Final Project Report

Meier Associates and Pacific Northwest Laboratory Staff Exchange: Transfer of Corrosion Monitoring Expertise to Assess and Develop In-Line Inspection Tools for Corrosion Control

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April 1995

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**Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute**

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Meier Associates and Pacific Northwest Laboratory Staff Exchange: Transfer of Corrosion Monitoring Expertise to Assess and Develop In-Line Inspection Tools for Corrosion Control

Staff exchanges, such as the one described in this report, are intended to facilitate communication and collaboration among scientists and engineers at Department of Energy (DOE) laboratories, in U.S. industry, and academia. Funding support for these exchanges is provided by the DOE, Office of Energy Research, Laboratory Technology Transfer Program. Funding levels for each exchange typically range from \$30,000 to \$50,000. The exchanges offer the opportunity for the laboratories to transfer technology and expertise to industry, gain a perspective on industry's problems, and develop the basis for further cooperative efforts through Cooperative Research and Development Agreements (CRADAs) or other mechanisms.

During the past five years, PNL has developed prototype instrumentation to automate the data collection required for electrochemical determination of corrosion rates and behavior of materials in various electrically conductive environments. The last version is labeled Sentry 100 for identification purposes. Now that at least prototype instrumentation is available, there is interest in determining if there is a market for the technology. One of the objectives of this staff exchange was to transfer the technology to an engineering firm (Meier Associates, Kennewick WA) and let the engineering firm assist with defining the industry applications of the technology. Additional objectives were as follows:

Purpose/Objective

The purpose of the proposed staff exchange was to transfer PNL corrosion monitoring expertise, assess the technology for application to industry, and to define in-line corrosion monitoring tools needed for corrosion control. The specific technology-assessment objectives were:

- evaluate linear polarization and Tafel data (Stern-Geary Approach) taken by PNL staff at Boise Cascade (e.g., black/white/filtrate liquors and digester systems) and at the Hanford Waste Tanks for commercial applicability;
- identify critical barriers to transferring the technology to industry;
- identify the type/design of automated data logging instrumentation and corrosion probes needed to economically bring electrochemical techniques to the market; and
- identify the technical services needed to implement advanced instrumentation for corrosion measurement in concert with a local engineering firm.

Summary of Tasks Performed

The Staff Exchange Program between PNL (Dr. Norman Olson) and Meier Associates began October 1993 and concluded June 1994. Electrochemical corrosion monitoring techniques were employed with a prototype instrumentation package by PNL to measure corrosion behavior and estimate corrosion rates of materials in various industrial environments of interest. See Appendix A for a

description of how the techniques were employed using the prototype instrumentation and Appendix B for application to estimating corrosion rates.

The work was divided into three tasks. First, Dr. Olson met periodically with three Meier Associates Staff (Mr. Terry Meier, President; Mr. Steve Strecker; Project Manager for Meier Associates Engineering Contract with Boise Cascade-Wallula and Mr. Mitchell Crowe, an engineer who assisted with on-site measurements at the Wallula mill) to review the technology and to get their input on how to present the technology. Corrosion monitoring results/successes obtained by PNL staff in double shell waste tanks at Hanford; and, Pulping Digesters, Filtrate Systems, and White Liquor Storage Tanks were reviewed with Meier Associates staff. This led to the development of the presentation materials given in Appendix C.

An example of a recent application of the technology and results as applied by PNL at a Pulp and Paper Mill are outlined in Table 1. In this experiment, it can be seen that different results were obtained in the three different white liquors over a period of time. These results verify the need for on-line monitoring instrumentation to define corrosion behavior for storage tank materials of interest. In this case, the four alloys were measured under laboratory conditions because on-line monitoring was not allowed. White liquor samples (1-3) were drawn at different times and the corrosion behavior and rates were measured in the Wallula laboratory at temperature using heaters ($\sim 190^\circ\text{F}$). Based on these results, the mill elected to replace the Type A36 white liquor storage tank with 304 stainless steel.

This review example was the most important to Meier Associates because of their contract for A&E services with the Boise Cascade-Wallula Mill. Dr. Olson assisted with the presentation to Boise Cascade on how the technology would help them identify a better material for their black liquor tank which was corroding rapidly at the condensate/black liquor interface. Again, we were not allowed to monitor the tank in-situ as desired and had to resort to the laboratory approach used in the previous example with white liquors. However, the problem did offer the chance to transfer the technology to Meier Associates staff under real conditions.

The technology was applied at the Boise Cascade Wallula Mill to a Black Liquor Storage Tank for which Boise Cascade was in the process of replacement planning. Dr. Olson transferred the technology to Mr. Strecker and Mr. Crowe during the course of the corrosion monitoring program as a technology transfer project. The results showed that 304 stainless steel would be the appropriate replacement material for the carbon steel tank and the tank was replaced by Meier Associates and Boise Cascade.

The data of interest upon which the decision was made are shown in Table 2. Note that the difference in E_{corr} between the black liquor and condensate would lead to galvanic corrosion at the interface. Unfortunately, the condensate solution was accidentally discarded by Mill staff prior to making the corrosion rate measurements. The Mill went ahead with the decision to build the new tank out of 304 stainless steel based on these results and consulting with other mills on their successful experience with 304 stainless steel in white and black liquor environments ($\text{pH} > 12$, $T \sim 190^\circ\text{F}$, $\text{NaOH}/\text{NaSO}_x$ solutions).

Meier Associates were comfortable taking the required data with the Sentry 100; however, they concluded they did not have the technical background to make the data interpretations required for the

materials selection process. Hence, technical services would be required from PNL or a spin-off company promoting the use of the technology application.

The second task was to attend the Technical Association of Pulp and Paper Industry Continuous Digester Corrosion Task Group Meeting in Richmond, BC March 18, 1994. At the meeting, Dr. Olson discussed the application of the technology with Pulp and Paper Engineers active in addressing corrosion problems in their industry. Meeting notes and attendees are given in Appendix D. Discussions with the participants were helpful in establishing the pulp and paper industrial market needs. Notable results from the meeting were that the Pulp and Paper Industry is interested in using the technology to monitor corrosion behavior, but the budgets to do so are too small to properly address the problems because of the poor economic condition of the industry.

A third task was to get feed back from potential points of sale on corrosion services needed to expand the application of the technology and from potential instrument manufacturers. Ten presentations were made outside the Eastern Washington area and numerous telephone discussions were conducted.

Technical presentations were made in concert with Meier Associates to Siemens Nuclear in Richland. In addition, presentations were made by Dr. Olson to Weyerhaeuser's Corporate Technology Center (Federal Way/WA), Pacific Western Services (Silverdale/WA), the Washington State Department of Ecology - Underground Storage Tank Division, ZETRON (Redmond, WA), Campbell Scientific Instruments (Logan, UT), CORTEST, Inc (Columbus, OH), Johnson Controls-World Services (Bangor/WA division), Corrosion Control Specialists (Kent, WA), and the Naval Undersea Warfare Center (Keyport/WA). Telephone discussions with Petrolite-Instruments (competing instrumentation) and Petroleum Industry representatives were also held. Two of the presentations led to a second related Staff Exchange Program (see Recommended Follow-On Work).

Significant Accomplishments

The significant accomplishments of this study were as follows:

Market: The real market for this technology is knowhow or technical services, rather than instrumentation sales. The true market would have to be created. It does not exist in terms of directly decreasing maintenance costs to make industry more competitive. Instead there may be an untapped market that is driven by environmental regulations, inspection regulations, and safety considerations (Table 3). While the expected customers may include industries such as Pulp and Paper, Chemical processing, and Hazardous Waste sites, the primary points of sale are the companies serving the industries where the technology would be applied. Hence vendors of equipment like pulping digesters, paper machines, etc to pulp and paper industry appear to be more logical points of sale than for example, direct sales to engineering or maintenance groups at a paper mill. Other primary points of sale are A/E engineering firms and performance based operation and maintenance companies (e.g., Johnson Controls World Services) who have the O&M contracts for industrial and federal sites. Their profits are augmented when they save money for their customers via their performance contracts.

The marketability of a company providing corrosion monitoring services would be enhanced by adding similar business elements such as environmental sensors that require automated data

acquisition and interpretation. Linking or networking with companies providing information services or database development could also increase potential profits.

Instrumentation Required to Economically Bring the Technology to Market: The initial thoughts were that the required instrumentation could be made user friendly and the customer would be the industry itself; e.g., sell the instrumentation and computer monitoring systems to a pulp and paper mill and their engineering and maintenance organizations would use the systems to minimize the cost of corrosion. The customers would buy the lowest priced systems if they were on the market. Instead the consensus of those involved with the project and the Industrial Companies, Federal Agencies, and Instrumentation contacts was that Technical Services (Knowhow) was the major business of the technology. In general, US Industry today is not staffed to handle data acquisition projects requiring technical interpretation. They outsource such requirements when needed. The competition is not other instrument companies, but other consulting companies.

The cost of the instrumentation and added bells and whistles are not considered major market factors (Table 4) by the people contacted in this study. However, the Sentry 100 prototype will significantly reduce the cost of data acquisition compared to present commercial competition.

Critical Barriers to Transferring the Technology: While the technology has been utilized extensively in the laboratory, there are no standard procedures for field use. There is not wide-spread acceptance of the technology for field use. In-situ monitoring for corrosion control is not a budgeted item for operation and maintenance for any of the industrial contacts used in this study. More successful field demonstrations are required and published results.

