NOTEBOOK NO. 831

ISSUED TO Watson

ON October 13 **20**06

DEPARTMENT Div 20

RETURNED ______________ **20**____

CONTROLLED COPY [redacted] 831

20.06002.01.212

Abbreviation Table

Abbreviation	Definition
	NAME SIGNATURE INITIALS
	SANDRA WATSON [signature] SW
	Nancy Hawkins [signature] NH
	3/26/07 - Notebook copied for QA (pp 1-14)

TITLE ____________ Book No. ________

10/13/06

~~This notebook is a continuation of SN 716E.~~ 10/13/06 SW

this notebook is a continuation of SN 817E.

1/18/07

STUDIES OF FLOW OF IN-DRIFT WATER THROUGH STRESS CORROSION CRACKS

1/19/07

the overall objective of the project is to document the experimental studies on flow of in-drift water through stress corrosion cracks. the experimental studies include procedure, set-up, data collection and results

June 18, 2007 NH

Recorded by: [signature] Date 1/19/07 Verified by: Date

Planned Work

- Experimental Work
  - Effect of thermal aging on contact angle between in-drift water and Alloy 22
  - Drop experiments for different crack width: 25, 50, 100, and 200 μm
  - Two temperature: 25 and 95 °C
  - Drop experiments with calcium chloride brines, alkaline brine, and neutral brine
  - Slanted surface with solution for which in-drift water enters the crack

2/1/07

Two temperature : 25C and 95C

This experiment will show the time lapse difference of flow on stress crack at two temperatures.

Control sample of DI water followed by three brine solns.

EQUIPTMENT -

ALLOY 22 BLOCK WITH CRACK, HOLES DRILLED FOR PROCCEDURE
(2) CHROMALOX HEATING ELEMENTS
OMEGA THERMOCOUPLE
WATLOW 93 CONTROLLER BOX
OAKTON INFRAPRO INFRARED THERMOMETER - AN# 010864
- SN# 2332580201
ULTRASONIC CLEANER

REAGENTS -

TYPE I DI WATER
NEUTRAL BRINE (SN 768/14)
ALKALINE BRINE (SN 768/14)
CaCl BRINE (SN 768/14)
ACETONE

SUPPLIES -

DISPOSABLE TRANSFER PIPETTES
KIMWIPES
STOPWATCH
HARD RUBBER STOPPERS ADDED 2/7/07 SW
ELECTRICAL TAPE ADDED 2/8/07 SW

SET UP OF EQUIPMENT - INITIAL

ALLOY 22 BLOCK WAS PLACED ON CLEAN SURFACE

HEATING ELEMENTS AND THERMOCOUPLE CONNECTED TO CONTROLLER BOX

HEATING ELEMENTS AND THEMOCOUPLE WERE INSERTED INTO ALLOY 22

CONTROLLER BOX WAS TURNED ON AND CONNECTIONS CHECKED

CONTROLLER TEMPERATURE ARBITRAIRLY SET TO 50C SO HEATING ELEMENTS CAN BE CHECKED

ALL CONNECTIONS WORKING

2/5/07

Before beginning this experiment it is necessary to ~~determint~~ SW 2/5/07 determine the internal block temperature required in order to maintain desired surface temperature

This will be accomplished by initially setting the controller box setting to the desired surface temperature and adjusting as necessary. Sufficient time will be allowed for temperature stabilization

Alloy 22 block is ultrasonically cleaned in acetone for 10 minutes and rinsed well using DI water. Block is then ultrasonically cleaned again using only DI water. Block is dried with compressed air

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Alloy 22 block is placed on clean, dry surface. Heating elements and thermocouple are inserted in to block

The initial temperature of the controller box is set to 95C. The block temperature will be allowed to stabilize over a period of one hour.

After 1 hour, the surface temperature was taken using infrared thermometer. Thermometer reading was 31C yet block very hot to touch.

Restart controller and reset temperature to 95C. After 20 minutes, infrared thermoter is reading 31C. After 1 hour, thermometer is reading 33C. Block is hot to touch

Shut down

2/6/07

Controller box turned on and set to 95C. Power lost.

Open controller box and replace fuse. Close box and restart.

Smoke observed from wires leading to one heating element. Fuse blew before unit could be shut off.

Visible damage to heating element wires

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Project No.

Book No. TITLE

2/7/07

Replacement heating element located and installed to controller box

Alloy 22 block raised off of counter surface using (4) hard rubber stoppers

* Add 50c and 75c to temperature points (Pavan Shukla)

Controller box set to 95C. After 1 hour, infared thermometer reading is 34C.

Contact thermometer manufacture (place black electrical tape on surface or paint surface dark color)

2/8/07

Alloy 22 block was cleaned using DI water and thoroughly dried with Compressed air

Block was placed on (4) hard rubber stoppers and heating elements and thermocouple were inserted.

(2) lengths of black electrical tape were placed on the surface of the block as per manufacturer of thermoters instructions.

Controller box turned on and set to 95C. After one hour the surface temperature reading on the electrical tape was 87C.

Controller temperature increased to 100C. After one hour the surface temperature was 92C

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[signature]	2/8/07		

2/9/07

Controller box turned on and set to 105C. After 1 hour surface temperature was 97C.

Controller set to 103C. After 1 hour, ~~surf~~ [2/9/07 SW] surface temperature was 95C.

Controller box temperature was decreased to 75C. Temperature was allowed to stabilize for a ~~period~~ [2/9/07] period of two hours and surface temperature was taken.

2/13/07

Controller box turned on and following procedure done 2/8/07 and 2/9/07, incremental controller box adjustments were done to determine temperatures necessary for desired surface temperatures

2/14/07

Below are the controller box settings required to maintain desired surface temperatures -

SURFACE TEMPERATURE	CONTROLLER BOX SETTING
95C	103C
75C	81C
50C	54C
25C	26C

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Project No. ____________

Book No. ____________ TITLE ______________________

2/20/07

Experimental procedure for determining flow rate of DI Water at 25C, 50C, 75C and 95 C.

Alloy 22 block is ultrasonically cleand in acetone for 10 minutes, then removed and rinsed well with DI water. Block is then ultra sonically cleaned in DI water for 10 minutes. Block is dried throughly with compressed air.

Alloy 22 block is placed on hard rubber stoppers and heating elements and thermocouple are inserted.

Controller box is set to temperature determined on previous page (831/7) and surface temperature is periodically checked. Once external temperature stability is achieved, an additional 10 minutes is allowed for certainty.

Using disposable pipette, one drop of DI water is dropped directly on the crack machined into Alloy 22 block. Simultaneously the stopwatch is started to record the time.

