							
Experiment	% Break-Down of Product			%NO ₃ Removed	Minimum % NO ₃ Removal	Coulombic Efficiency NH ₃	Coulombic Efficiency Total
	% NO ₂ and NH ₃ Formed	% NO ₂	% NH ₃				
DOEN-C71DI	4.75%	0.00%	100.00%	8.38%	42.03%	6.96%	6.96%
DOEN-C71	9.06%	52.30%	47.70%	7.57%	47.02%	5.01%	6.32%
DOEN-C71CV	4.13%	0.00%	100.00%	12.96%	50.62%	17.75%	17.75%
DOEN-C71CL	2.46%	0.00%	100.00%	4.17%	44.44%	4.35%	4.35%
DOEN-C71SO4	4.49%	0.00%	100.00%	6.25%	44.44%	6.43%	6.43%
DOEN-GF71DI	0.84%	0.00%	100.00%	2.67%	42.03%	1.80%	1.80%
DOEN-GF71	0.00%	#DIV/0!	#DIV/0!	-2.90%	47.02%	0.00%	0.00%
DOEN-GF71CV	2.20%	0.00%	100.00%	1.85%	50.62%	2.29%	2.29%
DOEN-GF71CL	0.00%	#DIV/0!	#DIV/0!	0.00%	44.44%	0.00%	0.00%
DOEN-GF71SO4	0.56%	0.00%	100.00%	0.38%	48.72%	0.43%	0.43%
DOEN-C91DI	5.07%	0.00%	100.00%	7.50%	44.44%	5.75%	5.75%
DOEN-GF91DI	0.52%	0.00%	100.00%	2.00%	44.44%	1.51%	1.51%

Target Initial	Target Initial	Reaction Time	Current (A)	Voltage (V)	pH	Vol. (L)	Temp. (°C)	TDS (ppm)	NaCl %	Cond. (µS)	[NO ₃] (as N) (ppm)	[NO ₃] (as N) (ppm)	[NH ₃] (as N) (ppm)	[SO ₄] (ppm)	[Cl] (ppm)	Desc.
7	100	02:36	0.10027	11.05	7.27	0.75	22	526	2.03	1051	25.67	0.00	0.00	44	23	Const. Current
Initial											113.66	0.00	0.00			
Std. Dev.			0.00046	0.09	0.08	0.00	1.73	16.74	0.06	32	4.62	0.00	0.00	1	1	
Final			0.10430	10.39	8.34	0.75	22	533	2.03	1065	20.45	0.00	0.00			
Std. Dev.			0.00745	0.67	0.24	0.00	1.73	22.94	0.06	45	3.79	0.34	0.50			Const. Potential
Initial	100	02:36	0.08390	10.00	7.63	0.75	20	545	2.10	1090	27.00	0.00	0.00	43	37	
Std. Dev.			0.00198	0.00	0.00	0.00	0.00	0	0.00	0	119.56	0.00	0.00	1	1	
Final			0.09025	10.00	8.61	0.75	20	553	2.10	1108	23.50	0.00	1.01			
Std. Dev.			0.00318	0.00	0.12	0.00	0.00	1	0.00	5	104.06	0.00	1.23			High [SO ₄]
Initial	100	02:36	0.10460	8.13	7.50	0.75	20	1108	4.30	2207	24.00	0.00	0.00	443	25	
Std. Dev.			0.00311	0.68	0.00	0.00	0	0	0.00	0	106.28	0.00	0.00	5	3	
Final			0.10215	7.86	8.28	0.75	20	1096	4.30	2194	22.50	0.00	1.06			
Std. Dev.			0.00233	0.77	0.02	0.00	0	13	0.00	25	99.64	0.00	1.28			High [Cl]
Initial	100	02:36	0.10095	9.83	7.41	0.75	20	866	3.40	1733	24.00	0.00	0.00	45	200	
Std. Dev.			0.00021	1.45	0.00	0.00	0	0	0.00	0	106.28	0.00	0.00	0	0	
Final			0.10275	9.18	8.30	0.75	21	876	3.40	1751	23.00	0.00	0.58			
Std. Dev.			0.00078	0.69	0.00	0.00	1	2	0.00	4	101.85	0.00	0.71			D.I. Water
Initial	100	13:00	0.02350	10.00	7.00	0.75	20	124	0.30	249	23.00	0.00	0.00	1	1	
Std. Dev.			0.00354	0.00	0.00	0.00	0	0	0.00	0	101.85	0.00	0.00	2	0	
Final			0.02460	10.00	7.01	0.74	20	135	0.50	270	21.50	0.00	1.07			
Std. Dev.			0.00156	0.00	0.28	0.01	0	2	0.00	5	95.21	0.00	1.30			D.I. Water
Initial	9	13:00	0.02850	10.00	9.10	0.75	20	150	0.50	300	24.00	0.00	0.00	0	0	
Std. Dev.			0.00071	0.00	0.00	0.00	0	0	0.00	0	106.28	0.00	0.00	0	0	
Final			0.02745	10.00	8.04	0.74	20	153	0.50	304	22.50	0.00	1.20			
Std. Dev.			0.00148	0.00	0.51	0.00	0	10	0.00	23	99.64	0.00	1.46			D.I. Water
											0.71	0.00	0.28			
											3.13	0.00	0.34			

