

308 --- Q200008310007
Scientific Notebook # 341

LABORATORY NOTEBOOK

CNWRA/SwRI

308 --- Q200008310007
Scientific Notebook # 341

NOTEBOOK NO. _____
ISSUED TO V. JAIN / P. DESHPANDE / M. Nugent
ON 5/26/99 19
DEPARTMENT CNWRA
RETURNED _____ 19

CNWRA
CORRECTION
COR 341

—SCIENTIFIC NOTEBOOK CO.—
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INSTRUCTIONS

1. **The primary purpose of this notebook is to protect your and the Company's Patent-Rights by keeping records of all original work in a form acceptable as evidence if any legal conflict arises.**
2.
 - When starting a page, enter the title, project number, and book number.
 - Use ink for permanence -- avoid pencil.
 - Record your work as you progress, including any spur-of-the-moment ideas which may be developed later.
 - Avoid making notes on loose paper to be recopied.
 - Record your work in such a manner that a co-worker can continue from where you stop. You might be ill and to protect your priority it could be urgent that the work continue while you are absent.
3.
 - Give a complete account of your experiments and the results, both positive and negative, including your observations.
 - Record all diagrams, layouts, plans, procedures, new ideas, or anything pertinent to your work including the details of any discussions with suppliers, or other people outside the Company.
 - Do not try to erase any incorrect entries; draw lines deleting them, note the corrections, sign and date the changes. This extra care is worthwhile because of the necessity of original data to prove priority of new discoveries.
4.
 - After entering your data, sign and date the entries.
 - Explain your work to at least two witnesses who are not co-inventors, and have them sign and date the pages in the place provided.
- Record the names of operators and witnesses present during any demonstration and have at least two witnesses sign the page. If no witnesses are present during an experiment of importance, repeat it in the presence of two witnesses.
5. Since computer programs can be patented these instructions apply to the development of computer software. In this case a description of the structure and operation of the program should be recorded in the notebook, together with a basic flow diagram which illustrates the essential features of the program. In the course of developing the code, the number of lines of code written each day should be recorded in the notebook, together with a statement of the portion of the flow diagram to which the section of code is directed.
6. This notebook and its contents are the exclusive property of the Company. It is confidential and the contents are not to be disclosed to anyone unless authorized by the Company. You must return it when completed, upon request, or upon termination of employment. It should be kept in a protected place.
If loss occurs, notify your supervisor immediately, and make a written report describing the circumstances of the loss.

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INTRODUCTION :

Hazardous and low activity wastes glass are planned for disposal in shallow sub-surface sites located in the vadose or unsaturated zone within the United States of America. An experimental technique has already been developed (by Pacific Northwest National Laboratory, Richland, Washington 99352) to test waste forms under unsaturated pressurized flow conditions, that is representative of the real conditions present at these sites. This technique is known as Pressurised Unsaturated Flow Test or PUF.

'PUF' TEST (PRESSURIZED UNSATURATED FLOW TEST)

AIM:

Determination of the chemical changes in water that has reacted with LAW Glass packed in a column under unsaturated pressurized conditions.

THEORY:

In putting together a system to evaluate the dissolution characteristics of barrier materials under the unsaturated pressurized conditions, our efforts incorporated the previous work done by the researchers of Pacific Northwest National Laboratory.

In operating a column under the unsaturated and pressurized conditions, the aim is to maintain a steady-state, vertical water flow while maintaining a uniform water content throughout the column. The principle for having such conditions is the Darcy's Law as modified by Richards:

$$J_w = -K(\Psi_m) \delta \Psi / \delta z$$

where

J_w = volumetric flux density, m/s

Ψ = water potential = matric potential (Ψ_m) + gravitational potential (Ψ_g), m

$K(\Psi_m)$ = conductivity as a function of matric potential, m/s

Z = length of column, m

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Date

Aug 20th 99

Invented by

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Date

08/20/99

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If the moisture content is uniform throughout the column, then

$$J_w = K(\Psi_m)$$

which simply states that under uniform water content conditions, the volumetric flux density of water is equal to the unsaturated hydraulic conductivity.

The hydraulic conductivity of a porous medium is a very important factor in obtaining unsaturated conditions. Unlike in the saturated conditions, where the large pores are responsible for conducting fluid, in the unsaturated conditions, it is the small pores that conduct the fluid. This is because the large pores drain first and become unable to transmit fluid.

Thus, the flow becomes more restricted through a smaller cross-sectional area resulting in an increase in flow path tortuosity. Gradually, as the void space opens, the matric potential reduces, which can be measured by an increase in suction and equated to the difference between the pressure in the water phase and the pressure in the air phase.

Thus, as the water content decreases, a point is reached after initial drainage at relatively high matric potentials where small decreases in water content result in large decreases in water potential. Hence, there is a greater decrease in hydraulic conductivity with smaller decrease in water content.

A small inflow rate is required to maintain low water contents inside the column, and hence, a flow rate of 2 ml/day is chosen to be of interest.

PUF TEST DESIGN:

In order to set up the equipment for the PUF (Pressurized Unsaturated Flow Test), the following components are being used:

- **PUF Column:** This column is fabricated with PEEK (PolyEtherEtherKetone, unfilled grade), which has outstanding chemical poperties. Its internal volume is 21.5 cc, and will be packed with a calculated amount of LAW Glass, in order to obtain a specific porosity.
- **Porous Titanium Plate:** This porous plate will have a pore size of 0.2 microns, and will be sealed at the bottom of the column. It will hold the glass powder above it. It will also let water to pass through it, and prevent the passage of air, as long as the applied pressure differential does not exceed the air entry relief pressure or bubble pressure. In this case, the bubble pressure is about 3 psi (corresponding to a pore size of 0.2 microns), which will enable the researchers to pressurize the column to about 2-2.5 psi. Titanium was chosen as it has good wetting properties and also has excellent corrosion resistance.

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		<u>Deshpande</u>	

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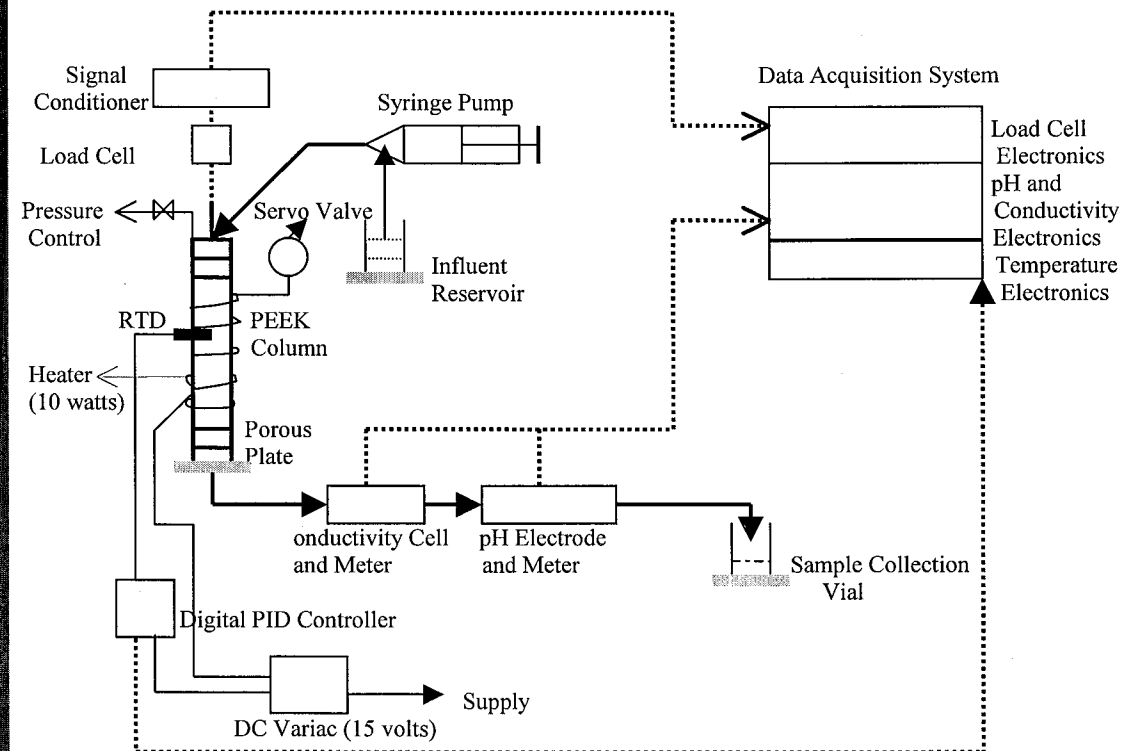
- **Kapton Heaters:** These will be wound around the heater to get a temperature of 90-100 ° C in about 20-40 minutes.
- **100 ohm Platinum RTD (Teflon Encapsulated):** This will sense the actual temperature inside the column. The Teflon encapsulation will prevent any contamination inside the column.
- **Temperature Controllers:** A digital PID Temperature controller with an SSR Output will be used to maintain the temperature at the set point.
- **DC Variac:** This is a variable transformer designed to give an output of 15 V to deliver wattage of 10 W to the heater.
- **Syringe Pump:** The syringe pump can handle upto 6 columns simultaneously, having the same or different flow rates of water. It can even be set to handle flow rates as small as 0.5 ml/day.

- **Teflon Tubes:** Teflon tubes will be used to connect the various components of the PUF Equipment. They are well-known for their excellent chemical properties, and hence, will prevent any contamination into the water.
- **Load Cell :** A precision load cell will be used to continuously monitor the weight of the column.
- **pH and Conductivity Cell and Meter:** An inline micro-flow through pH and Conductivity cell (with an internal volume of 20 and 80 microlitres respectively) will be used to measure the pH and conductivity continuously.
- **Date Acquisition System:** All the measuring instruments will be hooked on to the data aquisition system to record all the readings simultaneously.

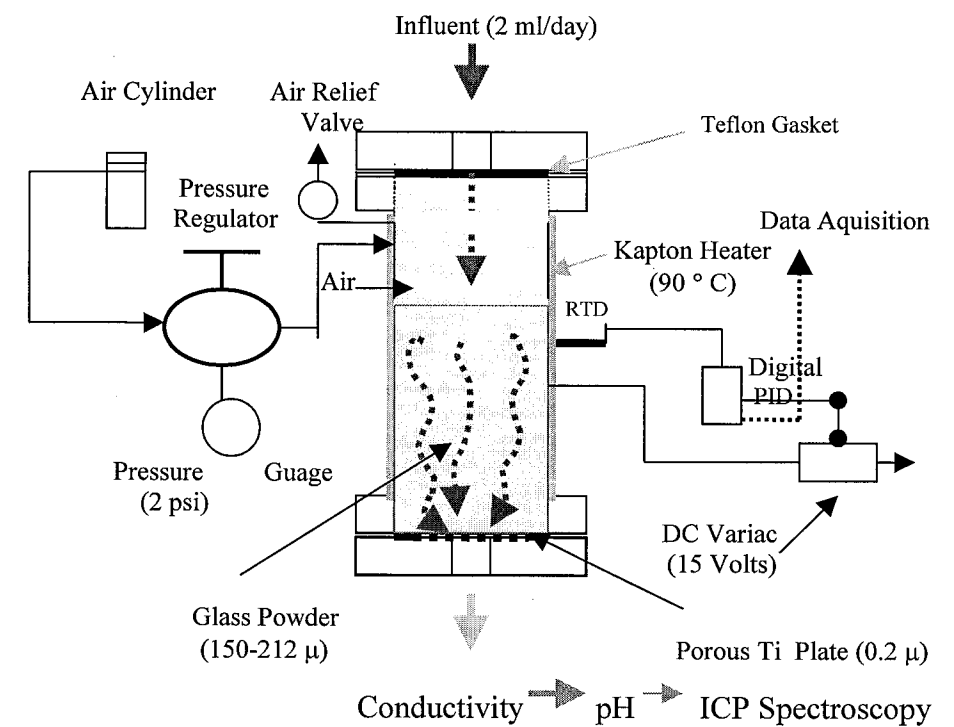
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'PUF' SCHEMATIC DIAGRAM



PEEK COLUMN (1.9 cm Φ X 7.6 cm height)



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SPECIFICATIONS AND MANUFACTURERS/DEALERS OF MAJOR PUF COMPONENTS.

- ① Fabricated PEEK Column (Poly Ether Ether Ketone-Unfilled Grade)
Internal Volume: 21.6 cm³ (1.9 cm ϕ x 7.62 ht)

Manufacturer:

Southwest Hi-Tech, Inc.
12040 Colwick
San Antonio, TX 78216

- ② Titanium Plate

Pore Size: 0.2 μ m.

Bubbling Pressure: 3-3.5 psig.

Dimensions: ϕ = 0.750", Thickness = 0.062".

Manufacturer:

Mott Corporation
84 Spring Lane
Farmington, CT 06032-3159
Ph: 800/289-6688

- ③ Kapton Heater

(1.98" x 3.82") HK5416R98.9L12B

Digital PID Controller

CT16112 SSR output

RTD100 Ω Platinum 0.00385 TCR (Teflon encapsulated) XS 100515 PD

Manufacturer:

Minco Products, Inc.
7300 Commerce Lane
Minneapolis, MN 55432-3177

Ph: 612/571-3121

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- ④ Variable Transformer

Part # ST3PN221B (3PN 221B 120V/2 25A)

Set voltage to 32 volts to give 10 W o/p to heater
to heat column to 90°C in 20-30 minutes.

Manufacturer:

ISE, Inc.

10100 Royalton Road
Cleveland, OH 44133
Ph: 440/237-3700

- ⑤ Pressure Gauge

Range: 0-5 psi

K5L-GF McDaniel Pressure Gauge, lower back mount.

Dealer:

SunSource - Houston
1833 Johanna
Houston, TX 77055
Ph: 713/548-5500.

- ⑥ pH Electrode and microflow thru cell.

Microflow thru pH system (internal volume \leq 100 μ L)
amplifier FTH-1A with standard electrode and flow cell
and BNC connector with inbuilt ATC probe.

