Presented in this report is an investigation of the design, construction, and operation of scaled-up cloth electrode assembly (CEA) air-cathode microbial fuel cells (MFCs). 40 mL MFCs were designed and constructed using either single or double CEAs with carbon cloth electrodes. The MFCs were evaluated using acetate media, municipal wastewater, and brewing wastewater with mixed culture originally enriched from municipal wastewater. A single CEA MFC using acetate reached a power density of 3450 mW/m² (860 W/m³), 192% of a similar 2.5 mL MFC. This was possibly due to increased platinum loading and an improved microbial community. Double CEA MFC power density losses were likely due to biofilm accumulation between CEA layers, changes in the microbial community, and greater areas for oxygen diffusion into media. The 12 W/m³ generated with municipal wastewater was much lower than that produced with acetate medium due to low chemical oxygen demand (COD) and pH buffer concentrations in the wastewater. 47% of COD in brewing wastewater was removed while producing a power density 81% lower than acetate (160 W/m³) due to the lack of sufficient pH control. Further efforts are needed to improve CEA fabrication to prevent unintended biofilm growth within the CEA structure and optimize the environmental and operational condition for wastewater treatment.

Keywords: microbial fuel cell, wastewater treatment

Corresponding e-mail address: nathanjfulton@gmail.com
Design, Construction, and Evaluation of a Scaled-Up
Air-Cathode Microbial Fuel Cell

by
Nathan J. Fulton

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I understand that my project will become part of the permanent collection of Oregon State University, University Honors College. My signature below authorizes release of my project to any reader upon request.

______________________________
Nathan J. Fulton, Author
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DEDICATION

This thesis is dedicated to Jennifer Harvester, who never gives up.
INTRODUCTION

Increasing worldwide demand for efficient and renewable energy sources has heightened international interest in hydrogen fuel cells [1]. Microbial fuel cell (MFC) technology provides a completely new approach for producing renewable energy from wastewater while accomplishing wastewater treatment simultaneously. Microorganisms in MFCs are capable of consuming a wide selection of environmental fuels, including human, animal, and industrial wastes [2]; a capability that could be utilized to produce energy during the biological stage of wastewater treatment processes. Such applications of MFCs show potential as a renewable energy source and wastewater treatment process.

A typical air-cathode MFC is composed of an anode, cathode, and proton exchange membrane. Electrons and protons are produced from microbes on the anode [3]. Electrons flow through the circuit forming electric current. Proton carriers (including pH buffers) facilitate the transfer of protons from the anode to the cathode, allowing oxygen to bind with protons and electrons on the cathode, forming water, as illustrated in Figure 1-1 [4-6]. The most significant sources of internal resistance have been determined by Fan et al. to be the cathode surface area and electrolyte [7].
Increasing the power density and decreasing production costs of microbial fuel cells are two of the most significant challenges for the practical use of MFCs in wastewater treatment and power generation. Recently, a significant increase in power density has been obtained in Dr. Liu’s lab at Oregon State University, achieving a power density of 1.5 KW/m3, a level that is comparable to the anaerobic digestion (Fan et al., 2008). However, this study was conducted in a small scale MFC with a total volume of 2.6 ml and using acetate medium solution. Investigating the effects of scale-up and the use of municipal and industrial wastewaters is an essential step toward the implementation of MFCs in municipal or industrial processes [8].
The research presented in this thesis confronted the aforementioned issues by designing, constructing, and evaluating a scaled-up air-cathode microbial fuel cell with similar design and evaluated using municipal and brewing wastewater. Municipal wastewater was chosen because it is highly available waste stream and could be used as a potential carbon source for industrial scale MFCs in the future, while brewing wastewater was chosen because it contains high strength and nutrient rich fuel for MFCs. To further mimic large-scale municipal and industrial wastewater treatment conditions, the MFCs were designed for continuous flow, allowing the reactor to be configured for a single pass through the MFCs, or for recirculation of the nutrient stream in both continuously fed or batch fed regimes.
2 MATERIALS AND METHODS

2.1 Design and Construction of a scaled-up single CEA MFC

The scaled-up single CEA (Cloth Electrode Assembly) MFC was initially designed with a pair of nested male and female frame pieces with the CEA located on the bottom of the female piece. The male piece was placed on top of the CEA and two gaskets; one gasket on top of the CEA, and another located between the two frame pieces. The frame pieces were compressed together by ten 18-8 s/s, 10-24 x 1 machine screws and bolts around the outer rim of the frame to avoid contact with the electrodes. A multi-channel influent distributor and effluent collector were integrated into the frames.