DATA

Target Initial pH	Target Initial [NO ₃]	Desc. of Exp.	Avg. Soln. Conductivity μS	Coulombic Efficiency %
7	100	DI	260	6.96%
7	100	Const. Current	1058	6.32%
7	100	High [SO ₄]	2200	6.43%
7	100	High [Cl]	1742	4.35%
7	100	Const. Potential	1099	17.75%

Coulombic Efficiency versus Average Solution Conductivity at Various Conditions

DOE Numbers - Copper - Sulphate and Chloride

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1S04	7	100	4.17%	8.33%	4.27%	8.59%	3.40%	5.58%	0.00%	0.00%	100.00%	100.00%
1CL	7	100	4.17%	4.17%	4.34%	4.36%	2.38%	2.54%	0.00%	0.00%	100.00%	100.00%

DOE Numbers - Graphite Felt - Sulphate and Chloride

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1S04	7	100	0.38%	0.38%	0.43%	0.42%	0.54%	0.58%	0.00%	0.00%	100.00%	100.00%
1CL	9	100	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

DOE Numbers - Copper - De-ionized Water

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	11.13%	5.62%	8.77%	5.15%	6.25%	3.25%	0.00%	0.00%	100.00%	100.00%
1	9	100	5.44%	9.56%	4.03%	7.47%	4.17%	5.98%	0.00%	0.00%	100.00%	100.00%

DOE Numbers - Graphite Felt - De-ionized Water

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	2.67%	2.67%	1.49%	2.10%	1.12%	0.56%	0.00%	0.00%	100.00%	100.00%
1	9	100	1.33%	2.67%	0.85%	2.16%	0.62%	0.41%	0.00%	0.00%	100.00%	100.00%

DOE Numbers - Copper Constant Current

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	4.35%	8.70%	3.54%	7.64%	2.61%	6.20%	58.74%	48.15%	41.26%	51.85%

DOE Numbers - Graphite Felt Constant Current

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	-4.35%	-4.35%	0.00%	0.00%	0.00%	0.00%	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

DOE Numbers - Copper Constant Voltage

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	11.11%	14.81%	15.58%	19.92%	2.91%	5.35%	0.00%	0.00%	100.00%	100.00%

DOE Numbers - Graphite Felt Constant Voltage

Run	Treatments	NO ₃ Removed	CE Total		CE NH ₃		Product		NO ₂		NH ₃	
	IPH	INO3C	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2	Run1	Run2
1	7	100	1.85%	1.85%	2.00%	2.57%	2.43%	1.96%	0.00%	0.00%	100.00%	100.00%

Experiment Name and Purpose	
Experiment Name:	DOENSO4-C71/DOENSO4-C71R
Date:	10/27/2004 -- 10/27/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 100ppm NO ₃ - High Sulphate

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds 2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	10/26/2004 23:04	0.75	7.50	20	2207	1108	4.3
Final	10/27/2004 01:40	0.75	8.26	20	2176	1086	4.3
<i>Repeat</i>							
Initial	10/26/2004 23:11	0.75	7.50	20	2207	1108	4.3
Final	10/27/2004 01:47	0.75	8.29	20	2211	1105	4.3