Corrosion Services Required: The corrosion services required are to provide the instrumentation to make the required measurements, make the measurements, report the results, and recommend remedial action. The corrosion services company could be independent or a separate division of an engineering firm. More rapid market development for a start-up company would occur if the company were networked with/or a division of a larger firm dealing with operation and maintenance issues on a worldwide bases (e.g., Johnson Controls Worldwide Services).

Significant Problems

No significant problems occurred during this staff exchange.

Industry Benefits Realized

The primary industry benefits realized through the exchange were improved guidance for a local pulp and paper company in replacing two processing tanks (for white liquor and black liquor) with tanks made of appropriate materials. It is difficult to place a dollar value on these benefits, but such tanks are major capital cost items in the industry. In addition, both the pulp and paper company and the engineering company that serves it received a practical, first-hand demonstration of the potential benefits of the PNL Sentry 100 corrosion monitoring technology.

According to Battelle studies (circa 1981), it has been estimated that corrosion costs in the US are greater than \$250 billion/year. The cost of corrosion is generally passed onto the consumer. Hence

there could be considerable cost savings to industry to get ahead of the game by monitoring corrosion behavior before failures happen. New drivers such as environmental regulations, inspection regulations, and safety liabilities should make the technology application more economically feasible in light of global competition.

Recommended Follow-On Work

Two recommendations for follow-on work resulted from this staff exchange. One was to broaden the PNL experience with the technology application beyond Pulp and Paper and Nuclear Waste Sites. Work on a second related staff exchange (PNL 22301) was initiated August 3, 1994 in Marine environments with Pacific Western Services, Silverdale Washington. This work will be completed in a year and is expected to address some of the technology limitations and provide additional field successes.

A second recommendation is to look for a CRADA partner to develop the commercial instrumentation. Initial discussions with Petrolite-Instruments indicated that they did not have the financial incentive (\$1 million) to upgrade their instrumentation to those of the Sentry 100 prototype. Prior to pursuing this recommendation, the current instrument market (programmable data loggers) should be reviewed for supportive technology applications to provide a basis for the commercial version of the Sentry 100. It is not clear if competitive instrumentation is a viable partnering approach. Candidate partnering companies such as Johnson Controls Instruments (who are not currently in the corrosion business) should be approached for immediate application to Johnson Controls World Services markets.

Appendix A

Features of the Sentry 100 Prototype Corrosion Data Scanner

APPENDIX A

FEATURES OF THE SENTRY 100 Prototype Corrosion Data Scanner

OVERVIEW: The Sentry 100 Corrosion Prototype Data Scanner is designed to automate the collection of potentiodynamic scans, open circuit potential or corrosion potential (E_{corr}), and the data required for the Stern-Geary approach to estimating corrosion rates. The scanner records data in a form convenient for importing into commercial spreadsheets or computer assisted graphing and analysis packages. The Sentry 100 records or logs data on 12 channels; four (4) scanning (working electrode) channels and eight general purpose (e.g., pH, temperature) channels.

The top view of the Sentry 100 is shown to the right. Menu screens are prompted from the key pad. Settings may be made from the key pad or the user friendly, mouse driven software provided with the Sentry 100. An operations manual is also provided with the system.

The Sentry 100 is based on an Intel 8032 chip (12-bit microprocessor) programmed in PLM, and 96K RAM. The Sentry 100 has eight general purpose channels (for temperature, pH, etc) and four (4) scanning electrochemical potential channels for working electrodes. Data are stored in the battery backed RAM. Permanent data storage and data analysis are accomplished by downloading into any computer equipment through the RS-232 port. Some of the Sentry 100 features may be outlined as follows:

- 160 Dot X 128 Dot Graphics Display
- 20 Button Key Pad with Soft Keys
- Real Time Clock
- 96K Bytes Battery Backed RAM
- 64K Bytes Programmable EPROM
- Analog PCB
- Low Noise 12 Bit Precision A/D
- 2 to +2 Volt Probe Drive to 0.204 amps

8 General Purpose Channels
 4 Scanning Electrochemical Probe Inputs/Outputs
 Auto Scaling
 LOTUS or Other Spreadsheet Data Output Format
 Low Power CMOS Design, and a
 High Impact Sealed ABS Case
 Overall Size is 10 X 6 X 4 Inches
 Voltage Resolution is +/- 1mV
 Current Sensitivity is 10nA

ESTIMATION OF CORROSION BEHAVIOR:

Potentiodynamic scan and E_{corr} measurements are used to estimate expected corrosion behavior. Utilization of a forward scan for this purpose is shown in the example to the right. After the forward scan is completed, the equilibrium corrosion potential is used to estimate the expected corrosion behavior. For example, one would expect passive corrosion behavior for an $E_{\text{corr}} = 0$ in the Figure to the right, where the anodic region is shown for a metal having one passive region. Each side of the passive region is bounded by increasing corrosion currents, active-passive to the left side and transpassive on the right side. Experience has shown, for example, that one may expect pitting and/or stress corrosion cracking in the active-passive and transpassive regions because a passive/protective surface layer will not form.

FORWARD SCAN

Three types of applied voltage versus current scans may be made on working electrodes in a given corrosive environment. These are the forward scan as shown above, reverse scan, and scans required to determine the Tafel Slopes required for the Stern-Geary analysis to estimate corrosion rates, which is referred to as Tafel Slope or Tafel Scans (See Appendix B). Scan rates may selected between 0.01(effective) and 99 mV/s using the Sentry 100, as well as start times and voltage ranges.

The reverse scan after a forward scan is used primarily to estimate the pitting potential and stability of passive regions. A reverse scan produces a forward scan (one direction) and then automatically reverses to the opposite direction after the forward scan has been completed. Pitting potentials are determined either by starting from a cathodic voltage as shown in the Figure to the right. Or more generally scans to determine pitting potential start from E_{corr} .

The start, reverse, and stop voltages are programmable in the Sentry 100.

A36 CARBON STEEL IN BZ FILTRATE FORWARD/REVERSE SCAN

The Stern-Geary approach for estimating corrosion rates is assisted with a scanning program to provide the data required to calculate Tafel slopes (shown in Figure to right) and a voltage change program (± 10 mV) to provide the current change data to complete the analysis once the slopes are known. The voltage change program is referred to as "delta I." An illustration of the method for estimating corrosion rates is given in Appendix B.

The scanner's RAM is large enough to hold one maximum size scan (reverse scan, 1mV/sec, -2.0V to +2.0V to -2.0V) for each scanning channel, 46 delta I scans and 1104 E_{corr} recordings for each channel. Logging 1104 E_{corr} records is enough storage capacity for 46 days when the data logging interval is one hour. E_{corr} (open circuit potential) for the four scanning channels is recorded in the Log Data subprogram.

A36 IN BZ FILTRATE 2mV/s TAFEL DATA (TAF012)

The Sentry 100 is operable either from the front panel keyboard, or, via the serial port, with either a user friendly program, or your favorite terminal emulator. The user friendly program is supplied with the Sentry 100 and is described in the Operations Manual.

The automated recording capability of the Sentry 100 gives the user considerable flexibility such as logging data remotely over long periods of time (as shown in the Figure to the right). Of course, the data may be retrieved at any time. The Sentry 100 has a clock and all data are recorded by channel and the time taken.

In the event of a power failure, the scanner upon power restoration, will check to see if it was in the waiting mode and if it was supposed to have taken data during the outage. If so, it will take log and delta I data and continue at the log and delta I intervals. Scans missed during the outage will be rescheduled for 1-1/2 hour intervals starting 1-1/2 hours after power restoration. The scanner will remain in the same serial port state (on-line or off-line) which it was in before the power failure.

BZ FILTRATE Ecorr DATA MARCH

PROBE AND WORKING ELECTRODE DESIGN: There are many configurations possible for probe or working electrode designs using the Sentry 100. The Sentry 100 cable connections can be wired to use separate or independent reference electrodes for each of the four working electrodes if all four scanning channels are used; or, the four working electrodes may be connected to a common reference electrode. This would allow one Sentry 100 to be used to probe up to four different locations with a single connector using a given working electrode alloy or four different working electrodes at one location, for example. While four working electrodes is the optimum probe design for automated scanning with the Sentry 100, more working electrodes may be monitored by a single Sentry 100 by manually changing wiring connections to the various electrodes as desired.

An example of a six electrode corrosion probe using a common reference electrode is shown

below. In this case, six working electrodes were embedded (cast) in Scotchcast along with the reference electrode and a thermistor to measure temperature. The outside of the casting is metal such as stainless steel which can serve as the counter electrode. Due to the sensitivity of the Sentry 100, one should start with small working electrode surface areas on the order of 1 cm² (acceptable current range is 200mA to 10nA). This probe design is ideal for immersion in Tanks, for example. Only four electrodes may be monitored at a time with the Sentry 100.

EXAMPLE OF MULTIPLE WORKING ELECTRODE CORROSION PROBE

Table 1: Corrosion Rates and Behavior of Four Metals in White Liquor Solutions at a Pulp and Paper Mill. Shows the variation that occurs in three white liquor compositions at different times (Reference: NJ Olson, Evaluation of Corrosion Behavior of Metals in Boise Cascade-Wallula White Liquor January 1992 (BNW 19364). The Mill switched from A36 to 304SS for future White Liquor Storage tank replacements based on these results.