Conductivity Probe

Cond-158B Conductivity Probe with flow thru cell and
analog o/p. $K=0.1$

Manufacturer:

Lazar Research Laboratories, Inc.
731 N. LaBrea Ave. Bldg 5
Los Angeles, CA 90038
800/824-2066

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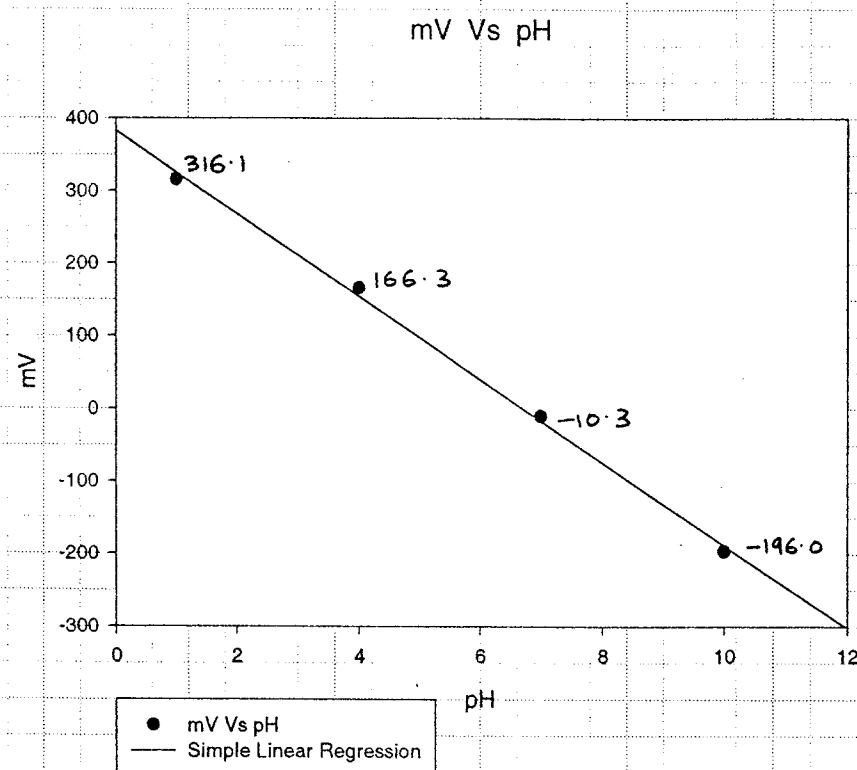
Westpaul

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pH Probe vs. mV o/p on pH meter.
(Data Acquisition for pH probe & meter)

<u>pH Buffer</u>	<u>pH meter (mV)</u>	<u>MT1. pH</u>	<u>MT6. V pH</u>
		<u>Data Acquisition</u>	
1	316.1	10.314	0.196 V.
4	166.3	7.164	0.010 V
7	-10.3	4.172	0.167 V
10	-196.0	1.625	0.316 V



$$\text{SLOPE} = -5.710 \text{ e} + 1$$

$$= -57.10$$

$$Y \text{ INTERCEPT} = 3.831 \text{ e} + 2 = 383.1$$

EQUATION:

$$Y = (-57.10)X + 383.1$$

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PUF: An Introduction

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LAZAR COND-158 B Conductivity Probe: $K=0.1$
(Data Acquisition System).

ScaleProbe: VJ 81100mV:

① Standard NaCl (1 g NaCl in 1000 ml DI)

1X	Scale!	927.0
10X		177.1
100X		17.8

② Standard KCl (0.745 g KCl in 1000 ml DI)

1X		849
10X		136
100X		13.4

③ Standard KCl (7.47287 g KCl in 1000 ml DI)

1X		934.7
10X		795.9
100X		116.9

④ Standard KCl (76.5654 g KCl in 1000 ml DI)

1X		931
10X		890
100X		361

Lazar Conductivity Probe:

Switch Position	Full scale conductivity range	Conductivity to millivolts equivalence
1X	100	1 micromho/cm = 10 millivolts
10X	1,000	1 micromho/cm = 1 millivolt
100X	10,000	1 micromho/cm = 0.1 millivolt

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START-UP PROCEDURE FOR THE MODIFIED PRESSURIZED UNSATURATED FLOW TEST**1. AIM**

This procedure describes the use of a Pressurized Unsaturated Flow (PUF) column and supporting apparatus to conduct pressurized unsaturated flow tests for the evaluation of dissolution/corrosion behavior of 316L stainless steel. Various solution chemistries may be considered. Crushed Yucca Mountain tuff particles will be used as the filling material for the column (also referred to as the reaction vessel).

The modified PUF is similar to the design of the PUF tests performed by McGrail, et al, (McGrail, P.B., C.W. Lindenmeier, P.F. Martin, and H.T. Schaef 1998. Corrsion testing of low activity waste glasses, PNNL-12014), with the following important exceptions:

- The intent of these studies is to investigate dissolution of the porous plate, whereas McGrail et al. were interested in the dissolution of the column-filling material.
- A stainless steel porous plate will be used instead of a titanium plate.
- There will be no radionuclides in this experiment.

2. PROCEDURE**2.0 Required Materials**

- 2.0.1 Some of the required equipment is described on pages 2 through 7 of this notebook, CNWRA controlled Copy XXX. Other materials and equipment required for these experiments include:

Sodium Chloride Manufacturer: Fisher Scientific Lot Number 9143
Metal Porous plates (0.2 nominal pore size, 316 L stainless steel) 913
Inert Tubing
Teflon O rings
Tanked Gas -- O₂/N₂ (CO₂-free air)

2.1 Performance Checks

Performance checks of all systems should be conducted prior to each test to ensure proper operation of all components.

- Calibrate pH electrode using standard buffer solutions and procedures.
- Calibrate conductivity sensor and monitor according to the procedures described by the manufacturer.
- Check the functioning of the pressure cylinders, pressure regulators, pressure gages and pressure inlet ports. It is important that the pressure in the column

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Procedure

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should never exceed the bubble pressure of the porous plate situated at the bottom of the column. Close valve to gas (or compressed air) and monitor pressure drop in the system. If pressure drop is noticed, check tubing connections and fittings and tighten where necessary.

- Check thermocouple circuits.
- Check heater connections. Ensure that the heating tape is properly secured on the column surface and is insulated to minimize heat loss.
- Check the temperature controller (Digital PID).
- Check the voltage on the DC variac. This is used to regulate the wattage on the heater to give the desired temperature in specified time.
- Check the calibration of the loadcell and its connections to the signal conditioner by weighing objects that have been measured on a separate calibrated scale.
- Check the Workbench software to ensure that Workbench is recording all relevant parameters correctly. Make sure that the current calibrations have been entered into the Workbench conversions. Also make sure that the frequency of data collection is appropriate for the anticipated length of the experiments.

2.2 Assembly and Start Up Procedure

The recommended startup sequence is to fill the reaction vessel with tuff, saturate the porous plate with the influent solution, begin the influent solution, apply the CO₂-free air at the desired pressure, and heat to the desired temperature.

- Obtain and record the mass of the porous plate.
- Wrap a thin strip of teflon tape around the edge of the porous plate to create a good seal between the plate and reaction vessel. Assemble the reaction vessel, including the porous plate. Measure the mass, add the tuff, and measure the mass again. Record the weight of the tuff added.
- Suspend the reaction vessel from the load cell, sealing all ports. Zero the load cell.
- Saturate the porous plate with the influent by attaching a vacuum pump to the air port and pulling the influent solution through the effluent line, the porous plate, and then through the tuff until the solution begins to come out the air port.
- Disconnect the vacuum pump and make all the necessary connections (making sure that all wires and tubing are coiled so that the reaction vessel is independent and the load cell reading is stable).
- Apply the heater to the desired temperature and set the CO₂-free air pressure to a pressure less than the porous plates bubble pressure by opening the air port. Begin the influent flow and begin recording the data on the computer.
- Care should be taken to make sure that the column and tubing are not moved during the test, as this will affect the load cell reading.

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2.3 Sample collection:

- 2.3.1 Aqueous samples should be collected periodically by removing the effluent line from the pH/conductivity line and attaching a sample vial to the effluent line. The sampling event, including time and date, should be recorded. The sample volume collected will depend on the volume required for various aqueous analyses, but should be greater than 1 ml. The type of aqueous analysis will depend on the solution chemistry.

2.4 Take-down procedure.

- 2.4.1 Stop the Workbench software from recording the data. Stop the influent pump. Turn the heater off, but leave all heater wiring hooked up so that the temperature decrease may be monitored. Turn off the air pressure system and allow the system to vent. Allow system to cool. Allow water to drain out.
- 2.4.2 Disassemble the reaction vessel and recover the tuff particles by carefully removing increments of tuff with a scraper. Record the depth of tuff removed from the reaction vessel and appearance, including wetness, color, presence of new phases, and any cementation. Remove the porous plate and note its appearance.
- 2.4.3 Gently rinse and ultrasonicate the porous plate in alcohol remove any loosely adhering particles.

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EXPERIMENT #1

Objective: perform flow through experiment at 90°C with Yuccamountain tuff and deionized water at a pressure less than the bubble point of the porous stainless steel plate.

Using (special equipment)

Yuccamountain tuff, sieved to 250 - 500µ size fraction, rinsed in acetone then deionized water repeatedly, until fines were removed.

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Melissa Nugent

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Input solution = nanopure water (weighed 3/1/2000)

Mass of bottle: 24.23 grams (no cap)

Mass of water added: 256.21 g

(note, scale was tared between measurements so the 'mass of water added' reflects only the water.)

Mass of water at end of experiment: 24.15 g

Date of measurement: 3/16/2000 - see page 20 of this notebook.

Porous plate: Mass: 6.0272 grams
measured before wrapping teflon tape around edgesReaction Vessel: Mass of top: 71.26 g (=top + fitting)
teflon ring, small hole/opening: 16.03 g

Mass of Rx vessel: 383.74 g (without top, without fitting for top, without nuts). includes bottom fitting + rxn cell bottom + rxn cell main body.

Mass of tef added: 24.23/24.24 grams.

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Exp 1

From Page No.

Calibration of Conductivity meter + sensor 3/1/2000 M. Nugent
follow procedure suggested by Lazar (see next page), see also page 9 of this notebook.1.0035 g of NaCl. Added nanopure water to 1001.1 grams (~1000 ppm)
This solution is called 'the standard'. The standard was then diluted to 500 ppm, 100 ppm, and 10 ppm.

Solution (ppm NaCl)	Conductivity Reading #1 (mV) (ts)	Conductivity reading #2 (mV) (ts)	Conductivity Reading #3 mV (ts)	see plot, page 17 of this notebook.
Nanopure Water	5.0 mV	5.2	5.44	
10 ppm	38.5	37.3	37.68	
100 ppm	50.6	49.5	49.75	
500 ppm	106.1	104.3	104.74	
1000 ppm	175.2	182.5	181.6	

3/1/2000 M. Nugent:

Calibration/Check load cell: weigh objects on a calibrated scale and the load cell.

Object	Mass on Calibrated Scale (grams)	Reading #1 Load Cell Mass (grams)	Reading #2 Load cell Mass (grams)
Hook	4.10	4.09	4.10
Scissors	120.36	120.35	120.35
Wrench	214.22	214.23	214.23
reaction vessel	162.59	162.57	162.59

Load cell reads within acceptable limits

See plot, page 17
this notebook,
CURR # 341.

* Calibration Information: Calibrated scale is calibrated by The S. R. I Calibration Lab, calibration ID 1440.

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PREPARED BY _____

SPECIFICATION

APPROVALS _____

LAZAR

Research Laboratories

NUMBER _____

TYPE _____

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PAGE 1 of 1

TITLE OPERATING INSTRUCTIONS: Model 158 and 157-C Conductivity Probes and 160 Conductivity Probe

Information potentially subject to copyright protection was redacted from this location. The redacted material is from an instruction for the models listed above.

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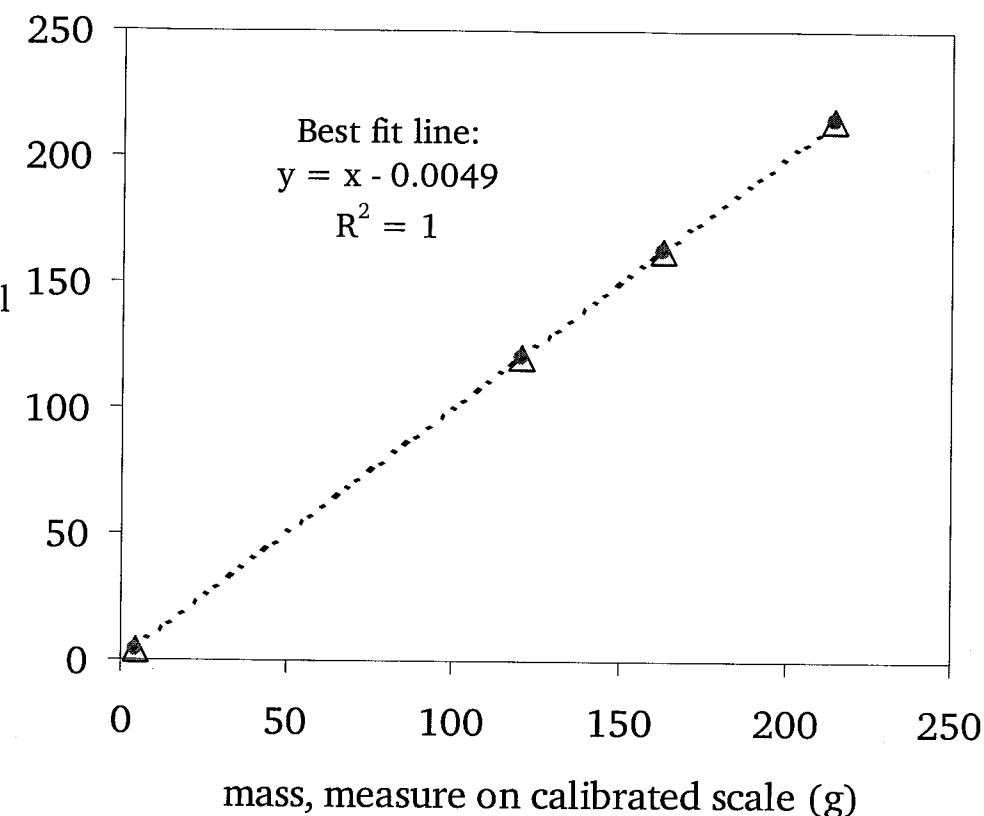
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M. Nugent3/1/2000TITLE Exp 1

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mass,
load cell
(g)

3/1/2000 Note: This calibration curve will not be used for load cell calibration. The load cell has been calibrated by SWRI Calibration (M31/2000) Materials Characterization Lab.

5/23/2000 See page 51 of this notebook CNWRA Controlled Copy # 341 for calibration information

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pH Calibration using pH=7 and pH=10 buffers, calibrate electrode and record millivolt readings for pH=7 and pH=10.

pH=7.0 millivolts = -21.4 } readings from pH meter
pH=10.0 millivolts = -188.0 }

So, $mv = -55.719 * (pH) + 368.63$. These readings were collected by removing the electrode from the effluent line and placing the electrode in bottles containing buffer and stirring vigorously.

Because the flow rate of effluent solution past the electrode will be very slow, also record the millivolt readings for 7 and 10 pH buffers under stagnant conditions, to register if there is any difference in millivolt readings.

pH = 7.02 millivolts = -12.5 (read from pH electrode)

pH = 10.02 millivolts = -178.0 (pH meter when meter showed 'ready')

$$So, mv = -55.351x + 376.07$$

↑
Where x = pH

This equation will be used in the computer software to record pH. (software is Workbench)

Start Experiment 3/2/2000

- Suspend reaction vessel (with porous plate and tuft particles). Zero the load cell after attaching air line, effluent line, influent line, and temperature sensors and ~~temperature~~ MN 3/2/2000 heater.

- Wet the porous plate. While wetting the porous plate, the reaction vessel detached from the load cell. After re-attaching the reaction vessel, the load cell reading is 7.2g. However, the orientation of the air line, heater, etc., is different, and the load cell reading of 7.2g cannot be attributed to the amount of water added. Continue to add influent water to the reaction vessel (adding water from the top). Apply vacuum through effluent line until solution exits into the effluent line.

Mass of water added = (Mass of rxn vessel, porous plate, tuft, water) - (Mass of rxn vessel, porous plate, tuft)

↑ from page 14 of this notebook

$$Mass of water = 418.27g - 407.98g = 10.29g$$

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Experiment 1

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3/2/2000

- reattach reaction vessel and re-zero load cell, after attaching all lines.

11:02 a.m. 3/2/2000: Begin heating. Note, pH reflects stagnant water in pH cell - is not reflective of experiment.

11:57 a.m. 70°C. Begin pumping influent water. Open air port to CO₂-free air tank. Water moves through immediately. P_{gauge} = 1.8 psi

1:55 p.m. T = 86.2°C

2:05 p.m. T = 91.5 and climbing! Load cell = -15.9 (water exited the system once air pressure was applied. Change P_{gauge} to 2.5 - water moves through system immediately, put P_{gauge} to 1.8. Less than 1 min (estimated from MN 3/2/2000) exited system. Adjust temperature feedback system to maintain T = 90°C.