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	8.61	0.1068	24.00	0.00	0.00	440.00	22.00
02:36	8.40	0.1005	23.00	0.00	0.81	450.00	27.00
<i>Repeat</i>							
00:00	7.65	0.1024	24.00	0.00	0.00	440.00	22.00
02:36	7.31	0.1038	22.00	0.00	1.30	440.00	27.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.61 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.86 mg
Mass Balance Final-Initial:	-0.14 mg		
<hr/>			
Repeat			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	16.50 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.98 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.48 mg
Mass Balance Final-Initial:	-0.52 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	76.39 mg
% NO ₃ Removed:	4.17%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	73.07 mg
% NO ₃ Removed:	8.33%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	6.25%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.74 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.043 mmol
% Product NO ₂ and NH ₃ :	0.96% by wt.
% Product NO ₂ and NH ₃ :	3.40% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.19 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.070 mmol
% Product NO ₂ and NH ₃ :	1.60% by wt.
% Product NO ₂ and NH ₃ :	5.58% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.28% by wt.
% Product NO ₂ and NH ₃ :	4.49% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.10365 A
Total Amt. of Coloumbs Passed:	967 C
Amt. of NO ₃ Decomposed:	3.32 mg
Moles of NO ₃ Decomposed:	5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.353E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	41.32 C
Coulombic Efficiency to NH ₃ :	4.27% C
Coulombic Efficiency:	4.27%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1031 A
Total Amt. of Coloumbs Passed:	962.05 C
Amt. of NO ₃ Decomposed:	6.64 mg
Moles of NO ₃ Decomposed:	1.071E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.071E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	82.64 C
Coulombic Efficiency to NH ₃ :	8.59% C
Coulombic Efficiency:	8.59%
Average Coulombic Efficiency to NH ₃ :	6.43%
Standard Deviation:	3.05%
Average Coulombic Efficiency:	6.43%
Standard Deviation:	3.05%

Experiment Name and Purpose	
Experiment Name:	DOENSO4-GF71/DOENSO4-GF71R
Date:	10/26/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 7 - Constant Current - 100ppm NO3 - High Sulphate

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO2	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	156	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds
	2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	10/26/2004 23:00	0.75	7.50	20	2207	1108	4.3
Final	10/27/2004 01:36	0.75	7.46	20	2197	1096	4.3
<i>Repeat</i>							
Initial	10/26/2004 23:20	0.75	7.50	20	2207	1108	4.3
Final	10/27/2004 01:56	0.75	7.83	20	2206	1100	4.3

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	7.72	0.1014	26.00	0.00	0.00	440.00	22.00
02:36	6.86	0.1029	25.90	0.00	0.14	460.00	27.00
<i>Repeat</i>							
00:00	8.87	0.1043	26.00	0.00	0.00	440.00	22.00
02:36	7.60	0.1063	25.90	0.00	0.15	440.00	27.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	19.50 mg	NO ₃ ⁻	19.43 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.11 mg
Total Nitrogen Souces Initial	19.50 mg	Total Nitrogen Souces Final	19.53 mg
Mass Balance Final-Initial:	0.03 mg		
<hr/>			
Repeat			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	19.50 mg	NO ₃ ⁻	19.43 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.11 mg
Total Nitrogen Souces Initial	19.50 mg	Total Nitrogen Souces Final	19.54 mg
Mass Balance Final-Initial:	0.04 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	86.35 mg
Amount of NO ₃ Final:	86.02 mg
% NO ₃ Removed:	0.38%
Min. % NO ₃ Removal Req.:	48.72%
<hr/>	
<i>Repeat</i>	
Amount of NO ₃ Initial:	86.35 mg
Amount of NO ₃ Final:	86.02 mg
% NO ₃ Removed:	0.38%
Min. % NO ₃ Removal Req.:	48.72%
<hr/>	
Average %NO ₃ Removal:	0.38%
Minimum % NO ₃ Removal:	48.72%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.13 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.007 mmol
% Product NO ₂ and NH ₃ :	0.15% by wt.
% Product NO ₂ and NH ₃ :	0.54% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.14 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.008 mmol
% Product NO ₂ and NH ₃ :	0.16% by wt.
% Product NO ₂ and NH ₃ :	0.58% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<hr/>	
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.15% by wt.
% Product NO ₂ and NH ₃ :	0.56% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.10215 A
Total Amt. of Coloumbs Passed:	953 C
Amt. of NO ₃ Decomposed:	0.33 mg
Moles of NO ₃ Decomposed:	5.353E-06 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.353E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	4.13 C
Coulombic Efficiency to NH ₃ :	0.43% C
Coulombic Efficiency:	0.43%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.1053 A
Total Amt. of Coloumbs Passed:	982.58 C
Amt. of NO ₃ Decomposed:	0.33 mg
Moles of NO ₃ Decomposed:	5.353E-06 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.353E-06 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	4.13 C
Coulombic Efficiency to NH ₃ :	0.42% C
Coulombic Efficiency:	0.42%
Average Coulombic Efficiency to NH ₃ :	0.43%
Standard Deviation:	0.01%
Average Coulombic Efficiency:	0.43%
Standard Deviation:	0.01%