	A36	A106	304SS	IN600
WHITE LIQUOR 1 (Electrodes Conditioned)				
(11/6-11/25/91)				
Ecorr(Mo* REF)	-0.354	-0.357	-0.073	-0.076
Corrosion Behavior	Active	Active	Passive	Passive
Beta (mV)	89	75	37	24
Average Corrosion Rate (MPY)	538	389	4	4
WHITE LIQUOR 2				
(11/25-12/05/91)				
Ecorr(Ag/AgCl* REF)	0.118	0.122	0.137	0.121
Corrosion Behavior	Passive	Passive	Active	Active
Beta (mV)	66	57	42	17
Average Corrosion Rate (MPY)	41	15	14	21
WHITE LIQUOR 3				
(12/05-18/91)				
Ecorr(Ag/AgCl* REF)	0.121	0.134	0.133	0.120
Corrosion Behavior	Passive	Passive	Active	Active
Beta (mV)	18	18	25	25
Average Corrosion Rate (MPY)	47	106	16	45

A36 = Carbon Steel Similar to the White Liquor Storage Tank Material

A106 = Carbon Steel

304SS = 304 Stainless Steel

IN600 = Inconel 600 (Nickel Base Alloy)

Table 2: Staff Exchange Experiment. Corrosion rates and behavior of four metals in Black Liquor and Condensate at Boise Cascade - Wallula Mill. The Mill replaced the A53 type Black Liquor storage tank with 304 stainless steel before the condensate corrosion rates were completed (see text).

	A53	304SS	A537	IN600
Area (CM**2)	3.43	1.61	1.41	3.23
BLACK LIQUOR				
Ecorr(VOLTS,Mo* REF)	0	0.004	0	-0.015
Corrosion Behavior	Passive	Passive	Passive	Passive
Average Corrosion Rate (MPY)	23	7	68	15
CONDENSATE				
Ecorr(VOLTS,Mo* REF)	-0.288	0.214	-0.256	0.188
Corrosion Behavior	Active	Passive	Active	Passive
Average Corrosion Rate (MPY)	Not Done	Not Done	Not Done	Not Done

A53 = Carbon steel similar to the Black Liquor storage tank.

304SS = 304 Stainless Steel

A537 = Carbon steel used in pressure vessels

IN600 = Inconel 600 (Nickel Base Alloy)

Table 3: Market Drivers, Primary Sites for Application, and Points of Sale for the Technology.

<u>Market Drivers</u>	<u>Primary Sites for Application Include:</u>	<u>Points of Sale</u>
<p>Environmental (Corrosion causes contamination)</p> <p>Regulations (State and Federal tank inspection requirements)</p> <p>Safety (Can't afford the liabilities of failures)</p>	<p>Pulp and Paper</p> <p>Marine</p> <p>Hazardous Waste Sites</p> <p>Petroleum (refineries, pipelines)</p> <p>Chemical Production</p>	<p>Engineering Service Firms</p> <p>Vendors (e.g., Pulping Digesters)</p> <p>Cathodic/Anodic Protection Firms</p> <p>Insurance Underwriters</p> <p>Performance Contractors (e.g., O&M contractors who make additional profit from savings-particularly those who serve government sites)</p>

Table 4: Comparison of Sentry 100 Attributes with Competition

ATTRIBUTE	COMPETITION ⁽¹⁾	SENTRY 100
# SCANNING CHANNELS	1	4
# GENERAL PURPOSE CHANNELS (pH, T, etc)	0	8
LOWEST CURRENT	10 ⁻⁶ A	10 ⁻⁸ A
AUTOMATED RECORDING	YES	YES
AUTOMATED PROGRAMMING	NO	YES
AUTOMATED DOWNLOADING OF DATA (e.g., PHONE)	NO	YES
INDUSTRIAL HARDENED (Can be used continuously in an industrial environment)	NO	YES
DATA COLLECTION RESUMES AFTER POWER FAILURE	NO	YES
DATA OUTPUT COMPATIBLE WITH COMMERCIAL SPREADSHEET PROGRAMS	YES	YES
DATA DELIMITERS CAN BE CHANGED	NO	YES
BATTERY BACK UP	YES	YES
SIZE	SUITCASE	10x4x6 in.
PRICE (1994)	\$15000	Est \$10000

NOTES:

- (1) Competition is a laptop computer attached to an electronic box which is carried in a suitcase - such as the Petrolite 4300 and EG&G Research Model 350.

Appendix B

Estimating Corrosion Rates from Tafel Data

APPENDIX B

ESTIMATING CORROSION RATES FROM TAFEL DATA

The purpose of this Appendix is to show an example of how to estimate corrosion rate using the Stern-Geary approach and the data obtained with the Prototype Sentry 100. It is assumed the reader is familiar with the approach and this Appendix is primarily offered to explain how the Sentry 100 logged data are utilized in the analysis rather than explain or defend the method. It is also assumed that the reader is familiar with the limitations of the method. For a more complete review of the method, the reader is referred to references that address electrochemical principles of corrosion such as Anodic Protection by Olen Riggs and Carl Locke, Plenum Press, New York, 1981, pps 205-226.

For the Stern-Geary approach, one needs to estimate a value for I_{corr} which is the actual corrosion current at E_{corr} , i.e.,

$$\text{Corrosion Rate} = K \cdot I_{corr} \quad \text{Eq. [B1]}$$

where, K is a constant defined for a given metal

$$K = 128,600 [\text{at.wt}/[(\text{valence})(\text{density})], \text{MPY}/(\text{Amp}/\text{cm}^2) \quad \text{Eq. [B2]}$$

MPY = mils/year

Calculating for Iron (Fe), for example

$$K = 457,000 \text{ MPY}/(\text{Amp}/\text{cm}^2) \quad \text{Eq. [B3]}$$

The Stern-Geary method to estimate I_{corr} from the Tafel slope data is as follows.

$$I_{corr} = \text{BETA}/R_p \quad \text{Eq. [B4]}$$

where, R_p = Polarization Resistance

$$1/R_p = dI/dE|_{E=E_{corr}}$$

$$\text{BETA} = \text{Ba} \cdot \text{Bc} / [2.3 \cdot (\text{Ba} + \text{Bc})] \quad \text{Eq. [B5]}$$

Appendix B

As an example, the Tafel Scan (slope) data for the A36 iron base alloy in the BZ filtrate was taken and can be imported into Lotus 1-2-3, EXCEL, or QUATTRO PRO (Trademarks) Spreadsheets. The data are plotted in Figure B1 and the linear regressions to determine B_a and B_c were performed in the spreadsheet analyses. The values for B_a and B_c and resultant $BETA$ are shown in Figure B1.

The slopes for this example are:

$$SLOPE_a = 1.8265$$

$$SLOPE_c = -1.0601$$

and the resultant values for B_a and B_c are:

$$B_a = 547 \text{ mV/Decade}$$

$$B_c = 943 \text{ mV/Decade}$$

Using Eq. [B5],

$$BETA = 151 \text{ mV}$$

Figure B1: Estimating Tafel Slopes

Appendix B

To obtain and estimate of $1/R_p = dI/DE)_{E=E_{corr}}$, we import the data from the Sentry 100 delta I program, as follows.

Channel Number	0	1	2	3
Time (hour,minute)	15,0	15,8	15,16	15,24
Date (month,day)	6,18	6,18	6,18	6,18
	<u>Volts,mA</u> 0.215,0.00050 0.205,0 0.194,-0.00020	<u>Volts,mA</u> -0.083,0.00324 -0.093,0. -0.103,-0.00348	<u>Volts,mA</u> -0.061,0.00294 -0.071,0 -0.081,-0.00392	<u>Volts,mA</u> -0.306,0.00424 -0.316,0 -0.326,-0.00229

The hold times at E_{+10mV} , E_{corr} , and E_{-10mV} were two minutes for the respective I(mA) recordings for the data in this report.

The A36 alloy is channel 3 (Area = 1 cm² so dI may be estimated as follows for the +/-10 mV or 20 mV range used for dE:

$$dI \sim 0.00424 - (-0.00229) \text{ mA/cm}^2 = 6.53 \cdot 10^{-6} \text{ A/cm}^2$$

Using Equations [B4]-[B5], the corrosion rate for the iron base alloy (A36) may be estimated as

$$MPY = 457,000(MPY/(A/cm^2) \cdot 151mV \cdot 6.53 \cdot 10^{-6} (A/cm^2)/20mV = 22.5 \text{ mils/year}$$

Once BETA is obtained and E_{corr} does not change, one only needs to continue the delta I program to estimate corrosion rates over time periods of choice.

Appendix C

Examples of Presentation Materials

Electrochemical Corrosion Control

- In-Situ Monitoring

Real Conditions

- Define the Problem(s)

Estimate Instantaneous Corrosion Rate

Predict Corrosion Behavior

Estimate Pitting Potential

Are There External Factors (e.g., Impressed Voltages) Contributing to Problem?

