

Scientific Notebook No. 549: Localized
Corrosion of Zircaloy-4 Alloy -- Continue in
Scientific Notebook No. 605 (10/07/2002
through 02/24/2004)

LABORATORY NOTEBOOK

Zr-4 Test

#549

(1)

CNWRA/SwRI

CNWRA
CONTROLLED
COPY 549

NOTEBOOK NO. _____
ISSUED TO Lietai Yang
ON _____ 20____
DEPARTMENT _____
RETURNED _____ 20____

Brian K. Darby · B. K. Darby - BKD

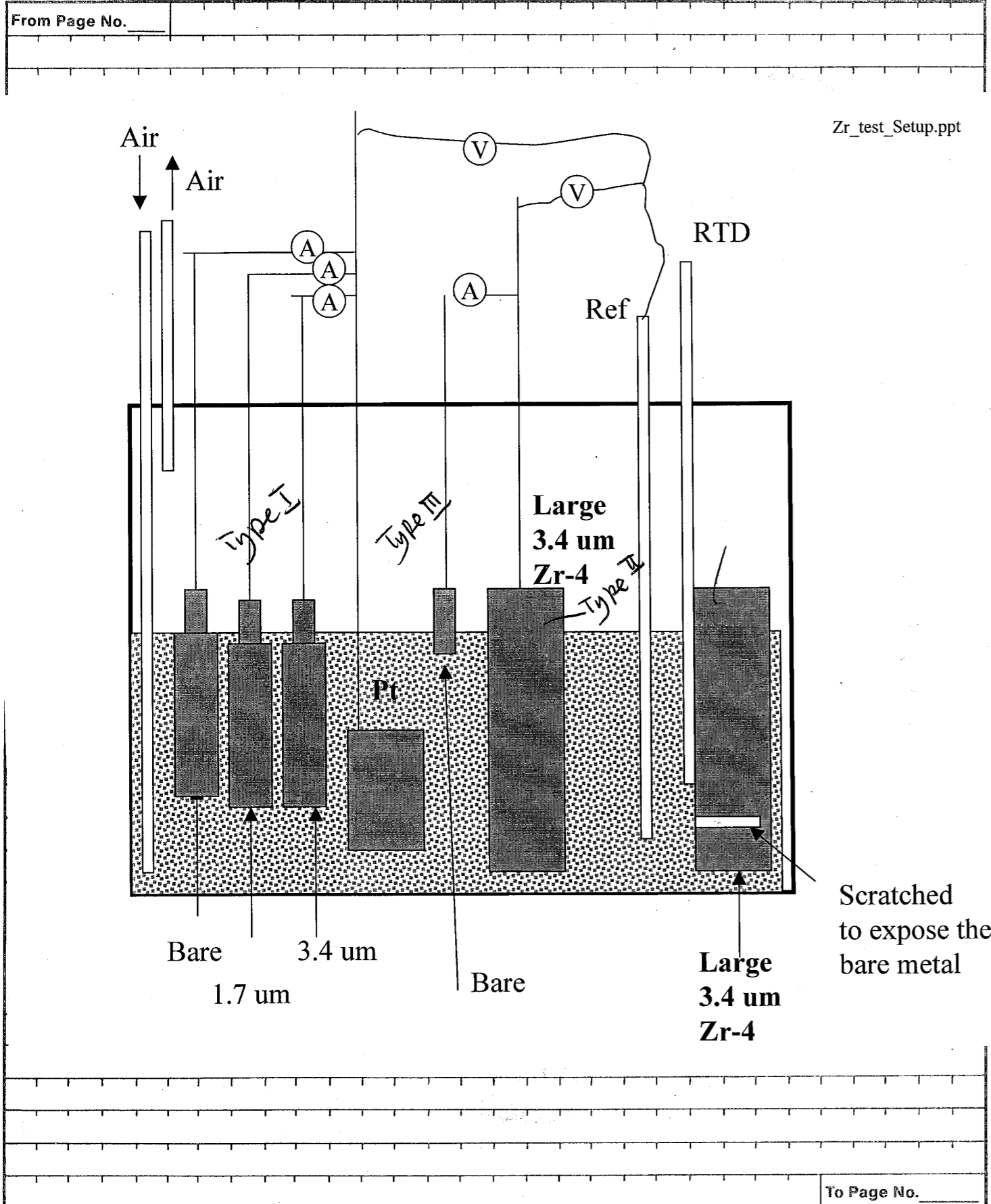
L. Yang J. Yang J. Y.

Don Bannon DRB

All electronic data files are stored
in NoteBooks / Notebook 549 & 605, Zr-4
J. Y. 11/17/03

SCIENTIFIC NOTEBOOK COMPANY
2831 LAWRENCE AVENUE
STEVENSVILLE, MICHIGAN 49127
(800) 537-3028 - <http://www.sneo.com>

11-10-2002 of dm fog of bot
126 files
126 files
Notebooks 549 & 605
Zr-4 - wpa - 2006 -
01-11
126 files



Scatched to expose the bare metal

Witnessed & Understood by me, _____ Date _____

Invented by _____ Date _____

Recorded by *J. J...* Date *10/7/02*

From Page No. _____

PURCHASING REQUISITION

REG. NO. **610159**

REQUISITION DATE: **5/28/02** ORDER DATE: _____ PURCHASE ORDER NUMBER: _____

DELIVER TO: **Lietai Yang/bidg. 57** PURCHASING SELECTED SUPPLIER

SHIP VIA: _____ SUPPLIER CODE: _____

F.O.B.: _____ PHONE: _____

TERMS: _____

LN.	QTY.	UNIT	DESCRIPTION	ORG	PROJECT	ACCT	%	DATE REQUIRED	EST. UNIT PRICE
A	25	Ft	Zr-4 annealed 0.5" bar stock, minimum order (\$950.00)	20	01402	571	100	6/6/02	\$950.00
1. Quality & Technical Requirements: Specification as per ASTM B351, item name and heat number properly identified on bar. 2. Material must be from same heat and from same bar. All 25-foot must be cut from the same heat & bar. 3. Materials will be used for the HLW project, independent confirmatory chemical analysis to be performed after receipt. <i>ing. V.S. 5/28</i>									
INTERNAL NOTES TO BUYER: Wah Chang is not on the ASI. However it is the manufacturer of the Zr-4 materials we used to buy from Metal Samples that is on the ASI. IF YES, CHECK THE APPROPRIATE PROPERTY TYPE (SEE BACK FOR EXPLANATION OF PROPERTY TYPES) 1. Government Project? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO 2. QUALITY ASSURANCE? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO a. ASL REQUIRED? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO b. QA CODES: Q1, Q3, Q4 c. INSPECTION CRITERIA: visual d. IS GOVT. PROPERTY BEING SENT TO SUPPLIER? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO 3. SOURCING NOTES IF YOU HAVE SELECTED A BRAND NAME OR PARTICULAR MANUFACTURER, WOULD AN EQUIVALENT BRAND OR PRODUCT ALSO SATISFY YOUR NEED? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO IF YOU HAVE SUGGESTED A SUPPLIER, AND NO OTHER SUPPLIER WILL MEET YOUR NEEDS, PLEASE ATTACH A MEMO OF EXPLANATION. REQUESTOR'S SIGNATURE: Lietai Yang EXT. NO. 2483 DEPT./DIVISION APPROVAL: <i>[Signature]</i> DATE: 5/29/02 ADMIN. APPROVAL: _____ DATE: _____ 4. REPAIRS a. IS THIS REQ. FOR A REPAIR? <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO b. IS THE REPAIR ON OR OFF CAMPUS? <input checked="" type="checkbox"/> ON <input type="checkbox"/> OFF c. IF OFF CAMPUS PROVIDE SHIPPING TICKET NO. _____									

DATE: _____ BUYER SIGNATURE: *[Signature]*

CONTRACT REVIEW APPROVAL: _____

SEE INSTRUCTIONS ON REVERSE SIDE

Witnessed & Understood by me, _____ Date _____

Invented by _____ Date _____

Recorded by *J. J...* Date *10/7/02*

PACKING LIST



Wah Chang
An Allegheny Technologies Company

PO Box 460
1600 NE Old Salem Road
Albany, OR 97321-0460
ph: 541-967-6977 f: 541-967-6994

SHIP TO:
SOUTHWEST RESEARCH INSTITUTE
6220 CULEBRA ROAD
SAN ANTONIO, TX 78238-5166
United States

Page: 1 of 1

PURCHASE ORDER 2866435	SALES ORDER NUMBER 115510	DATE SHIPPED 03-SEP-02
BILL OF LADING NO. 25834711-3	GOVT CONTRACT NO.	PICK NUMBER 1904116
FREIGHT CARRIER	GOVT. PRIORITY	INSPECTION OWC
FEDEX FREIGHT	EXPORT LICENSE NO.	PAYMENT TERMS NET 30 DAYS
FCA-COLLECT	CAS CODE	SALES PERSON HALL R.F.
DELIVERY POINT HUNTSVILLE, AL		

SO LINE	SHIP LINE	CUST ITEM	GRD QTY	UNIT OF MEASURE	ITEM DESCRIPTION	PIECES	SFC NUMBER	HEAVY NUMBER	CITY SHIPPED	NET WT LBS	GROSS LBS
1	1		1	LOT	ZIRCALOY-4 ROD ANNEALED & CENTERLESS GROUND (57 MICROINCH AA MAX) Diameter: 0.526-0.531 IN Length: RL (25 FEET TOTAL) ECCN: 1C234 License Type: No Lic LINE NOTES MAY BE CUT TO LENGTHS FOR UPS SHIPMENT. MATERIAL MUST BE FROM SAME HEAT. GOVERNMENT CONTRACT NO.: NRC0297009 Packaged In: WOODEN CRATE Number: 1 Net Weight: 15 LBS Gross Weight: 62 LBS Export Notes: THESE COMMODITIES ARE SUBJECT TO EXPORT CONTROLS. PRIOR UNITED STATES GOVERNMENT APPROVAL MAY BE REQUIRED FOR THEIR EXPORT AND/OR RE-EXPORT.	3	1223217	243687	1	15	0

9/15/02
J. Gas

TITLE _____
From Page No. _____
WHH LHHNG FAX: 5419170704 May 00 02 14:05

Wah Chang of 2
An Allegheny Technologies Company
P.O. BOX 460
ALBANY, OREGON 97321-0136
(541) 926-4211 FAX (541) 967-6994

TO: SOUTHWEST RESEARCH INSTITUTE
ADDRESS: 6220 Culebra Road
San Antonio, TX 78238-5166

ATTENTION OF: Jim Silvers

IN REGARD TO YOUR
Purchase Order No.: 2866435
Sales Order No.: 115510
Item No.: 1
Description: Zircaloy-4 Rod
Dimensions: 0.526"-0.531" Dia. x R/L
Specifications: ASTM B351-92 (Gr. R60804) and P.O.
Date Certified: August 30, 2002
Date Shipped: 9.9.02
Quantity Shipped: 3 pcs.
Weight (lbs.): 15
Heat No.: 243687 Zr4
SFC No.: 1223217

Should be 2866435
J. Gas 9/16/02

THE TEST REPORT FOLLOWS:

Material Condition: Annealed and centerless ground.