Experiment Name and Purpose	
Experiment Name:	DOENCL-C71/DOENCL-C71R
Date:	10/26/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 100ppm NO ₃ - High Chloride

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds 2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl %
Initial	10/26/2004 19:13	0.75	7.41	20	1733	866	3.4
Final	10/26/2004 21:49	0.75	8.30	21	1754	877	3.4
<i>Repeat</i>							
Initial	11/02/2004 23:22	0.75	7.41	20	1733	866	3.4
Final	11/03/2004 01:58	0.75	8.30	20	1748	874	3.4

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.85	0.1008	24.00	0.00	0.00	45.00	200.00
02:36	9.67	0.1033	23.00	0.00	0.56	45.00	200.00
<i>Repeat</i>							
00:00	8.80	0.1011	24.00	0.00	0.00	45.00	200.00
02:36	8.69	0.1022	23.00	0.00	0.60	45.00	200.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.42 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.67 mg
Mass Balance Final-Initial:	-0.33 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.25 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.45 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.70 mg
Mass Balance Final-Initial:	-0.30 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	76.39 mg
% NO ₃ Removed:	4.17%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	76.39 mg
% NO ₃ Removed:	4.17%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	4.17%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.51 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.030 mmol
% Product NO ₂ and NH ₃ :	0.66% by wt.
% Product NO ₂ and NH ₃ :	2.38% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.55 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.032 mmol
% Product NO ₂ and NH ₃ :	0.71% by wt.
% Product NO ₂ and NH ₃ :	2.54% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.69% by wt.
% Product NO ₂ and NH ₃ :	2.46% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.10205 A
Total Amt. of Coloumbs Passed:	952 C
Amt. of NO ₃ Decomposed:	3.32 mg
Moles of NO ₃ Decomposed:	5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.353E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	41.32 C
Coulombic Efficiency to NH ₃ :	4.34% C
Coulombic Efficiency:	4.34%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.10165 A
Total Amt. of Coloumbs Passed:	948.52 C
Amt. of NO ₃ Decomposed:	3.32 mg
Moles of NO ₃ Decomposed:	5.353E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	5.353E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	41.32 C
Coulombic Efficiency to NH ₃ :	4.36% C
Coulombic Efficiency:	4.36%
Average Coulombic Efficiency to NH ₃ :	4.35%
Standard Deviation:	0.01%
Average Coulombic Efficiency:	4.35%
Standard Deviation:	0.01%

Experiment Name and Purpose	
Experiment Name:	DOENCL-GF71/DOENCL-GF71R
Date:	10/26/2004 -- 10/26/2004
Test Type / Purpose:	Initial pH 9 - Constant Current - 100ppm NO ₃ - High Chloride

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	156	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
Assume:	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.1 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	9331 seconds 2.6 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	10/26/2004 19:08	0.75	7.41	20	1733	866	3.4
Final	10/26/2004 21:44	0.75	7.92	21	1758	875	3.4
<i>Repeat</i>							
Initial	11/02/2004 23:07	0.75	7.41	20	1733	866	3.4
Final	11/03/2004 12:07	0.75	7.94	20	1752	876	3.4