The initial single CEA design was abandoned due to leakage complications and manufacturing complexity. The design used for the scaled-up double CEA MFC was modified with a rubber sheet to replace the top CEA. The improved, modular design eliminated leakage, improved durability, and significantly decreased design and fabrication complexity. The multi-channel influent distributors and effluent collectors were also abandoned due to clogging, and were modified to a straight dual-channel design to further simplify construction. See Figure 2-1 for the improved single CEA MFC design assembly. Additional schematics of the MFC are located in Appendix A.
Frame pieces were constructed from acrylic sheeting, while gaskets were cut from 0.07” rubber sheeting. Carbon cloth was used for both anode and cathode construction. A 25% polyester, 75% rayon cloth was used to separate the cathode and anode. Further information on the preparation of the cathode and anode can be seen in Section 2.3.
2.2 Design and Construction of a scaled-up double CEA MFC

The scaled-up double CEA MFC was designed with considerations from the initial single CEA MFC design, significantly decreasing fabrication time, materials, and leakage. Three identical 0.25” thick frames were produced with a 2.25x10” opening for the CEA. The width of the frame sides were 7/8” while the inlet and outlet sides were 1” wide. Inlet and outlet channels were drilled through the narrow ends of each frame with a #30 bit and were placed 0.6” to either side of the MFC centerline.

The first CEA was placed on top of a 0.07” thick rubber gasket which was cut to the inner edge of the frame. The next frame piece was placed on top of the first CEA, while the second CEA was placed on top of the second frame. Another gasket was placed on top of the second CEA and the final frame piece was placed upon the gasket. Figure 2-2 shows the assembly schematics for the double CEA MFC. Additional schematics of the MFC are located in Appendix A.
2.3 CEA preparation

Due to lower material costs and higher power density generation than MFCs using PEMs, CEAs were used in the scaled-up MFCs [2,6,9]. The cathodes were prepared by first adding diffusion layers, then by adding PTFE layers, and finally by adding a catalyst layer. 2.5 mg of carbon powder per cm² of cathode area and 30% PTFE at 15 ul per mg of carbon powder was
mixed for 2-4 hours. The mixture was uniformly brushed onto one side of the cathode and dried. This was repeated until 2.5 mg per cm$^2$ of the mixture had been applied to the cathode.

One layer of 60% PTFE was brushed onto the same side of the cathode. The cathode was dried and then heated at 380°C for 30 minutes. This process was repeated until 4 mg per cm$^2$ of the PTFE was present on the cathode.

Onto the other side of the cathodes, a catalyst layer of 1 mg platinum per cm$^2$ of cathode was applied [10]. Platinum powder was mixed for 5-8 hours with 7 uL of 5% Nafion per mg of platinum powder. Individual layers of the mixture were applied to the cathode and allowed to dry until all of the solution had been applied.

The carbon cloth anodes were degreased by soaking in ethanol and then rinsed in de-ionized water. Each CEA was constructed by taping a titanium wire to the outer edge of the cathode, while simultaneously taping the cathode to the rubber gasket. The rayon/polyester cloth was simultaneously taped to the anode with a titanium wire about the edge of the anode. The two layers were then stacked together, with the rayon/polyester cloth between the anode and the cathode. The electrical tape used on the layers of the CEA also served as a gasket, preventing leakage around the titanium wires and cloth layers.

2.4 Operation of a scaled-up single and double CEA MFCs on Acetate

Acetate media used in the evaluation of the scaled-up single CEA MFC design contained 15 g/L sodium acetate, 0.62 g/L NH4Cl, 0.26 g/L KCl, 5.85 g/L NaH2PO4·H2O, 15.5 g/L Na2HPO4·7H2O [2], and 12.5 mL/L of mineral and vitamin solutions as defined by Lovley and Phillips [11]. Media at approximately 7.1 mL/hour was fed into a recycle loop running at 1.2
L/hour as illustrated in Figure 2-3. Acetate media diluted to 25 mM was added to the recycle reservoir prior to MFC startup.