2:17 p.m. T = 90.5°C

2:58 p.m. T = 89.0°C. Begin to collect sample #1.

Mass of Empty Bottle: 5.21g (no cap).

3:58 p.m. Stop sampling because no water came out effluent line. This maybe because the pressure was temporarily increased @ 2:05 pm. 3/4/2000 3:30 pm pH cell is not full. Air port detached (from tank) - reattached.

3/6/2000 2 p.m. pH cell is filled.

3/6/2000 10:35 a.m. Load cell = -16.7g pH = 6.62, pH cell is full. Begin collecting sample #2.

Mass of Empty Bottle: 5.23g (no cap)

4:00 p.m.: No water in sample bottle. Remove bottle. Notice that air port detached. Reattached it. Noticed water moved through system.

3/7/2000 9 a.m. air port is attached. ~~3/7/2000~~ ^{MN 3/7/2000} Begin to sample #3

Mass of Empty Bottle: 5.20g

3:30 pm Stopped sampling. Mass of full Bottle: 5.29g. Not enough sample?

4:59 air port detached! Load cell = -15.2g.

Changed the CO₂-free air tank. Reattached air port - water moving through C = 19.2 mV (on conductivity meter).

3/8/2000 10:10 a.m. air port detached (it was attached at 9 a.m.). Re-attached.

This problem will be addressed after the experiment - until then it will be monitored. Water moved through effluent line after ~~3/8/2000~~ re-attaching, pH = 7.04, conductivity = 12.5 mV.

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3/8/2000 cont 11⁰⁴ a.m. electrode (pH) cell is full pH = 6.99. Conductivity is ~4.8 (oscillating between 4.7 - 4.8, more often on 4.8).

3/9/2000 10²⁰ a.m. pH = 6.38, Conductivity = 5.1. Collected a sample #4 beginning now. Mass of bottle: 5.24g

2³⁵ p.m. Stop sampling. Stop recording on Workbench, look at data.

Re-start Workbench. Continue to monitor puf air-port (tightened fitting).

3/14/2000 10⁰⁰ a.m. puf air-port detached overnight - reattached. Load cell = -34.8g

3/15/2000 8³⁰ p.m. Take down experiment. Stopped pumping influent. Turned header off. Pumped air into system. Allow tuff to dry overnight.

3/16/2000 Weigh the tuff and porous plate.

Mass of porous plate: 6.0459g. Note, plate was ultrasonicated in acetone and dried in air before weighing.

Mass of tuff: tuff was removed from top to bottom in increments. Visual inspection of tuff: does not appear to have changed in particle size, color, or "mineralogy". The tuff has not consolidated.

Top of rxn vessel: Mass, increment 1: 3.26g

Mass, increment 2: 3.79

Mass, increment 3: 4.68

Mass, increment 4: 8.8g

Mass, increment 5: 3.62

Total Mass: 24.15g

Note, the mass after the experiment is less than the mass before the experiment. This is probably due to difficulties associated with removing the tuff particles quantitatively.

3/16/2000 Visual inspection of the 316L Stainless Steel Plate: The plate does not appear to be corroded on the top part, which was in contact with the tuff particles. The bottom part of the plate, however, has a faint 'halo' of corrosion, which is located where the plate contacts the fitting on the bottom portion of the reaction vessel.

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M Nugent

Date

3/2/2000

Recorded by

TITLE

Exp. 1

From Page No.

Results:

Masses of samples will be recorded here.

Sample Name;	Mass of Bottle + Sample	Mass of Bottle (also recorded on page 19-20)	Mass of Sample (calculated)
#1	5.21g	5.21g	0 grams
#2	5.23g	5.23g	0 grams
#3	5.29g	5.20g	0.09 grams
#4	5.94g	5.24g	0.7 grams

Note, aqueous samples will not be analyzed for this experiment because of the low volumes collected.

Note, pH, load cell, and conductivity plots will not be presented here. This is because of MN 3/17/2000 because, for pH, the pH chamber was partly or fully empty at several points during the experiment. Because the experiments were not observed overnight, all overnight data may be suspect (i.e., the pH readings may not be accurate). The same argument applies for the conductivity data. The load cell data is also not plotted. This is because the air-port detached and was re-attached numerous times.

3/18/2000 Note: pH, conductivity, and load cell data are plotted for a portion of the experiment - the first ~100 hrs.

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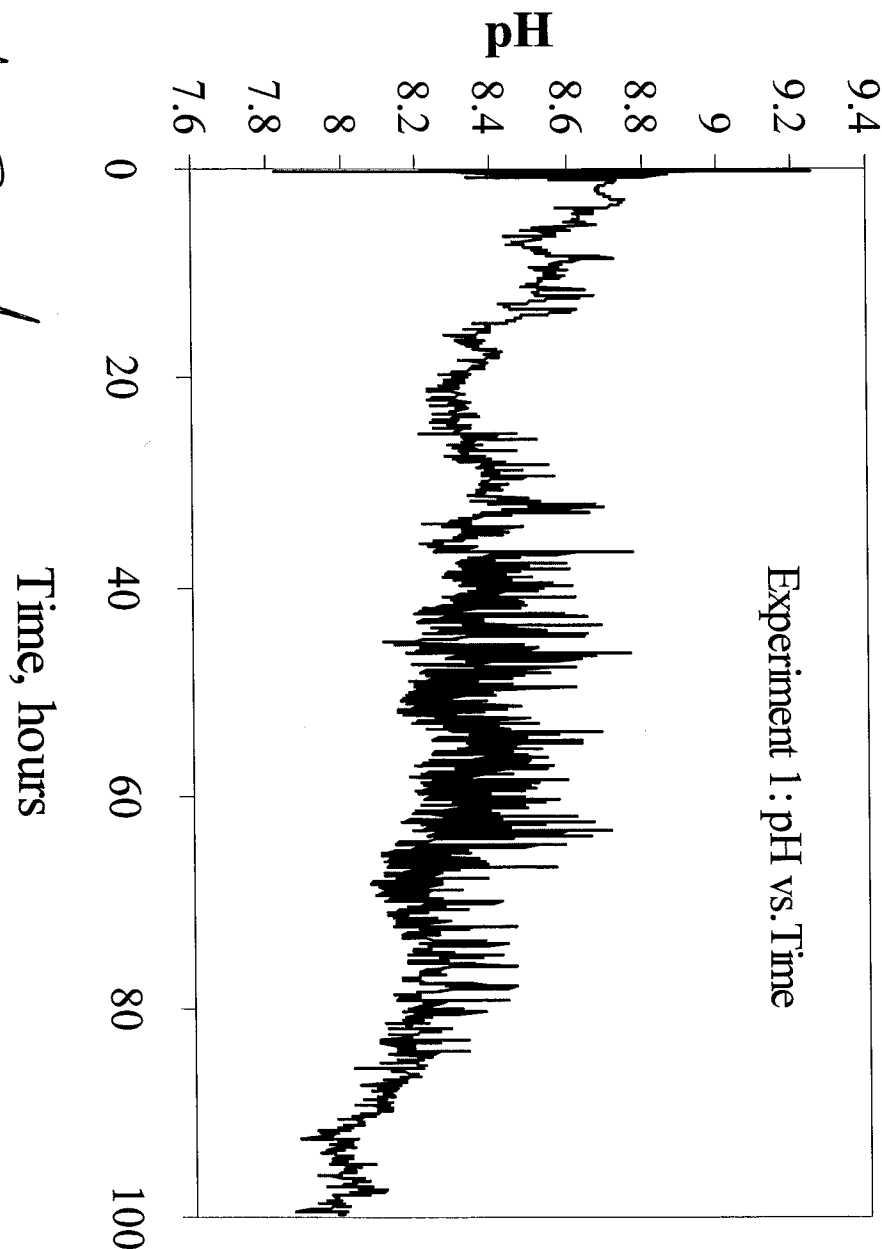
M Nugent

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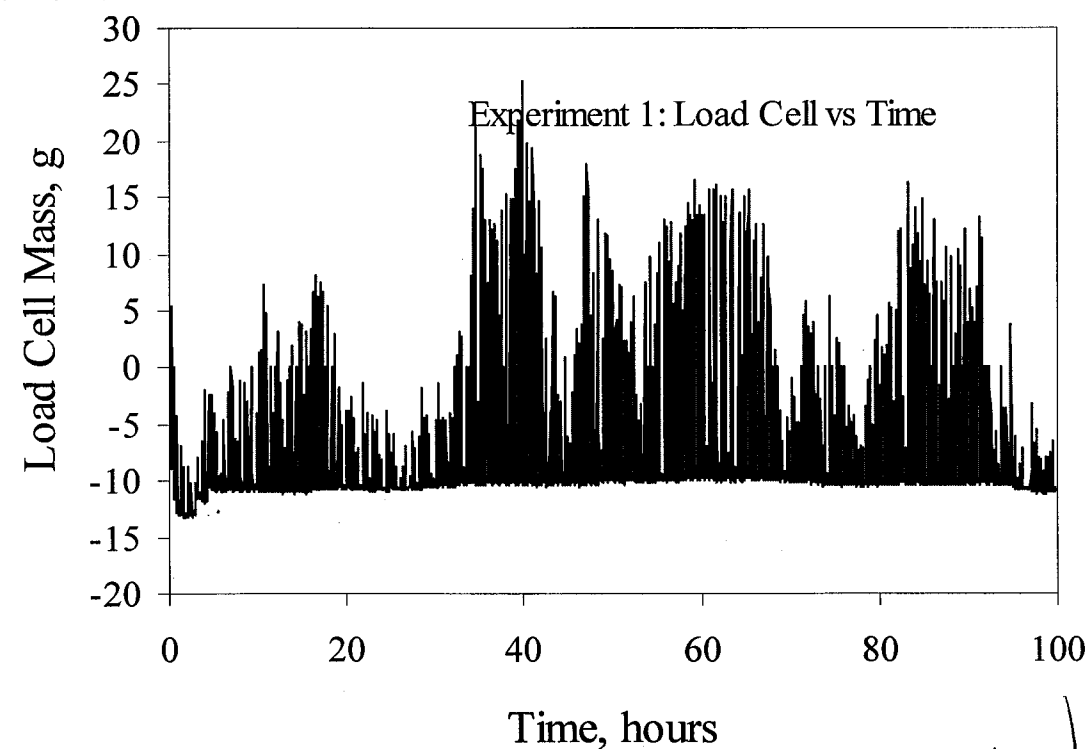
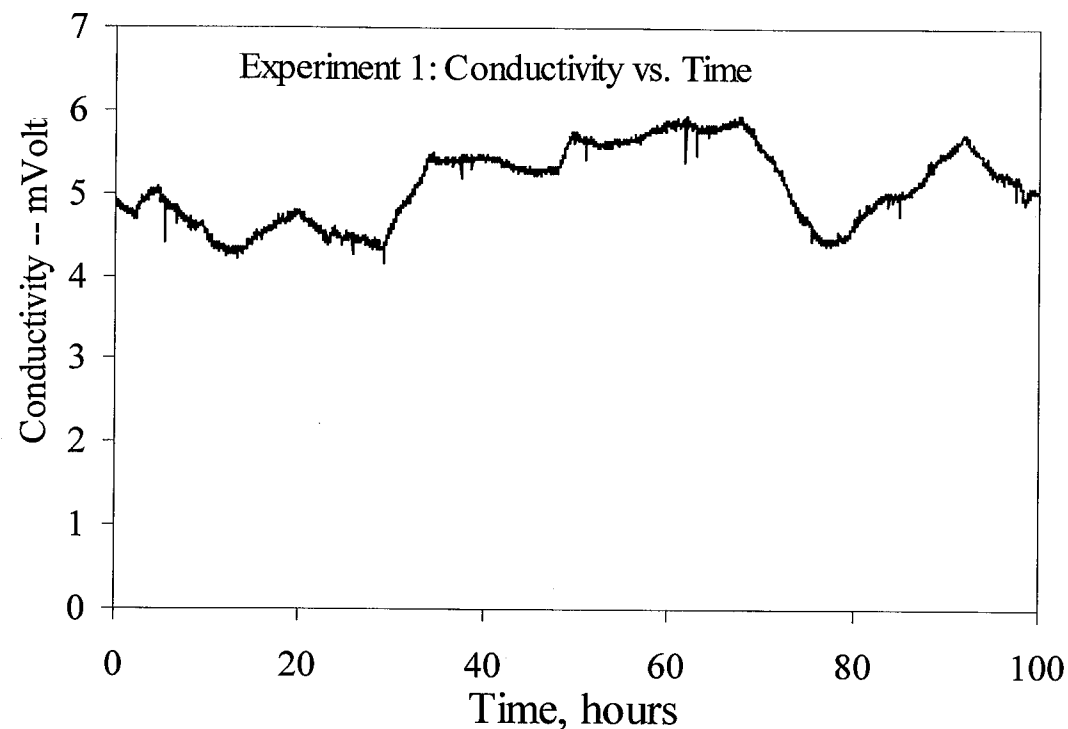
Invented by Melissa Nugent
 Recorded by Melissa Nugent

Date _____

3/2/2000

TITLE Experiment 1

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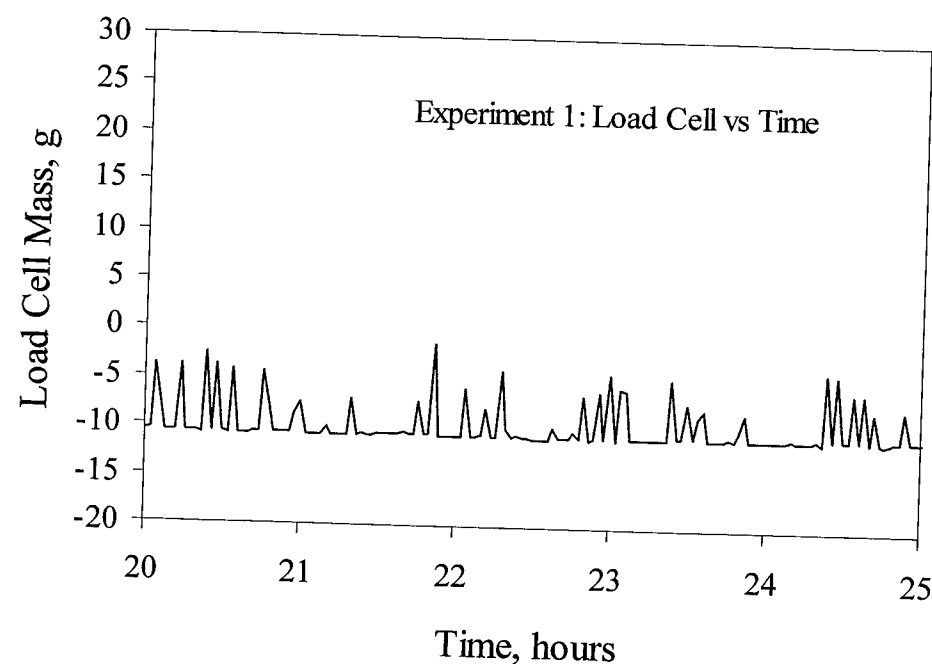
Invented by Melissa Nugent
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Note, the noisiness of the load cell data is due to short time, frequent disturbances. These occur during the experiment, at day and at night, and may be related to the 1st run 3/18/2000 instrumentation.

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3/2/2000

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Experiment 2

From Page No.

EXPERIMENT #2

Objective: perform an unsaturated flow through column experiment using the 1st particles, at 90°C, with 1M NaCl and a stainless steel, porous plate, so that the plate is corroded.