Automated Data Logging and Collection

- Define the Solution

Change Materials (e.g., Stainless Steel Versus Carbon Steel)

Change the Corrosion Environment

Anodic or Cathodic Protection

- Benefits

Reduced Maintenance Costs

Schedule Longer Life Replacements Before Failure

In-Situ Corrosion Surveillance

CORROSION MONITORING

ELECTROCHEMICAL APPLICATIONS

- **PROBE CONSISTS OF WORKING, REFERENCE, AND COUNTER ELECTRODE(S),**

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

- **ESTIMATE INSTANTANEOUS
CORROSION RATE**
- **PREDICT CORROSION
BEHAVIOR
(e.g., Stress Corrosion
Cracking)**
- **ESTIMATE PITTING POTENTIAL**
- **AUTOMATED DATA LOGGING
AND PROGRAMMING
ARE REQUIRED**

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

- INSTANTANEOUS VS
AVERAGE CORROSION
RATES

VALUE OF INSTANTANEOUS MEASUREMENTS
MPY: INSTANTANEOUS VS AVERAGE

CORROSION POTENTIAL ELECTROCHEMICAL APPLICATIONS

- STERN - GEARY APPROACH
TO DETERMINE BETA FOR
CORROSION RATE ESTIMATES

A36 IN BZ FILTRATE
2mV/s TAFEL DATA (TAF012)

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

**TAFEL APPROACH (STERN-GEARY)
MAY OVER ESTIMATE CORROSION
RATE BECAUSE**

- 1) ALL ELECTRONS ARE NOT
ASSOCIATED WITH
CORROSION**
- 2) TAFEL MODEL MAY NOT
BE CORRECT**
- 3) DID NOT WAIT LONG
ENOUGH - EQUILIBRIUM NOT REACHED
e.g., Parabolic Behavior**

PARABOLIC CORROSION BEHAVIOR

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

- POTENTIODYNAMIC SCAN
(LINEAR POLARIZATION)
DEFINES POTENTIAL
CORROSION BEHAVIOR

FORWARD SCAN

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

- OPEN CIRCUIT POTENTIAL (CORROSION POTENTIAL IS DENOTED BY E_{corr}) MUST BE MEASURED OVER LONG PERIODS OF TIME

BZ FILTRATE E_{corr} DATA
MARCH

CORROSION MONITORING

ELECTROCHEMICAL APPLICATIONS

- **USING THE SCAN, CORROSION BEHAVIOR CAN BE MAPPED AS A FUNCTION OF TIME BY RECORDING E_{corr}**

CORROSION MONITORING ELECTROCHEMICAL APPLICATIONS

- PITTING POTENTIAL AS
DETERMINED FROM SCANS

A36 CARBON STEEL IN BZ FILTRATE
FORWARD/REVERSE SCAN

CORROSION MONITORING

SENTRY 100

- WASTE TANK

*Sentry 100 Corrosion Data Scanner
(Example of Application)*

CORROSION MONITORING

SENTRY 100

- POTENTIAL CANDIDATES
FOR CORROSION MONITORING
IN PULP AND PAPER PLANTS

SENTRY 100 CORROSION DATA SCANNER
ALLOWS CORROSION MONITORING AT
MULTIPLE LOCATIONS FROM A
CENTRAL COMPUTER SYSTEM
(PULP AND PAPER EXAMPLE)

Appendix D

TAPPI Continuous Digester Corrosion Task Group Minutes

June 22, 1994

**Re: TAPPI CONTINUOUS DIGESTER CORROSION TASK GROUP
MINUTES**

Dear Task Group Member,

Enclosed please find a copy of the minutes of the all-day Task Group meeting held at Bacon Donaldson Consulting Engineers on March 18, 1994. Please read them, and let me know if there are any errors or omissions.

The next meeting of the TAPPI Continuous Digester Corrosion Task Group will be during the TAPPI Engineering Conference, to be held September 19-22 in San Francisco.

Task Group members are encouraged to report cases of digester cracking or thinning. It is through sharing experience that this committee best fulfills its mandate to inform digester owner/operators of materials and corrosion problems and their solutions.

If you would like to make a short presentation (e.g., 5-15 minutes) at the next Task Group meeting, please let me know and I will include you on the agenda.

Yours truly,

Dr. Angela Wensley, P.Eng.
TASK GROUP CHAIRPERSON

ADVANCING TECHNOLOGY AND PROFESSIONAL ACHIEVEMENT IN THE PAPER AND RELATED INDUSTRIES

Please Reply To:

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MINUTES

TAPPI Continuous Digester Thinning Task Group

CA 920402.04

9:00 am - 5:00 pm, March 18, 1994

Bacon Donaldson Consulting Engineers

Richmond, BC, Canada

1.0 INTRODUCTIONS

Task group chairperson Angela Wensley welcomed the 46 attendees to a day-long meeting devoted to the issue of rapid thinning of continuous digesters.

2.0 ANTI-TRUST STATEMENT

Angela Wensley stated that the meeting would be held in accordance with TAPPI anti-trust guidelines.

3.0 MINUTES OF PREVIOUS MEETING

The minutes of the previous meeting, held on September 22 1993 during the TAPPI Engineering Conference in Orlando, Florida, had been mailed out to the task group in October 1993. The minutes were approved with one change: Dave Bennett said that the vapour dome corrosion mechanism explanation for the Michigan digester had nothing to do with the Union Camp mill.

4.0 DIGESTER THINNING SURVEYS

The results of surveys of continuous digester owner/operators in both North America and outside North America were presented. Both surveys employed the same questionnaire, which was drawn up by this task group.

4.1 NORTH AMERICAN SURVEY

Angela Wensley re-presented the results of a survey of 95 North American continuous digesters. These results were previously given as a paper during the 1993 TAPPI Engineering conference. The paper has been published in the conference proceedings, so the detailed data are not given here. About 25% of the respondents reported thinning, either as the result of slow corrosion over the life of the digester (> 2.5 mm), or more rapid thinning in a single year (> 0.5 mm). No single factor was identified as the cause of the thinning problem. Although digesters running MCC or EMCC appeared to experience more thinning problems, rapid thinning nonetheless occurred in digesters not running MCC or EMCC.

4.2 KAMYR AB SURVEY

Erik Maspers presented the results of a survey of 44 digesters outside North America (23 in Nordic countries). Maspers' overheads are included as Page D-17. Ten digesters reported corrosion, either slow (7 digesters) or rapid (6 digesters). The percentage of thinning digesters was thus similar in both surveys.

A significant difference from the North American survey was the preference for cleaning by hydroblasting outside North America, rather than acid cleaning: only 13 digesters reported acid cleaning, and 5 of those employed (less corrosive) sulfamic acid; further, some mills had discontinued acid cleaning.

Another difference from the North American survey was the finding of corrosion in young digesters. Nordic digesters have an average startup of 1986. Otherwise the two surveys were similar: digesters with MCC, EMCC or ITC were more prone to thinning than conventionally-operated digesters, yet no single cause for the thinning problem was identified.

In subsequent discussion, Dave Bennett pointed out that acid cleaning is seldom performed in Scandinavia, yet digesters there are experiencing rapid thinning. This strongly suggests that acid cleaning is not responsible for the rapid thinning phenomenon. Bennett also stated that hydroblasting is an effective surface preparation for crack inspection, but questioned how piping was cleaned. Ian Munro replied that, in a New Zealand mill, the piping was blanked off and acid cleaned. Martti Huttunen stated that some Scandinavian mills have valves which permit cleaning of piping separately from the digester.

Bob Grantham asked whether there was any correlation of corrosion with over-production. It was pointed out that in digesters where the dummy plates had been removed to get extra capacity, there may no longer be any upflow.

Scott Melton asked if anyone had observed corrosion behind dummy plates. No such observations were reported. Angela Wensley said that there had been reports of digester shell corrosion after removal of dummy plates to increase capacity.

5.0 DIGESTER THINNING REPORTS

Several reports of digester thinning were made to the task group.

5.1 AF-IPK REPORT

Angela Wensley read a letter from Leif Kiessling of AF-Industrins Processkonsult AB, in Stockholm, Sweden, sent to the task group on November 24 1993. The letter is attached as Page D-19. Kiessling reported on the inspection of 10 digesters running MCC or ITC. A significant relation was found between prolonged delignification processes and increased corrosion.

5.2 PAPER BY SVEN LAHTI

Angela Wensley read a memorandum from Patrik Lownertz, dated December 20 1993, which summarized a translation of a paper by Sven Lahti (an inspector with the Swedish Plant and Pressure Vessel Inspectorate, given at the SPCI Pulping Section autumn meeting, November 10-11 1993. The memorandum is included as Pages D-20,21. Lahti reports that serious corrosion damage has occurred in digesters employing MCC over the last 2-3 years. "Catastrophic" erosion corrosion in flash cyclones was also reported.

5.3 DOMTAR WINDSOR QUEBEC MILL

Doug Singbeil of PAPRICAN reported, on behalf of Rejean Beaudoin, on the thinning problem in the continuous digester in Domtar's Windsor mill. His overheads are included as Page D-23. The digester, part of a 2-vessel MCC hydraulic system, is about 5 years old. Corrosion was first observed after a six month EMCC trial in 1991. As an MCC digester, white liquor was added at the top and in the middle; as an EMCC digester, white liquor was added in the top (150°C), middle (160°C), and bottom (130°C). The corrosion was observed between the wash and extraction screens.

The investigation of this corrosion problem included in situ tests with A285C carbon steel corrosion coupons (on the inside of manway doors) and with a corrosion potential monitoring probe. The coupons were not in the area of worst corrosion, and the probe was located below the extraction screens but above the area of worst corrosion.

Coupons in the top and bottom have consistently experienced low corrosion rates (< 0.5 mpy). The top coupons were heavily (calcium) scaled; the bottom coupons were shiny and as-new, and covered with an oily substance (possibly an extractive) which apparently prevented them from seeing water. The middle coupons, however, have experienced corrosion at rates up to 20 mpy, and were covered with a black corrosion product.

A second EMCC trial was run from February to October 1993, and there is a verbal report that there is significantly more corrosion than previously.