A Type 602-N decade resistance box (rheostat) by General Radio Co. was used to provide a circuit load between the anode and cathode. A Keithley 2700 Multimeter/Data Acquisition System was used to record current and voltage in intervals of 2 minutes. Figure 2-4 illustrates the circuit layout. The media was fed into the recycle loop by means of a peristaltic VWR variable flow mini-pump, while the recycle was circulated by a Masterflex L/S Digital Standard Drive peristaltic pump by Cole-Parmer Instrument Company. Resistance was manually
adjusted to maintain a potential between 0.25 V to 0.5 V in order to achieve maximum power production. If adequate resistance is not applied to the circuit, significant potential loss occurs due to the limited mass transfer of protons. Resistances higher than necessary can bottleneck the flow of electrons through the circuit, thus limiting the electrochemical gradient which transports protons to the cathode.

![MFC Circuit Layout](image)

Figure 2-4 MFC circuit layout

2.5 Operation of a scaled-up double CEA MFC on Municipal Wastewater

Municipal wastewater (post primary clarification) was acquired from the Corvallis wastewater reclamation plant in Corvallis, Oregon. 2 L samples were transported on ice and
refrigerated before use. The 2 L samples were stored in a cooler with ice throughout the
duration of the experiment. Municipal wastewater was fed into the recycle loop as described in
Section 2.4. CEA assemblies were washed to remove biofilm from previous experiments and
municipal wastewater at a 1/4th dilution was added to the recycle reservoir before startup of the
MFC. A Keithley 2700 Multimeter/Data Acquisition System was used to record current and
voltage every 4 minutes. Further operation procedures were conducted as described in Section
2.4.

2.6 Operation of a scaled-up double CEA MFC on brewing wastewater

Brewing wastewater washout was acquired from the Block 15 Brewery boil kettle in
Corvallis, Oregon and was fed into the recycle loop as described in Section 2.4. The MFC was
started on acetate (as described in Section 2.4) and was transferred to brewing wastewater for
the first experiment, while the second experiment was started on brewing wastewater with a
1/4th dilution added to the recycle reservoir and a 100 mM phosphate buffer. Biological
inoculation was not added to the MFC in the second experiment. The MFC was transitioned to a
batch-fed regime during the second experiment, administering approximately 30 mL of brewing
wastewater each day and periodically injecting phosphate buffer according to pH. CEA
assemblies were washed to remove biofilm from previous experiments. A Keithley 2700
Multimeter/Data Acquisition System was used to record current and voltage every 4 minutes.
Further operation procedures were conducted as described in Section 2.4.
2.7 Analyses

MFC operating voltage and current was monitored by a Keithley 2700 Multimeter/Data Acquisition System in conjunction with a Windows XP based computer with Microsoft Excel. Chemical oxygen demand (COD) was determined by a closed reflux, colorimetric method as described by 5220 D. in the Standard Methods for the Examination of Water and Wastewater [12]. Colorimetric readings were recorded from a Shimadzu PharmaSpec UV-1700 UV-visible spectrophotometer, while COD samples were digested in an Omegalux LMF-3550 oven.
3 RESULTS AND DISCUSSION

3.1 Scaled-up single CEA MFC

The scaled-up microbial fuel cells were tested with various nutrient sources in order to identify efficiency losses due to scale-up, to identify methods of operation for the use of industrial and municipal waste streams in a scaled-up reactor, and to determine significant holdbacks associated with scaled-up microbial fuel cell systems with both synthetic and industrial wastewaters. Testing a scaled-up single CEA MFC on a controlled and commonly used substrate is required for characterizing the loss of power density due to scale-up. This is achieved by comparing power density results of a scaled-up, single CEA MFC to a smaller single CEA MFCs with similar construction. Studying power density in the scaled-up single CEA MFC also serves as a benchmark for comparison with other scaled-up fuel cells with different CEA configurations, such as the double CEA MFC discussed in Section 3.2. The scaled-up single CEA MFC was initially designed with nesting frame pieces which held the CEA into place, illustrated by Figure 3-1. However, when the nutrient feed system was installed and the MFC filled with media, continuous leaking occurred and could not be repaired. Leak repairs were hindered by the inability to access to the outside edge of the CEA and inner gasket, thus points of leakage could not be located. Media that had leaked into the space between the two gaskets also prevented the crystallization of minerals at leak points which would have otherwise sealed small leaks. Because of the complexity of the design, holes had to be drilled through both pieces of the frame for the wires connected to the anode and cathode, creating small channels linking the outside of the MFC to the space between the two gaskets. This channel allowed media that had leaked past the first gasket to bypass the second gasket. Due to the nestling design of the frame
pieces, the outer edge of the cover frame piece and the inner edge of the CEA frame piece were thin and eventually failed after prolonged use.