Special Equipment: the system was adjusted so that the air-port will not detach. The conductivity probe was removed from the effluent line, because the conductivity of this experiment will be too high for the probe's operational range. All other equipment is described by the procedure, except for the procedure for wetting the frit, which is described on this page (25) of this notebook (see below).

Mass of porous plate: 5.9551 grams

Mass of tuff added to reaction vessel: 22.86 grams

Tuff was added to just below air-port.

Prepare 1M NaCl solution: 29.23 grams of NaCl [Fisher ACS grade, lot # 914913] added to 500 mL nanopure. Total mass 500.21 grams.

Mass of 1M NaCl put in influent line bottle: 288.71 grams

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pH calibration: done under stagnant conditions on 3/23/2000.

pH = 6.94, -15.1 millivolts

pH = 10.01, -181.2 millivolts

will record mV data and convert it to pH after collection of data using this calibration.

Start-up Experiment #2 : 3/23/2000

• Suspend reaction vessel (including tuff and porous plate) from load cell, attach lines, zero load cell. Collecting bottle = Effluent #1 Exp2

• Wet the tuff and porous plate using 1M NaCl solution.

4:35 pm = Applied air pressure, mass to -4.5 on the load cell. I assume the mass reading is negative due to a change in the orientation of the reaction vessel, not a loss of material from the reaction vessel.

Pressure (Pgauge) from the tank set to 1.8 psi. Started heating.

• 5:37 pm T = 89.9°C. mass = -9.3 grams. Unfortunately, the shift in the reaction vessel did cause the load cell reading to change (indicating the load cell is not independent from the various lines feeding in and out of the system) and the mass readings will be difficult to interpret absolutely.

• 5:40 pm. Start pumping influent solution. Change to MN 3/23/2000 bottle @ end of effluent line to: Effluent #2.

3/24/2000 9:45 a.m. air-port is attached, but the support for the air port (which holds the air tubing up) detached from the wall, and the load cell reading is -25g. Reattached support, load cell = -9.8g. Readjusted to approximately the same set up for the support, and load cell reading = -9.3g.

3:25 pm Support slipped again, load cell = -7.8. This must be fixed - but after the experiment. Began MN 3/24/2000 Began sampling. Exp2.S1

4:59 pm Stopped sampling. pH ~ 6.78 Some bubbles in effluent line

3/27/2000 8:45 a.m. Begin collecting sample Exp2.S2. Increase Pgauge to 1.9

10:09 a.m. Stop sampling. Return Pgauge to 1.8 psi.

3/29/2000 8:45 a.m. Some bubbles in effluent line. Decrease Pgauge to 1.2 for the remainder of the experiment

10:30 a.m. Start collecting sample Exp2.S3 Pgauge → 1.5 psi

2:16 p.m. Stop sampling. Pgauge → 1.2 psi

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3/23/2000

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Experiment 2

From Page No.

3/30/2000 4:58 p.m. Change tank. Start collecting sample Exp2.S4.

Increase Pgauge to 1.8 psi.

5:43 pm Stop sampling. P → 1.2 psi

4/3/2000 9:55 a.m. pH = 6.08, pH cell is full. Begin collecting sample

Exp2.S5. Increase Pgauge to 1.8 psi.

2:28 p.m. Stop collecting sample. Decrease Pgauge to 1.2 psi.

4/4/2000 12:11 p.m. Start collecting sample Exp2.S6. Pgauge → 1.8 psi

3:43 pm Stopped sampling. Pgauge → 1.2 psi

4/6/2000 pH = 6.47 12:22 p.m. Begin sampling. Pgauge → 1.8 psi

6:07 pm stop sampling. Replace collection bottle on effluent line.

Note, sample name is Exp2.S7 for sample collected on 4/7/2000. MN 4/6/2000

4/7/2000 Stop experiment 8:50 a.m.

Stop data collection, turn off heat, stop influent, run air through system, detach air-port. Allow tuff to dry.

Mass of 1M NaCl influent line solution left in bottle: 259.99 grams without cap on bottle.

Amount of 1M NaCl solution run through experiment =

281.71g - 259.99g = 21.72 grams.

4/8/2000 Porous plate rinsed briefly with nanopure water and allowed to dry. There is obvious 'rust' on surface of plate, but only in contact with the reaction vessel MN 4/8/2000 but only where the plate was in contact with a fitting found 4/8/2000 on the reaction vessel.

The tuff has NaCl deposited on it, but does not appear to have changed. It is still unconsolidated. MN 4/8/2000

Upon removal of the tuff, approximately 2/3 of the way down the reaction vessel column, the salt had dried and cemented the tuff particles together. The entire mass of tuff was removed together and weighed together, then rinsed, dried and re-weighed (to remove salt).

Tuff Mass before rinsing: 22.51 grams

Tuff Mass after rinsing: 22.47 grams.

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Results:

porous plate appears to be slightly rusted on top side (the side in contact with the tuft), and the plate has a 'corrosion halo' on the bottom side - due to contact with the fitting on the bottom of the reaction vessel.

The porous plate will be sent for SEM/EDX analysis - see page 43 of this notebook. The porous plate was rinsed briefly in alcohol and ultrasonicated briefly in alcohol and dried before SEM/EDX

Masses of the samples collected will be recorded here:

Sample Name:	Mass of Bottle (gr)	Mass of Bottle + Sample (grams)
Exp2S1	5.20g	5.99g
Exp2S2	5.26	6.17grams
Exp2S3	5.19	6.01grams
Exp2S4	5.18	6.26grams
Exp2S5	5.23	5.93grams
Exp2S6	5.19	6.11grams
Exp2S7	5.23	6.20grams

Samples were sealed, but not acidified or altered in any way.

Note, The pH, load cell, and conductivity data were collected each second of the experiment. The data files are very large and need to be compressed. The data will not be presented here.

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Experiment 3

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Experiment 3

Objective: Repeat Experiment # 2 of this notebook, but use 1000ppm KCl ^{mn 5/16/2000} instead of 1M NaCl, and 0ppm Mn, instead of 1M NaCl, and 0ppm Mn. Try to use inert sand (Zirconia) and tuff-equilibrated water.

Special Equipment: conductivity probe is not being used. Zirconia sand will replace the tuff particles. Tuff-equilibrated nanopure (equilibrated @ 90°C for 3 weeks) will be adjusted to 1000ppm KCl with KCl.

Mass of stainless steel porous plate: 6.0592 grams

Mass of zirconia sand added to reaction vessel: 47.01 grams

Note, vessel filled to just below puf-port (air-port)

Prepare 1000ppm KCl solution with 5ppm Manganese:

- Filter equilibrated water to remove tuff.

- Make stock solution for Mn: want 0.002m/L solution of Mn.

$0.002 \text{ m/L} \times 158.9 \text{ g/m} \times 100 \text{ mg/1000} \mu\text{L} = 0.0316 \text{ grams } \text{K}_2\text{MnO}_4 \text{ for } 100 \text{ mL of solution.}$

Actually added: 0.0324 grams K_2MnO_4 [Lot #885967 Fisher ACS grade] ^{mn 5/16/2000}

Added nanopure to 100.0256 grams. Estimated concentration: ~1,107 ppm

Use this solution to spike the tuff-equilibrated water to 5ppm Mn.

- Prepare 100ppm KCl tuff-equilibrated solution.

Fisher ACS grade

Actually added 0.2242g KCl [Fisher Lot #885967]

Lot 904466

Added tuff-equilibrated nanopure to 102.3199 grams.

- Prepare 15mL of influent solution.

15.3025 mL of KCl -tuff-equilibrated water + 69 μL of Mn stock solution for a total mass of 15.3737 grams was prepared. This will be used on the influent line.

This solution will be replaced (re-prepared) during the experiment.

pH calibration after calibration, take readings under stagnant conditions

pH = 7.00 - 12.0 millivolts

pH = 10.01, -183.4 millivolts

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Melissa Nygant

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Recorded by

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Start up Experiment #3

• Suspend reaction vessel (including zirconia sand and stainless steel plate), attach all lines - influent, effluent, heating, air - port. Attach a collecting bottle on effluent line. Exp3 Effluent #1.

• Wet the porous plate. Wetting the plate seems difficult - The zirconia sand is exiting the reaction vessel into the pump. I cannot apply the pump vacuum for long enough to pull water through the porous plate. EXPERIMENT ABORTED.

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TITLE

Experiment 4

From Page No.

Experiment 4

Objective Repeat Experiment 3 but successfully wet the porous plate.

Special Equipment: see Experiment 3 of this notebook.

Mass, stainless steel porous plate: 6.0172 grams.

Mass of zirconia sand added to reaction vessel: 48.25 grams

filled reaction vessel to just below air - port.

Prepare Influent Solution

~~use 1000ppm Ce tuff equilibrated water prepared in experiment 3 of this notebook.~~ MN 5/17/2000

prepare 100ml of 1000ppm Ce tuff equilibrated water (which has been filtered)

Actually added 0.2248 grams K Ce

Added tuff equilibrated water to 102.1089 grams.

prepare - 15ml of Mn-spiked, 1000ppm Ce tuff-equilibrated influent solution. 15.3047 grams 1000ppm Ce tuff-equilibrated water, added 69 μ L to a total mass of 15.3748 grams.

pH calibration after meter is calibrated, take readings in stagnant buffers.

pH = 7, -12.1 mV

pH = 10.01, -183.0 mV

Start up Experiment #4 5/17/2000

• Suspend reaction vessel (including zirconia and plate), attach lines.

Zero the load cell, attach a collecting bottle on effluent line. Bottle is labelled Effluent #1 Expt4.

• wet the porous plate. The porous plate was wet using the same procedure described in Experiment 3 of this notebook, but the tube going to the vacuum pump was folded to make it more difficult for the zirconia sand and/or water to enter the pump. The vacuum was not applied for as long as in Experiments 2,3 of this notebook, but water did come through the plate, suggest MN 5/17/2000. Meaning the

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plate is wetted. Some sand was lost, however.

4:30 p.m. Start heating. Attached the air-line but did not pressurize.

4:40 p.m. Load cell reading = -11.4 grams. Stable reading with all lines attached. Re-adjusted lines, load cell reading is now stable at -9.4 to 9.5.

4:48 p.m. Start pump. $T = 68.5^{\circ}\text{C}$. Apply vacuum ^{MN} 5/22/2000 bottle air pressure @ 1.8 psi. Readjusted lines.

5/18/2000

9:05 a.m. Experiment running fine. No water in effluent.

11:40 p.m. Load cell reading = -13.1 grams.

2:24 p.m. Still no effluent. This may be because the plate is not fully wetted. Detach air-port, seal air-port, and allow experiment to continue as a saturated (saturated with respect to water) experiment. Changed Effluent bottle to Effluent #2 Expt. Note, no sample in Effluent #1 Expt.

5/20/2000

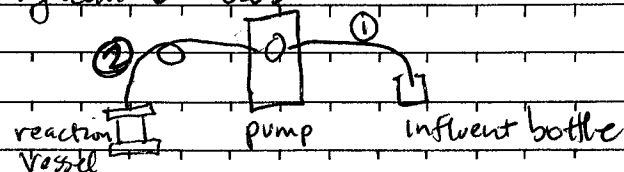
3:25 p.m. Very little effluent solution in effluent line. pH cell is approximately 1/2 full.

5/22/2000

3:00 p.m. Still very little effluent in effluent line. Stop pumping solution into reaction vessel. The influent bottle is more full than this morning and has what appears to be zirconia sand in it.

3:35 Stopped data collection. Turned off temperature. Removed plug from air-port. Nothing came out line. The reaction vessel itself was not under pressure from excess water in it.

Performed test on pump to see why pump is 'refluxing'. For the purposes of this test, will refer to line (1) (the line which runs from the influent bottle to the pump) and line (2) (the line which runs from the pump to the reaction vessel) as described on the diagram below.



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Experiment 4

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• Detach line (2) from reaction vessel. Run pump @ 20 ml/day.

No solution is delivered from out of line (2).

• remove line (1) from the influent ~~bottle~~ ^{MN} 5/22/2000 bottle.

This is very difficult to do, so line (1) and the influent bottle may have created a vacuum in the line. The bottle was originally nearly full, and was attached so that the bottle would not detach, but that air could get in to replace the volume of solution removed by the pump. It appears that the bottle and connecting cap locked to prevent air getting in, and this may have caused the 'reflux'. Run pump with nothing attached to line (1) and nothing attached to line (2). Pump works fine - air entered line (1) and exited line (2). The reflux must have been due to a vacuum between line (1) and the influent bottle.

End test.

Removed zirconia sand - an estimated 15-20% of sand ~~at~~ ^{MN} 5/22/2000 was lost - probably during the porous plate wetting process.

The porous plate has a 'black corrosion product' in some areas. This black corrosion product is located on the top part of the porous plate. On the bottom, there is a slight halo of reddish corrosion product where the Teflon fitting on the reaction vessel contacted the porous plate.

Visual inspection by microscope reveals pits.

5/24/2000

Note, no pH-time, load cell-time plots will be presented because the pH cell was empty for much of the experiment.

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From Page No.

Batch Experiment #1

Objective: to check if 316L stainless steel will corrode in a permanganate solution, while measuring solution potential, at 90°C.

Materials:

Potassium permanganate Fisher ACS grade, Lot # 904466

Sodium chloride Fisher ACS grade Lot # 975121

Thermocouple S/N E98293

316L Stainless steel - polished to 600 grit. Heat # D80746

Reaction vessel - glass

Lids and stoppers for reaction vessel

Thermometer

Pipettor and pipette tips.

Note, pipettor calibration (SWPI)

Number: S/N 360050

Mass of 316L Stainless Steel Before Reaction:

11.70819 grams

1. Prepare 1M NaCl solution - add 750ml to reaction vessel. Close reaction vessel and heat to 95°C. pH before heating = 5.739. Wait until solution is at temperature ~~to record~~ MN 5/23/2000 begin adding Mn.

2. Prepare ~10,000ppm Mn solution. Using 100ml of nanopure water, add

Actually, placed 1.0020 grams of K_2MnO_4 in bottle and filled to 100.04 grams. This is 10,020 ppm Mn.

3. After solution is at 90°C in reaction vessel, add small increments of Mn - to increase the Mn concentration by ppm 5ppm each time. Each addition of Mn will be made by a pipettor, and after the potential is calmed, will add 0.375ml increments. Upon first addition the pipette tip dropped into the experiment. It was removed, but the E (Volts) recorder had to be temporarily interrupted.

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Time Amt Mn solution Added (ml)

450.68 sec 0.375 ml

- restarted

1813 - after re-start 0.375 ml

4028 = 12:28 pm

0.375 ml

106 pm = 6:160

0.375 ml

6:317

0.375 + 0.375 = 0.750 ml

11:38

1.125 ml

23,435 sec

1.125 ml

Solution allowed to sit overnight at 90-95°C, then the stainless steel was removed from solution, ultrasonicated in alcohol, and dried under a hot-air dryer.

Mass of stainless steel after reaction: 11.6777 grams.

The stainless steel is quite pitted. Before cleaning, it was covered in a black film. This film was easily removed by the cleaning procedure used.

The solution in the reaction vessel was allowed to cool, and the pH was measured again at room temperature. pH_{final} = 8.190.