When the drop of water is no longer visable over the crack, the stopwatch is stopped and the time recorded.

Since only DI water is being used it is not ~~neeet~~ [2/20/07 SW] necessary to clean block between drops

Recorded by:	Date	Verified by:	Date
Sandra Watson	2/20/07		

TITLE ______________________________ Book No. ____________

DI WATER DROP Summary

	25C	50 C	75C	95C
DROP #1	61min 03 sec	8min 42sec	2min 47 sec	1min 14sec
DROP #2	56min 49 sec	9min 01 sec ~~9min 01 sec~~ 2/21/07	2min 54sec	1min 18sec
DROP #3	57min 33sec	8 min 51 sec	2min 41sec	1min 21 sec
DROP #4	49 min 40 sec	9 min 12 sec	2 min 22 sec	1min 22sec
DROP #5	50 min 51 sec	9min 00 sec	2 min 49sec	1 min 16 sec
DROP #6	66min 12 sec	8 min 58 sec	2 min 34 sec	1 min 14sec
DROP #7	60 min 31 sec	8 min 45sec	2min 36 sec	1 min 19 sec
DROP #8	59 min 18 sec	8 min 53 sec	2 min 51 sec	1 min 28 sec
DROP #9	56 min 21 sec	8min 49 sec	2min 44 sec	1 min 22 sec
DROP #10	52min 18 sec	8min 36 sec	2 min 33sec	1 min 20sec

SW 2/26/07

Recorded by: Sande Watts

Date 2/26/07

Verified by:

Date

Project No. ____________

Book No. ____________ TITLE ____________

3/2/07

EXPERIMENTAL PROCEEDURE FOR DETERMINING FLOW RATE OF ALKALINE BRINE, NEUTRAL BRINE AND Ca-Cl BRINE IS THE SAME AS FOR DI WATER WITH ONE CHANGE. THE ALLOY 22 BLOCK MUST BE ULTRASONICALLY CLEANED WITH DI WATER AFTER EACH DROP TO REMOVE RESIDUE. AS A RESULT, ENOUGH TIME MUST BE ALLOWED TO ELAPSE BETWEEN EACH CLEANING AND SUBSEQUENT DROP FOR TEMPERATURE STABILIZATION

3/2/07 SW

Recorded by:	Date	Verified by:	Date
Sandie Watts	3/2/07		

TITLE ____________________ Book No. ____________

SUMMARY OF BRINES AT 95 C

	CaCl Brine	Neutral Brine	Alkaline Brine
Drop#1	1min 32 sec	1min 15 sec	1min Ø1 sec
Drop#2	1min 38 sec	1min 22 sec	Ømin 59 sec
Drop#3	1min 45 sec	1min 28 sec	1min Ø8 sec
Drop#4	1min 29 sec	1min 19 sec	1min Ø3 sec
Drop#5	1min 44 sec	1min 21 sec	Ømin 53 sec
Drop#6	1min 38 sec	1min 26 sec	1min Ø5 sec
Drop#7	1min 3Ø sec	1min 29 sec	1min 1Ø sec
Drop#8	1min 41 sec	1min 2Ø sec	1min Ø3 sec
Drop#9	1min 33 sec	1min 23 sec	Ømin 58 sec
Drop#10	1min 3Ø sec	1min 22 sec	1min Ø sec

3/7/07 SW

Recorded by: Sandi Watt | Date 3/6/07 | Verified by: | Date

Project No.

Book No. TITLE

Summary of Brines at 75 C

	Ca-Cl Brine	Neutral Brine	Alkaline Brine
Drop#1	2min 45sec	2min 50sec	3min 15sec
Drop#2	2min 54sec	2min 56sec	2min 58sec
Drop#3	3min 06 sec	2min 51 sec	3min 09 sec
Drop#4	2min 41sec	3min 10 sec	3min 21 sec
Drop#5	2min 30 sec	3min 2 sec	3min 26 sec
Drop#6	2min 56 sec	2min 42 sec	3min 30 sec
Drop#7	2min 44 sec	2min 39 sec	3min 17 sec
Drop#8	2min 51 sec	2min 49 sec	3 min 22 sec
Drop#9	2 min 55 sec	2 min 56 sec	3min 25 sec
Drop# 10	3min 01 sec	3min 03 sec	3min 19 sec

3/12/07 SW

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[signature]	3/12/07		

TITLE

Summary of Brines at 50c

	Ca-Cl Brine	Neutral Brine	Alkaline Brine
Drop #1	9min 10 sec	7min 31sec	9min 07 sec
Drop #2	7min 58 sec	8min 03 sec	9 min 21 sec
Drop #3	8min 41sec	7min 53 sec	8min 43 sec
Drop #4	7min 14 sec	4 min 18 sec	
Drop #5	7min 20 sec		
Drop #6	7 min 03 sec		
Drop #7	6min 48 sec		
Drop #8	6min 36 sec		
Drop #9	6min 40 sec		
Drop #10	6min 29 sec		

3/23/07 SW

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Project No. ____________

Book No. ____________ TITLE ______________________

Summary of Brines at 25C

	Ca-Cl Brine	Neutral Brine	Alkaline Brine
Drop#1			
Drop#2			
Drop#3			
Drop#4			
Drop#5			
Drop#6			
Drop#7			
Drop#8			
Drop#9			
Drop#10			

NO DATA ENTERED 3/23/07 SW

3/23/07

Changes made to experimental procedure, set-up, and data desired. Need to restart.

Recorded by: Sandra Waters

Date 3/27/07 26 3/26/07 SW

Verified by:

Date

3/27/07

Two temperature : 25c and 95c (Original experimental plan)

Modified to the following -

Drops at 4 temperatures : 25c, 50c, 75c, 95c

- This experiment will show the amount of time required for complete (visible) evaporation of a single, 15 µl drop of DI water on a heated block of Alloy 22 at four different temperatures in a controlled environment.

Equipment -
- Alloy 22 block
- (2) chromalox heating elements
- omega thermocouple
- watlow 93 controller box
- Oakton infrapro infrared thermometer (AN# 010864) (SN# 2332580201)
- Norlake scientific environmental chamber
- plexiglass sheild

Supplies -
- Eppendorf reference pipetter
- disposable pipette tips
- stopwatch
- hard rubber stoppers
- electrical tape

Reagents -
- DI water

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Book No. ____________ TITLE ____________

3/27/07

Set up -

Alloy 22 block was throughly cleaned in DI water and dried with compressed air.