INGOT CHEMISTRY ANALYSIS: RESULTS IN PERCENT

Element:	Spec. Rng.	1	2	3	4	5	6
Cr:	0.07-0.13	0.12	0.12	0.12	0.12	0.12	0.12
Fe:	0.18-0.24	0.21	0.22	0.22	0.22	0.22	0.22
Fe+Cr:	0.28-0.37	0.34	0.34	0.34	0.34	0.34	0.34
O:	0.09-0.16	0.13	0.13	0.13	0.14	0.13	0.14
Sn:	1.20-1.70	1.32	1.32	1.31	1.31	1.33	1.31
Zr:		B A L A N C E					

INGOT IMPURITIES ANALYSIS: RESULTS IN PPM

Element:	Spec. Max.	1	2	3	4	5	6
Al:	75	31	33	36	30	46	29
B:	0.5	0.3	0.3	0.3	0.3	0.3	0.4
C:	270	181	144	156	172	179	183
Cd:	0.5	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Co:	20	<10	<10	<10	<10	<10	<10
Cu:	50	<25	<25	<25	<25	<25	<25
H:	25	4	4	<3	3	4	3
Hf:	100	50	56	53	53	62	47
Mg:	20	<10	<10	<10	<10	<10	<10
Mn:	50	<25	<25	<25	<25	<25	<25
Ni:	50	<10	<10	<10	<10	<10	<10
P:	80	25	28	28	31	27	28
S:	70	<35	<35	<35	37	38	<35
Si:	120	92	98	95	92	96	94
Ti:	3.5	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Zn:	100	<50	<50	<50	<50	<50	<50

J. Gas 9/16/02

From Page No. _____

WHH CHANG FAX: 541 928 4211

Should be 2866435



Wah Chang of 2

An Allegheny Technologies Company

Purchase Order No.: 2866435
Sales Order No.: 115510
Item No.: 1
Heat No.: 243687 Zr4
SFC No.: 1223217

J. Yang 9/6/02

P.O. BOX 480
ALBANY, OREGON 97321-0136
(541) 928-4211 FAX (541) 987-6991

PRODUCT CHEMISTRY ANALYSIS: RESULTS IN PERCENT

Element	Spec.	Max.	1	2
H:	0.0025	<0.0003	0.0003	
N:	0.0080	0.002	0.002	
O:	0.09-0.16	0.13	0.13	

ROOM TEMPERATURE TENSILE TEST: RESULTS

Test:	Spec. Min.:	Long. #1	Long. #2
Tensile Strength - Ksi:	60	77.6	77.3
Yield Strength (0.2% Offset) - Ksi:	35	52.8	56.4
Elongation in 1" - (%):	14	25	24

CORROSION TEST: RESULTS

Run No.: 8002-9
Method: ASTM G2
Procedure: LP-CT-8 Rev. 0
Parameters: 3 days/750°F/1500psi

Sample:	Spec. Max. mg/dm ²	Wt. Gain mg/dm ²	Visual Appearance
1:	22	16	Accept., no visible corrosion defects.
2:	22	16	Accept., no visible corrosion defects.

CERTIFIED BY: P. J. Sudar, Quality Assurance Dept.:

P. Sudar 8/30/02

mbc

WC: A Registered/Certified ISO 9002 Company.

Format: A1.06.04.19.BLNK 1800.P1-2
Document: C1.04.20.24

J. Yang 9/8/02

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From Page No. _____

EDS Analysis Conducted at Div. 18, 9/6/02

jfs2710
[ANALYSIS REPORT]

GENERAL CONDITIONS

Result File : JFS2710R
File Version : 1
Background Method : Fit
Decon Method : Gaussian
Decon ChiSquared : 75.09
Analysis Date : 6-SEP-2002
Microscope : SEM
Comments : ZR-4 P.O. ~~2866435~~

2866435

J. Yang 9/6/02

ANALYSIS CONDITIONS

Quant. Method : ZAF/ASAP
Acquire Time : 300 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%	K-Ratio	Cnts/s	Atomic%
Al	Ka	0.92	0.0066	32.15	3.03
Cr	Ka	0.15	0.0014	3.14	0.26
Fe	Ka	0.25	0.0025	4.28	0.40
Zr	La	97.65	0.9647	1525.53	95.54
Sn	La	1.03	0.0069	9.88	0.77
Total		100.00			

To Page No. _____

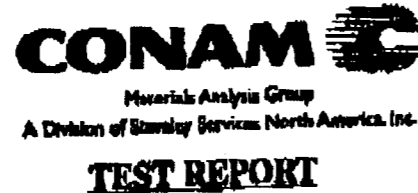
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Date _____

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194 International Blvd
Glenville Heights, IL 60139
Telephone + 1 630-681-0008
Facsimile + 1 630-671-5530
http://www.conamrap.com

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[Handwritten signatures]

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*Pages 1 - 9 copied for QA
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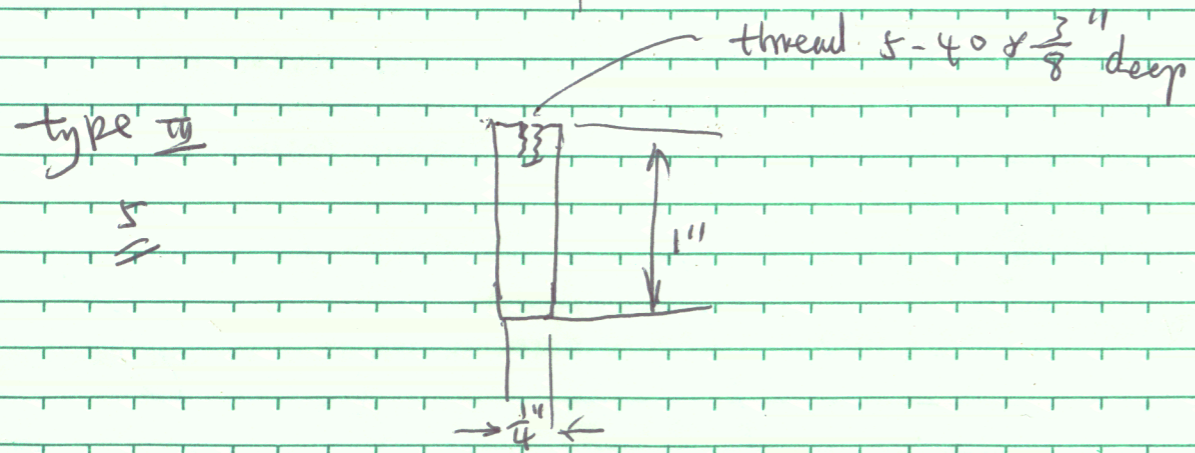
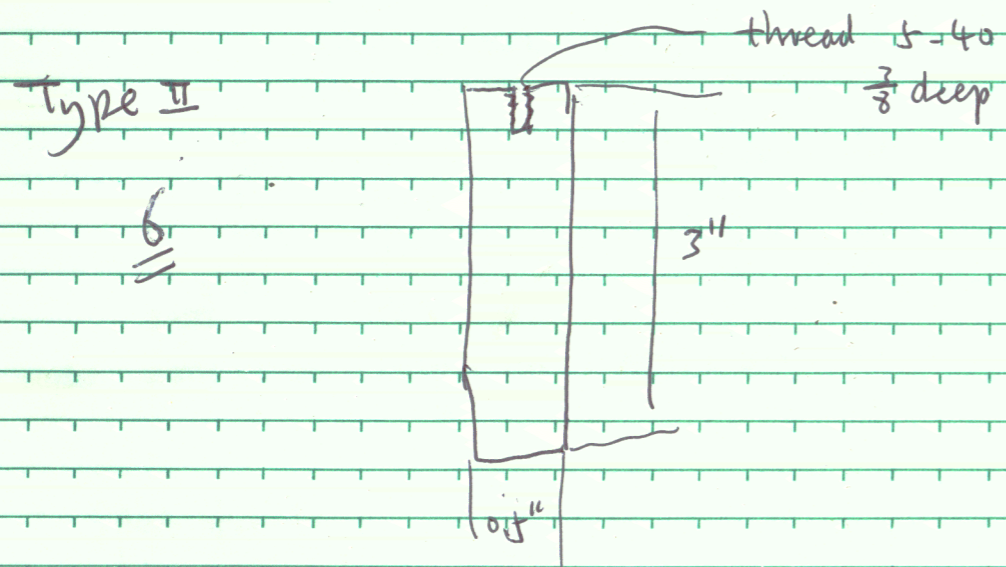
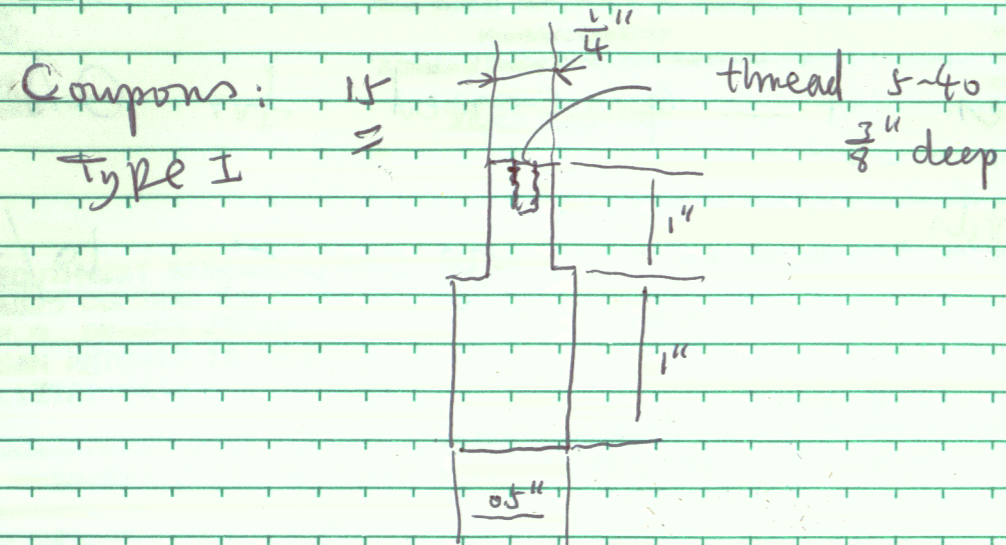
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All materials from P.O. 2386435 (page 5)