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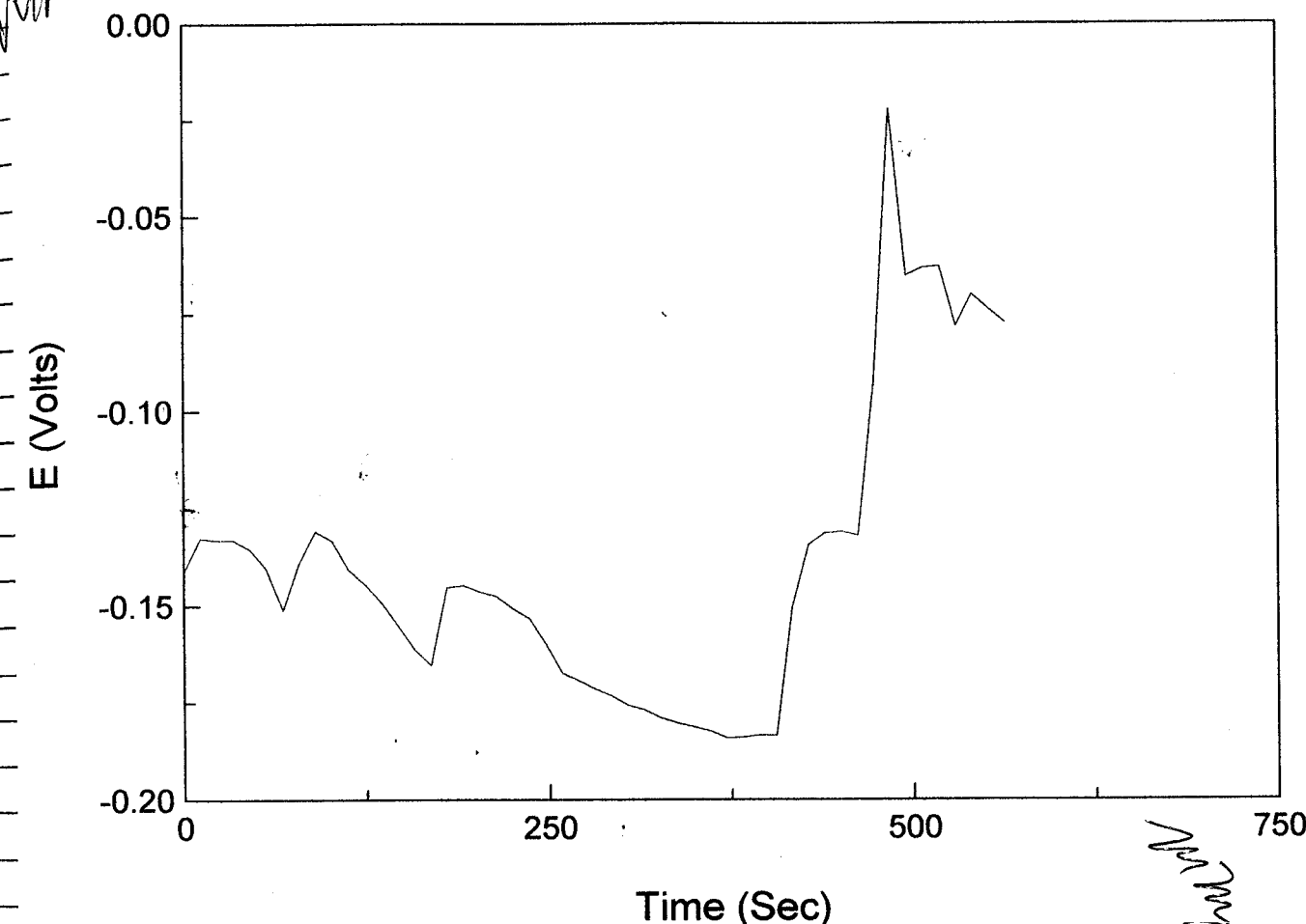
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TITLE

Batch experiment #1

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↑ This is a plot of E (volts) vs. time for the solution at 90°C. The first increment of Mn was added at 450.68 seconds, and the pipette ~~tip~~ ^{2/2/2000} was accidentally added - which meant that E recording had to be stopped in order to remove it. The E vs. time plot is continued on the next page of this notebook.

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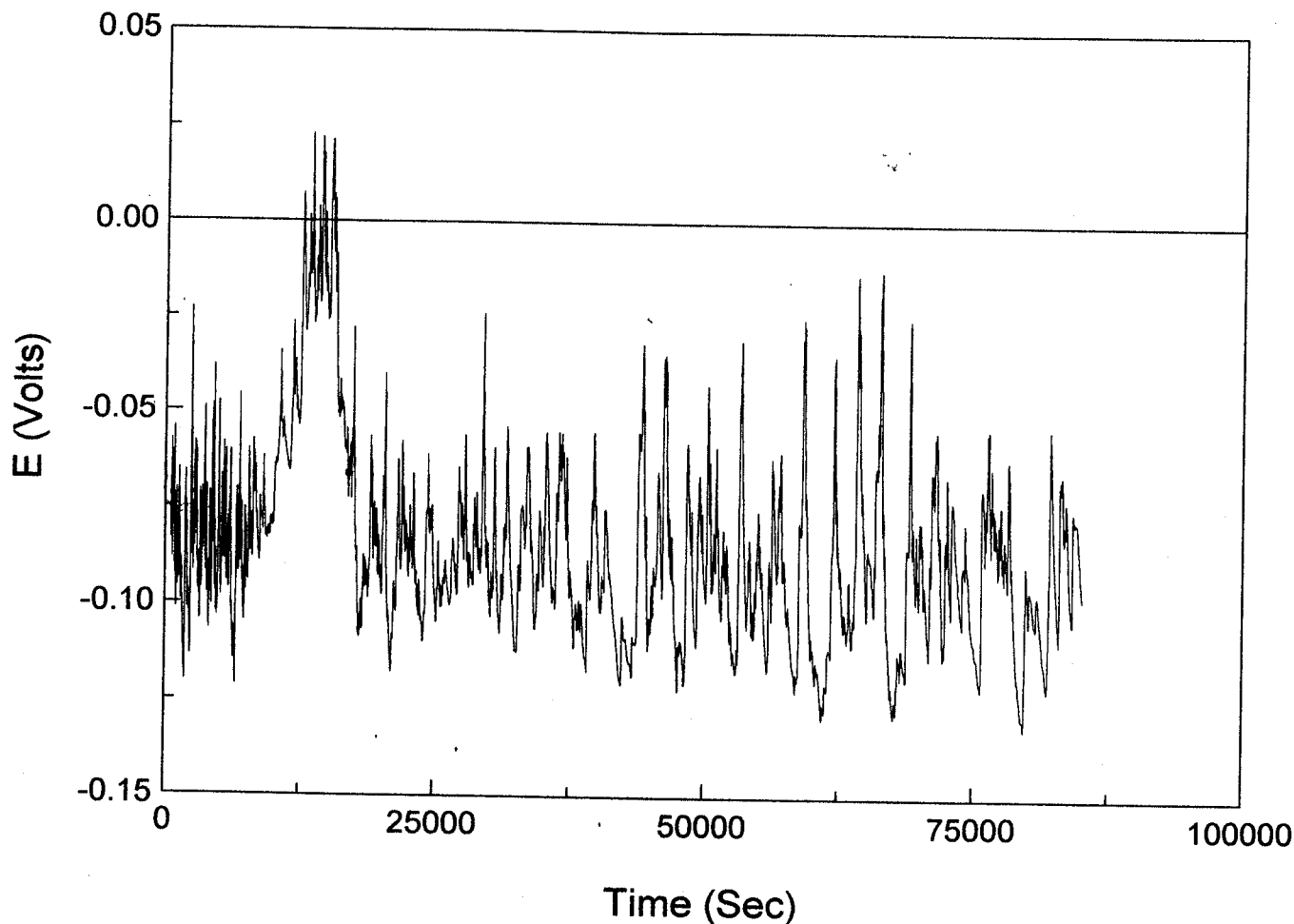
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↑ The above plot of E (volts) vs. time (seconds) records all additions of Mn in Batch Experiment #1, except the first addition of Mn, which is recorded on page 41 of this notebook.

Because of the noisiness of the E (volts) signal from the first addition of Mn until the end of the experiment, the stainless steel probably pitted immediately. This indicates that a 5ppm Mn, 1M NaCl solution at 90°C will corrode 316L stainless steel.

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5/23/2000

SEM Analyses

SEM (Scanning Electron Microscopy) and EDS analyses were performed on an unreacted 316L stainless steel porous plate, and the 316L stainless steel porous plate from Experiment 2, after it had been reacted. MN 5/20/2000

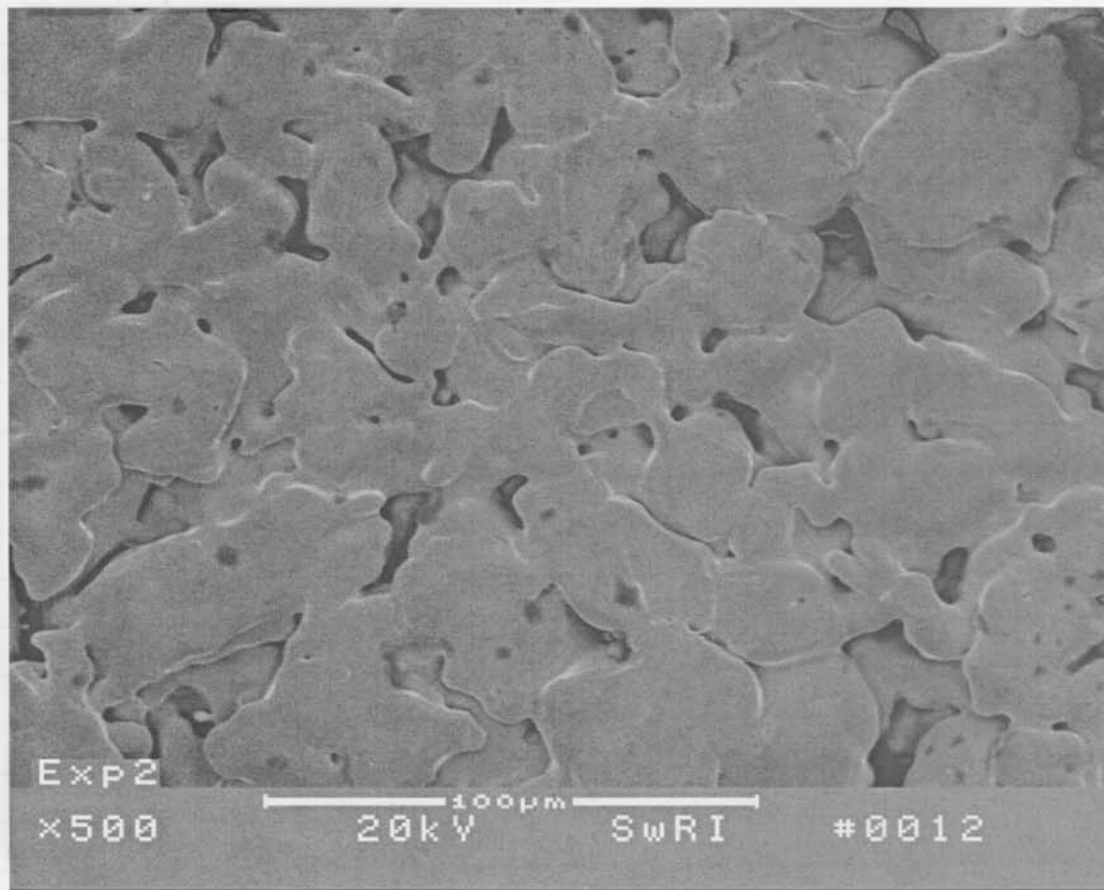
Analysis were performed on ~~MN 5/20/2000~~ May 8, 2000, but are recorded on 5/20/2000, because the final results were all recieved on 5/20/2000.

Analysis of The Unreacted 316L stainless steel porous plate. MN 5/20/2000

Note: Analysis were not performed on the unreacted stainless steel.

Analysis of The porous plate from Experiment 2

The plate was inspected in an area that had no obvious rust (from visual inspection) - This is called 'Exp2 Away from 'rusted location. in EDS Analysis



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EDS Analysis for SEM picture on page 43 of this book.

File: C:\SIGMA\QUASAR\14025715.RPT

[ANALYSIS REPORT]

GENERAL CONDITIONS

Result File : 14025715
 File Version : 1
 Background Method : Fit
 Decon Method : Gaussian
 Decon ChiSquared : 3.54
 Analysis Date : 8-MAY-2000
 Microscope : SEM
 Comments : EXP2 Away from "rusted location"

ANALYSIS CONDITIONS

Quant. Method : ZAF/ASAP
 Acquire Time : 100 secs
 Normalization Factor: 100.00

SAMPLE CONDITIONS

kV : 20.0
 Beam Current : 150.0 picoAmps
 Working Distance : 29.5 mm
 Tilt Angle : 0.0 Degrees
 TakeOff Angle : 35.0 Degrees
 Solid Angle*BeamCurrent: 0.8

Element	Line	Weight%	Cnts/s	Atomic%
O	Ka	1.55	16.49	5.17
Na	Ka	0.10	0.82	0.23
Si	Ka	1.00	26.07	1.89
P	Ka	0.06	1.68	0.10
Cl	Ka	0.10	3.35	0.15
Cr	Ka	17.47	459.44	17.93
Fe	Ka	64.94	1148.45	62.05
Ni	Ka	12.05	146.47	10.96
Mo	La	2.73	34.99	1.52
Total		100.00		

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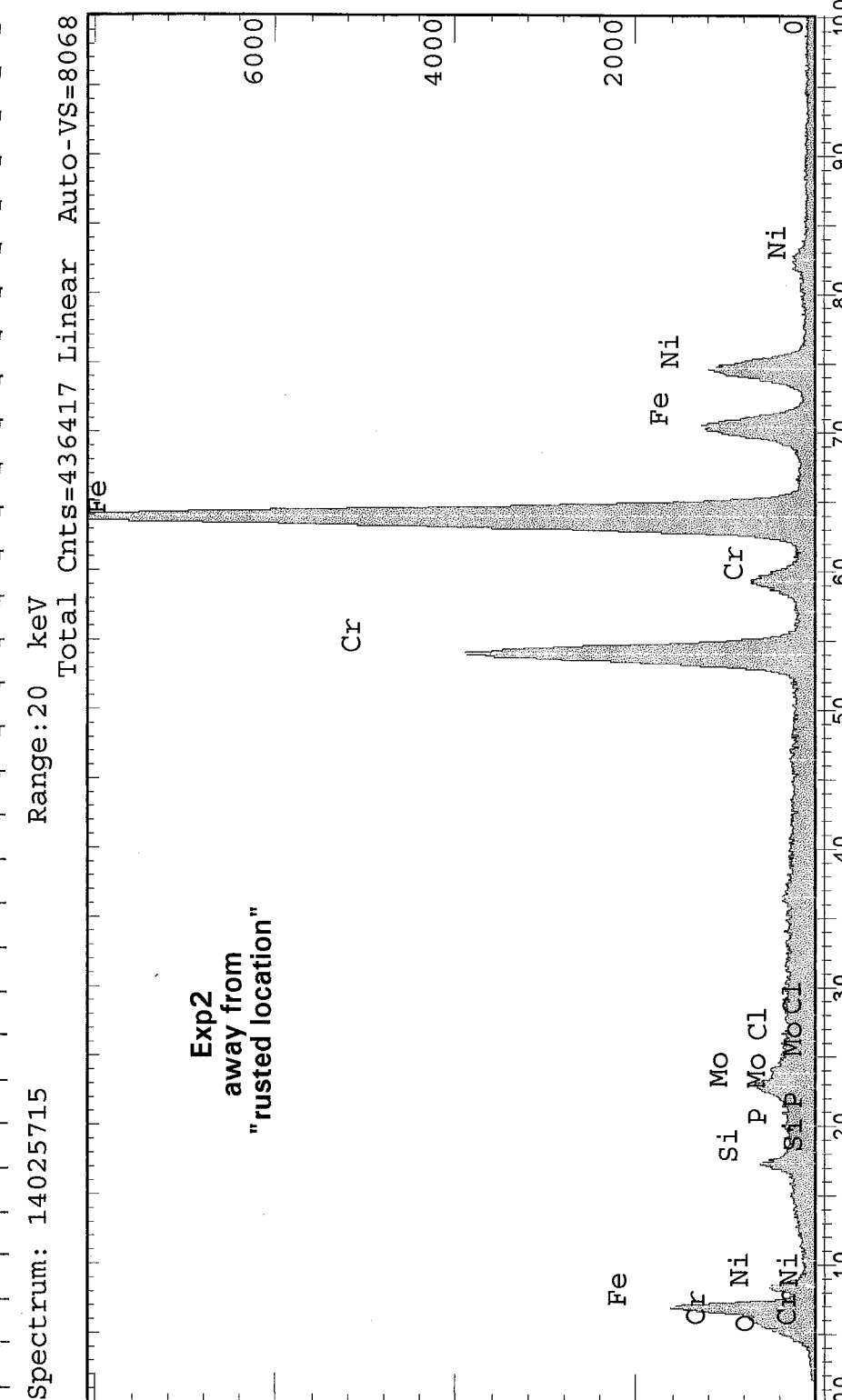
Recorded by

M Nugent

5/20/2000

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EDS spectra for EDS analysis for SEM picture on page 43 of this book.