Block was placed on (4) hard rubber stoppers to elevate it from work surface

Heating elements and thermocouple were placed in holes predrilled in block and connected to controller box

Black electrical tape was placed along sides of block for temperature measurements

two-sided plexiglass cover was placed over block set-up to minimize air flow

Experimental procedure -

Using eppendorf pipetter and clean pipette tip, 15 μl of DI water is dropped as a single drop onto the surface of the alloy 22 block. Simultaneously the stop watch is started to record the time. The drop is continuously observed until it has visibly disappeared and at that point the stopwatch is stopped. The elapsed time is recorded on the chart on p. 17 of this notebook. Using the infrared thermometer, the surface temperature of the block is then taken and recorded under the time entry. The temprature is placed in paranthesis for easier visibility.

this procedure is repeated for each drop

99c block → 95c surface
77c block → 75c surface
51c block → 50c surface
25c block → 25c surface

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[signature]	3/27/07		

DI water drop summary - 15 µl

	95c ~~25c~~	75c ~~50c~~	50c ~~75c~~	25 ~~85c~~
Drop # 1	56" (96c)	2'08" (75c)	7'04" SW 3/27/07 (~~55c~~)(50c)	
Drop # 2	55" (96c)	2'09" (75c)	7'02" SW 3/27/07 (~~55c~~) (50c)	
Drop # 3	56" (95c)	2'10" (75c)	7'06" (50c)	
Drop # 4	55" (96c)	2'13" (74c)	7'11" (49c)	
Drop # 5	57" (96c)	2'09" (75c)	7'17" (49c)	
Drop # 6	55" (97c)	2'11" (75c)	7'11" (50c)	
Drop # 7	53" (97c)	2'12" (75c)	7'13" (50c)	
Drop # 8	56" (96c)	2'11" (76c)	7'08" (51c)	
Drop # 9	56" (95c)	2'14" (75c)	7'10" (50c)	
Drop # 10	57" (95c)	2'13" (74c)	7'14" (50c)	

No Data entered - drop size modified 4/3/07 SW

Environmental chamber conditions - 75°F, 69% humidity (START)

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Book No. ________ TITLE ________

4/2/07

Pavan Shukla has requested a change in 'drop size' from 15 µl to 30 µl.

4/3/07

Restart experiment (initial SN 831/15) using 30 µl drop of DI water

Equiptment, Supplies, reagents identical

Addition to set up / procedure -
Prior to placing drop on block, the surface temperature is verified using infrared thermometer and the following guideline:

Controller box setting	Surface reading
99c	95c
77c	75c
51c	50c
25c	25c

Also, conditions in the environmental chamber are noted at both beginning and end of experimental procedure

Recorded by: Sandi Watt Date 4/3/07 Verified by: Date

TITLE ____________ Book No. ____________

4/4/07 DI water drop summary - 30 µl drop

	95c	75c	50c	25c
Drop#1	1'37" (95c)	3'40" (75c)	11'18" (50c)	2:18'49" (25c)
Drop#2	1'34" (95c)	3'42" (74c)	11'20" (50c)	2:04'33" (25c)
Drop#3	1'35" (94c)	3'44" (74c)	11'18" (50c)	1:59'42" (25c)
Drop#4	1'34" (95c)	3'43" (75c)	11'21" (49c)	2:12'09" (25c)
Drop#5	1'36" (95c)	3'44" (75c)	11'24" (49c)	~~2:10'18"~~ 4/23/07 SW 2:10'18" (25c)
Drop#6	1'35" (95c)	3'41" (75c)	11'20" (50c)	N/A
Drop#7	1'34" (95c)	3'43" (76c)	11'19" (50c)	N/A
Drop#8	1'34" (94c)	3'40" (76c)	11'18" (51c)	N/A
Drop#9	1'36" (94c)	3'43" (75c)	11'21" (50c)	N/A
Drop#10	1'35" (95c)	3'41" (75c)	11'23" (50c)	N/A

Beginning conditions - 74 F air temp, 70% humidity
Ending conditions - 76 F air temp 69% humidity

Recorded by: Sarah Watt Date 4/23/07 Verified by Date

 Project No. ____________

Book No. ____________ TITLE ______________________________

The experiment on the previous page is repeated using three (3) solutions

- Alkaline brine (SN 768/14)
- Ca·Cl brine (SN 768/14)
- Neutral brine (SN 768/14)

Alloy 22 block is rinsed well with DI water and dried between each drop to remove residuals.

June 18, 2007 NH

Recorded by:	Date	Verified by:	Date
Nancy Martin	6/17/07		

TITLE ______________________________ Book No. ____________

Alkaline Brine drop summary - 30µl size - Smooth surface

	95c	75c	50c	25c
Drop#1	1'32" (95c)	3'41" (75c)	12'18" (51c)	2:28'19" (25c)
Drop#2	1'30" (95c)	3"42" (75c)	12'19" (50c)	2:30'07" (25c)
Drop#3	1'32" (94c)	3'45" (75c)	12"27" (50c)	2:08'56" (25c)
Drop#4	1'29" (95c)	3'42" (74c)	12'22" (50c)	2:03'43" (25c)
Drop#5	1'33" (96c)	3'43" (75c)	12'28" (49c)	2:17"33" (25c)
Drop#6	1'32" (96c)	3'40" (76c)	12'20" (50c)	N/A
Drop#7	1'33" (95c)	3'42" (76c)	12'30" (50c)	N/A
Drop#8	1'30" (95c)	3'41" (75c)	12'25" (50c)	N/A
Drop#9	1'31" (95c)	3'41" (76c)	12'27" (50c)	N/A
Drop#10	1'32" (95c)	3'42" (75c)	12'21" (50c)	N/A

initial 76F/70h
end 75F/67h

Recorded by:	Date	Verified by:	Date
Nancy Martin	6/7/07		

 Project No. ____________

Book No. ____________ TITLE ____________

Ca·Cl Brine drop summary - 3Ø μl size - smooth surface

	95c	75c	5Øc	25c
Drop#1	1'31" (95c)	3'37" (75c)	13'15" (5Øc)	2:16'16" (25c)
Drop#2	1'31" (95c)	3'37" (75c)	13'22" (5Øc)	2:23'31" (25c)
Drop#3	1'32" (96c)	3'36" (75c)	12'57" (5Øc)	2:04'29" (25c)
Drop#4	1'29" (95c)	3'37" (75c)	13'11" (5Øc)	2:27'4Ø" (25c)
Drop#5	1'33" (95c)	3'34" (76c)	13'25" (5Øc)	2:19'2Ø" (25c)
Drop#6	1'32" (95c)	3'35" (75c)	13'29" (5Øc)	N/A
Drop#7	1'31" (95c)	3'38" (75c)	13'40" (5Øc)	N/A
Drop#8	1'32" (94c)	3'37" (75c)	13'36" (5Øc)	N/A
Drop#9	1'3Ø" (95c)	3'38" (74c)	13'19" (5Øc)	N/A
Drop#1Ø	1'33" (95c)	3'36" (75c)	13'29" (5Øc)	N/A