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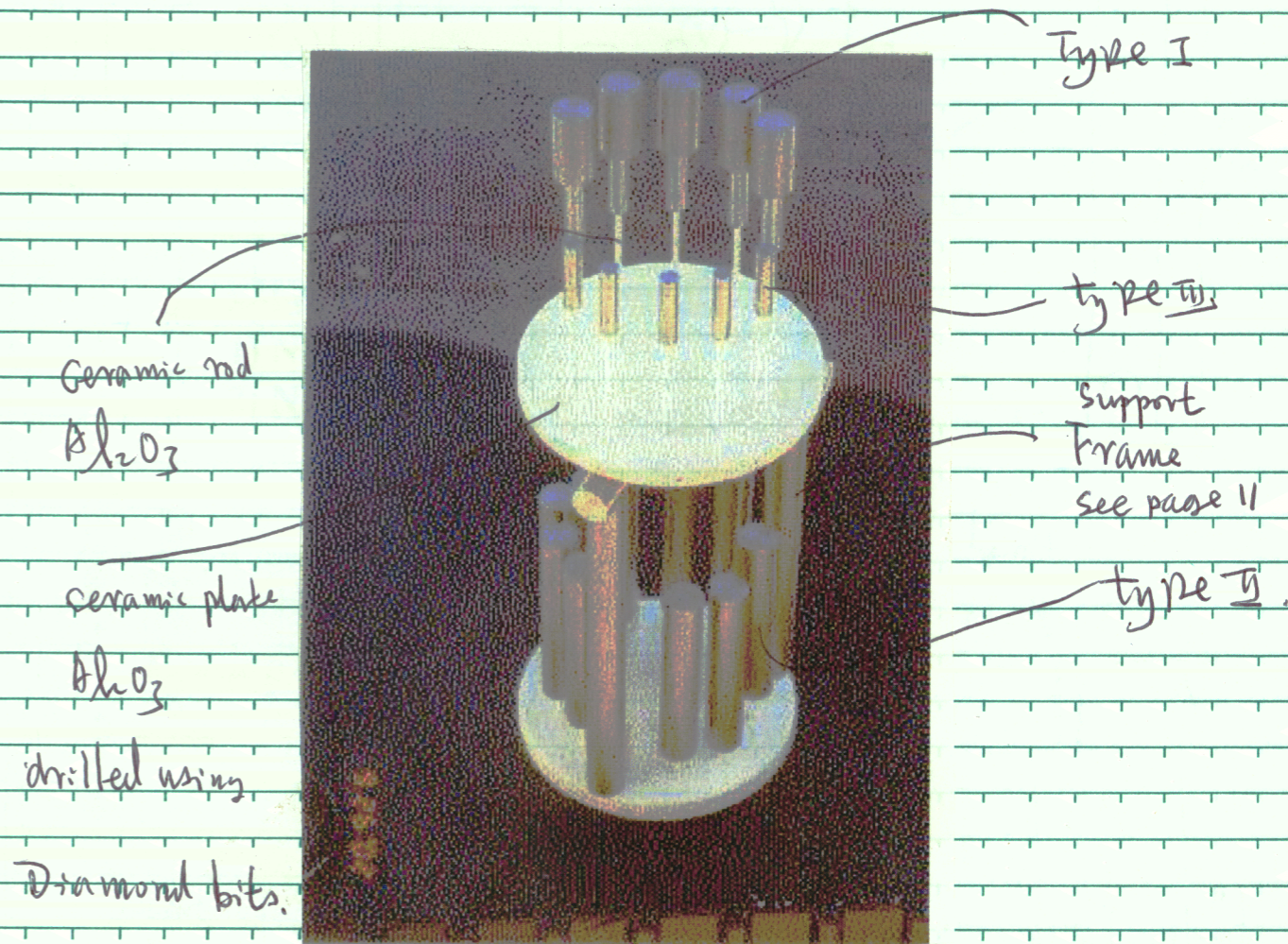
J. Yang

1/23/03

From Page No. _____

Hydrothermal oxidation:

using Hastelloy C Autoclave.



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Date

Invented by

Date

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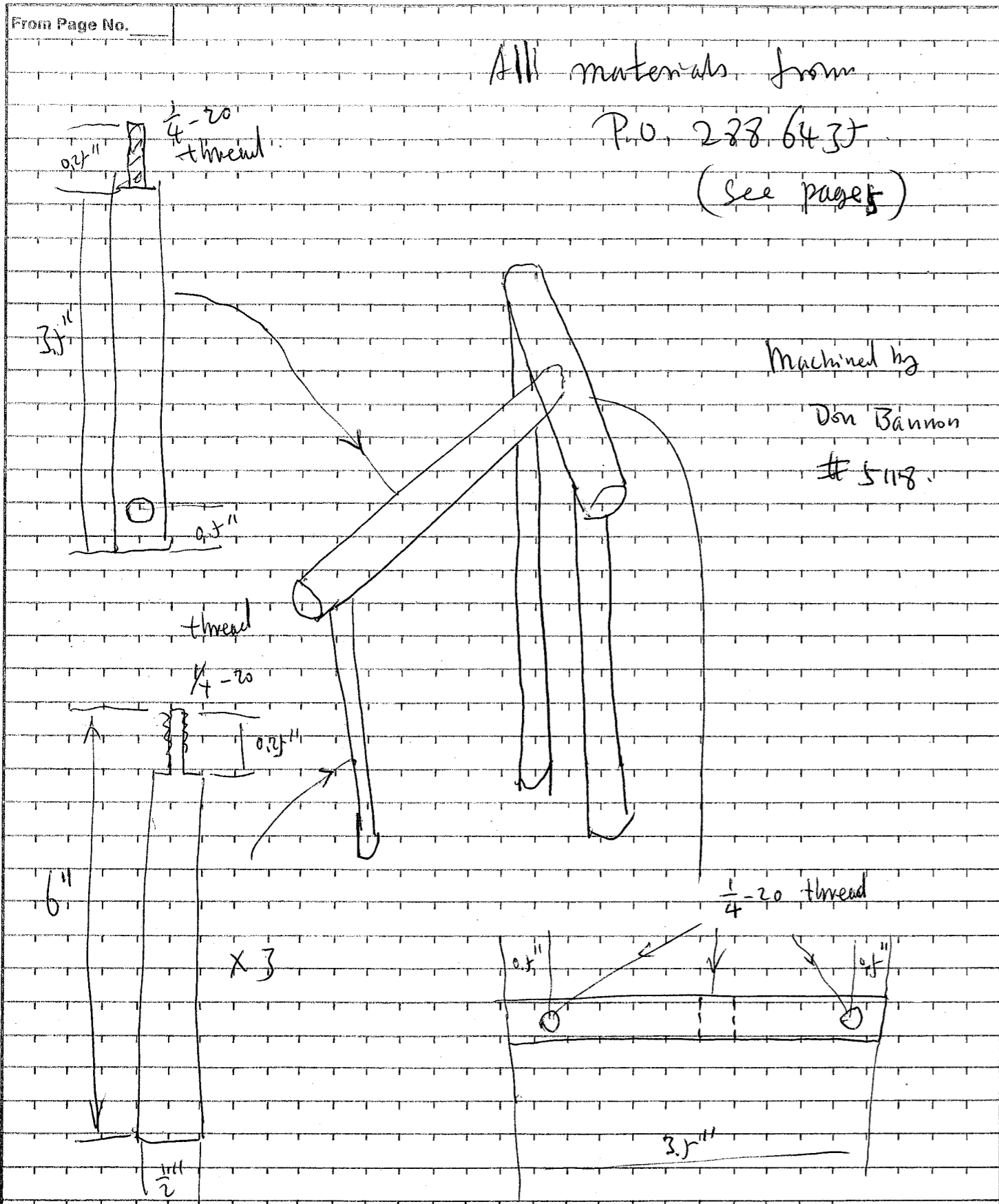
J. Yang

1/23/03

To Page No. _____

All materials from
P.O. 228 643J
(see pages 5)

Machined by
Don Bannon
5118



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Date _____

Invented by _____

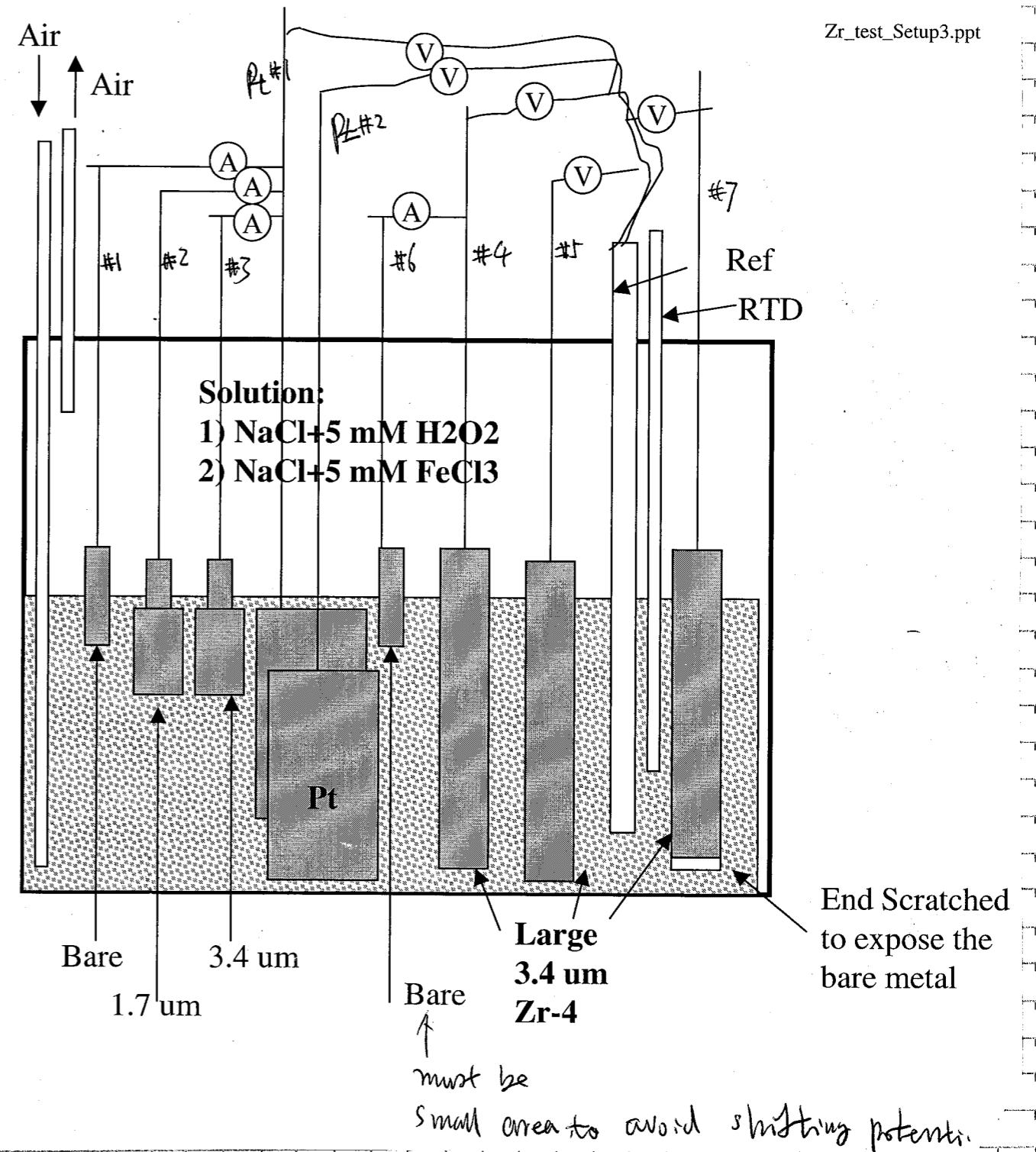
Date _____

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J. Young 1/23/03

First and second cells:

Zr_test_Setup3.ppt



Bare
↑
must be
small area to avoid shifting potenti.