![Diagram of a simple flat frame design](image)

**Figure 3-1** Initial single CEA MFC design

After the shortfalls of the initial MFC design were realized, a new scaled-up CEA MFC was designed to allow access to the CEA and gasket edges while the MFC was fully assembled. Other design considerations included the need to strengthen the frame near the CEA and bolts, to reduce fabrication complexity, and to make the system modular, such that the frame assembly could be arranged to make single CEA MFCs, double CEA MFCs, and stacks of MFCs. The result was a simple flat frame design composed of three identical frame pieces. Single CEA MFCs were constructed by installing a CEA on one side of the center frame and a rubber sheet on the other, as illustrated in Figure 3-2. Figure 3-3 shows how double CEA MFCs were assembled by installing a CEA on either side of the MFC.
The improved design also allows for the compact stacking of MFCs by permitting two cathodes of separate MFCs to share a common frame, as shown in Figure 3-4. The influent and
Effluent holes on the cathode-side frames also allow for the control of gas concentrations which interact with the cathodes. Leaking was minimal during the first few days of operation and ceased shortly thereafter. The MFC proceeded without further incident of leaks throughout multiple experiments, some of which involved disassembly and reassembly of the MFC.

Figure 3-4  MFC stack assembly
Figure 3-5 shows the power density response of a single CEA MFC running in continuous flow with recycle on 100 mM Acetate media in the feed stream and a 100 mM phosphate buffer. The experiment was conducted over the time period of approximately one month. Power generation peaked at approximately 3450 mW/m² and a volumetric power density of 860 W/m³. The 40 mL scaled-up single CEA MFC with an anode surface area of 100 cm² produced approximately 192% of the power density achieved by a 2.5 mL single CEA MFC with similar construction and buffer concentration but lower platinum loading, which produced up to 1800 mW/m² [6].

![Figure 3-5 Single CEA MFC](image-url)
3.2 Scaled-up double CEA MFC

Recognizing power generation differences between small MFCs, scaled-up double CEA MFCs and scaled-up single CEA MFCs help identify potential sources of power generation loss and indicate the magnitude of the loss, thus contributing to the redesign of MFCs for higher power production and further scale-up. Figure 3-6 illustrates the power density generated by a double CEA MFC utilizing the same feed solution as the experiment discussed in Section 3.1. According to the results obtained in Section 3.1, each CEA should have run at approximately 3450 mW/m$^2$. CEA2 ran at approximately 1700 mW/m$^2$, comparable to the results of a 2.5 mL double CEA MFC, which achieved 1800 mW/m$^2$ [2]. However, CEA1 ran at approximately 1500 mW/m$^2$, resulting in a volumetric power density of 800 W/m$^3$ for the double CEA MFC design, 60 W/m$^3$ less than the single CEA MFC design. This could be the result of increased diffusion of oxygen into the media due to increased CEA area, a shift in the makeup of the microbial community, biofilm accumulation between the layers of CEA1, and of cathode age, as it was previously used in the single CEA MFC described in Section 3.1. The decrease in power density between days 4 and 5 was due to a temporary pump failure.
3.3 Performance of scaled-up double CEA MFC using municipal wastewater

Municipal wastewater is a highly available waste stream and could be used as a potential carbon source for industrial scale MFCs in the future. Understanding operational procedures required to successfully utilize municipal wastewater in MFCs is an essential step toward the application of the technology in wastewater reclamation plants. Observing the power densities generated by the double CEA MFC with municipal wastewater and comparing results to power densities generated by acetate media helped identify issues associated with carbon source, buffers, and nutrients for MFC systems. Figure 3-7 illustrates the power density generated by the double CEA MFC on municipal wastewater, followed by the transfer to 100 mM acetate media. Power density while using municipal wastewater peaked at 20 mW/m² for
CEA1 and 29 mW/m² for CEA2 (12 mW/m³). COD tests indicate that the wastewater had been degraded to below 200 mg/L (Table 3-1), thus was an insufficient source of biodegradable material for the MFC configuration used in this test. This could have been caused by degradation of nutrients within the feed tubing, where the media flow rate is only 7.1 mL/hour. Additional information regarding the COD test is located in Appendix B.