The corrosion potential monitoring results suggested a possible correlation of increased corrosion with lower sulfidity. In May and June 1993, when the average white liquor sulfidities were 24% and 22%, the digester was passive (defined as having a potential above -50 mV versus Ag) 94% of the time. In July and August, when the average sulfidities were 17% and 19%, the digester was passive for 63% and 72% of the time. In September, however, when the average sulfidity was 18%, the digester was again passive for 94% of the time.

Laboratory polarization curves in mill white liquor were obtained at 170°C, 150°C, and 120°C. The potential range for the active nose shifts to lower potentials as the temperature is increased; indeed, the potential range of the active nose (-900 to -1000 mV versus SCE) at 120°C is the same as that of the passive zone at 170°C. Caution must be exercised in interpreting probe potential results as being "active" or "passive."

Corrosion potentials measured for A285C electrodes measured with respect to a silver reference electrode in different liquors (wash, white and probe liquor from an EMCC trial) at 120°C do not have the same relative differences when measured with respect to SCE at the same temperature. The reason for this is that the silver electrode potential is not constant, but rather varies with sulfide concentration. Again, one must be wary of the interpretation of potential measurements.

Comments and Questions

Hugh White commented that in EMCC the total chemical demand of the bleach plant is reduced, but there is use of more white liquor, which necessitates the purchase of more fresh (50%) caustic.

Ben Sen commented that the sulfidity was low during the EMCC trial because there was no salt cake addition; only salt cake from the ClO_2 generator is re-used.

Angela Wensley asked if transpassive corrosion had been observed in digester liquor. Doug Singbeil replied that he has seen increased corrosion as the potential is moved from -800 to -400 mV versus SCE.

Singbeil also emphasized the need for a digester operation model which would show temperature, flows, and other parameters. Dave Bennett said that simulations of the digester have been done. The alkali profile for MCC digesters suggests that the lower welds are now susceptible to cracking. Ron Bain said Kamyr Inc. has a computer model in Glens Falls. If the model were made available to mills, they could attempt to validate it by measuring temperature profiles.

5.4 NORANDA MILL

Alex Nadezhdin reported on corrosion of two digesters, both 800 tpd. His overheads are included as Page D-25. One is conventional; the other (13-14 years old) has been running MCC since Spring 1991.

Corrosion in the MCC digester occurs below the extraction screens. Corrosion rates for the MCC digester previously reported to the task group (deduced from vertical UT surveys to be around 100 mpy) are now known to have been erroneously high; however, the corrosion rates are still high (about 40 mpy). Visual evidence of the corrosion in the wash zone has been confirmed by views of stainless steel weld overlaid nozzles (the overlay was applied before MCC) standing proud about 1 mm from the carbon steel digester shell after running MCC for 19 months.

Corrosion in the conventional (non-MCC) digester is slightly above the extraction screens, and occurs over a narrower area than the corrosion in the MCC digester. The conventional digester has historically experienced corrosion between the cooking and extraction zones. There has not been much change in shell thickness in more recent years (1991, 1992, 1993).

A carbon steel rod cathode (25 mm diameter) extending down the length of the conventional digester was found to experience 5 mm of thinning in

exactly the same area where the thickness survey found thinning of the digester shell.

Comments and Questions

Ben Sen asked what liquors were used for washing the pulp. Nadezhdin replied that the new digester has a diffuser, but that he didn't know what goes into the digester. Wash water or liquor in the bottom is presumed to go up, but it is not known if it reaches the extraction screens.

5.5 WESTERN PULP LIMITED PARTNERSHIP MILL

Derek Smith reported on corrosion in the WPLP mill in Squamish, BC. The digester was built in 1965 and was originally rated at 300 tpd, and is now operating at 425-450 metric tpd. The digester usually runs with about 30% western red cedar. Historically, corrosion was not a problem, although some Inconel® overlay had been applied in the extraction screens. The dummy plates between the extraction and cooking screens were replaced. A new row of extraction screens was added (alternating screens and dummy plates), and a year later the dummy plates had virtually disappeared ($> 0.200''$ metal loss; exposed to liquor from both sides); also, there was significant loss of shell thickness below the extraction screens and thinning of the shell behind the lower two rows of screens. The fourth row of screens was removed.

In Spring 1991 about 800 square feet of shell were affected. Carbon steel weld overlay was applied to a thickness of $3/16''$. Five corrosion monitoring probes were installed. Polarization scans were done in situ with digester operation changed back to original variables; no effect on corrosion could be discerned.

In Fall 1991 up to $0.120''$ was lost from the carbon steel overlay. The corroded overlay was very rough and the corroded weld beads had a sharp profile. An anodic protection system was installed, and the fourth row of extraction screens was re-installed. More carbon steel weld overlay was applied.

In Spring 1992 corrosion rates were found to be reduced by about $2/3$. Some carbon steel weld overlay was done behind the screens.

In Fall 1993 the bottom of the digester from the bottom dome liner up to the bottom of the extraction screens was overlaid with type 309LSi stainless steel. The cathodes were not replaced over the stainless steel weld overlay.

Comments and Questions

Alex Nadezhdin asked if the fourth screen drew countercurrent liquor. Smith replied that there was no upflow, and that this was verified with tracer studies. A diffusion washer was installed in 1989; formerly the batch and continuous digesters had separate washing lines.

Some changes in polarization behaviour could be related to different operators (e.g., different ways of pumping black liquor into the top of the digester).

Hank Wink said that weld deposits made with E7010 electrodes stood up much better compared with E7018.

In response to a question as to whether plug movement was impaired by the rough surface of the corroded welds, Smith replied that there had been no complaints from the operators.

The screens and backing bars are stainless steel. The top two rows are slot type; the bottom two are stave type. When the fourth row was added, there was an increase in volume extraction. They went to switching valves.

The diffuser is atmospheric, not pressure. Addition of the diffuser did not result in increased production rate.

Temperatures vary around the circumference of the digester. A temperature profile is attached as Page D-27. The location of greatest corrosion does not correspond to either the highest or lowest temperature locations. Dave Bennett asked if such a temperature distribution was evidence of upflow, and Smith replied that the digester is overloaded and shouldn't have upflow (this was confirmed by a tracer study). Hugh White commented that the temperature minimum is likely related to feeding one wash nozzle preferentially.

5.6 CORROSION TESTING IN WPLP DIGESTER LIQUOR

Angela Wensley reported on corrosion testing performed at Bacon Donaldson, in autoclaves at 170°C containing extraction liquor from the Squamish BC digester. The results are attached as Page D-30. Tests were performed under potentiostatic control, at +150, +100, +50, 0, and -100 mV versus a molybdenum reference electrode. Three materials were evaluated: A516-Gr.70 carbon steel, type ER309LSi stainless steel weld overlay, and type 304L stainless steel.

The results clearly show that carbon steel resists corrosion (relatively) only in a narrow range of potentials (+50 to +100 mV versus Mo). At lower potentials, the carbon steel exhibited corrosion rates as high as 288 mpy, comparable with the worst corrosion actually experienced in the digester. At higher potentials carbon steel experienced transpassive corrosion.

The ER309LSi stainless steel weld overlay, on the other hand, was profoundly resistant to corrosion at all potentials tested (showing even better corrosion resistance than type 304L stainless steel).

Comments and Questions

Wensley stated that she considered potentiostatic testing in laboratory autoclaves to be the best way to study the phenomenon of digester corrosion. Actual digester liquors can be used, and certain variables altered (e.g., temperature, dilution, sulfidity, organics, etc.) under carefully controlled conditions. If the corrosion is due to some property of the liquor, the potentiostatic polarization method should determine this.

5.7 SWEDISH DIGESTER

Erik Maspers showed photographs of a digester in a mill that Sven Lahti wrote about in his paper (mentioned in Section 5.2 above). Just above the extraction screens there were depressed areas about 0.5 mm deep. In 1993 the corrosion had grown 0.5 mm deeper, and also had spread in area. The flash cyclones suffered severe erosion corrosion, and were lined with stainless steel in October 1993.

Comments and Questions

Ben Sen said that he has seen rapid flash tank corrosion and rapid evaporator corrosion (fourth effect) in mill with rapid digester corrosion.

5.8 REPORT BY MARTTI HUTTUNEN

Huttunen reported on corrosion in three digesters. His overheads are included as Page D-31.

In the first digester, where the corrosion rate was about 75 mpy, a survey of corrosion potential from bottom (plate 1) to top (plate 21) showed potentials varied between -115 and -40 mV vs Mo.

In another digester, with a probe in the cooking zone, the anodic protection potential was +80 mV vs Mo (compared with the free potential,

which varied down to -160 mV vs Mo). There was an inverse effect of production rate on corrosion potential in the cooking zone - at low production rates the potential was high; at high production rates the potential was low. Plotting minimum and maximum corrosion rates as a function of potential revealed that there was a steady increase in corrosion rate as the potential decreased from +100 to -300 mV vs Mo. A polarization curve from the cooking zone is also shown.

The third digester had corrosion probes installed from top to bottom of the vessel. The potential in the bottom had been about 20 mV vs Mo (see charts for probes 1 and 3); however, in the last 8 months the bottom potential went as low as -150 mV at times. The sudden change correlated when switching from hardwood to softwood cooking (see subsequent charts for probes 1 and 3).

Huttunen also showed a graph of previously published data by J.A.F. Gardener, indicating that certain wood species (red cedar, Douglas fir) can be extremely corrosive.