To Page No. _____

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Date _____

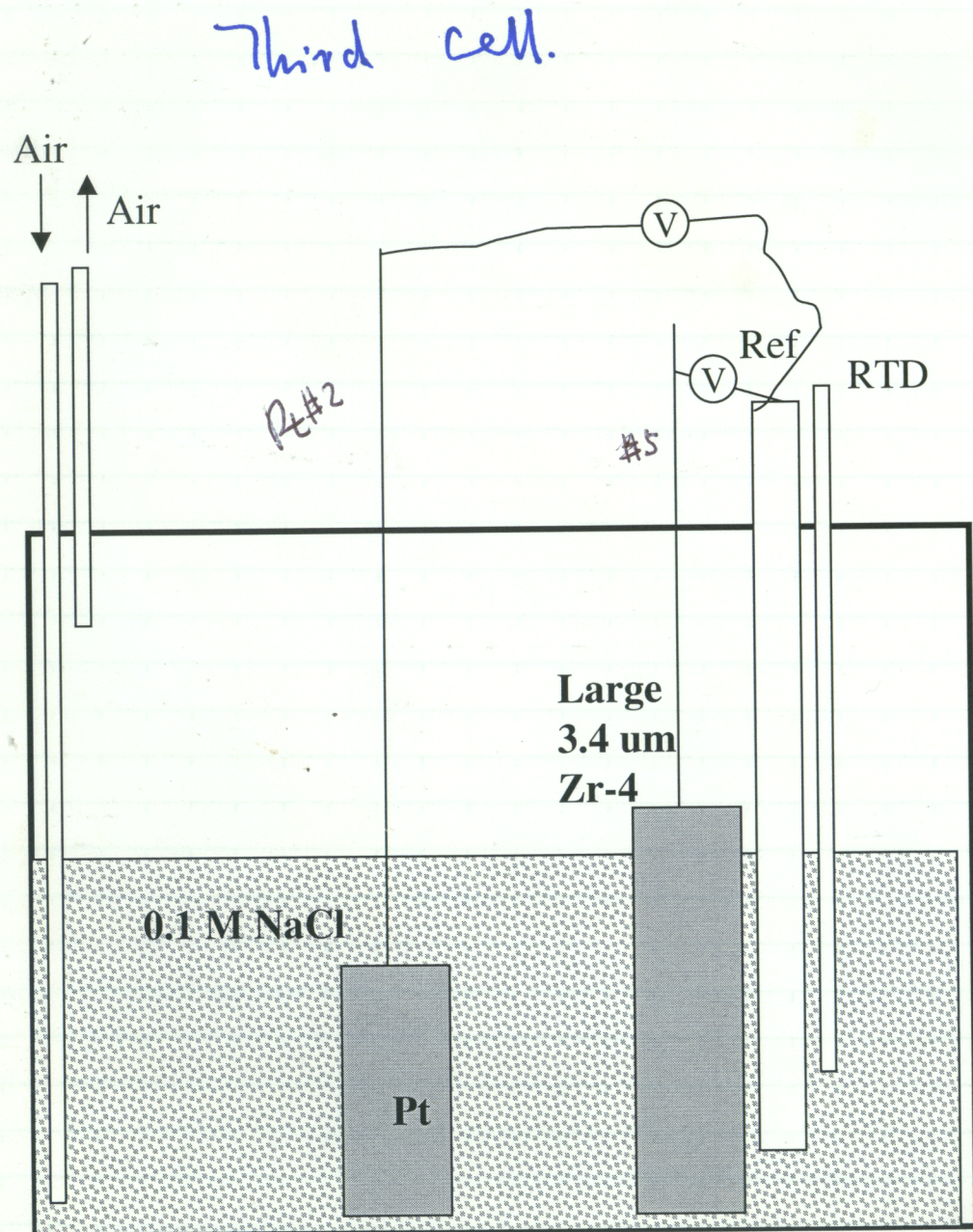
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J. Young 1/23/03

From Page No. _____



Zr_test_Setup4.ppt

0.1 M NaCl

Large
3.4 um
Zr-4

Pt

All other coupons taken out.

suggested by Gustavo.

To Page No. _____

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Invented by _____

Date _____

Recorded by *J. Yang*

1/23/03

0.1 M NaCl, CO₂-Free Air Bubbled, 50 Days Experiments

Coupling type	FeCl ₃ , 5 mM, pH=5	H ₂ O ₂ , 5mM	Plain	
1	Bare coupled with Pt	Bare coupled with Pt		Measure E(couple) and coupling current
2	1.7 um oxide coupled with Pt	1.7 um oxide coupled with Pt		Measure E(couple) and coupling current
3	3.4 um oxide coupled with Pt	3.4 um oxide coupled with Pt		Measure E(couple) and coupling current
4	Bare coupled with 3.4 um oxide	Bare coupled with 3.4 um oxide		Measure E(couple) and coupling current
5	3.4 um oxide Specimen with Bare Scratch (Internal Coupling)	3.4 um oxide Specimen with Bare Scratch (Internal Coupling)		Measure E(ocp)
6	Pt and large 3.4 um Zr (no Coupling)	Pt and large 3.4 um Zr (no Coupling)	Pt and large 3.4 um Zr (no Coupling)	Measure E(ocp)

Coupon / Electrode Summary

Electrodes	NaCl+ 5 mM FeCl ₃ , pH=5	NaCl+ 5 mM H ₂ O ₂	Plain NaCl	total
Type III, bare Zr	2 (1 to Pt, 1 to Large Zr)	2 (1 to Pt, 1 to Large Zr)		4
Type I, 1.17 um Zr	1 (to Pt)	1 (to Pt)		2
Type I, 3.4 um Zr	1 (to Pt)	1 (to Pt)		2
Type II, 3.4 um Zr	2 (for coupling, 1 for ECP)	2 (for coupling, 1 for ECP)	1 (for ECP)	5
Type II, 3.4 um Zr with Polished End (simulate scratch)	1 (internally coupled)	1 (internally coupled)		2
large Pt	2 (1 for coupling, 1 for ECP)	2 (1 for coupling, 1 for ECP)	1- for ECP	5
SCE Ref	1	1	1	3
			Sum	23

File: Test_Matrix_Zr-4_B / Tab: Connections

electrode ID

Electrodes in Each Cells

Electrodes	NaCl+ 5 mM FeCl ₃ , pH=5			NaCl+ 5 mM H ₂ O ₂			Plain NaCl			total
	Connections	I Chan	V Chan	Connections	I Chan	V Chan		I Chan	V Chan	
Type III, bare Zr	1 to Pt	I-1 Ch1	-	to Pt	I-3 Ch17	-	-	-	-	2
Type III, bare Zr	6 to Large Zr	I-2 Ch9	-	to Large Zr	I-4 Ch25	-	-	-	-	2
Type I, 1.17 um Zr	2 to Pt	I-1 Ch2	-	to Pt	I-3 Ch18	-	-	-	-	2
Type I, 3.4 um Zr	3 to Pt	I-1 Ch3	-	to Pt	I-3 Ch19	-	-	-	-	2
Type II, 3.4 um Zr	4 for coupling	Return, I-2	45 (II-1)	for coupling	Return, I-4	34	-	-	-	2
Type II, 3.4 um Zr	5 as control	-	44 (II-1)	as control	-	33	as control	-	36	3
Type II, 3.4 um Zr with Polished End (simulate scratch)	7 internally coupled	-	43 (II-1)	internally coupled	-	48 (II-1)	-	-	-	2
large Pt	4-1 for coupling	Return, I-1	42 (II-1)	for coupling	Return, I-3	47 (II-1)	-	-	-	2
large Pt	as control for ECP	-	41 (II-1)	as control for ECP	-	46 (II-1)	as control for ECP	-	35	3
SCE Ref. Electrode	Referen	-	Return	Referen	-	Return	Referen	-	Return	3

Witnessed & Understood by me, _____

Date _____

Recorded by *J. Yang*

1/23/03

From Page No. _____

Surface Areas for Electrodes

Sample	Dia	Height	inches		Total A (in ²)
			Sec A	Cyl A	
Previous	0.25	1.915	0.049063	1.503275	1.5523375
Type I	0.5	1	0.19625	1.57	1.76625
Type II	0.5	3	0.19625	4.71	4.90625
Type III	0.25	1	0.049063	0.785	0.8340625
Type III-Half	0.25	0.5	0.049063	0.3925	0.4415625
Type III-Quarter	0.25	0.25	0.049063	0.19625	0.2453125

Pt Plate W and L)	1	3		3	6
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Sample	Dia	Height	cm		Total A (cm ²)
			Sec A	Cyl A	
Previous	0.635	4.8641	0.316532	9.698529	10.01506062
Type I	1.27	2.54	1.266127	10.12901	11.3951385
Type II	1.27	7.62	1.266127	30.38704	31.6531625
Type III	0.635	2.54	0.316532	5.064506	5.381037625
Type III-Half	0.635	1.27	0.316532	2.532253	2.848784625
Type III-Quarter	0.635	0.635	0.316532	1.266127	1.582658125

Pt Plate W and L)	2.54	7.62		19.3548	38.7096
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J. Yang.
3/7/03