Table 3-1  COD test results for municipal wastewater

<table>
<thead>
<tr>
<th>Description</th>
<th>Date of Sampling</th>
<th>Sample dilution ratio</th>
<th>response (A)</th>
<th>COD mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Municipal WW in</td>
<td>4/19/09</td>
<td>1</td>
<td>0.014</td>
<td>37</td>
</tr>
<tr>
<td>Municipal WW in</td>
<td>4/19/09</td>
<td>1</td>
<td>0.000</td>
<td>0</td>
</tr>
<tr>
<td>Municipal WW out</td>
<td>4/19/09</td>
<td>1</td>
<td>0.026</td>
<td>69</td>
</tr>
<tr>
<td>Municipal WW out</td>
<td>4/19/09</td>
<td>1</td>
<td>0.000</td>
<td>0</td>
</tr>
<tr>
<td>Municipal WW in</td>
<td>4/29/09</td>
<td>1</td>
<td>0.061</td>
<td>162</td>
</tr>
<tr>
<td>Municipal WW in</td>
<td>4/29/09</td>
<td>1</td>
<td>0.002</td>
<td>6</td>
</tr>
<tr>
<td>Municipal WW out</td>
<td>4/29/09</td>
<td>1</td>
<td>0.011</td>
<td>28</td>
</tr>
<tr>
<td>Municipal WW out</td>
<td>4/29/09</td>
<td>1</td>
<td>0.057</td>
<td>149</td>
</tr>
</tbody>
</table>

The following use of acetate media and phosphate buffer indicated that the low municipal wastewater concentration or lack of proton carriers (pH buffer) caused the low power generation. Possible solutions to the low power density recorded with municipal wastewater include using a higher feed flow rate, shorter feed tube length, frequent restocking of fresh wastewater samples, and a refrigerated container for the feed. Starting municipal wastewater experiments in MFCs with an acetate media and phosphate buffer start-up would further improve power density by establishing a strong electrochemically active microbial community.

The acetate power generation showed a decrease in power density when compared to the
results of Sections 3.1 and 3.2, possibly due to the further loss of platinum catalyst by the washing of biofilm from the CEAs between experiments, and by the accumulation of biofilm between the anode and cathode, and a less electrochemically active.

Figure 3-7  Double CEA with municipal wastewater

3.4 Performance of scaled-up double CEA MFC using brewing wastewater

Brewing wastewater is a COD rich, non-toxic nutrient stream and is extensively available worldwide, but few studies have made use of brewery wastewater in MFCs [13-15]. Brewing wastewaters have been confirmed as an appropriate substrate for other forms of anaerobic treatment, but are typically treated by expensive aerobic processes, creating a demand for alternatives [16]. The high COD content of brewing wastewater was the central motive for use
in the scaled-up double CEA MFC, as complications with power production from municipal wastewater were rooted in its lower COD concentration. Brewing wastewater was fed into the double CEA MFC after being started on a 100 mM acetate media with 100 mM phosphate buffer. Additional buffer was not added, as the MFC was assumed to maintain sufficient bicarbonate buffer concentrations through the capture of carbon dioxide in the recycle reservoir. Figure 3-8 shows that the power production quickly peaked at 390 mW/m² for CEA1 and 260 mW/m² for CEA2 (160 W/m³). Both CEAs promptly decreased in power density thereafter due to acid accumulation within the MFC, which had lowered the pH of the recycled media to approximately 3.3. The initial intention of this experiment was to measure the power density and COD oxidation response to changes in feed flow rates. However, power production did not stabilize until after the pH had severely inhibited power generation.

![Figure 3-8](image-url)  
**Figure 3-8** Double CEA with acetate to brewing wastewater
COD analysis of the samples taken at day 24 demonstrated that the average removal of COD was 50 mg/hour, 1 kg/h-m\(^3\) of reactor volume, or approximately 47% (Table 3-2), indicating that the microbial community had shifted from a electrochemically active culture to an acidophilic community capable of oxidizing COD, but less capable of exocellular electron transfer.