Huttunen suggested that an evaluation of digester corrosion and protection should involve the following steps:

- documentation of history (corrosion, process, etc.)
- gather new information (potential monitoring, coupons, etc.)
- investigate alternatives (AP, weld overlay, etc.)
- implementation (planning, specifications, QC, etc.)
- new design (AP-system, material selection, process spec., etc.)

Comments and Questions

Ben Sen commented that the Squamish digester had operated for a very long time pulping western red cedar, without corrosion. Sen also mentioned that there are 5 polysulfide pulping digesters in the world, 3 are of carbon steel construction, yet none is reportedly experiencing corrosion.

6.0 DISCUSSION OF DIGESTER THINNING

Several possible causes of the phenomenon of rapid thinning of carbon steel digesters were discussed.

6.1 MCC, EMCC, ITC

Dave Bennett commented that mills are going to MCC with digesters not designed for MCC, and that we need to know where to look and how to look for the problem.

Huttunen showed a figure comparing the E.A. and lignin profiles in 2-vessel MCC and conventional cooking. The captions were in Finnish. Erik Maspers subsequently provided a similar figure (in English) comparing the E.A. and lignin profiles in MCC and isothermal MCC cooking (Both figures are attached as Page D-35). Hugh White commented that the E.A. is higher at the end of the cook with isothermal cooking.

6.2 MILL CLOSURE

A comment was made that mill closure and MCC are occurring simultaneously, and that the corrosion seen in MCC digesters may actually be the result of closure.

Ben Sen mentioned that volatile corrosive phenolics are now recycled.

6.3 WASHING

Dave Bennett commented that changes in the source of wash water in the bottom of the digester may have an effect on the corrosion.

Derek Smith asked if all mills have a consistent source of wash water. Ben Sen said that mills with diffusion washers have no blow tanks, and that volatile organics are no longer flashed off, but rather put back into the process streams.

Hugh White said they replaced the drum washers at their St. Helens mill with a more efficient design, but no diffuser was added. He mentioned that upflow from the wash zone in a digester can reach the extraction screens. The wash liquor in such a case is usually the first stage filtrate.

Bob Grantham said that the Squamish digester is in overproduction, and has no upflow. He also said that some mills have used contaminated condensate as wash water.

6.4 OXYGEN REACTOR

Hugh White asked if the corrosion problem could be related to mills having an oxygen reactor. Dave Bennett replied that they have digesters in 4 mills with oxygen reactors, and that there is no corrosion. The vapour dome corrosion problem at Quinnesec (previously reported to this Task Group) was not related to oxygen reactors, as it occurred 2 years before the reactor was installed.

6.5 AOX

Martti Huttunen asked whether the corrosion was related to AOX. Dave Bennett replied that he would be surprised if mills gave out AOX data.

6.6 FLASH TANKS

Jim Reid stated he has seen severe flash tank corrosion in mills where the digester is not corroding.

6.7 PROCESS DATA

Hank Wink said that more process data are needed. A suggested list included:

- tonnage (design)
- tonnage (running)
- vessel type (e.g., single vessel hydraulic)
- cooking temperature
- white liquor strength (lb/ft³)
- white liquor sulfidity
- A.A.
- cook zone availability (A.A., lb/ft³)
- chip furnish
- extraction ratio target

7.0 INSPECTION

Wensley commented that the Task Group recommended inspection for thinning by, as a minimum having two vertical lines from the bottom head to about 3 m above the extraction zone, with readings taken every 0.5-1 meter, with the minimum thickness in an 80 mm diameter spot being recorded at each location.

Without reproducible thickness testing locations to compare data from year to year it can be extremely difficult to determine corrosion rates.

Alex Currie said Coast Testing do two vertical bands, and if thinning is found, they go around the digester circumferentially at that elevation.

Jim Reid of INDT Ltd. commented that it is the mills that direct the NDT companies.

Hank Wink said that the B-scan method is used at Grande Prairie, and that they do 8 bands top-to-bottom. They have found as much as 45 mils difference in thickness around the circumference.

8.0 CORROSION PREVENTIVE MEASURES

Methods employed to prevent rapid thinning of digesters have included anodic protection, corrosion-resisting weld metal overlay, and thermal spray.

8.1 ANODIC PROTECTION (AP)

Dave Bennett said that AP is usually installed to protect against SCC; however, the second AP system ever installed (in an Alabama mill) was put in to protect against rapid bottom thinning. Ian Munro said the system was still operating, but he had no recent corrosion rate data (he recalled 5 mpy).

Hank Wink said that the Grande Prairie digester has been anodically protected since 1987. The stitch welds on the hardware bars (welds attaching the cathode support frames to the digester shell) in the bottom of the digester had experienced cracking. The AP protects the carbon steel shell at the edge of Inconel 625® overlay. PAPRICAN has done coupon testing in the digester; the highest corrosion rates are about 10 mpy.

Derek Smith said that AP is more effective at reducing the corrosion rate on base metal than it is on carbon steel weld overlay.

Ian Munro commented that there is a minimum corrosion rate in the lower part of digesters where AP cannot reduce it any further. The "non-zero" corrosion rate is 10-20 mpy, which is borderline. The "motion" of the corrosion potential in the extraction zone is great compared with the impregnation zone. Craig Reid said that carbon steel electrodes exposed in the extraction zone of the Harmac digester corroded at about 60 mpy when the digester was cooking cedar (short duration exposure), and that AP tests could not reduce the corrosion rate to zero.

Angela Wensley said that one cannot rely on corrosion potential measurements to know if rapid thinning is occurring; the potential data have to be backed up with actual corrosion rate measurements.

8.2 CORROSION-RESISTING WELD OVERLAY

Wensley referred to the potentiostatic polarization testing (Appendix VII) she carried out in Squamish digester liquor, which showed that ER309LSi stainless steel weld overlay resisted appreciable corrosion attack over the entire range of possible corrosion or anodic protection potentials which may exist in that digester.

Wensley reminded the attendees that there was a TAPPI Technical Information Sheet (TIS 0402-03) for corrosion-resisting weld overlay in digesters. She recommended increasing the minimum alloy content values for overlay to be applied in rapidly corroding digesters.

Craig Reid said that 30' of the extraction zone of the Harmac digester was weld overlaid in 1967 with stainless steel by Alloy Cladding. Corrosion probe testing in the 1980's revealed that corrosion potentials for carbon steel electrodes in the extraction zone plummeted when switching from hem-bal to cedar; cedar cooking was subsequently discontinued. In 1991 they extended the overlay to the bottom of the digester, including areas behind screens and headers. Since then they have gone back to cooking cedar.

8.3 THERMAL SPRAY

Bob Readal showed two overheads (Page D-37). One was a list of digesters in which Di-Arc 360 arc spray had been applied. The second was a figure showing the change in the thickness of the Di-Arc in a Louisiana digester as a function of time. The coating had been applied to the lower weld of the upper transition. Coating thickness has decreased from 77 mils in 1986, to about 50 mils in 1992, indicating an average wear rate of about 5 mpy. It is believed the coating wears particle by particle, not by corrosion. Ultrasonic imaging has revealed no SCC of the weld below the Di-Arc.

A digester in Florida is now completely protected with Di-Arc thermal spray (6000 ft²); the work began in 1989 and was completed in 1993. The screens were removed so that the shell behind could be sprayed.

Readal said he has not observed undercutting of the thermal spray. A typical band to protect a weld seam from SCC is full thickness for a width

of one foot, and then feathers out to nothing beyond that. These feathered edges "shrink" back each year.

Readal also said they are looking at a high chromium alloy (25-30% Cr) for better abrasion resistance in the bottom cone area of batch digesters (this coating is currently in a batch digester).

Following the meeting, a report of a Di-Arc coating failure was sent to the Task Group by George Sniff of Westinghouse (Pages D-39,40). The cause is believed to be a compromised surface preparation.

9.0 SUMMARY

At the end of the day, Wensley summarized pertinent points from the meeting. These included:

- Digesters with MCC, EMCC and ITC may be at risk of rapid corrosion; however, the Squamish digester (which has very high corrosion rates) runs conventional cooking.
- The highest corrosion rates tend to be around the extraction screens.
- Carbon steel weld overlay is not an effective solution to the rapid thinning problem.
- While improper acid cleaning can result in corrosion, the thinning phenomenon cannot universally be attributed to acid cleaning; in particular, non-North American digesters do not as a rule acid clean, yet may be experiencing rapid thinning.
- Species changes such as switching from hardwood to softwood, or from hem-bal to cedar, may play a role in the rapid thinning process.
- The question as to whether the corrosion can be related to sulfidity remains unanswered; data from the Domtar digester suggest that low sulfidity liquors may be more aggressive.
- Increased production rates and pushing the wash zone out of the digester may increase corrosion rates.
- Temperature may be an important factor in the rapid corrosion phenomenon; in particular, higher temperatures lower in the digester as the result of MCC, ITC or conventional cooking where overproduction has pushed the wash zone outside the digester.

- Upflow could be responsible for variations in temperature around the digester circumference.
- Anodic protection can be effective, but in rapidly corroding digesters may not reduce the corrosion rate to zero.
- Corrosion-resisting weld overlay has a good track record in digesters, and can be highly resistant to corrosion provided it meets alloy content requirements.
- Properly-applied thermal spray has a good track record in digesters, but may experience wear at a slow rate.

10.0 ADJOURNMENT

The Task Group meeting was adjourned at 5:00 pm, following which refreshments were served.