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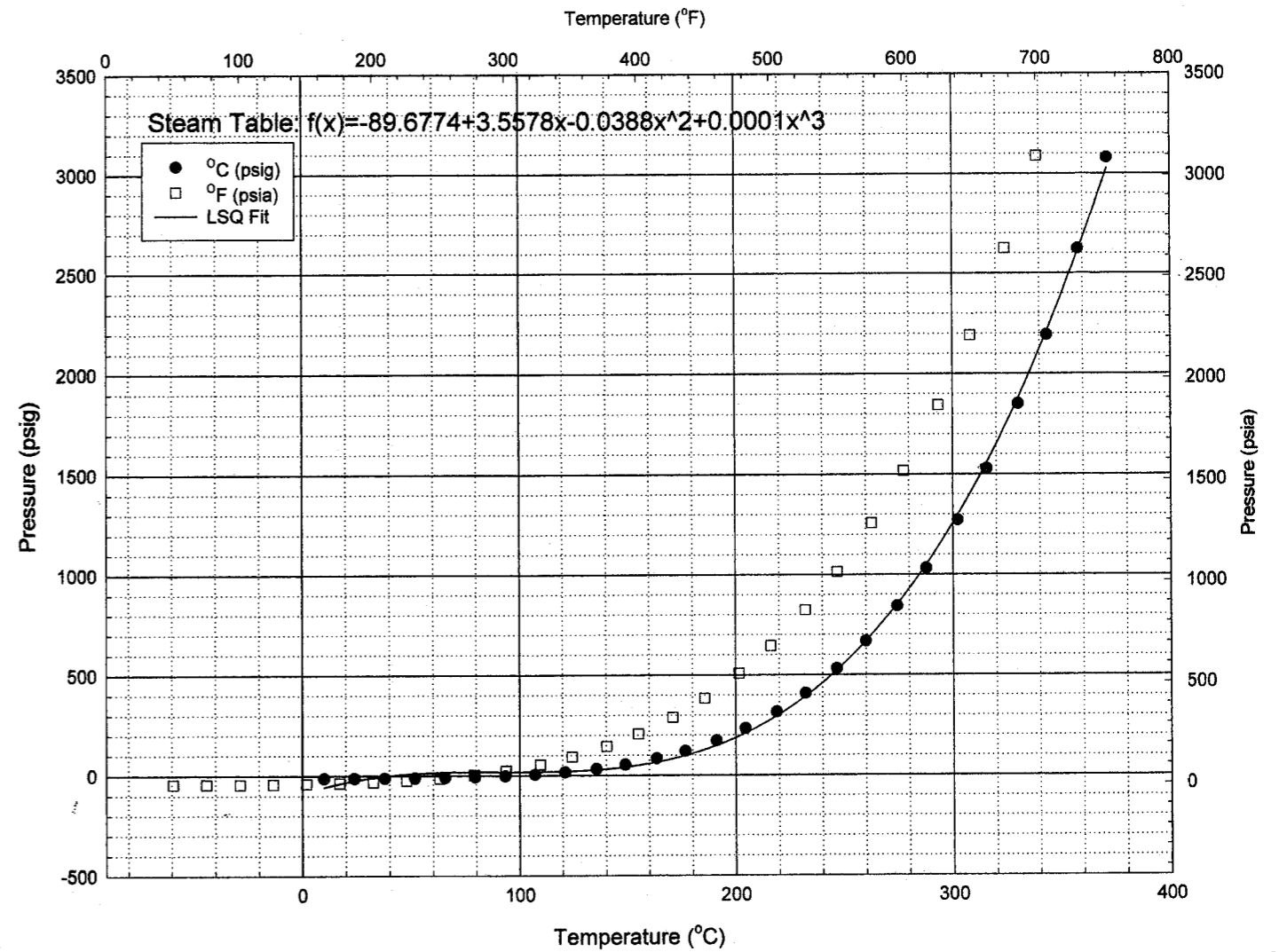
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Source: Perry's Chem. Eng. Handbook
6th Edition, pages 3-237 - 3-243.

J. Yang
4/7/06

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

B. Ford

2/12/03

Zirconium Exposure					
Date	time	Set point/Heater C	Internal Temp	Pressure	
				0	
10/22/02	8:22	50	24	24	0
10/22/02	8:40	75	49	26	0
10/22/02	9:00	100	77	31	0
10/22/02	9:15	125	103	37	0
10/22/02	9:30	150	129	45	0
10/22/02	9:45	175	154	56	0
10/22/02	10:00	200	179	65	0
10/22/02	10:15	225	203	77	0
10/22/02	10:30	250	231	89	0
10/22/02	10:45	275	256	102	0
10/22/02	12:00	275	275	158	0
10/22/02	12:25	300	275	172	150
10/22/02	12:45	300	304	184	200
10/22/02	1:00	300	301	194	225
10/22/02	1:30	300	300	209	300
10/22/02	2:00	300	300	221	375
10/22/02	2:30	300	300	323	450
10/22/02	2:45	300	300	236	490
10/22/02	3:15	300	300	243	550
10/22/02	3:45	300	300	249	600
10/23/02	7:12	300	300	274	850
10/23/02	8:00	300	320	280	1050
10/23/02	9:00	300	320	285	1625
10/23/02	10:17	300	320	288	1675
10/23/02	12:00	300	320	291	1625
10/24/02	7:27	300	320	293	1700
10/24/02	9:45	300	320	293	1680
10/24/02	Internal TC is questionable- Pressure is more in line with heater TC lowering temperature to make pressure agree with 300C				
10/24/02	10:13	300	297	288	1100
10/24/02	10:46	305	300	286	1000
10/24/02	12:13	305	305	282	975
10/24/02	12:42	310	305	282	1000
10/25/02	7:48	315	310	284	1000
10/29/02	7:15	315	312	286	1100
10/30/02	7:20	315	315	288	1050
11/5/02	7:46	Started cooldown to remove specimens #1-4 type 1			

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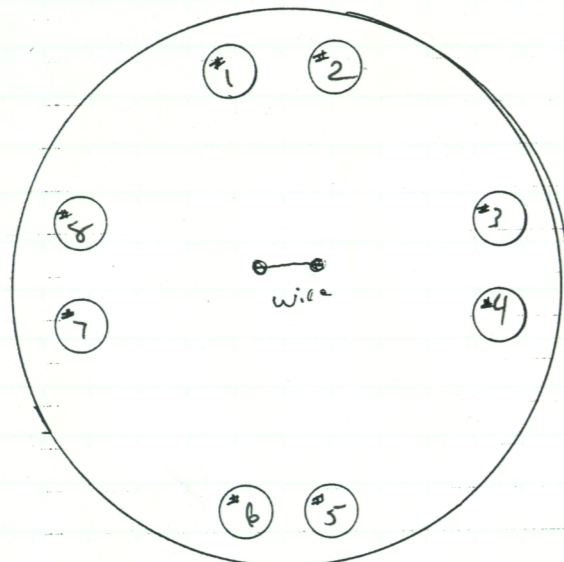
Zirconium Exposure second exposure					
Date	time	Set point/Heater C	Internal Temp	Pressure (psi)	
11/7/02	9:06	50	22	22 (2)	0
11/7/02	9:30	75	49	24	0
11/7/02	9:55	100	76	31	0
11/7/02	10:25	125	101	42	0
11/7/02	12:25	150	125	76	0
11/7/02	12:50	175	151	84	0
11/7/02	1:35	200	175	101	0
11/7/02	2:05	325	201	113	0
11/7/02	2:30	250	227	124	0
11/7/02	3:00	275	251	141	0
11/7/02	3:30	300	276	158	0
11/8/02	7:14	315	300	273	850
11/8/02	8:40	310	305	279	1225
11/8/02	9:11	310	310	280	1300
11/8/02	9:28	310	310	208	1375
11/8/02	10:00	305	310	281	1400
11/8/02	10:26	305	310	280	1325
11/8/02	11:58	305	305	278	1150
11/8/02	2:30	305	305	278	1000
11/9/02	10:40	315	305	277	970
11/10/02	10:10	310	315	287	1780
11/11/02	7:15	310	310	282	1050
11/12/02	7:10	310	310	282	1050
11/15/02	3:15	310	310	282	1050
Cell /Autoclave was stable until 12/13/02 when we lost our heating mantel - cell was cooled over the weekend and restarted on 12/16/02 precautions were taken not to disturb the specimens during this procedure Repressure checked the cell and system started heating					
12/16/02	11:21	325	277	188	200
12/17/02	7:00	310	310	296	1500
12/17/02	8:00	305	310	291	1100
12/17/02	3:45	300	300	286	1100
12/18/02	7:15	300	300	286	1100
12/18/02	3:12	300	300	286	1050
12/19/02	7:15	300	300	286	1050
12/21/02	1:30	300	300	286	1100
autoclave was stable for the rest of oxidation procedure					
1/20/03	Start cool down procedure for removal of Zirconium specimens				

Witnessed & Understood by me, _____ Date _____ Invented by _____ Date 2/12/03
 Recorded by B. K. J.

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Ceramic plates with Zirconium stand see pg # 11 for Pic.

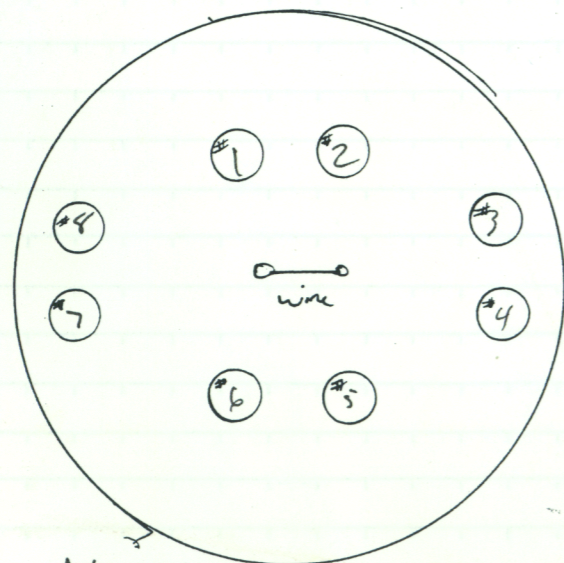
TOP



Top Plate Specimen Type I
 Circle #1-4 Removed 14 days
 Circle #5-8 Removed 12 weeks

4
4

Bottom



Bottom Plate All Specimens Type II
 All Specimens 12 weeks

8

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Witnessed & Understood by me, _____