Spectrum: 14025715

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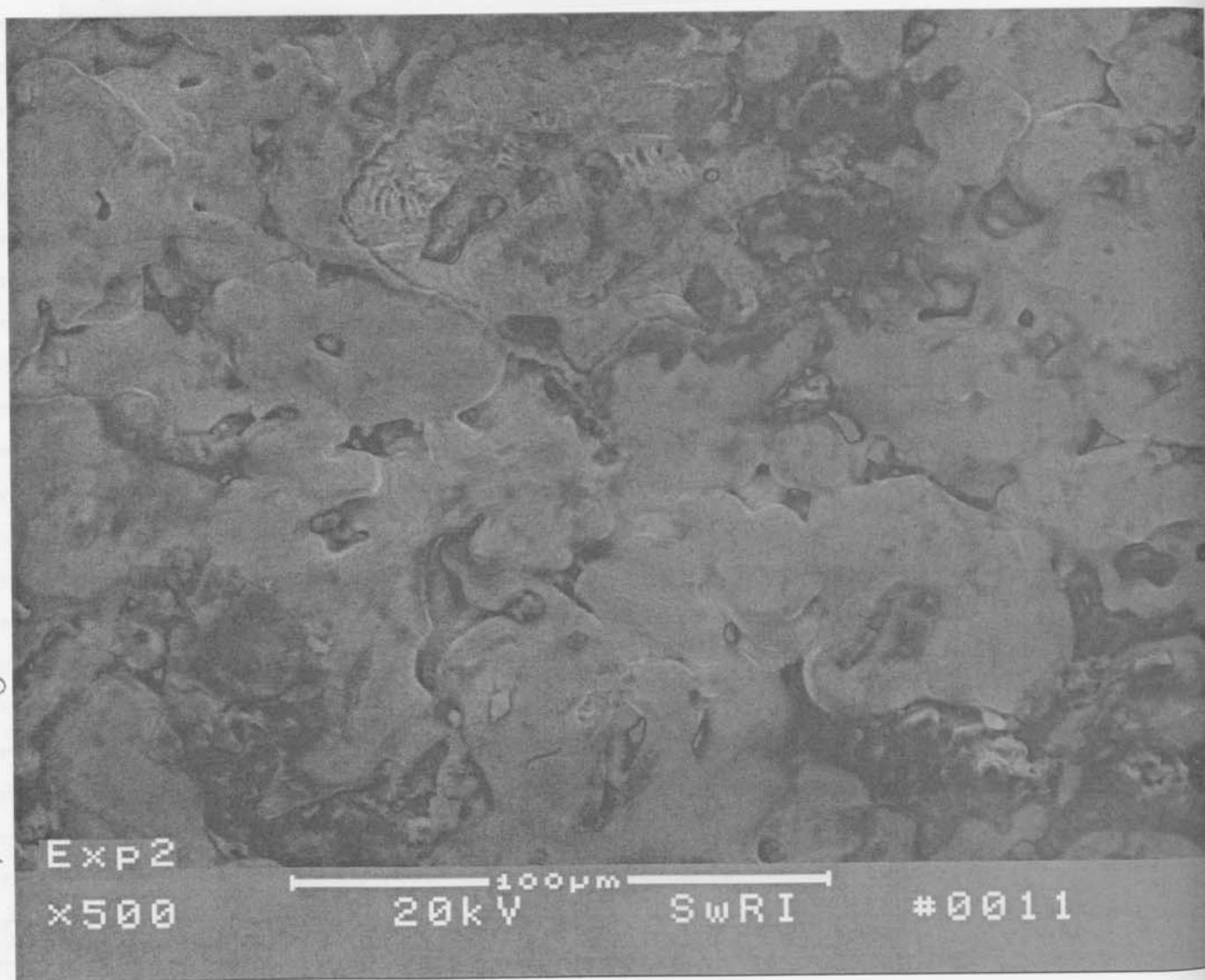
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Below on this page is an SEM image from an area that appeared, by visual inspection, to be corroded. EDS analyses were performed in 'light area in "rust location"' - a lighter area in the picture below, and in 'dark area in "rust location"' - the darker areas below.



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SEM analysis

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EDS analysis on SEM image on page 46 of this book

File: C:\SIGMA\QUASAR\14025714.RPT

[ANALYSIS REPORT]

GENERAL CONDITIONS

Result File : 14025714
File Version : 1
Background Method : Fit
Decon Method : Gaussian
Decon ChiSquared : 4.32
Analysis Date : 8-MAY-2000
Microscope : SEM
Comments : EXP 2 Light area in "rusted location"

ANALYSIS CONDITIONS

Quant. Method : ZAF/ASAP
Acquire Time : 100 secs
Normalization Factor: 100.00

SAMPLE CONDITIONS

kV : 20.0
Beam Current : 150.0 picoAmps
Working Distance : 29.5 mm
Tilt Angle : 0.0 Degrees
TakeOff Angle : 35.0 Degrees
Solid Angle*BeamCurrent: 0.8

Element	Line	Weight%	Cnts/s	Atomic%
---------	------	---------	--------	---------

O	Ka	4.56	51.52	13.99
Na	Ka	1.14	10.32	2.42
Si	Ka	1.17	33.44	2.04
P	Ka	0.20	6.20	0.32
Cl	Ka	0.36	13.30	0.51
Cr	Ka	16.98	477.59	16.03
Fe	Ka	60.50	1154.67	53.17
Ni	Ka	11.71	154.38	9.79
Mo	La	3.39	47.39	1.74

Total		100.01		
-------	--	--------	--	--

M. Nugent

Witnessed & Understood by me,

Date

Invented by

M. Nugent

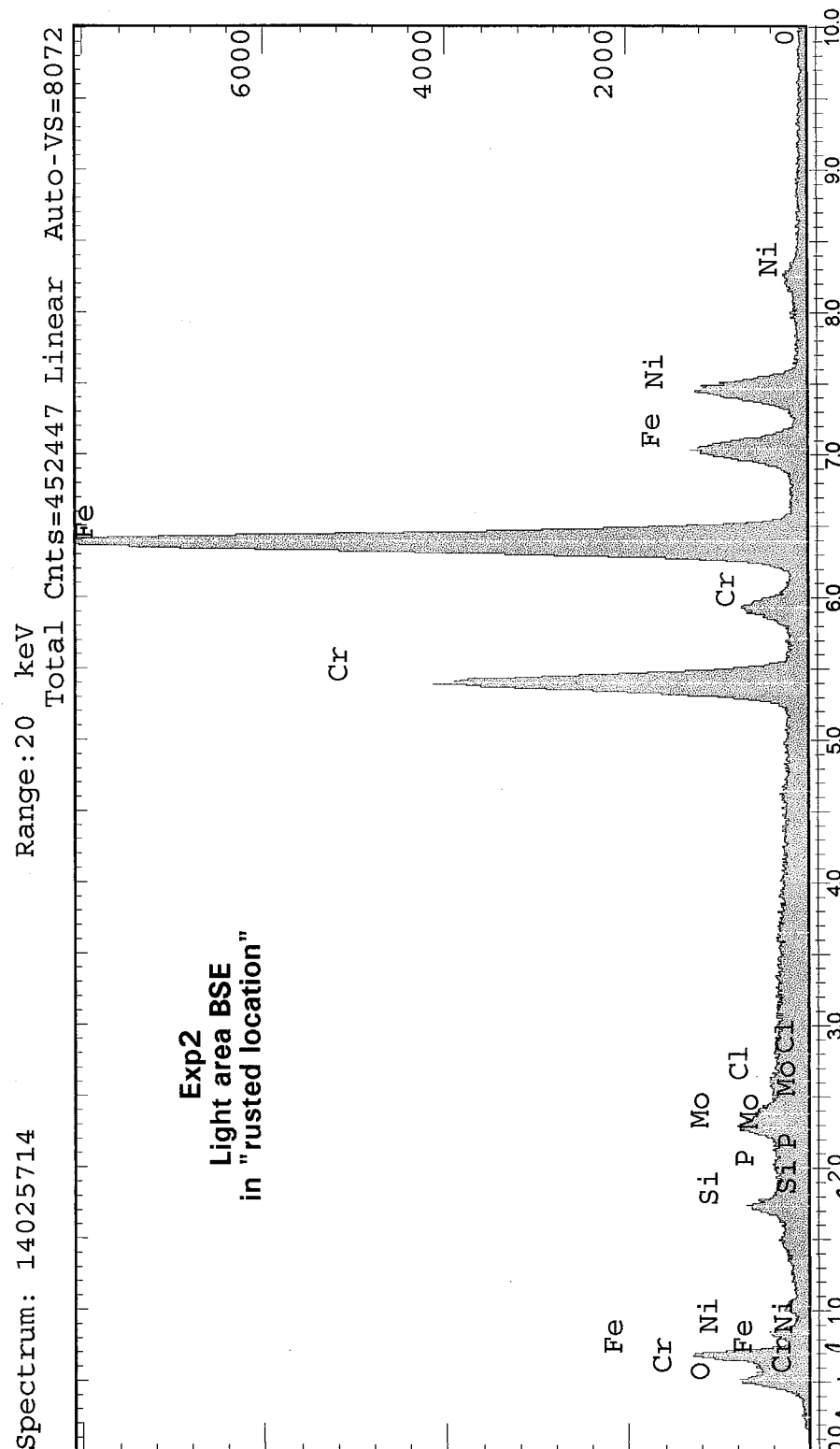
Date

5/20/2002

Recorded by

From Page No.

EDS analysis on SEM image (this is the EDS spectra) on page 46 of this book.



To Page No.

Witnessed & Understood by me,

Date

Invented by

M Nugent

Date

5/20/2000

Recorded by

TITLE

SEM analysis

From Page No.

EDS analysis on SEM image on page 46 of this book.

File: C:\SIGMA\QUASAR\14025713.RPT

[ANALYSIS REPORT]

GENERAL CONDITIONS

Result File : 14025713
File Version : 1
Background Method : Fit
Decon Method : Gaussian
Decon ChiSquared : 3.89
Analysis Date : 8-MAY-2000
Microscope : SEM
Comments : Dark area in "rusted" location

ANALYSIS CONDITIONS

Quant. Method : ZAF/ASAP
Acquire Time : 100 secs
Normalization Factor: 100.00

SAMPLE CONDITIONS

kV : 20.0
Beam Current : 150.0 picoAmps
Working Distance : 29.5 mm
Tilt Angle : 0.0 Degrees
TakeOff Angle : 35.0 Degrees
Solid Angle*BeamCurrent: 0.8

Element	Line	Weight%	Cnts/s	Atomic%
O	Ka	17.05	164.89	40.28
Na	Ka	2.22	18.48	3.65
Si	Ka	1.14	28.88	1.53
Cl	Ka	2.54	79.88	2.70
Cr	Ka	16.57	382.59	12.05
Fe	Ka	49.85	789.98	33.73
Ni	Ka	7.46	83.01	4.80
Mo	La	3.18	38.73	1.25
Total		100.01		

To Page No.

Witnessed & Understood by me,

Date

Invented by

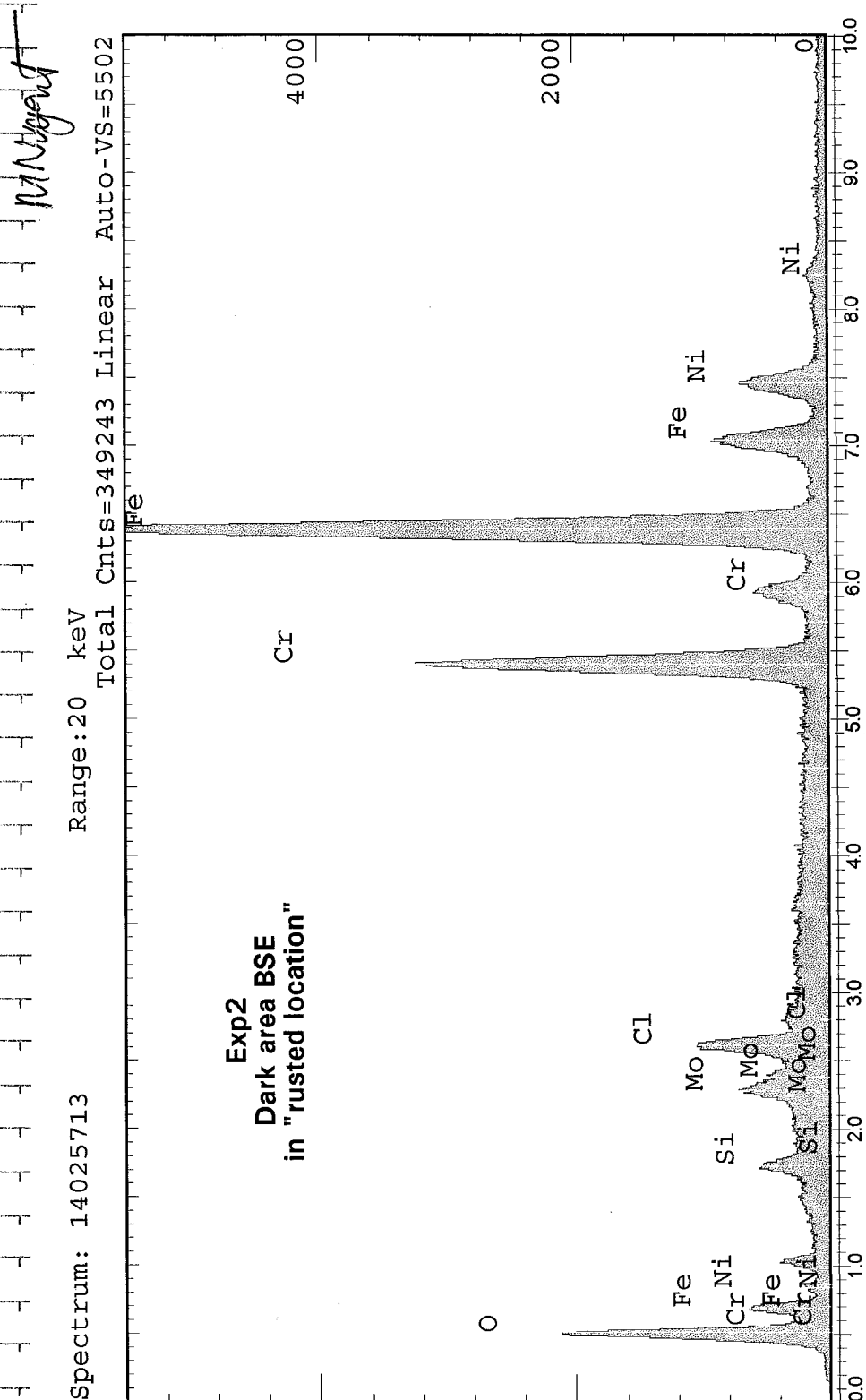
M Nugent

Date

5/20/2000

Recorded by

From Page No. Spectra for EDS analysis on SEM image on page 46 of this book.