The next meeting of the TAPPI Continuous Digester Thinning Task Group will be during the week of September 19-22, in conjunction with the 1994 TAPPI Engineering Conference in San Francisco.

CONTINUOUS DIGESTER THINNING SURVEY

Fourty four owner/operators of kraft continuous digester responded to a survey of the Kvaerner Pulping Technologies in 1993.

The data have been arranged in the same way as Angela Wensley did in her presentation at the 1993 TAPPI Engineering Conference.

The numbers inside brackets refers to digesters in the Nordic countries i.e Finland, Norway and Sweden.

1. With Corrosion: 10 (4) digesters had shell corrosion > 2.5 mm and/or > 0.5 mm in any given year.
2. Without Corrosion: 31 (17) digesters had shell corrosion < 2.5 mm and < 0.5 mm in any given year.
3. Don't Know: 3 (2) digesters where the total amount and the rate of shell corrosion were not known.

10 (4) Digesters reported corrosion:

7 (2) digesters > 2.5 mm total

6 (3) digesters > 0.5 mm in any given year

Mill Location

	With Corrosion	Without Corrosion	Don't Know	Total
Europe	8 (4)	24 (17)	2 (2)	34
South America	1	5	1	7
Australia and New Zealand	1	1	1	2
Asia		1		1
Total	10	31	3	44

Digester Age with Standard Deviations

	With Corrosion	Without Corrosion	Don't Know
Age (Startup)	1975 (1986)	1974 (1971)	1977 (1977)
Std. Deviations	10 (4)	9 (7)	3 (4)

Method Used to Determine Extent of Thinning

	With Corrosion	Without Corrosion	Don't Know
UT	9 (2)	23 (10)	
"NDT"	2 (1)	2 (2)	
Others		6 (4)	
Not Reported	1 (1)	1 (1)	3 (2)

Acid Cleaning

	With Corrosion	Without Corrosion
Muriatic Acid	3	5 (2)
Sulfamic Acid	2	3 (1)

Process Changes

	With Corrosion	Without Corrosion	Don't Know
No Reply	1 (1)	18 (10)	1 (1)
No Process Changes	4	3 (3)	
MCC, ITC etc.	3 (3)	2	1
Temperature Changes	1 (1)	1	
Sulfidity Changes	2 (1)	6 (4)	1 (1)
Mill Closure		2	

Cleaning Frequency

	With Corrosion	Without Corrosion
2 times a year	2	4 (2)
1 time a year	1,5	3 (1)
1/2 time a year	1,5	
< 1/2 time a year		1

Screen Changes

	With Corrosion	Without Corrosion	Don't Know
Within 2 Years of Rapid Corrosion	2		
No Changes	8 (4)	22 (11)	1
Changes		10 (6)	2 (2)

24 November 1993

Leif Kiessling
Materials Technology & Inspection
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telefax +46 8 653 91 36

Dr. Angela Wensley
Bacon Donaldson
Consulting Engineers
12271 Horseshoe Way
Richmond, BC, Canada, V7A 4Z1

Dear Angela,

I would like to contribute to your group by some of our latest observations from digesters in Sweden and other countries.

We have inspected about ten digesters running MCC (modified) or isothermal cooking (ITC) and found a significant relation between the prolonged delignification processes and increased corrosion activities. The general view is a zone with pitting corrosion between MCC screens and extraction screens. Another observation is a depassivation in the washing zone. The cover plates near the screens were more affected than the nearby shell. This could be an effect of the locally higher flow rates and maybe also supported by the erosion/corrosion from the pulp. In two digesters UT survey has revealed shell thinning of 0.5-1 mm/y near the extraction screens, for first period of operation with prolonged delignification. However, the attacks seem to slow down after this period and the surface of the thinned area is now partly passivated again. We hope to initiate a corrosion study where the influences of MCC and ITC on shell wastage and depassivation will be studied.

We have just furnished a corrosion study in a batch digester running conventional cooking. The influence of various process parameters for a batch digester will be monitored by a corrosion probe located below the extraction screens. The digester shell is made of mild steel without any stainless protection or cladding. It has been attacked by severe pitting corrosion in the shell and welds near the circulation nozzles in the extraction screens. No result is available yet.

Yours faithfully,

ÅF-INDUSTRINS PROCESSKONSULT AB
Materials Technology & Inspection

Leif Kiessling

D-19

ÅF-INDUSTRINS PROCESSKONSULT AB

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A PARTICIPANT IN THE STRATEGIC ALLIANCE OF H.A. SIMONS LTD. AND ÅF-INDUSTRINS PROCESSKONSULT AB

MEMORANDUM

H.A. SIMONS LTD.

FROM: PATRIK LOWNERTZ

DATE: 20 December 1993

SUBJECT: CORROSION IN MCC DIGESTERS

FILE NO.:

TO: DAVID CARTER

CC: NICK FLIPSE
PETER GLEADOW
CALVIN HASTINGS
DON HERSCHMILLER
WALLY MACKAY
KEN SERENIUS
Doug Singbeil, Paprican
Ken McChesie
Eol Loberg

This is an excerpt and translation from a paper given by Sven Lahul, an inspector with the Swedish Industrial Plant and Pressure Vessel Inspection, at the SPCI Pulping Section autumn meeting November 10-11, 1993.

...

(SA, Sundsvall)

"General corrosion in digester and pre-impregnation vessels have with a few exceptions been almost negligible in the past. 20-25 years old digesters show hardly any loss of thickness. Some digesters have however shown some corrosion in the cooking zone.

Requirements for reduction in the use of chlorine in pulp bleaching has led the development toward lower kappa numbers. Conventional cooking has via modified continuous cooking (MCC) arrived at something called isothermal cooking (ITC). Many mills in Sweden has up-graded their digesters to MCC/ITC.

Will these new digesting processes result in additional corrosion on the digester plant pressure vessels?

We start to get results from our inspections that all point in the same discouraging direction. Digesters that over the last 3-4 years have been employing MCC digesting show serious corrosion damage in the counter-current cooking zone. 2-2.5 mm general loss of material thickness has been measured. As the corrosion allowance is about 5 mm this means that repairs will have to be made maybe as early as after six years of operation.

If the corrosion in the digesters is serious, the erosion corrosion in the flash cyclones is catastrophic. I will not speculate in how large the corrosion rate is in mm/year but can state that more than 10 mm loss in material thickness has been measured over a three-year period. Carbon steel is a clearly unsuitable material of construction for flash cyclones in a MCC/ITC process. I recommend that you who have not taken action regarding the cyclones start planning, preferably for a replacement. Stainless material has shown good resistance towards erosion corrosion.

As far as we can see, the carbon steel digesters in MCC/ITC service will only have a few years of operation left before the corrosion allowance has been used up and therefore planning of corrective measures must be done now. The magnitude of the problem is much larger than for the flash cyclones due to the size of the digester vessels. I want to mention three possible measures:

1. Overlay welding of a stainless layer (the Uddcomb method).

MEMORANDUM (Cont'd . .)

Page 2 of

This method is relatively time consuming, but made over a number of years it can be a realistic alternative.

2. *Metal spraying of a protective layer.
The method has been tried in a number of digesters with varying results.*
3. *Installation of anodic corrosion protection.
This method has been used in a number of digesters in, among other countries, Finland for protection against stress corrosion and should be effective also against general corrosion.*

To summarize I can state that the problem with stress corrosion now has been supplemented with serious general corrosion and erosion corrosion that probably can be referred to the use of the MCC/ITC process... With reference to this we must strongly consider whether the intervals between inspections should be shortened from the normal three years to one year with complete crack inspection of welds, including those hidden behind screens."

In view of these alarmingly high rates of corrosion found in Swedish MCC digesters I feel it is important that we find out more about this issue and make our clients aware of these findings as applicable.

Patrik Lownertz

COUPON CORROSION RATES (mpy)					
	OCT 91 APR 91	APR 92 OCT 92	OCT 92 APR 92	APR 93 OCT 93	
9th Floor	0.06	0.12	0.03	0.02	heavily scaled
5th Floor	-	10	6	20	black corrosion product
2nd Floor	0.040	0.11	0.08	0.32	shiny, brand new

EMCC OPERATION: OCT 90 - APR 91
FEB 93 - OCT 93

Coupons: A285 Grade C
isolated from digester

PROBE POTENTIAL				
% of time in each condition				
MONTH	<-70	<-50	PASSIVE	% SULPHIDITY
May	3	6	94	24
June	2	6	94	22
July	17	37	63	17
August	11	28	72	19
September	3	6	94	18

- 5 year old digester
 - Corrosion observed for 91 after EMCC trial
 - In situ tests: corrosion coupons
potential measurements
 - Lab tests: white liquor
black liquor
probe liquor
wash liquor
- polarization curves
corrosion potential

LABORATORY TESTS			
	NaOH	Na ₂ S	Na ₂ CO ₃
White Liquor	160	---	---
Black Liquor	16	---	---
Probe Liquor	8	---	---
Wash Liquor	5	---	---
Concentrations as g/L Na ₂ O

COMMENTS & SUGGESTIONS

- 1.) Be wary of how you interpret potential measurements.
- 2.) Need an easy pictorial way to represent changes in digester operation
- temp, flows, velocity CFD?
- 3.) Coupon data can supplement, but not substitute for shell thickness measurements.
- 4.) Shiny = rapid active corrosion

Figure 10. Digester "A" lower cathode diameter as surveyed in September 1993

Figure 11. Corrosion of the centre pipe above the cathode support

Aver. Wall Thickness Change (92 to 93)
1/1000", loss shown positive

Figure 4. Average thickness loss since fall of 1992 along the circumference at different elevations

Kamyr Temperature Distribution

Figure 4. Photographs of extraction nozzles. Stainless portion of the shell surface "stands proud" by at least 1 mm, 1 year of MCC.