Date _____

Invented by _____

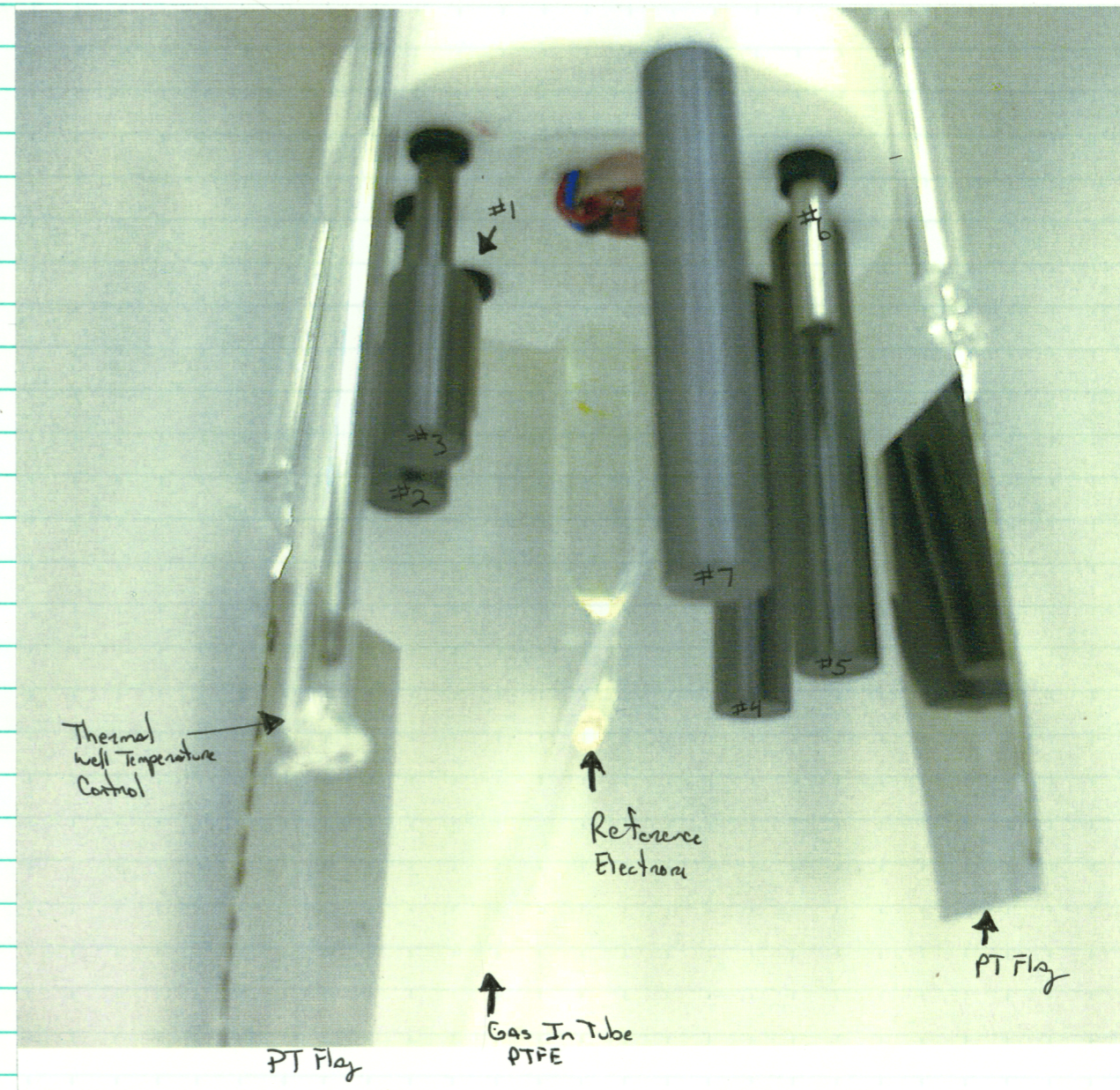
Date _____

Recorded by _____

2/12/03

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Specimens In Cell - Cell Configuration



#1-7 Specimens Both Cells Are Configured The Same
 (see pg #13)

Picture on Next Page #22 Is A Zoom In shot of
 Cell Configuration.

To Page No. _____

Witnessed & Understood by me, _____

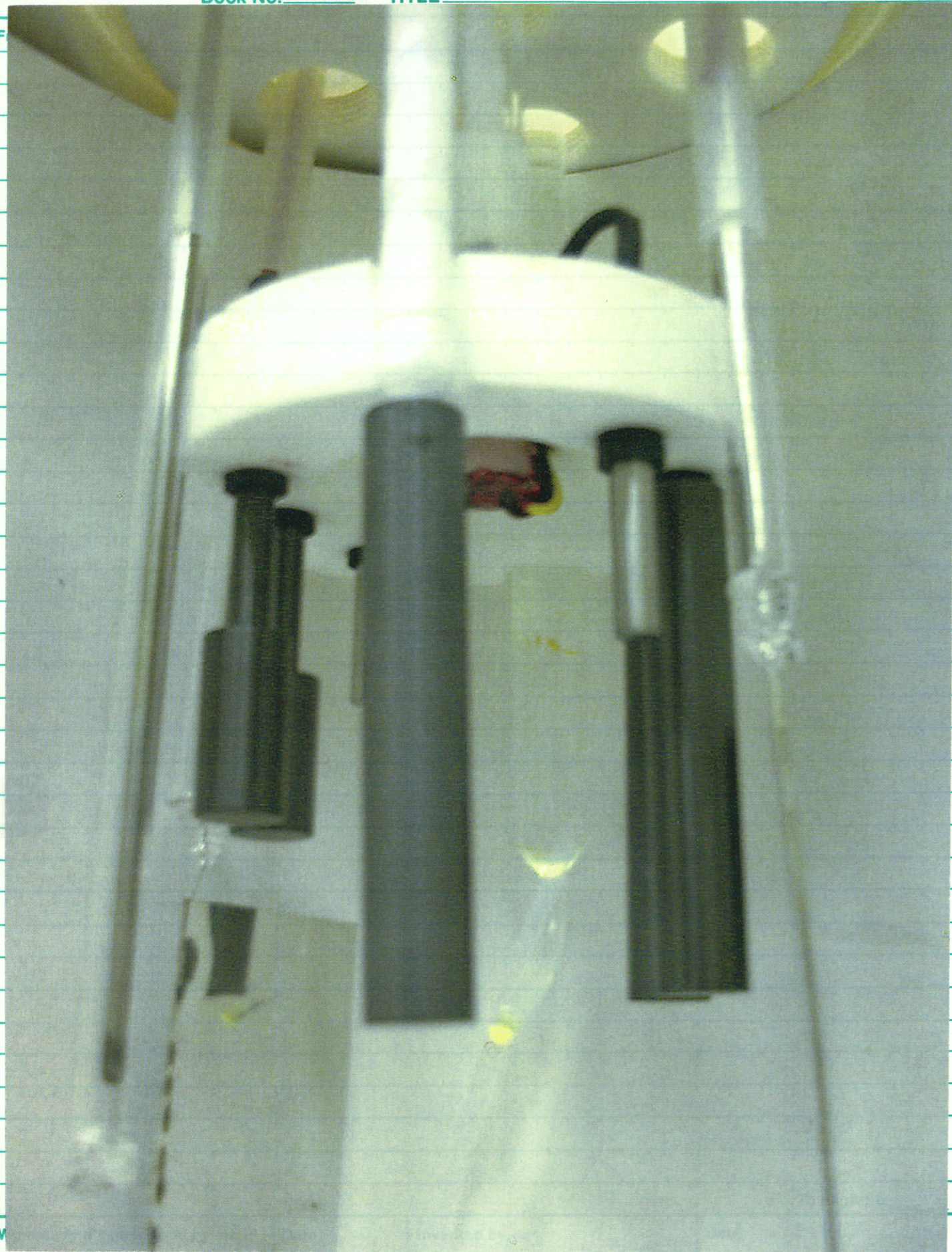
Date _____

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Date _____

Recorded by _____

2/12/03



Recorded by

B. K. S.

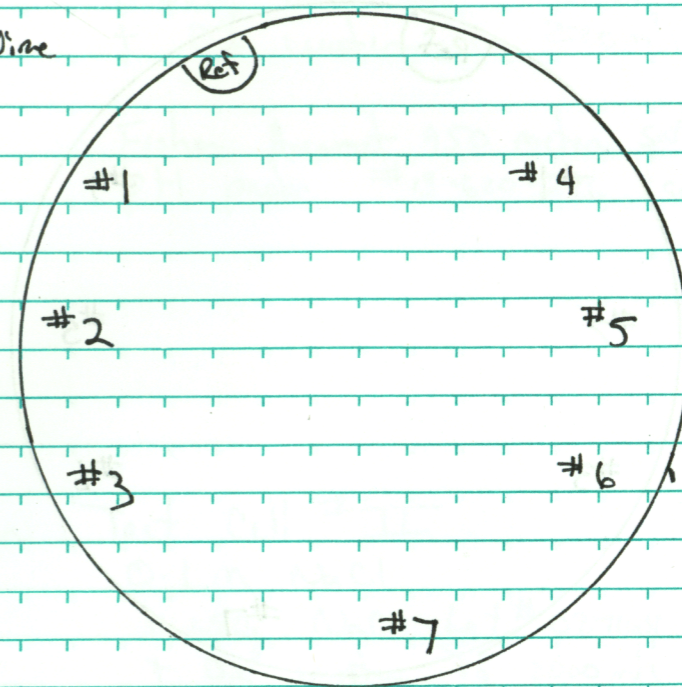
2/12/03

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Cell # I
Reference:

Connection Color Wire

- #1 = Red
- #2 = Blue
- #3 = Grey
- #4 = Brown
- #5 = Yellow
- #6 = Black
- #7 = Ron Brass



Specimens weight Taken with Sartorius Genius SN# 12809099
cal 11/15/02 due 5/15/03

Specimen #1	Type III Base	
Start wt =	4.7114g	End wt = 4.70180g
Specimen #2	Type I 1.7µm	
Start wt =	27.97456	End wt = 27.96904g
Specimen #3	Type I 3.4µm	
Start wt =	29.28712	End wt = 29.29545g
Specimen #4	Type II	
Start wt =	69.46392	End wt = 69.4917g
Specimen #5	Type II	
Start wt =	68.98238	End wt = 69.32894g broken Specimen Rod In Specimen
Specimen #6	Type III Base	
Start wt =	4.70223	End wt = 4.70579g
Specimen #7	Type II	
Start wt =	69.78889	End wt = 69.79223g