Recorded by: Nancy Marlin

Date 6/7/07

Verified by:

Date

Project No. ______

TITLE ______ Book No. ______

Neutral Brine drop summary - 3∅ µl size - smooth surface

	95c	75c	50c	25c
Drop#1	1'35" (95c)	3'26" (75c)	12'56" (5∅c)	2:31'∅8" (25c)
Drop#2	1'33" (95c)	3'28" (75c)	12'41" (51c)	2:26'39" (25c)
Drop#3	1'33" (95c)	3'30" (75c)	12'39" (51c)	2:38'12" (25c)
Drop#4	1'34" (95c)	3'32" (74c)	12'52" (5∅c)	2:1∅'19" (25c)
Drop#5	1'32" (96c)	3'31" (75c)	12'58" (5∅c)	2:∅3'56" (26c)
Drop#6	1'33" (95c)	3'29" (75c)	12'5∅" (5∅c)	N/A
Drop#7	1'34" (95c)	3'28" (76c)	12'44" (5∅c)	N/A
Drop#8	1'33" (95c)	3'26" (75c)	12'49" (5∅c)	N/A
Drop#9	1'31" (95c)	3'27" (75c)	12'57" (49c)	N/A
Drop#10	1'32" (95c)	3'28" (74c)	13'02" (5∅c)	N/A

Recorded by: Nancy Mauls [signature] Date 6/7/07 Verified by: Date

Project No. ______

Book No. ______ TITLE ______

June 7, 2007

NH

Recorded by: Nancy Ualri [signature]	Date 6/7/07	Verified by:	Date

TITLE ______________________________ Book No. ____________

June 7, 2007

NH

Recorded by:	Date	Verified by:	Date
Nancy Martin	6/7/07		

Project No. ____________

Book No. ____________ TITLE ____________

DI water drop summary - 30 µl - cracked surface

	95c	75c	50c	25c
Drop #1	1'30" (95c)	3'40" (75c)	11'40" (50c)	1:58'34" (25c)
Drop #2	1'32" (95c)	3'44" (75c)	11'36" (50c)	2:26'17" (25c)
Drop #3	1'32" (95c)	3'41" (74c)	11'42" (50c)	2:15'28" (25c)
Drop #4	1'31" (95c)	3'46" (74c)	11'49" (50c)	1:59'53" (25c)
Drop #5	1'33" (95c)	3'46" (75c)	11'32" (51c)	2:10'41" (25c)
Drop #6	1'32" (95c)	3'45" (75c)	11'51" (50c)	N/A
Drop #7	1'31" (95c)	3'47" (75c)	11'43" (49c)	N/A
Drop #8	1'30" (96c)	3'44" (75c)	11'40" (50c)	N/A
Drop #9	1'33" (95c)	3'41" (75c)	11'28" (50c)	N/A
Drop #10	1'32" (95c)	3'43" (74c)	11'39" (50c)	N/A

Recorded by: Nancy [signature] Date 6/7/07 Verified by: Date

Project No. ____________

TITLE ____________ Book No. ____________

Alkaline Brine drop summary - 300 µl - cracked surface

	95c	75c	50c	25c
Drop#1	1'34" (95c)	3'38" (95c)	13'19" (50c)	2:10'09" (25c)
Drop#2	1'35" (94c)	3'40" (75c)	13'12" (50c)	1:52'37" (25c)
Drop#3	1'33" (95c)	3'40" (75c)	13'26" (50c)	2:17'40" (25c)
Drop#4	1'33" (95c)	3'41" (75c)	13'20" (50c)	2:23'31" (25c)
Drop#5	1'31" (95c)	3'37" (76c)	13'31" (50c)	2:18'43" (25c)
Drop#6	1'32" (96c)	3'36" (75c)	13'39" (50c)	N/A
Drop#7	1'33" (95c)	3'39" (75c)	13'28" (50c)	N/A
Drop#8	1'34" (95c)	3'38" (75c)	13'40" (50c)	N/A
Drop#9	1'32" (95c)	3'42" (74c)	13'35" (51c)	N/A
Drop#10	1'33" 95c	3'41" (75c)	13'29" (50c)	N/A

Recorded by: Nancy Martin   Date 6/7/07   Verified by: Date

 Project No. ________

Book No. ________ TITLE ________

Ca-Cl Brine drop summary - 30μl - cracked surface

	95 c	75 c	50c	25c
Drop#1	1'32" (95c)	3'44" (75c)	13'09" (51c)	1:55'39" (25c)
Drop#2	1'33" (95c)	3'45" (75c)	13'11" (51c)	2:12'43" (25c)
Drop#3	1'31" (94c)	3'37" (75c)	13'26" (50c)	2:19'18" (25c)
Drop#4	1'33" (95c)	3'41" (75c)	13'22" (50c)	1:56'32" (25c)
Drop#5	1'34" (95c)	3'49" (75c)	13'31" (50c)	2:06'29" (25c)
Drop#6	1'34" (95c)	3'48" (75c)	13'29" (50c)	N/A
Drop#7	1'31' (95c)	3'47" (75c)	13'20" (50c)	N/A
Drop#8	1'32" (95c)	(3'50")* (74c)	13'19" (50c)	N/A
Drop#9	1'31" (96c)	(3'53")* (74c)	13'23" (50c)	N/A
Drop#10	1'34" (94c)	(3'48")* (75 c)	13'33" (50c)	N/A

* inadvertantly placed paranthesis

Recorded by: Nancy Martin

Date 6/7/07

Verified by:

Date

TITLE

Project No.

Book No.