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ⁴⁻ ppm	Cl ⁻ ppm
00:00	9.22	0.1011	24.00	0.00	0.00	45.00	200.00
02:36	8.39	0.1026	24.00	0.00	0.00	45.00	200.00
<i>Repeat</i>							
00:00	8.76	0.1083	24.00	0.00	0.00	45.00	200.00
13:00	8.21	0.1077	24.00	0.00	0.00	36.00	200.00

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	18.00 mg
Mass Balance Final-Initial:	0.00 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	18.00 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.00 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	18.00 mg
Mass Balance Final-Initial:	0.00 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	79.71 mg
% NO ₃ Removed:	0.00%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	0.00%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	- mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	- mg
Total Moles of NH ₃ and NO ₂ Formed:	0.000 mmol
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by wt.
% Split of NH ₃ :	#DIV/0! by wt.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.00% by wt.
% Product NO ₂ and NH ₃ :	0.00% by mol.
% Split of NO ₂ :	#DIV/0! by mol.
% Split of NH ₃ :	#DIV/0! by mol.

Coulombic Efficiency	
Time for Experiment:	9331 s
Current Passed:	0.10185 A
Total Amt. of Coloumbs Passed:	950 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	9331 s
Current Passed:	0.108 A
Total Amt. of Coloumbs Passed:	1007.77 C
Amt. of NO ₃ Decomposed:	0.00 mg
Moles of NO ₃ Decomposed:	0.000E+00 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	0.000E+00 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	- C
Coulombic Efficiency to NH ₃ :	0.00% C
Coulombic Efficiency:	0.00%
Average Coulombic Efficiency to NH ₃ :	0.00%
Standard Deviation:	0.00%
Average Coulombic Efficiency:	0.00%
Standard Deviation:	0.00%

Experiment Name and Purpose	
Experiment Name:	DOEN-C71DI/DOEN-C71RDI
Date:	11/01/2004 -- 10/01/2004
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 100ppm NO ₃ - De-ionized Water

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.02 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/01/2004 21:00	0.75	7.0	20	248.9	124.4	0.3
Final	11/02/2004 10:00	0.73	6.81	20	266.8	133.4	0.5
<i>Repeat</i>							
Initial	11/01/2004 21:22	0.75	7.0	20	248.9	124.4	0.3
Final	11/02/2004 10:22	0.74	7.2	20	273.7	136.8	0.5

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.0260	23.00	0.00	0.00	0.00	0.68
13:00	10.00	0.0257	21.00	0.00	1.40	3.20	0.65
<i>Repeat</i>							
00:00	10.00	0.0210	23.00	0.00	0.00	0.00	0.68
13:00	10.00	0.0235	22.00	0.00	0.74	0.00	0.65

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	15.33 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.02 mg
Total Nitrogen Souces Initial	17.25 mg	Total Nitrogen Souces Final	16.35 mg
Mass Balance Final-Initial:	-0.90 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	16.28 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.55 mg
Total Nitrogen Souces Initial	17.25 mg	Total Nitrogen Souces Final	16.83 mg
Mass Balance Final-Initial:	-0.42 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	67.89 mg
% NO ₃ Removed:	11.13%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat</i>	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	72.09 mg
% NO ₃ Removed:	5.62%
Min. % NO ₃ Removal Req.:	42.03%
Average %NO ₃ Removal:	8.38%
Minimum % NO ₃ Removal:	42.03%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	1.24 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.073 mmol
% Product NO ₂ and NH ₃ :	1.80% by wt.
% Product NO ₂ and NH ₃ :	6.25% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.67 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.039 mmol
% Product NO ₂ and NH ₃ :	0.92% by wt.
% Product NO ₂ and NH ₃ :	3.25% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.36% by wt.
% Product NO ₂ and NH ₃ :	4.75% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.02585 A
Total Amt. of Coloumbs Passed:	1206 C
Amt. of NO ₃ Decomposed:	8.50 mg
Moles of NO ₃ Decomposed:	1.370E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.370E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	105.78 C
Coulombic Efficiency to NH ₃ :	8.77% C
Coulombic Efficiency:	8.77%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.02225 A
Total Amt. of Coloumbs Passed:	1038.10 C
Amt. of NO ₃ Decomposed:	4.30 mg
Moles of NO ₃ Decomposed:	6.924E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	6.924E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	53.44 C
Coulombic Efficiency to NH ₃ :	5.15% C
Coulombic Efficiency:	5.15%
Average Coulombic Efficiency to NH ₃ :	6.96%
Standard Deviation:	2.56%
Average Coulombic Efficiency:	6.96%
Standard Deviation:	2.56%