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

B. K. S.

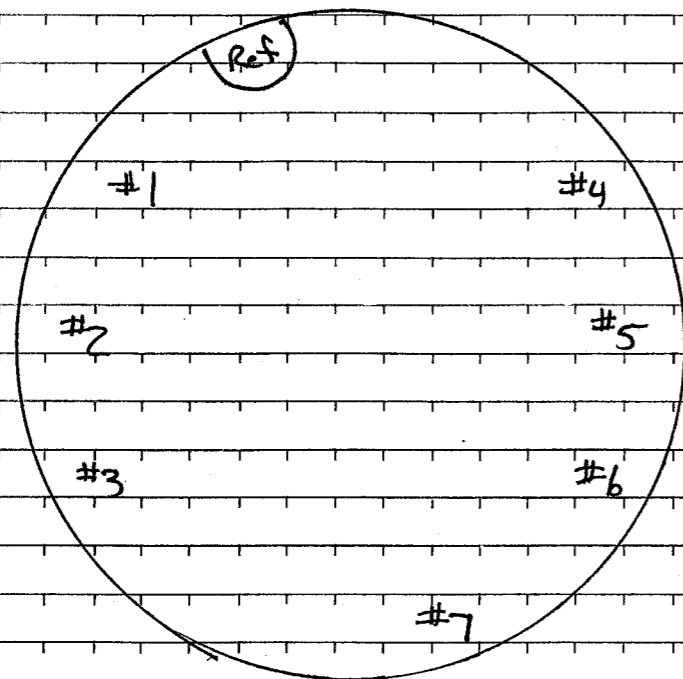
2/12/03

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Cell # II
Reference =

Connection Color Wire

- #1 = white
- #2 = Purple
- #3 = Pink
- #4 = Green
- #5 = Orange
- #6 = TAN
- #7 = Red Brass



Specimen weights taken with Sartorius Genius SN# 12809099 cal 11/15/02
Duc 5/15/03

#	Specimen #	Type	Bar	Start wt	End wt
1	1	Type III	Bar	4.77221	4.76520g
2	2	Type I	1.7um	28.00591	28.00897g
3	3	Type I	3.4um	29.43229	29.43809g
4	4	Type II		63.43525	63.44221g
5	5	Type II		69.41992	69.42513g
6	6	Type III	Bar	4.77165	4.76551g
7	7	Type II		63.54453	63.80305g

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 Recorded by B. K. D.

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Solution for Test Cell # I

0.1 M NaCl
11.688g NaCl Lot # 027168
+ DI water to 2000mls

pH start = 5.378
pH End = 4.751

Fisher Accumet 950 meter SN# 3340 cal 8/7/02 over 7/03
pH probe # 13-620-296 SN# 2291257P6

Solution for Test Cell # II

0.1 M NaCl
11.690g NaCl Lot # 027168
+ DI water to 2000mls

pH start = 4.573
pH End = 4.932

Fisher Accumet 950 meter SN# 3340 cal 8/7/02 over 8/7/03
pH probe # 13-620-296 SN# 2291257P6

Solution for Test Cell # III

0.1 M NaCl
11.691g NaCl Lot # 027168
+ DI water to 2000mls

pH start = 5.581
pH End = 4.997

Fisher Accumet 950 meter SN# 3340 cal 8/7/02 over 7/03
pH probe # 13-620-296 SN# 2291257P6

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 Recorded by B. K. D.

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Cell # III

Test Cell # III

See pg # 14 for Cell Configuration

And pg # 25 for Solution

Specimen Type II

Start wt: 69.28397g Saltonius Genius sn# 12809899
 6/10/03 Taken End wt = 69.28721g cal 11/15/02 pur 5/15/03

Reference: Fisher SN# 0249099 13-620-52

Temperature = 95°C Hg Thermometer sn# H98-182
 cal 5/1/03 pur 5/1/04

* Note End at Specimen Examination Has small Buildup of Material on Specimen
 After cleaning will change Solution And Restart Test. See pg # 25 for
 New Solution

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		Recorded by <i>B. J. D.</i>	2/12/03

TITLE _____

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2/11/03 Cell I, II Assembled

13:30 Temp. being raised.

14:45 91°C. Heater shut down to wait
 for Cell II.

16:00 All connections to Multi Cell instrument
 are done. (see page 15)

16:00 Program started.
 Data file: Tebba.txt — Results see pages 30-32
 Config file: Zr-Test

16:31 Found. Forget to connect return of II-1 to Reference
 in Cell - II. — corrected. L y. 2/11/03

2/12/03

10:56 Cell III Assembled

II - Pt-2 and III - #5 - 3.4 um connected
 to computer.

All air bubbling well.

Verifying connections by shortcircuiting each electrode.

III - #5 good - 14 μ V.
II - Pt-2 good. 2.657 μ V

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Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>J. Yest</i>	2/12/03

From Page No. _____			
#34 - II - #4	data. 588.	- 117 mV	problems!
#33 - I - #5	good.	0.011 μ V	
#34 - II - #4	again.	- 32733 μ V	problem.
		10.92 μ V	after cleaned the chipper.
#48 - II - #7	good.	0.586 μ V	
#47 - I - Pt1	good	0.036 μ V	
#46 - II - Pt2	+323 mV	computer 323414 μ V	good.
#45 - I - #4	-31.2 mV	computer -30375 μ V	✓
#44 - I - #5	shorten this	0.416 μ V	good
#43 - I - #7	-39.0 mV	computer. -42817 μ V.	o.k.
#42 - I - Pt1	+178.5 mV	computer +178906 μ V	good.
#41 - I - Pt2	+320.1 mV	computer 320130 μ V	good.

The tests are complex potentials. Values are reasonable.

Electrode areas: see page 66.

Type I $\leftarrow \begin{matrix} 0.5" \times 1" \\ 0.25" \times 0.25" L \end{matrix} \rightarrow \begin{matrix} 2.15 \text{ cm}^2 \\ 2.78 \text{ cm}^2 \\ 2.84 \end{matrix} \rightarrow 24.38 \sim 24.4 \text{ cm}^2$
 Type II $0.5" \times 2.25" \rightarrow \frac{2.25"}{3"} \times 62.04 \text{ cm}^2 = 46.5 \text{ cm}^2$
 Type III: $0.25" \times 0.25" L \rightarrow \frac{2.78}{2.85} \text{ cm}^2$
 sig. 2/13/03

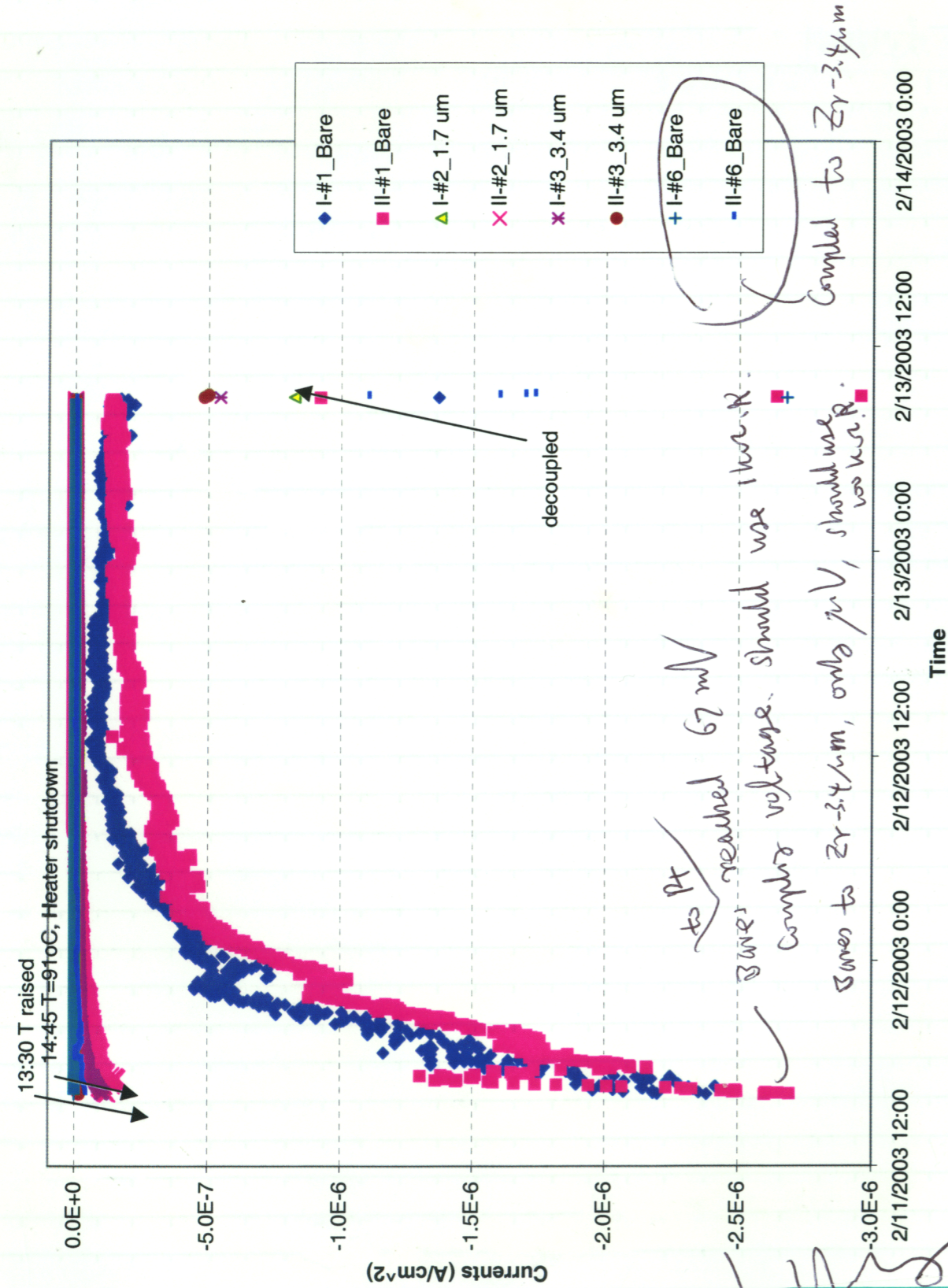
Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by J. V. Y.	2/13/03

From Page No. _____		
2/13/03	8:51	Decouple all the coupled electrodes, #1108
		The returns of these channels to Ref. Electrode.
	9:18	Found ch I-1 Broken, — fixed.
	11:26	prog. stopped.
	11:27	New file started. Feb13a
	14:13	All E of Bare sample, -120 \rightarrow -220 mV/sec
	14:22	cell II opened, #1, #6 Bare coupon repolished grit.
	14:44	cell II closed.
	14:49	cell I opened, #1, #6 Bare coupon repolished grit
	14:59	cell I closed.
		All E of Bare sample, -700 \rightarrow -580 mV/sec.
	15:24	E of #7 in I and II cells are low! -200 mV. measure with Dmm shows the same value!