Neutral Brine drop summary - 30 µl - cracked surface

	95c	75c	50c	25c
Drop#1	1'35" (95c)	3'34" (75c)	12'29" (51c)	2:20'23" (25c)
Drop#2	1'33' (95c)	3'30" (75c)	12'49" (50c)	1:41'58" (25c)
Drop#3	1'36" (94c)	3'31" (75c)	12'36" (50c)	2:14"19" (25c)
Drop#4	1'35" (95c)	3'35" (75c)	12'41" (49c)	2:09'31" (25c)
Drop#5	1'34" (95c)	3'38" (75c)	12'56" (50c)	2:39'17" (25c)
Drop#6	1'35" (95c)	3'36" (75c)	12'53" (50c)	N/A
Drop#7	1'36" (95c)	3'35" (75c)	12'48" (50c)	N/A
Drop#8	1'33" (96c)	3'34" (75c)	13'04" (50c)	N/A
Drop#9	1'32" (96c)	3'31" (7c5)	12'50" (50c)	N/A
Drop#10	1'34" (95c)	3'36" (75c)	12'42" (50c)	N/A

Recorded by: Nancy Martin

Date 6/7/07

Verified by:

Date

Analysis of Contact Angles on Alloy 22

Initially Assigned To: Nancy Hawkins and Scott Hutzler

Objective: Measure the contact angle of four solutions on a planar, alloy 22 specimen at varying temperatures to determine flow probability.

Sample: Alloy 22 Metal Block

Reagents:
Alkaline Brine (SN 768/14)
CaCl Brine (SN 768/14)
Neutral Brine (SN 768/14)
DI water
Acetone

Equipment:
(2) Chromalox Heating Elements
Omega Thermocouple
Watlow 93 controller box
Oakton InfraPro Infrared Thermometer (model #35629-20; sn: 2332580201)
Standard Goniometer (model #: 250-F1; sn: 607113)
Auto Dispenser (model #: 110-22-100; sn: 607113)
DROPimage Computer Software (Ramé-Hart Instrument Co.)
Ultrasonicator
added 7/25/07 NH — calibrated precision combo calibration device (An: 013057 sn: 12)

Supplies:
Disposable transfer pipette tips
DI water
Kimwipes
Gloves
Electrical tape
Compressed air

Basic Outline of Procedure:

- Clean alloy 22 block according to the procedure in SN 831/4
- Calibrate the standard goniometer with the ball bearing provided by the manufacturer
- Position alloy block on the stage and center/focus it on the live view on the computer
- Place clean pipette tip on the auto dispenser and pull 30µL of liquid into the pipette
- Position the pipette over the alloy surface and adjust the distance from the pipette tip to the surface where desired
- Release the liquid onto the surface
- Measure the contact angle by masking unwanted portions of the drop with the appropriate cursor lines using computer software
- Measure the contact angles using computer software
- 10 measurements are taken for each drop. An average of the left and right contact angle is displayed as well as an average of the combined contact angles.

Recorded by: Nancy Hawkins | Date 6/8/07 | Verified by: | Date

6/8/07

Cleaned alloy 22 block according to SN 831/4.

Preliminary analysis was performed using the standard goniometer and DROPimage software to learn the methods necessary to run a contact angle experiment. The equipment is owned and located in Division 8, building 140.

PRELIMINARY TEST METHODS

Adjustments and Calibration

- Adjust the entire apparatus with the two knobs closest to the bench with the bubble (circle) level on the base.
- Make sure the stage is level at all angles with a hand-held bubble level.
- Put alloy block on stage and lock the stage into the desired vertical position. On the software's' live image the block should be viewed as a silhouette, not looking down on the surface.
    - To create a silhouette, adjust the stage to the desired height.
    - Focus the solid surface using the depth adjustment wheel under the camera
- Put a fresh pipette tip on the auto dispenser and position the tip at the desired distance from the block. *The closer the tip to the block the less likely the drop will detach from the pipette tip.*
    - If the tip is not in focus it will not appear on the screen.
    - When the tip is in focus it wall be square shaped, not tip shaped.
    - To move the pipette tip towards or away from the block surface adjust the screw facing down on the pipette stand.
- Place calibration tool on the stage. Focus the ball bearing and take a picture of the live feed.
    - Click calibration menu → new calibration → sphere: diameter should = 4
    - Aspect ratio should be close to 1 (preliminary results = 1.005).

Setting up a Method

- File → New Experiment Wizard… → CA → Name the Method (preliminary name = CA-20).
    - Droplet = water
    - External = air
    - Solid = steel
    - No volume step (default)

Running a Sample

- Device control → drop volume control
    - volume step = 30μL (*Refers to the volume pulled into the pipette*)
    - syringe volume = 250 (*Refers to the auto dispenser*)
    - full stroke = 240.0 sec (*Refers to how quickly the solution is pulled in and dispensed. The longer the time the less likely for a splash*).
    - syringe level 58% (*Refers to the piston in the auto dispenser*)

- Transfer solution that will be used to measure the contact angle into a clean beaker

- Pull solution into pipette by clicking "input step" on the screen. With a Kim wipe remove any solution residue that may have attached to the pipette tip. *Be gentle to make sure the tip is not loosened.*

- Be sure the tip is focused on the screen. Position the pipette over the block in the prearranged position and release the drop by clicking "output step"

- Once the auto dispenser is finished (listen carefully), carefully pull the pipette tip away from the stage, detaching from the drop.

- Center the drop in the live feed screen.

- Take a picture of the drop.

- Bring cursors into view (green "play button" icon). Be sure the green and red vertical cursors are on either side of the center tick marks. Also, the red horizontal line must be above the tick marks, and the green horizontal line below the tick marks. The spaces between the green and yellow vertical lines, above the red line, and below the green horizontal line are not included in the measurement.

Recorded by:	Date	Verified by:	Date
Nancy Hawkins	6/8/07		

TITLE ______ Book No. ______

Data Generated

10 measurements are reported for each drop (approximately 2 min.)

1. Left contact angle
2. Right contact angle
3. Combined contact angles

At the end of each set, the measurements are averaged.

Reports

After the data is collected on the contact angle screen, select the following menus on the saved pictures (not the live feed) toolbar:

Results → Setup Report (make sure everything is selected) → Show Report → Save Report

Once the initial report is generated any new reports will replace the existing report under the Div. 20 folder on the C drive.

The following programs are shown in the Div. 20 folder:

RTF – raw file (current, complete report)
JPG – images
LOG – minimal results reported

For preliminary purposes DI water contact angles were observed on the alloy block. Two CaCl-Brine drops were also examined to note any changes to contact angle with a different solution.

The data collected from the preliminary analysis is summarized with supporting comments on the next page.