Experiment Name and Purpose	
Experiment Name:	DOEN-C91DI/DOEN-C91RDI
Date:	11/02/2004 -- 11/02/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 100ppm NO ₃ - De-ionized Water

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Copper		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
Assume:	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.02 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/02/2004 22:57	0.75	9.10	20	300	150	0.5
Final	11/03/2004 11:57	0.74	7.68	20	287.3	145.4	0.5
<i>Repeat</i>							
Initial	11/02/2004 23:22	0.75	9.10	20	300	150	0.5
Final	11/03/2004 12:22	0.74	8.40	20	320	160	0.5

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.0290	24.00	0.00	0.00	0.00	0.24
13:00	10.00	0.0285	23.00	0.00	1.00	0.54	0.73
<i>Repeat</i>							
00:00	10.00	0.0280	24.00	0.00	0.00	0.00	0.24
13:00	10.00	0.0264	22.00	0.00	1.40	0.66	0.77

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.02 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.74 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.76 mg
Mass Balance Final-Initial:	-0.24 mg		

<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	16.28 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	1.04 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.32 mg
Mass Balance Final-Initial:	-0.68 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	75.37 mg
% NO ₃ Removed:	5.44%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	72.09 mg
% NO ₃ Removed:	9.56%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	7.50%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.90 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.053 mmol
% Product NO ₂ and NH ₃ :	1.18% by wt.
% Product NO ₂ and NH ₃ :	4.17% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	1.26 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.074 mmol
% Product NO ₂ and NH ₃ :	1.72% by wt.
% Product NO ₂ and NH ₃ :	5.98% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	1.45% by wt.
% Product NO ₂ and NH ₃ :	5.07% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.02875 A
Total Amt. of Coloumbs Passed:	1341 C
Amt. of NO ₃ Decomposed:	4.34 mg
Moles of NO ₃ Decomposed:	6.995E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	6.995E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	53.99 C
Coulombic Efficiency to NH ₃ :	4.03% C
Coulombic Efficiency:	4.03%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.0272 A
Total Amt. of Coloumbs Passed:	1269.05 C
Amt. of NO ₃ Decomposed:	7.62 mg
Moles of NO ₃ Decomposed:	1.228E-04 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.228E-04 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	94.76 C
Coulombic Efficiency to NH ₃ :	7.47% C
Coulombic Efficiency:	7.47%
Average Coulombic Efficiency to NH ₃ :	5.75%
Standard Deviation:	2.43%
Average Coulombic Efficiency:	5.75%
Standard Deviation:	2.43%

Experiment Name and Purpose		
Experiment Name:	DOEN-GF71DI/DOEN-GF71RDI	
Date:	11/01/2004 -- 11/01/2004	
Test Type / Purpose:	Initial pH 7 - Constant Voltage - 100ppm NO3 - De-ionized Water	

Experimental Conditions		
No. Electrodes:	2	
Electrode Spacing:	34	mm
Cathode Material:	Graphite Felt	
Cathode Size:	60x100	mm
Cathode Area Immersed In Solution:	12000	mm ²
Anode Material:	Ti/TiO2	
Anode Size (w x h):	60 x 100	mm
Anode Area Immersed in Solution:	12000	mm ²
Stirrer Speed:	max	
Total Experimental Time:	778	minutes

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitroge	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.02 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds
	13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/01/2004 21:15	0.75	7.0	20	248.9	124.4	0.3
Final	11/02/2004 10:15	0.73	5.94	20	262.3	131.1	0.4
<i>Repeat</i>							
Initial	11/01/2004 21:10	0.75	7.0	20	248.9	124.4	0.3
Final	11/02/2004 10:10	0.73	5.91	20	254.6	127.4	0.4

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	10.00	0.0345	23.00	0.00	0.00	0.00	0.68
13:00	9.97	0.0382	23.00	0.00	0.26	1.90	0.77
<i>Repeat</i>							
00:00	10.00	0.0280	23.00	0.00	0.00	0.00	0.68
13:00	10.01	0.0238	23.00	0.00	0.13	0.71	0.69