To Page No.

Witnessed & Understood by me,

Date

Invented by

M. Nugent

Date

5/20/2000

Recorded by

TITLE Load cell calibration

From Page No.

Load cell Calibration

Load cell calibrations through the Materials Characterization Lab should be performed annually.



Material Characterization Lab

Sheet ___ of ___

Calibration of 1000g LC. Serial No. D40641

Amplifier: Cooper Serial No. 8300613

Voltmeter: Serial No. Cert. Date:

Standard Used: See Attached Sheet Serial No. Cert. Date:

Frame No. By F. Canalep Tens/Comp/other

Date: 11-4-99 Temp: 74 F. Rel. Humidity 38%

Calibration Volts: A: B: 745.2

Grams	Run 1	Run 2	Run 3	Avg.	Error
0	0.0	0.0	0.2	.07	
100	99.9	100.0	100.2	100.03	
200	199.9	200.0	200.2	200.03	
300	300.0	300.1	300.2	300.10	
400	400.0	400.1	400.2	400.10	
500	500.0	500.1	500.3	500.13	
600	600.0	600.2	600.3	600.17	
700	700.2	700.3	700.4	700.30	
800	800.2	800.4	800.4	800.33	
900	900.3	900.4	900.4	900.37	
1000	1000.3	1000.5	1000.6	1000.47	

Y-intercept R² Standard Dev. of Error

Slope α

Poly Fit Coeff. a₀ a₁ a₂ a₃ a₄

$$y = a_0 + a_1(x)^1 + a_2(x)^2 + a_3(x)^3 + a_4(x)^4$$

To Page No.

Recorded by

M. Nugent

5/23/2000

From Page No.

Load Cell Calibration

1000gr-CALIBR.XLS

[illegible]

To Page No.

Witnessed & Understood by me,

Date _____

Invented by

Date _____

Recorded by

by
M Nygerit

5/23/2000

TITLE

Load cell calibration

From Page No.

Load Cell Calibration

Information potentially subject to copyright protection was redacted from this location. The redacted material is from the following reference:

Rice Lake Weighing Systems
230 West Coleman Street
Rice Lake, Wisconsin 54868

Report of Mass Values : 1g & 5g, Class 1, 2g, Class 2,
10g-500g, Class S conducted on 07/21/1997.

Witness

Recorded by

M Nugent

5/23/2020

From Page No.

Load Cell Calibration

Southwest Research Institute

PO# 47548

TEST NUMBER 293056B

PAGE 26

7/21/97

TABLE I.
EXPANDED
UNCERTAINTY
(G)

ITEM	MASS (G)	UNCERTAINTY (G)	VOL AT 20 (CM3)	COEFF. OF EXP
500 g C870	500.00162196	0.00006370	63.77571584	0.00004500
200 g C868	200.00071192	0.00003821	25.51029439	0.00004500
200 g C869	200.00049771	0.00003821	25.51026706	0.00004500
100 g C867	100.00036379	0.00003536	12.75514819	0.00004500
50 g C866	50.00011315	0.00002100	6.37756532	0.00004500
20 g C864	20.00004588	0.00001059	2.55102621	0.00004500
20 g C865	20.00002053	0.00001059	2.55102297	0.00004500
10 g C863	10.00003215	0.00000857	1.27551428	0.00004500
5 g C862	4.99999017	0.00000537	0.62892959	0.00004500
2 g C861	2.00002452	0.00000299	0.25157541	0.00004500
2 g C860	2.00001324	0.00000299	0.25157399	0.00004500
1 g C859	0.99999694	0.00000265	0.12578578	0.00004500

NOTE : THE UNCERTAINTIES ARE CALCULATED ACCORDING TO NIST
TECHNICAL NOTE 1297 IMPLEMENTED JANUARY 1, 1994, SEE REFERENCE
NO. 14. THE EXPANDED UNCERTAINTY IS 2 TIMES THE ROOT SUM SQUARE
OF THE TYPE A AND TYPE B UNCERTAINTIES.

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M. Nugent

5/23/2000

TITLE

Load Cell Calibration

From Page No.

Load Cell Calibration

Information potentially subject to copyright protection was
redacted from this location. The redacted material is from
the following reference:

Rice Lake Weighing Systems
230 West Coleman Street
Rice Lake, Wisconsin 54868

Report of Mass Values: 50g-5kg, Class S, 10kg, Class I,
S.S. Weights conducted on 05/27/1997.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M. Nugent

5/23/2000

From Page No. _____

Load Cell Calibration

Southwest Research Institute

PO# 47548
TEST NUMBER 293056DPAGE 26
7/21/97

ITEM	MASS (G)	TABLE I. EXPANDED UNCERTAINTY		VOL AT 20 (CM3)	COEFF. OF EXP
		(G)	(G)		
1 kg C871	1000.00418404	0.00010594		127.55155160	0.00004500

NOTE : THE UNCERTAINTIES ARE CALCULATED ACCORDING TO NIST
TECHNICAL NOTE 1297 IMPLEMENTED JANUARY 1, 1994, SEE REFERENCE
NO. 14. THE EXPANDED UNCERTAINTY IS 2 TIMES THE ROOT SUM SQUARE
OF THE TYPE A AND TYPE B UNCERTAINTIES.

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M. Nugent

5/23/2000

TITLE

Surface Area analysis

From Page No. _____

Surface area analysis on the porous plates (316L)
were performed by BET surface area analysis.
Three 316L stainless steel porous plates were
sent for analyses.

The masses of the plates, as well as the surface
area analyses, are presented on the next three
pages of this notebook, CNWRA Scientific Notebook
controlled copy # 341.

Surface area analyses were performed by Bob
Farick of Dunsen & [522-2653]

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

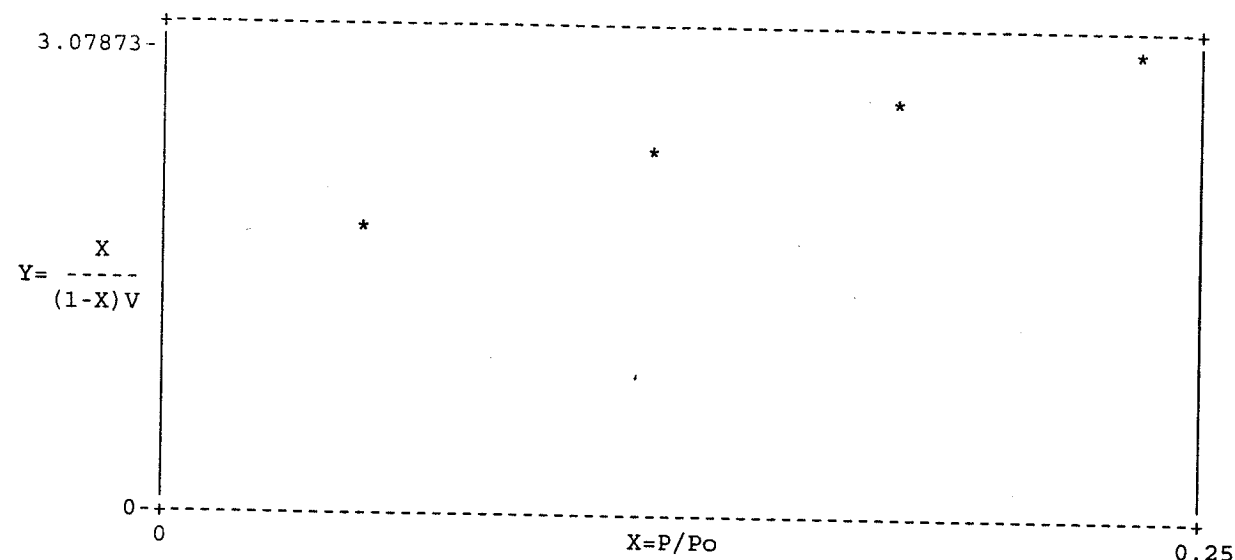
M. Nugent

6/12/2000

From Page No.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300BET SURFACE AREA ANALYSIS
REPORT DATE: 05-07-00SAMPLE I.D.: COUPON#2
SAMPLE WEIGHT: 6.0015 g
MOL. CROSS-SECTIONAL AREA: 0.162 nm²
SAMPLE TEMPERATURE: 0.00 CADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/Po	Y=X/[(1-X)V]
4.990	0.16	0.03	0.0489	1.92993
11.990	0.33	0.05	0.1176	2.42327
17.990	0.48	0.08	0.1764	2.67827
24.000	0.60	0.10	0.2354	3.07873

BET SURFACE AREA: 0.57 +/- 0.03 m²/g
SLOPE: 6.0012 +/- 0.3813
INTERCEPT: 1.6600 +/- 0.0611
C: 4.62
Vm: 0.13 cm³/g
CORRELATION COEFFICIENT 0.9960

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

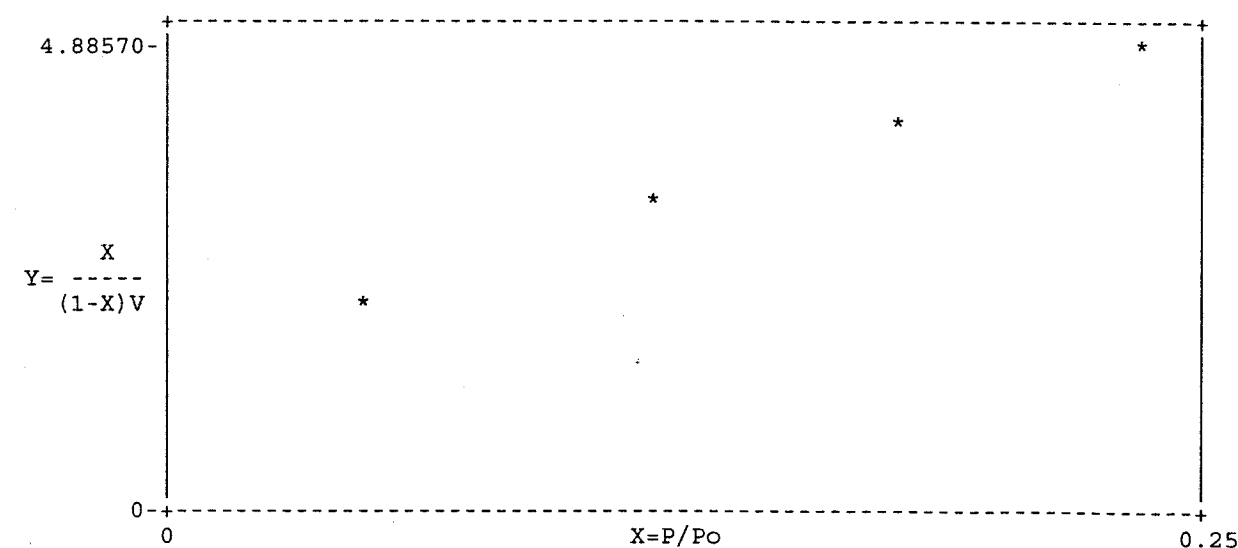
M Nugent

6/2/2000

From Page No.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300BET SURFACE AREA ANALYSIS
REPORT DATE: 5/30/2000SAMPLE I.D.: COUPON #3
SAMPLE WEIGHT: 6.0318 g
MOL. CROSS-SECTIONAL AREA: 0.162 nm²
SAMPLE TEMPERATURE: 0.00 CADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/Po	Y=X/[(1-X)V]
4.990	0.15	0.02	0.0489	2.06899
11.990	0.25	0.04	0.1176	3.21486
17.990	0.31	0.05	0.1764	4.16794
24.000	0.38	0.06	0.2354	4.88570

BET SURFACE AREA: 0.26 +/- 0.01 m²/g
SLOPE: 15.2437 +/- 0.7496
INTERCEPT: 1.3806 +/- 0.1201
C: 12.04
Vm: 0.06 cm³/g
CORRELATION COEFFICIENT 0.9976

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M Nugent

6/2/2000

From Page No.

M. Nugent

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300BET SURFACE AREA ANALYSIS
REPORT DATE: 06-05-00

SAMPLE I.D.: COUPON-1

SAMPLE WEIGHT: 6.0590 g

MOL. CROSS-SECTIONAL AREA: 0.162 nm²

SAMPLE TEMPERATURE: 0.00 C

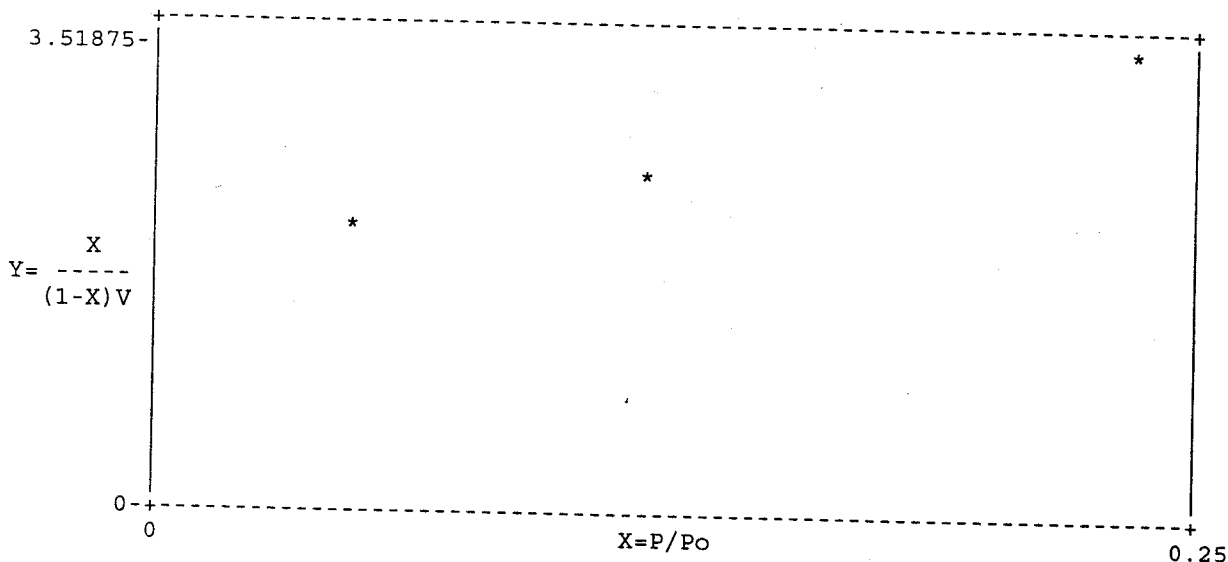
ADSORBATE: Nitrogen

BAROMETRIC PRESSURE: 760 mmHg

SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/Po	Y=X/[(1-X)V]
4.990	0.15	0.02	0.0489	2.07832
11.990	0.32	0.05	0.1176	2.52294
24.000	0.53	0.09	0.2354	3.51875

BET SURFACE AREA: 0.46 +/- 0.03 m²/g
 SLOPE: 7.8058 +/- 0.5194
 INTERCEPT: 1.6610 +/- 0.0802
 C: 5.70
 Vm: 0.11 cm³/g
 CORRELATION COEFFICIENT 0.9978



To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M. Nugent

6/12/2000

SEM analysis

3MN 6/21/2000 2001402571

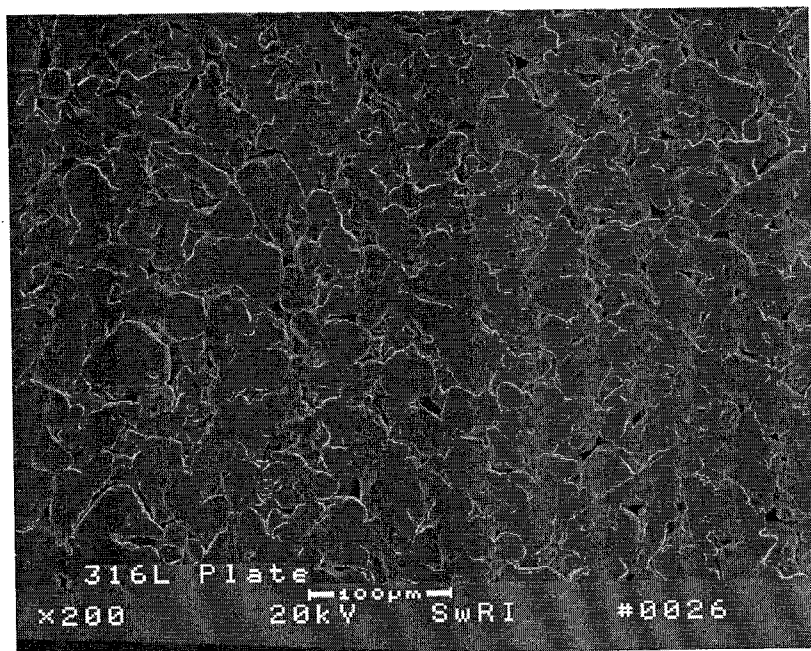
341

Form Page No.