Recorded by: Nancy Martin | Date 6/8/07 | Verified by: | Date

 Project No. ____________

Book No. ____________ TITLE ____________

Drop #	Solution	Average Contact Angle	Comments
1	DI	69.57	One drop was transferred from pipette tip to surface
2	DI	68.68	One drop was transferred from pipette tip to surface
3	DI	78	The liquid was dropped on itself in increments of approximately 5 microliters
4	DI	65	One drop was transferred from pipette tip to surface
5	DI	64.55	One drop was transferred from pipette tip to surface
6	DI	65.34	One drop was transferred from pipette tip to surface
7	DI	72.23	One drop was transferred from pipette tip to surface
8	CaCl Brine	68.75	The liquid was dropped on itself in increments of approximately 5 microliters
9	CaCl Brine	71.5	The liquid was dropped on itself in increments of approximately 5 microliters

An issue regarding which drop is preferred for contact angle measurements arose from the preliminary analysis. The two drops in question is the one continuous drop transferred directly from the pipette tip to the block surface, and the drop that dripped from the pipette tip to the block surface in smaller increments. Even so, both drops contained the same amount of solution (30μL) in the end.

6/12/07

Cleaned alloy 22 block according to SN 831/4.

Analysis was performed to compare the contact angles of five drops that were continuous from the pipette tip to the alloy block, and five drops that dripped to form one ~~6~~ 6/12/07 NH drop on the alloy block. Both kinds of drops contained 30μL. The contact angle comparisons can be found on the next page.

Recorded by: Nancy Martin (signature) | Date 6/8/07 | Verified by: | Date

TITLE ______________________ Book No. ___________

Drop Comparisons
6/12/2007

Dripped Drop

Drop #	Right CA	Left CA	Total CA
1	71.87	70.05	70.96
2	72.95	72.76	72.85
3	71.63	72.21	71.92
4	60.02	57.58	58.80
5	54.57	65.30	59.94
Average	66.208	67.58	66.894

Continuous Drop

Drop #	Right CA	Left CA	Total CA
1	69.66	68.2	68.93
2	79.63	77.75	78.69
3	72.17	73.76	72.97
4	71.97	71.43	71.7
5	64.42	63.88	64.15
Average	71.57	71.004	71.288

Out of the total contact angle measurements of the ten drops, there is a 4.394 unit difference between the continuous drop and the dripped drop.

6/20/07

A calibration procedure based on the method provided by Ramé-Hart Instrument Co. was written for approval as a Technical Operating Procedure. The following pages explain the purpose of using a goniometer, the responsibilities involved in the calibration methods, the procedure used to calibrate the goniometer, and the records where verification data can be found. In addition to the written procedures, Figure 1 and Figure 2 are examples of how to properly position the calibration ball and cursors within the "live image" screen.

Recorded by: Nancy Martin Date 6/12/07 Verified by: Date

PROCEDURE FOR VERIFICATION OF THE PERFORMANCE OF A STANDARD AUTOMATED GONIOMETER AND ASSOCIATED IMAGE ANALYSIS SOFTWARE

1. PURPOSE AND APPLICABILITY

1.1 The purpose of this procedure is to provide a method for the verification of the performance of a standard automated goniometer and the associated image analysis software. This procedure establishes controls required by Quality Assurance Manual (QAM), Section 12, "Control of Measuring and Test Equipment."

1.2 Goniometers are used to measure the contact angle between a liquid, or a vapor, and a solid surface. Surface tension can also be measured to predict the spreading of the liquid or vapor over the solid surface. However, reference is made in this document only to the procedures applicable for contact angle measurements between a liquid and a solid surface. Understanding the contact angle will provide information on the probability of flow of the liquid through the material

2. RESPONSIBILITIES

2.1 Principal investigators are responsible for the implementation of this procedure.

2.2 Personnel performing contact angle tests are responsible for complying with the requirements of this procedure.

3. PROCEDURE

3.1 Contact angle measurements are digitally collected with the standard automated goniometer using the DROPimage software included with the system. Calibration is attained using a certified precision combo calibration device provided by the manufacturer.

3.2 The standard automated goniometer shall be calibrated periodically as specified in QAP-019, "Control of Measuring and Test Equipment". The initial calibration interval shall be one month, but shall be adjusted as needed when the instrument is first set up, moved, when new components are added to the system, and/or the setup has significantly changed.

3.3 Before calibrating the equipment, ensure the live image is visible on the PC screen and the sample stage is level. Using compressed air, remove any dust particles that have attached to the calibration ball.

3.4 To calibrate the instrument, use the sphere calibration tool (a calibrated steel ball bearing secured to a glass slide) on the precision combo calibration device. Place the calibration ball assembly vertically on the stage so the steel ball is imaged through the glass and positioned in the middle of the live image screen. Use the depth adjustment wheel under the camera to focus the ball as shown in Figure 1.

Recorded by: Nancy Martin	Date 6/20/07	Verified by:	Date

3.5 Select the "new sphere calibration" option on the calibration drop down menu to attain a complete profile of the sphere. Enter the diameter of the ball (4mm) and the number of runs (10) required for accurate calibration results. On the live image screen, position the vertical green line through the center of the ball, and the horizontal line through the area below the ball to center the image as shown in Figure 2.

3.6 Once all of the parameters are entered and the cursors are properly aligned, begin the calibration sequence. The calibration values will include the vertical (x) pixel, horizontal (y) pixel and aspect ratio (y/x) obtained from 2nd degree polymonials fitted to the four extreme regions (upper, lower, left, right). Because the video camera has "square pixels", the aspect ratio will be close to 1.0, but will often differ slightly because of small inaccuracies in the hardware. Once the measured values are generated the values are stored in a DROP.CAL file.

3.7 In addition to the required periodic calibration process, the calibration should be checked before each use to verify precision. Select "check calibration" on the view drop down menu. Enter the ball diameter (4mm) and position the cursors as shown in Figure 2. A dialog box will appear containing the stored values from the last calibration, the currently tested values and the percent deviation. If the deviation is ± 0.2% the equipment should be recalibrated.

3.8 The procedure outlined in Sections 3.4-3.7 shall be performed and documented prior to conducting contact angle measurements. When the performance of the standard automated goniometer has been verified, a performance verification label shall be placed on the instrument. An example performance verification label is shown in Figure 3. The label should include the verification date and the date the next scheduled verification should be performed. Performance verification of the standard automated goniometer should be performed once a month or whenever the instrument has been adjusted.

4. RECORDS

Documents containing verification data, including scientific notebooks, shall be controlled as QA records in accordance with QAP-12, "Quality Assurance Records Control".