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	16.79 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.19 mg
Total Nitrogen Souces Initial	17.25 mg	Total Nitrogen Souces Final	16.98 mg
Mass Balance Final-Initial:	-0.27 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	17.25 mg	NO ₃ ⁻	16.79 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.09 mg
Total Nitrogen Souces Initial	17.25 mg	Total Nitrogen Souces Final	16.88 mg
Mass Balance Final-Initial:	-0.37 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	74.35 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	42.03%
<i>Repeat</i>	
Amount of NO ₃ Initial:	76.39 mg
Amount of NO ₃ Final:	74.35 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	42.03%
Average %NO ₃ Removal:	2.67%
Minimum % NO ₃ Removal:	42.03%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.23 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.014 mmol
% Product NO ₂ and NH ₃ :	0.31% by wt.
% Product NO ₂ and NH ₃ :	1.12% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.12 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.007 mmol
% Product NO ₂ and NH ₃ :	0.16% by wt.
% Product NO ₂ and NH ₃ :	0.56% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.23% by wt.
% Product NO ₂ and NH ₃ :	0.84% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.03635 A
Total Amt. of Coloumbs Passed:	1696 C
Amt. of NO ₃ Decomposed:	2.04 mg
Moles of NO ₃ Decomposed:	3.283E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.283E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	25.34 C
Coulombic Efficiency to NH ₃ :	1.49% C
Coulombic Efficiency:	1.49%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.0259 A
Total Amt. of Coloumbs Passed:	1208.40 C
Amt. of NO ₃ Decomposed:	2.04 mg
Moles of NO ₃ Decomposed:	3.283E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.283E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	25.34 C
Coulombic Efficiency to NH ₃ :	2.10% C
Coulombic Efficiency:	2.10%
Average Coulombic Efficiency to NH ₃ :	1.80%
Standard Deviation:	0.43%
Average Coulombic Efficiency:	1.80%
Standard Deviation:	0.43%

Experiment Name and Purpose	
Experiment Name:	DOEN-GF91DI/DOEN-GF91RDI
Date:	11/02/2004 -- 11/03/2004
Test Type / Purpose:	Initial pH 9 - Constant Voltage - 100ppm NO ₃ - De-ionized Water

Experimental Conditions			
No. Electrodes:	2		
Electrode Spacing:	34	mm	
Cathode Material:	Graphite Felt		
Cathode Size:	60x100	mm	
Cathode Area Immersed In Solution:	12000	mm ²	
Anode Material:	Ti/TiO ₂		
Anode Size (w x h):	60 x 100	mm	
Anode Area Immersed in Solution:	12000	mm ²	
Stirrer Speed:	max		
Total Experimental Time:	778	minutes	

Constants	
Density of Water @ 20°C	998.23 g/L
Molecular Mass of NaNO ₃	84.95 g/mol
Molecular Mass of NO ₃	62.04 g/mol
Molecular Mass of NO ₂	46.05 g/mol
Molecular Mass of NH ₃	17.04 g/mol
Molecular Mass of N	14.01 g/mol
Faradays Constant F	96485 C/mol

Experiment Time Calculation	
Using Faraday's Law: time = (number of electrons*Faradays Constant*moles)/Current	
<u>Assume:</u>	
No. of electrons to make all NO ₃ to NH ₃	8 electrons
Concentration of NO ₃ in Initial Solution	100 mg/L or ppm
Concentration of NO ₃ in Initial Solution as Nitrogen	22.58 mg/L
Volume of Initial Solution	0.75 L
Current	0.02 A
Theoretical Time to Decompose all Nitrate in Solution	
Experiment Run Time	46656 seconds 13.0 hours

Solution Conditions							
	Exp. Time hrs.	Volume L	pH	Temp. °C	Conductivity µS	TDS ppm	NaCl% %
Initial	11/02/2004 23:15	0.75	9.10	20	300	150	0.5
Final	11/03/2004 12:15	0.74	6.68	20	284.9	142.1	0.5
<i>Repeat</i>							
Initial	11/02/2004 23:07	0.75	9.10	20	300	150	0.5
Final	11/03/2004 12:07	0.73	6.82	20	288.7	144.3	0.5