2/18/03 T = 232 for all cells.
 9:00 E of #7, in I and II cells still low!
 venting started.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by J. V. Y.	2/18/03

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Witnessed & Understood by me, _____

Date _____

Invented by _____

Recorded by _____

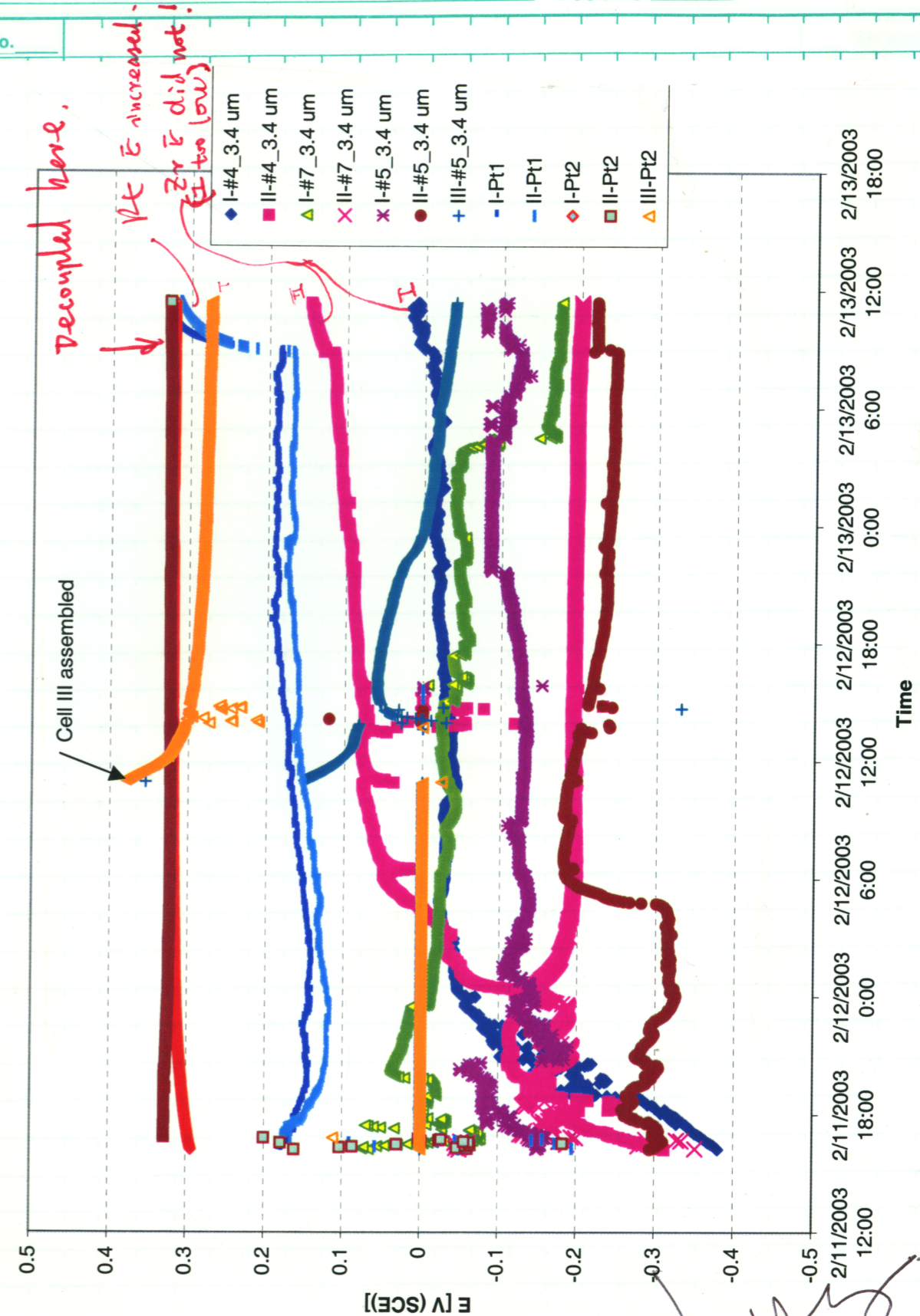
Date _____

Handwritten signature and date: 2/13/03

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File: Feb11a00_01 / Tab: Potential



Witnessed & Understood by me, _____

Date _____

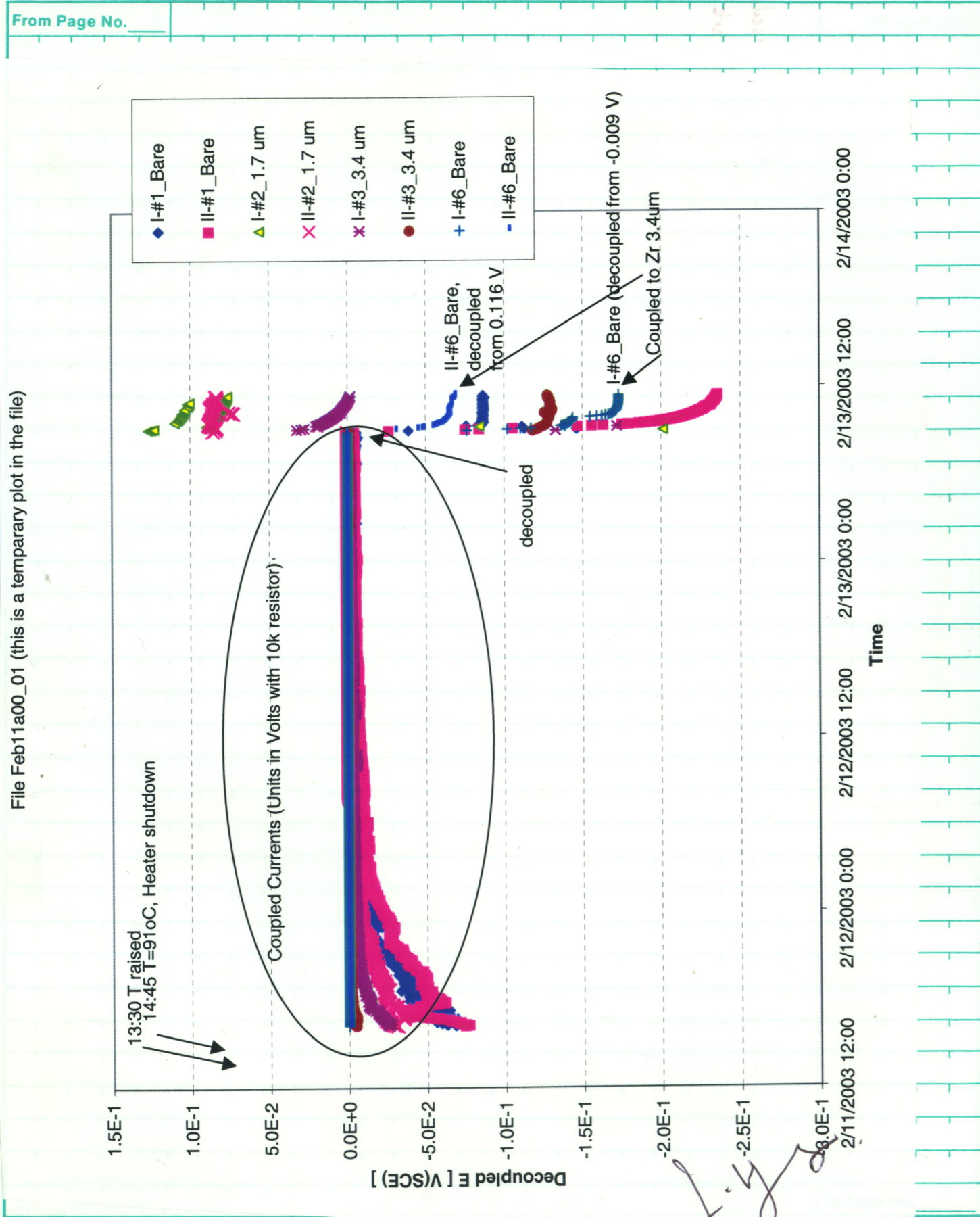
Invented by _____

Recorded by _____

Date _____

Handwritten signature and date: 2/13/03

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Witnessed & Understood by me, _____ Date _____
Invented by _____ Date _____
Recorded by J.Y. 3/13/03

From Page No. _____

2/13/03
9:28. $T_{cell I} = 60.4^{\circ}$, $T_{cell II} = 63^{\circ}$, $T_{cell III} = 44^{\circ}$

10:35 $T_{cell I} = 91.3^{\circ}$, $T_{cell II} = 92^{\circ}$, $T_{cell III} = 92^{\circ}$

2/26/03
14:10 Found program stopped by its own at 11:37 p.m. 2/24/03 !!
Air was gone!

14:20 program restarted. 03-0226a with a new computer.

Temp: I II III IV (glass)
91.7 $^{\circ}$ 93.6 $^{\circ}$ 92.8 $^{\circ}$ 94 $^{\circ}$

I - #3_3.4 um
I - #7_3.4 um gives 1.48E4?
I - #5
I - #4 > sometimes also!

cell I reference Junction/Bridge moved!
bubbles going up!

14:30 Ref. E. taken out to fix bubble problem.
14:33 problem fixed!

14:30 Air bottle changed.

2/28/03
8:34. Found program halted due to bad connection of IEEE cable.

Witnessed & Understood by me, _____ Date _____
Invented by _____ Date _____
Recorded by J.Y. 2/28/03

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8:35 program restarted

10:13 I-#7, +138.71 mV, II-#7 176.50 mV
 III-#5, 170. mV

10:17 Raised I-#7, II-#7 by ~ 0.2 in

10:23 Raised III-#5, 1/8" of bare electrodes in cell I
 Wiped up liquid level in II cell ^{immersed}

12:36 So 1/8" of bare electrodes immersed.

14:10 potential of I-#7, II-#7, III-#5 had no change

14:28 coupons coupled as specified in Page 15
 Resistors for Pt Coupling: 1k Ω
 Resistors for Zn-ZnAm Coupling: 10k Ω

14:34 after 14:24, All returns connected to common joints

3/3/03

10:34 noticed bare II-#1 barely on liquid line
 due to evaporation
 bare III-#6 only 2mm below water line
 water refilled to cell I.
 II-#1 immersed 0.5 cm,
 II-#6 immersed 0.7 cm.

To Page No. _____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by J. Yeg	3/3/03

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II-#

I-#1 immersed 0.3 cm

I-#6 immersed 0.3 cm.

more water added to cell I

I-#1 and I-#6 cell have 0.7 cm immersion depth

13:16 processed data, Found, channel 42 oscillating too much.

Found out that channel 42 was disconnected / came off.
 be plugged in channel 42.

3/6/03

11:50 more distilled H₂O added to I and II cells

3/7/03

12:08 Found computer was down
 (because the power cable was temp. off)

new file 03-0307a.

3/12/03

15:32 program quit for connections to the Mic Test

16:12 program restarted 03-Mar12a

3/14/03 12:28 program restarted 03-Mar14b.

To Page No. _____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by J. Yeg	3/12/03