7/13/07

Cleaned alloy 22 block according to SN 831/4

Calibrated the goniometer with the calibrated precision combo calibration device recieved from Ramé-Hart Instrument Co. on 7/2/07. The calibration values were as follows:

vertical pixel: 9.514 μm
horizontal pixel: 9.510 μm
aspect ratio: 0.9996

Recorded by: Nancy Martin
Date 6/20/07
Verified by:
Date

Book No. ____________ TITLE ________________________________

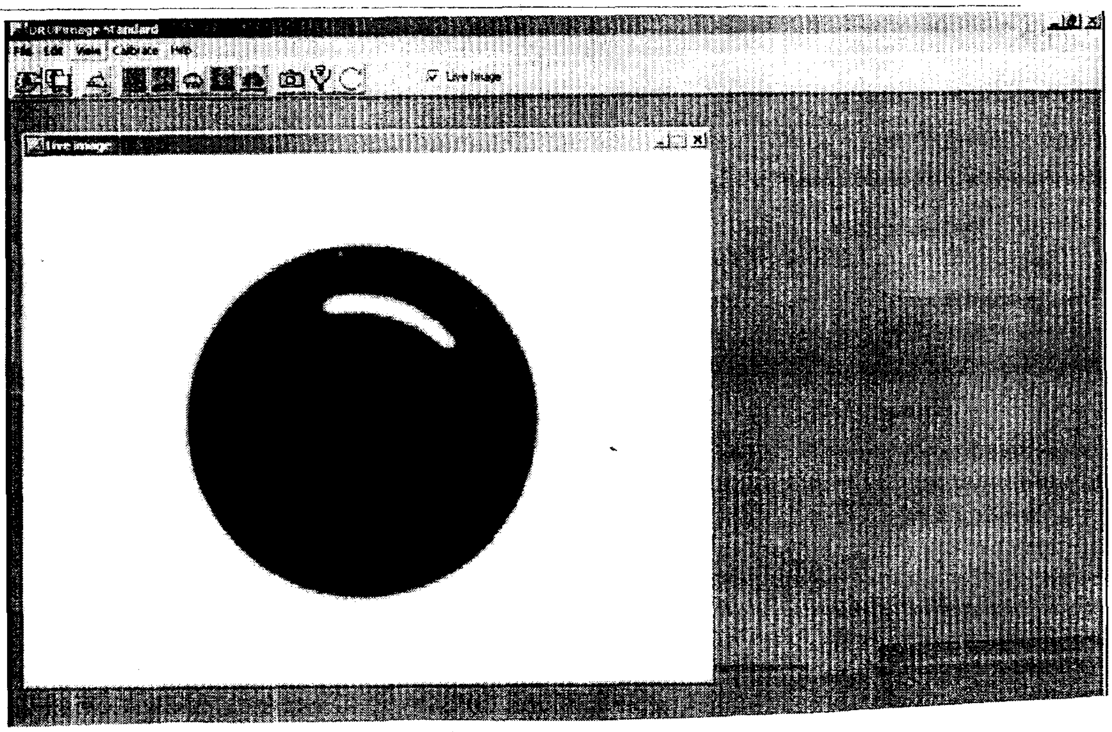


Figure 1: Live image of calibration ball centered and focused on the standard automated goniometer stage on the DROPimage software.

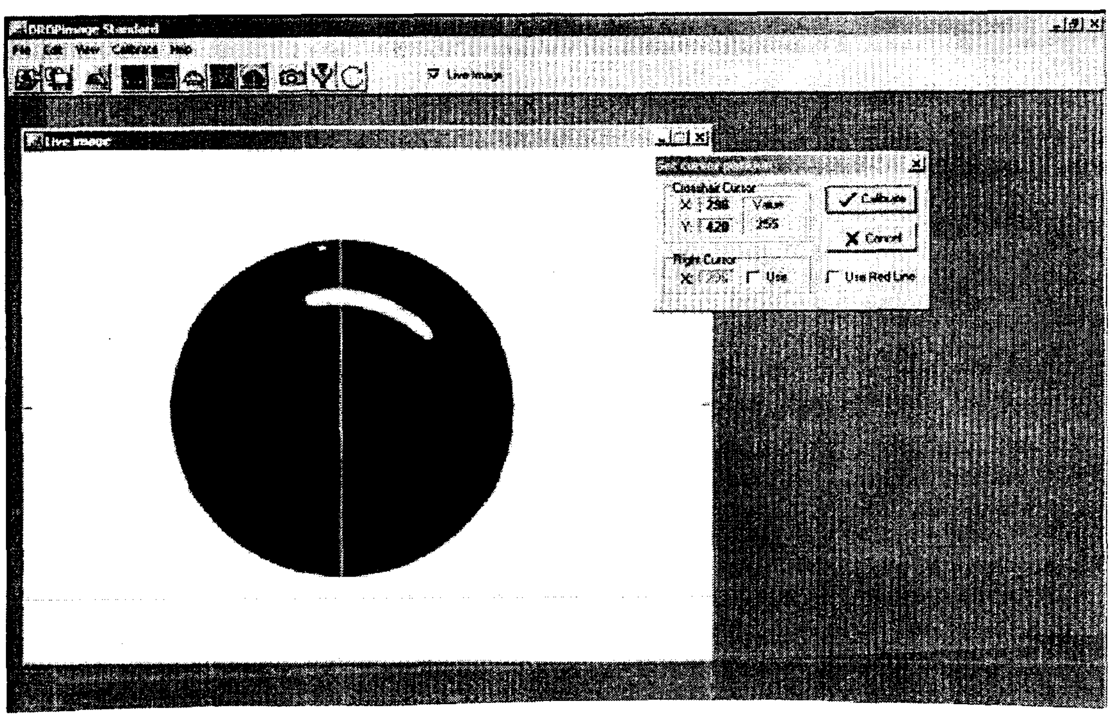


Figure 2: Required cursor alignment to center calibration ball in the live image window. Vertical line shall be through the center of the ball and the horizontal line through the area below the ball.

Recorded by:	Date	Verified by:	Date
Nancy Malin	6/20/07		

7/25/07

Rinsed the lines of the auto dispenser using DI water and the directions below.

1. Make sure the beaker is full of DI water and remove any air bubbles that may have accumulated in the fresh water with a sonicator.
2. Turn on auto dispenser.
3. When turning on the PC it asks you if you want to reset the auto dispenser.
   a. Click yes if you wish to flush out the lines with fresh water.
   ~~b. Click no if you are satisfied with the current setting.~~ NH 8/2/07
4. Place a beaker on the stage to collect water that is emitted from the tip.
5. Make sure the *Drop Volume* option is selected under the *Device Control* drop down menu.
   a. If it is not highlighted select *Options* in the *Edit* drop down menu and select *Drop Volume* in the *Installed Options* box.
6. In the *Drop Volume* dialog box change the *Full Stroke Time* to 10 sec. so the water will move through the lines quicker.
7. Input 4 as the number of rinse cycles.
8. Press *Start* to begin the rinse cycles.
9. Change the *Volume Step* to 150µl.
10. Select *Input Step* to draw 150µl of air into the line to avoid the mixing of sample fluid and DI water. At this time the *Syringe Level* should go up to about 60% ± 5%.
11. Before beginning an experiment change the *Volume Step* back to 30µl and the *Stroke Time* to 240µl.