Raw Data							
Elpsd. Time hrs.	Voltage V	Current A	NO ₃ (as N) ppm	NO ₂ (as N) ppm	NH ₃ (as N) ppm	SO ₄ ²⁻ ppm	Cl ⁻ ppm
00:00	9.96	0.0322	24.00	0.00	0.00	0.00	0.24
13:00	9.98	0.0342	24.00	0.00	0.15	1.30	0.83
<i>Repeat</i>							
00:00	10.01	0.0247	24.00	0.00	0.00	0.00	0.24
13:00	10.02	0.0278	24.00	0.00	0.10	0.78	0.79

Mass Balance on Nitrogen			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.76 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.11 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.87 mg
Mass Balance Final-Initial:	-0.13 mg		
<hr/>			
<i>Repeat</i>			
<u>Nitrogen Souces Initial (as mg Nitrogen)</u>		<u>Nitrogen Souces Final (as mg Nitrogen)</u>	
NO ₃ ⁻	18.00 mg	NO ₃ ⁻	17.52 mg
NO ₂ ⁻	0.00 mg	NO ₂ ⁻	0.00 mg
NH ₃	0.00 mg	NH ₃	0.07 mg
Total Nitrogen Souces Initial	18.00 mg	Total Nitrogen Souces Final	17.59 mg
Mass Balance Final-Initial:	-0.41 mg		

NO ₃ Removal Efficiency	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	78.65 mg
% NO ₃ Removed:	1.33%
Min. % NO ₃ Removal Req.:	44.44%
<i>Repeat</i>	
Amount of NO ₃ Initial:	79.71 mg
Amount of NO ₃ Final:	77.58 mg
% NO ₃ Removed:	2.67%
Min. % NO ₃ Removal Req.:	44.44%
Average %NO ₃ Removal:	2.00%
Minimum % NO ₃ Removal:	44.44%

NO ₂ /NH ₃ Generation	
Total Wt. of NH ₃ and NO ₂ Formed:	0.14 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.008 mmol
% Product NO ₂ and NH ₃ :	0.17% by wt.
% Product NO ₂ and NH ₃ :	0.62% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Repeat</i>	
Total Wt. of NH ₃ and NO ₂ Formed:	0.09 mg
Total Moles of NH ₃ and NO ₂ Formed:	0.005 mmol
% Product NO ₂ and NH ₃ :	0.11% by wt.
% Product NO ₂ and NH ₃ :	0.41% by mol.
% Split of NO ₂ :	0.00% by wt.
% Split of NH ₃ :	100.00% by wt.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.
<i>Average</i>	
% Product NO ₂ and NH ₃ :	0.14% by wt.
% Product NO ₂ and NH ₃ :	0.52% by mol.
% Split of NO ₂ :	0.00% by mol.
% Split of NH ₃ :	100.00% by mol.

Coulombic Efficiency	
Time for Experiment:	46656 s
Current Passed:	0.0332 A
Total Amt. of Coloumbs Passed:	1549 C
Amt. of NO ₃ Decomposed:	1.06 mg
Moles of NO ₃ Decomposed:	1.713E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	1.713E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	13.22 C
Coulombic Efficiency to NH ₃ :	0.85% C
Coulombic Efficiency:	0.85%
<hr/>	
<i>Repeat</i>	
Time for Experiment:	46656 s
Current Passed:	0.02625 A
Total Amt. of Coloumbs Passed:	1224.72 C
Amt. of NO ₃ Decomposed:	2.13 mg
Moles of NO ₃ Decomposed:	3.426E-05 mol
Amt. Of NO ₃ Decomposed to NO ₂	0 mol
Amt. Of NO ₃ Decomposed to NH ₃	3.426E-05 mol
Number of Electrons to NO ₂ :	2 mol
Number of Electrons to NH ₃ :	8 mol
Coulombs to NO ₂ :	0.00 C
Coulombs to NH ₃ :	26.45 C
Coulombic Efficiency to NH ₃ :	2.16% C
Coulombic Efficiency:	2.16%
Average Coulombic Efficiency to NH ₃ :	1.51%
Standard Deviation:	0.92%
Average Coulombic Efficiency:	1.51%
Standard Deviation:	0.92%