Table 3-2  COD test results for brewing wastewater

<table>
<thead>
<tr>
<th>Description</th>
<th>Date of Sampling</th>
<th>Sample dilution ratio</th>
<th>response (A)</th>
<th>COD mg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brew in (from feed tube)</td>
<td>6/7/09</td>
<td>0.05</td>
<td>0.300</td>
<td>15783</td>
</tr>
<tr>
<td>Brew in (from feed tube)</td>
<td>6/7/09</td>
<td>0.05</td>
<td>0.262</td>
<td>13777</td>
</tr>
<tr>
<td>Brew out</td>
<td>6/7/09</td>
<td>0.05</td>
<td>0.148</td>
<td>7757</td>
</tr>
<tr>
<td>Brew out</td>
<td>6/7/09</td>
<td>0.05</td>
<td>0.148</td>
<td>7789</td>
</tr>
</tbody>
</table>

CEAs were cleaned and tested with brewing wastewater in a second experiment without an acetate startup or microbial inoculation. This was done to determine if microorganisms naturally occurring in the brewing wastewater were capable of exocellular electron transfer. Figure 3-9 shows no significant power production, with a maximum power density of 14 mW/m\(^2\) for CEA1 and 20 mW/m\(^2\) for CEA2 (8.5 W/m\(^3\)), thus indicating that the biofilm formed on the MFC anodes were likely comprised of electrochemically inactive microbes.
Figure 3-9  Double CEA MFC with brewing wastewater without inoculation
FUTURE RESEARCH

Scaled-up MFCs introduce a multitude of engineering problems which must be solved before further scaling-up. Biofilm growth between the anode and the cathode was observed after prolonged operation of the MFC, likely inhibiting the mass transfer of proton carriers between the anode and cathode. Unintended gaps between CEA layers also inhibit mass transfer of protons due to larger mass transfer distances and due to the biological growth within the gaps. Losses of power density due to scale-up from the single to double CEA MFCs were possibly due to changes in the microbiological composition within the MFC, the deformation of CEAs, as seen in Figure 4-1, creating the aforementioned gaps between CEA layers. Further research on CEA fabrication processes is required to reduce CEA layer spacing, to prevent washout of the platinum catalyst, and to improve cathode shape and rigidity. Future research will focus on the bonding of CEA layers with various materials, such as hydrogels, thus preventing the separation of CEA layers in scaled-up MFCs, reducing washout of platinum catalyst, and preventing unintended biological growth. Another possible source of internal resistance in larger MFCs include the greater distance required for electrons to travel along the electrodes [8].
The introduction of municipal wastewater resulted in low power densities due to insufficient COD concentration, possibly caused by insufficient cooling for preservation of the wastewater influent, and/or by an insufficient feed flow rate. Future experimentation with municipal wastewater will include a refrigerated feed and a higher ratio of feed to recycle flow rate. Brewing wastewater was also low in power production, likely due to the lack of pH buffer in the first experiment, and the dominance of bacteria incapable of exocellular electron transfer in the second experiment. During the first experiment with brewing wastewater, COD samples indicated that the MFC was biologically active despite the lack of power production, removing COD at a rate of 1 kg/m³-h. Future experiments involving brewing wastewater will include methods of pH control and an inoculation of exoelectrogens. Future research may involve the modeling and prediction of bicarbonate buffer concentration in continuous flow MFCs due to the biological production of carbon dioxide. Future research in acidophilic bacteria capable of exocellular electron transfer could significantly decrease operation costs for MFC wastewater treatment in breweries by eliminating the need for pH adjustment [17].
REFERENCES


APPENDICES
Appendix A: MFC Design

Figure A 1  Assembled double CEA MFC
Figure A 2   MFC modular frame design
Figure A 4  MFC assembly
Appendix B: COD Test

Error! Reference source not found. shows sample descriptions, instrument response, and COD, calculated using the standard curve shown in Figure B 1.

Table B 1   Tabulated COD data and results

<table>
<thead>
<tr>
<th>Description</th>
<th>Date of Sampling</th>
<th>Sample dilution ratio</th>
<th>response (A)</th>
<th>Calculated COD mg/L</th>
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<td>13777</td>
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<td>0.148</td>
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y = 2.6291x  
$R^2 = 0.9847$

Figure B 1   Standard curve for closed reflux, colorimetric COD test