Figure 1. The difference between shell thickness measured in 1992 and that measured in 1993 in a vertical survey along 0° and 180° lines. The values in the graph have been averaged over each individual plate designated by letters as shown in the digester drawing on the left.

NOMINAL minus MEASURED wall THICKNESS 0-degree line during last three years

Figure 2

1990 1991 1992

NOMINAL minus MEASURED wall THICKNESS 180-degree line during last two years

Figure 3

1991 1992

CORROSION TESTING
in
W.P.L.P. DIGESTER LIQUOR

		Corrosion Rate in mpy		
Potential mV vs Mo	Temp. °C	A516 Gr. 70	ER309LSi	304L
+150	170	49.7	0.03	0.52
+100	170	7.1	0.01	0.07
+50	170	6.0	0.01	1.04
0	170	288	0.30	1.89
-100	170	254	0.05	0.22

Liquor Composition (gpl)

NaOH 7.1
Na₂S 18.0
Na₂S₂O₃ 3.7
Na₂SO₄ 5.2
Na₂CO₃ 22.1

PROTECTED VS UNPROTECTED

Corrosion Rate vs. Potential

SAVCOR CONSULTING INC.
200-1130 West Pender St.
Vancouver, B.C. V6E 4A4

Effect of Wood Extractives (Coupon Trials)

From: J.A.F. Gardner Paper

Kuva 89: Alkali- ja ligniiniprosenttien vertailu

Lignin and alkali profiles.

Isothermal MCC

MCC

Arc Spray Coating Applied In Continuous Digesters

Location	Work Scope	Area (Sqft)	Manual/Auto	'85	'86	'87	'88	'89	'90	'91	'92	'93	'94
LA	Circumferential (Circ.) Weld Seam	20	Manual		◆								
AR	Circ. & Vertical Weld Seams	160	Manual							◆			
AR	Circ. & Vertical Weld Seams	20	Manual								◆		
AR	Circ. & Vertical Weld Seams	50	Manual									◆	
PA	General Area & Weld Seams	330	Auto							◆			
ME-A	General Area & Weld Seams	1110	Manual/Auto							◆			
ME-B	Circ. & Vertical Weld Seams	160	Manual								◆		
FL	General Area & Weld Seams							◆					
FL	General Area & Weld Seams								◆				
FL	General Area & Weld Seams									◆			
FL	General Area & Weld Seams										◆		
FL	General Area & Weld Seams: Total	6000	Manual/Auto									◆	
OK-1	Circ. & Vertical Weld Seams (All)	1240	Manual									◆	
OK-IV	Circ. & Vertical Weld Seams (All)	450	Manual									◆	
OK-2	Circ. & Vertical Weld Seams (All)	1070	Manual									◆	

Arc Spray Applied In 1986 To Prevent Stress Corrosion Cracking Half Circumferential Weld Seam (18' x 18" High Band)

Alloy 625 applied to the lower weld of the upper transition.

**Westinghouse
Electric Corporation**

Di - Arc 360 Operations

2057 Hwy. 87
Alabaster, AL 35007
(205) 664-4602

May 15, 1994

Dr. D. A. Wensley
Bacon, Donaldson & Associates, Ltd.
12271 Horseshoe Way
Richmond, B. C.
Canada V7A4Z1

Dear Angela:

At the recent meeting of the Digester Thinning Task Group of TAPPI, Bob Readal reported that in over eight years of application of the Di-Arc Protective Alloy Coating Systems to the interior surfaces of continuous digesters for corrosion and stress corrosion cracking prevention that we have experienced no problems. Unfortunately, we have just experienced a loss of the coating to one of two seams in a continuous digester after two and one-half years of exposure.

A review of the job logs of that particular application indicates that we were pulled off of the job for safety considerations in the middle of the application when a feed screw was removed at the top of the digester. Apparently, we did not have enough Di-Arc Coating on the surface to protect the blast and/or contamination fouled the surface. We finished the application without re-blasting the surface. The consequence is that the coating wasn't there at the inspection. Naturally, we have learned from this experience and it will not happen again. Incidentally, we are replacing it for our customer.

The fortunate part of this experience is that no corrosion and/or stress corrosion cracking has occurred at this seam. Apparently, there was enough of the Di-Arc Protective Alloy Coating on the surface to protect the digester seams for a period in excess of two years. The coating on the other seam is intact and doing its job as we would expect. We consider this incident to be an application problem and not a fundamental problem with the coating as it is applied by Westinghouse.

Page 2
Bacon, Donaldson & Associates, Ltd
Richmond, B. C.

I would appreciate your inserting a note in the minutes of the Task Group minutes and report this to our colleagues so they will know the details of this incident. Thank you for your assistance.

Cordially,

George L. Sniff
National Product Manager
Di-Arc Operations

ATTENDANCE RECORD

Committee Digester ThinningPresiding Officer Angela WensleyMeeting Place Bacon Donaldson Town Richmond, BC Date Mar. 18, 1994

NAME (Please Print)	COMPANY	PHONE #	MEMBER		With in print Committee	Address Change
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CRAIG REID	MacMillan Bloedel Ltd STREET 4225 Kincaid St	PHONE # 604 439 8511				
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	W. NYACK NY 10994	FAX # 578-7175				
Alex Mudezhlin	Noranda Technology Centre 240 Hymns blvd	PHONE # 514 650-4306	✓	✓		
	Pointe-Claire HAR 1G5	FAX # 514 650-9379	X	X		
Bob Charlton	Levelton Associates 1-12791 Clarke Pl.	PHONE # 604 278-1411	X	X		
	Richmond, B.C. V6V 2H9	FAX # 604 278-1042				
Brad Smith	Fletcher Challenge ELK FALLS	PHONE # 604 287-5559				
	P.O. Box 2000 CAMPBELL RIVER BC	FAX # 604 287-5480				

ATTENDANCE RECORD

Committee Digester ThinningPresiding Officer Angela WensleyMeeting Place Bacon Donaldson Town Richmond, BC Date Mar. 18, 1994

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Scott MELTON	FLETCHER CHALLENGE STREET	PHONE # 257-5200				
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PLANT SERVICES MANAGER	Squamish, BC VON 3G0	FAX # 892-6603	NO	NO		
BOB READAL	Westinghouse Distr 2057 Hwy 87	PHONE # 205 664 4602				
MET. ENG.	ALABAMA AL 35007	FAX # 205 664 4662				
NORM OLSON	Battelle - Pacific Northwest Labs Battelle Blvd. Richland WA 99352	PHONE # 509 372 1570				Yes
Manager, Industrial Modernization		FAX # 509 372 1585	NO	NO		
GARRY RUSTAN	CANADIAN PACIFIC Forest Pro DUKE ST.	PHONE # (807) 223-9428	NO	NO		
SR. PROF. ENGINEER	DRYDEN ONTARIO P8N 2Z7	FAX # (807) 223-9215				

~~Committee Digestor Thinning~~
~~Presiding Officer Angela Wensley~~

Meeting Place Bacon's Woodlawn Town Richmond BC Date March 18, 1984

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Douglas Singhal	Chief Engineer	601-222-3200	601-222-3200	601-222-3200	601-222-3200	601-222-3200	✓	✓	✓
Margaret Grogg	Helicopter Eng	Weytheiser Co	WTC 1C39	924-6045	924-6045	924-6045	✓	✓	✓
Bob Harrison	PO Box 96077	Factory Mutual	(206)	454-3931	454-3931	454-3931	✓	✓	✓
ENGINEER	3333 1st Ave	WA 98009-6977	637-2399	637-2399	637-2399	637-2399	✓	✓	✓
TERRY MCELROY	INVESTIGATIVE NAT	604	459 1711	459 1711	459 1711	459 1711	✓	✓	✓
T.M. Reid	INDUSTRIAL NOT LTO	8955 Fairview Ct.	439 1711	439 1711	439 1711	439 1711	✓	✓	✓
Jim Oeri	Western Pipe	Western Pipe	892-6689	892-6689	892-6689	892-6689	✓	✓	✓
	892-6689	892-6689	892-6689	892-6689	892-6689	892-6689	✓	✓	✓

ATTENDANCE RECORD

Committee Digester Thinning

Presiding Officer Angela Wensley

Meeting Place Bacon Donaldson Town Richmond, BC Date Mar 18, 1994

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ATTENDANCE RECORD

Committee Digester Thinning

Presiding Officer Angela Wensley

Meeting Place Bacon Donaldson Town Richmond, BC Date Mar 18, 1994

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Mike Grogan	Weyerhaeuser Canada Ltd	P.O. Box 800				604/828-7510	604/828-7465		✓	
Reliability Engineer										
Sue Dyer	Weyerhaeuser Canada Ltd	Box 800				604-528-7510				
Reliability Engineer										
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Frank Easten	The Methylene Co.	11615 Bony Street								
President										
MIKE PLACE	9533 STEIN AD					206-366-4059			✓	
General Manager										
Dycker Rodin	CUSTOE, WA	91240								
Payson										

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Mgr - Eng. Services						EAX#				
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		RICHMOND									
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		B.C.									
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