SEM Analysis on unreacted 316L Stainless Steel plate

SEM (scanning electron microscopy) and EDS were performed on an unreacted 316L Stainless Steel plate.

These analyses were performed by Fawn Daby (x 6577) of Division 6 at Southwest Research Institute. The analyses were performed on June 15, 2000.



mw

Witnessed & Understood by me,

Date:

Invented by

Reviewed by

M. Nugent

File No.

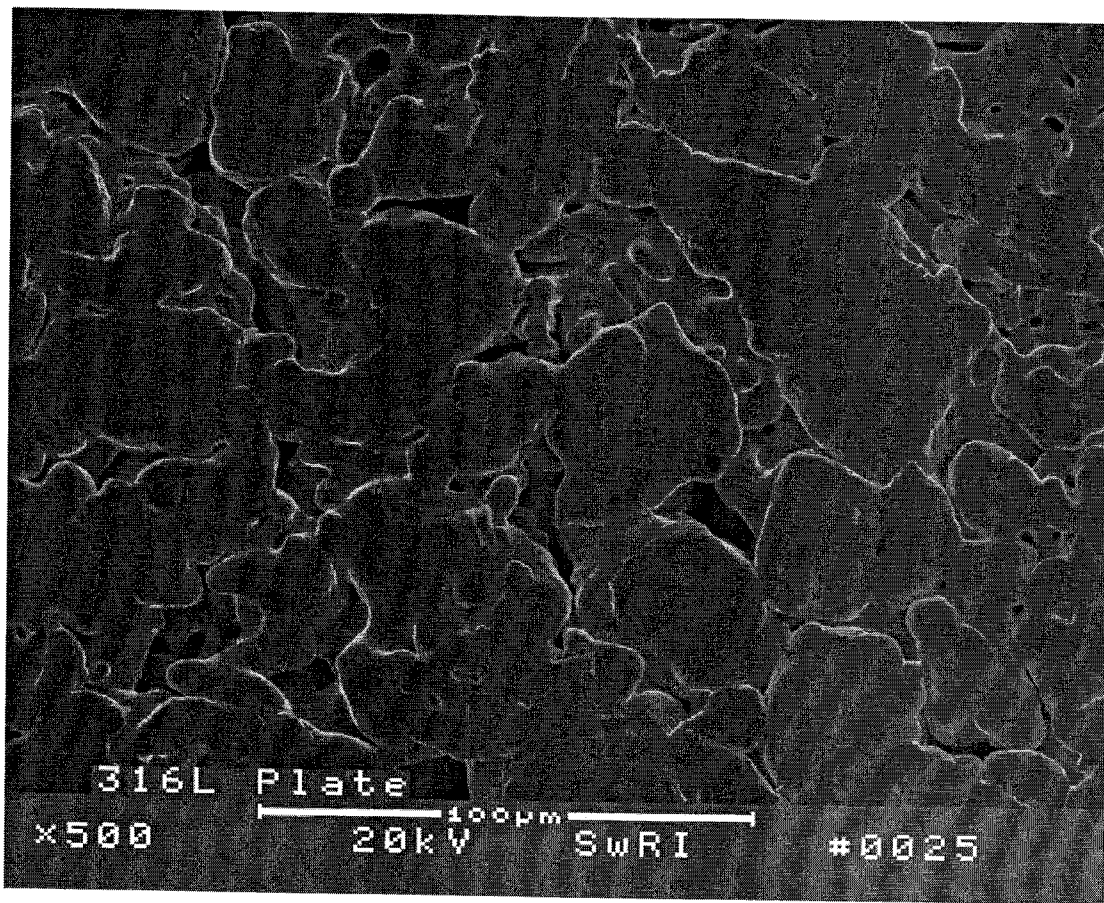
6/21/2000

Project No. 2001402571

Book No. 341

TITLE SEM analysis

From Page No.



To Page No.

Witnessed & Endorsed by me,

Date

Invented by

Date

Recorded by

M. Nugent

6/21/2000

TITLE

SEM analysis

Project No. 200102571

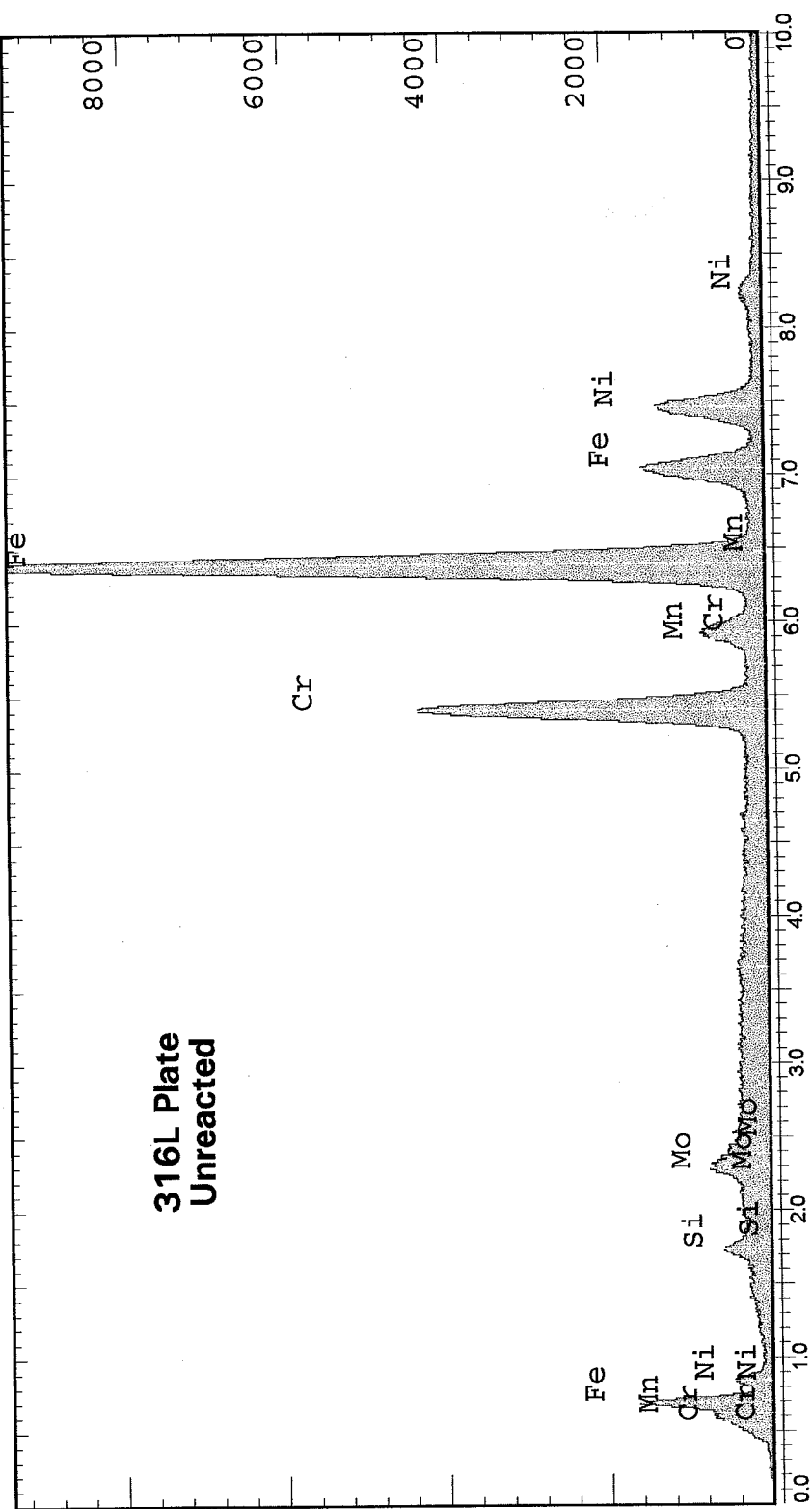
Book No. 341

63

From Page No.

gpm

Spectrum: 1402571D Range: 20 keV Total Cnts=494667 Linear Auto-VS=9381



To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M. Nugent

6/21/2000

From Page

File: C:\SIGMA\QUASAR\1402571D.RPT

[ANALYSIS REPORT]

GENERAL CONDITIONS

Result File : 1402571D
File Version : 1
Background Method : Fit
Decon Method : Gaussian
Decon ChiSquared : 2.12
Analysis Date : 15-JUN-2000
Microscope : SEM
Comments : 316L plate unreacted

ANALYSIS CONDITIONS

Quant. Method : ZAF/ASAP
Acquire Time : 100 secs
Normalization Factor: 100.00

SAMPLE CONDITIONS

kV : 20.0
Beam Current : 150.0 picoAmps
Working Distance : 29.5 mm
Tilt Angle : 0.0 Degrees
TakeOff Angle : 35.0 Degrees
Solid Angle*BeamCurrent: 0.8

Element	Line	Weight%	Cnts/s	Atomic%
---------	------	---------	--------	---------

Si	Ka	0.97	29.45	1.92
Cr	Ka	17.29	531.67	18.49
Mn	Ka	0.00	0.00	0.00
Fe	Ka	66.62	1375.24	66.33
Ni	Ka	12.24	173.26	11.59
Mo	La	2.87	42.85	1.66

Total		99.99		
-------	--	-------	--	--

Page: 1

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M Nugent

6/21/2000

From Page No.

Material Certification for 316L Stainless Steel
plates used in PVF Experiments.

The 316L stainless steel plates were ordered from Mott, Inc.
Material Certification sheets were obtained from Mott Corp.
and are presented on the following page (page 66 of
this scientific notebook, CNWRA Controlled Copy #341)

Note, a Mott representative states that the Lot # is the same
as the heat number, but refuses to provide the name of the
company that provided the 316L stainless steel and the Lot number.

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Melanie Nugent 6/30/2000

From Page No.

ATOMIZED METAL POWDERS

PROCESS/QUALITY CONTROL DATA SHEET

CUSTOMER: MOTT		PRODUCT: P316L LOT #: 114199		DATE: 11/8/99 PO #: 91837 QUANTITY: 2669 LBS.	
CHEMICAL COMPOSITION (Weight %)					
C : 0.028 ✓	Ni: 12.90 ✓	Cr: 16.88 ✓	Fe: BAL ✓	Mo: 2.33 ✓	Si: 0.67 ✓
Mn: 0.42 ✓	Co:	Cu:	W:	S: 0.013 ✓	Cb:
B:	O:	P: <0.01 ✓	:	:	:
<div>CUSTOMER <u>Southwest Research</u> CUSTOMER P/N <u>NA</u> CUSTOMER P/O <u>XENON75</u> P/O LINE ITEM <u>NA</u> MOTT CPN <u>1003750-01-002</u> MOTT S/O <u>49675</u> FROM JOB# <u>49675-01-00</u> DTG <u>9-99</u> QTY SHIPPED <u>100</u> INSP. <u>John Moore</u> DTG <u>9-99</u></div>					
NOTES AND OTHER DATA: <u>Product shipped 12-9-99</u>					
APPROVED BY MNC QC INSP. <u>CC</u> DATE <u>11/12/99</u> <u>John C. White</u>					

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Melina Nugent

6/30/2000

From Page No.

Chemical characterization of the 316L stainless steel plates was performed by Conam Kavin to make sure that the 316L stainless steel plates, obtained from Mott Corp., are within ASTM standards.

The chemical analysis, performed on one sample set to Conam Kavin, are presented on the following page (page 68 of this scientific notebook, CNWRA controlled copy # 341).

The checks indicate that the analyses for Si, P, Cr, Mn, S, Mo, C, and Ni are within the ASTM standard (A699) for 316L stainless steel.

Note, contact information for Conam Kavin is given on the following page (P68 of Scientific Notebook CNWRA # 341).

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Melina Nugent

7/8/2000

From Page No.

CONAM192 Internationale Boulevard
Glendale Heights, IL 60139-2094
Telephone +1 630-681-0008
Facsimile +1 630-681-0009**CONAM**
KAWIN194 Internationale Blvd., Glendale Heights, IL 60139
Telephone +1 630-681-0008
Facsimile +1 630-871-5520SOUTHWEST RESEARCH INST. 7010
6220 CULEBRA RD
P.O. DRAWER 28510
SAN ANTONIO TX 78284
MELISSA NUGENT

P.D. # 50138

DESCR 07-05-00
HEAT# 114199
316L STAINLESS STEEL
REPORT DATE: 07/13/2000

LAB NO: 0710-060 / 01

JOB NO: 07/13 #20

ASTM A666-99

CHEMICAL ANALYSIS

Si	.94 ✓	Mn	.21 ✓	C	.018 ✓
P	.006 ✓	S	.001 ✓	Ni	13.00 ✓
Cr	16.48 ✓	Mo	2.29 ✓		

THE ABOVE CHEMICAL TEST RESULTS CONFORM TO ASTM A699 TYPE 316L STAINLESS STEEL
TEST METHODS: ASTM E 663 ; ASTM E 1019 ; ICP ; ASTM E 354 ;

WKID


G. A. INSPECTOR

ALL CHEMICAL TEST RESULTS ARE REPORTED IN WEIGHT PERCENT UNLESS OTHERWISE NOTED.

PAGE 1 OF 1

THIS REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL,
WITHOUT THE WRITTEN APPROVAL OF CONAM KAWIN, INC.

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

M Nugent

7/8/2000

TITLE

From Page No.

Closing Statement

These initial studies have indicated that the PUF set-up, in its current design, is not suitable for studying the internal waste package chemistry. The experimental set-up will therefore be redesigned exclusively to study corrosion using well defined pores in stainless steel.

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Melissa Nugent

8/21/2000