Calibrated the goniometer with the calibrated precision combo calibration device verified by the calibration laboratory on SwRI campus 7/24/07. The calibration values are as follows:

vertical pixel: 9.121 µm
horizontal pixel: 9.127 µm
aspect ratio: 1.0007

Recorded by: Nancy Marlin Date 7/25/07 Verified by: Date

7/26/07

Checked the calibration on the goniometer that was performed yesterday and only had a 0.1% deviation (an acceptable deviation is less than 1.0%). However, the calibration was ran to record one measurement instead of 10.

In order to get 10 measurements, the instrument had to be recalibrated and produced the following values:

vertical pixel: 9.275 μm
horizontal pixel: 9.283 μm
aspect ratio: 1.0008

Five drops of DI water and CaCl Brine was measured with no complications. The measurements were collected at room temperature.

7/27/07

Checked the calibration on the goniometer and there was a 3.6% deviation from yesterday.
Recalibrated the equipment:

vertical pixel: 9.390 μm
horizontal pixel: 9.399 μm
aspect ratio: 1.0010

Five drops of neutral brine and alkaline brine solution were measured with some complications. A lot of dust particles had accumulated on the alloy block surface distorting the actual shape of the drops. Used compressed air to remove the dust.

Recorded by: Nancy Martin Date 7/27/07 Verified by: Date

TITLE ______ Book No. ______

7/31/07

Checked the calibration that was performed Friday and there was a -3.2% deviation (% deviation may be due to a change in room temperature).
Recalibrated the equipment:
vertical pixel: 9.092 µm
horizontal pixel: 9.101 µm
aspect ratio: 1.0009 µm

Five drops of DI water and CaCl Brine was measured at 25°C. Because the room was 20°C it took about one hour to heat the block. The temperature controller was set to 35°C to maintain a surface temperature between 24.6°C and 25.1°C.

By comparison, the contact angles of the DI water and CaCl Brine decreased by 20° with an increase of surface temperature. Data will be shown at the end of the experiment (SN 831/49).

8/1/07

Checked the calibration performed on 7/31/07 and there was a 0% deviation between the stored values and the new ~~s~~ values meaning a new calibration is not necessary.

Five drops of Alkaline Brine and Neutral Brine solution were measured at 25°C. It took about one hour to heat the block with the temperature controller set at 35°C.

On average the Alkaline Brine solution had an 8° decrease in contact angle with an increase in temperature. Neutral Brine only had a 3° decrease in contact angle with an increase in temperature.

Recorded by:	Date	Verified by:	Date
Nancy Martin	8/1/07		

Project No. ____________

Book No. ____________ TITLE ______________________________

Data is presented at the end of the experiment (SN 831/49).

Attempted to begin 95°C portion of the experiment. Calibration had 2% deviation and was recalibrated:

vertical pixel: 9.275 μm
horizontal pixel: 9.286 μm
aspect ratio: 1.0012

Gradually increased the temperature control box beginning at 75°C, 105°C, 115°C, 125°C. After one hour of heating at an internal temperature of 125°C, the surface temperature was 45°C according to the infrared thermometer. Will resume this portion of the experiment when more time can be devoted to heating.

8/2/07

Calibration was >1% deviation requiring new calibration. The values are as follows:

vertical pixel: 9.397 μm
horizontal pixel: 9.395 μm
aspect ratio: 0.9997.

Insulated the block with glass cloth electrical tape described as having class "B" insulation operating @ 130°C (266°F). However, the block surface temperature would plateau between 40°C and 50°C. Limitations to any insulation used on the block is that it cannot obstruct the camera's view of the surface and it must fit on the goniometer stage.

8/3/07

Checked the calibration values and they did not exceed 1% deviation from the previous values.

Recorded by:	Date	Verified by:	Date
Nancy Martin	8/3/07		

TITLE

Book No.

In order to heat the block to 95°C the block was wrapped in a thermal blanket careful not to obstruct the camera's view of the surface. The temperature controller box was set to 130°C, heating up the block surface very quickly. Between 95-100°C DI water and neutral brine solution was dropped onto the surface. Due to the extreme heat, the liquid burned off immediately not allowing enough time to measure the contact angle.

The next test will be to determine at what temperature the solutions can form a drop and a contact angle be measured before burning off.

8/8/07

New calibration:
vertical pixel: 9.222 μm
horizontal pixel: 9.220 μm
aspect ratio: 0.9998

To determine the maximum temperature in which a contact angle can be measured, I began between 75°C and 80°C. When the drop hit the surface it began to bubble, but I was able to capture an image. However, the light reflected from the bubble caused the software to measure the contact angle through the drop instead of around it.

The same test was performed at 65°C-70°C, but the results were unreliable. The previous brine drops had left a residue on the surface pulling the new drops in that direction. The temperature the drop was exposed to could not be certain at the new location because the block surface is not uniformly heated.

The block surface needs to be cleaned and remove the brine residue in order to get accurate results at

Recorded by:	Date	Verified by:	Date
Nancy Martini	8/8/07		

this temperature.

8/9/07

New calibration
vertical pixel: 9.436 µm
horizontal pixel: 9.433 µm
aspect ratio: 0.9996

Started the drop analysis at 70°C to be sure the Surface temperature was accurate from the previous day. However, bubbles were still present within the drop distorting the contact angle.

Tried to collect data at 50°C. Bubbles were still present. Attempted to wait for bubbles to disappear but at that point the drop was too flat for a measurement to be made.

Since the block temperature is constantly fluctuating as well as the infrared thermometer, the surface temperature will be observed from now on with the thermocouple. It produces accurate reading and does not require a black background as does the infrared thermometer.

Tried to collect data at 52°C as indicated by the thermocouple. Again, there were too many bubbles to make a measurement.

8/13/07

New Calibration
vertical pixel: 9.203 µm
horizontal pixel 9.202 µm
aspect ratio: 0.9999

Collected contact angle measurements of DI water at 50°C. The surface temperature of the block was determined by the thermocouple. When the thermocouple was not in use, it was reverted back into the block to measure the internal temperature and limit the overall temperature increase of the block.

Five drops of DI water was observed. The contact angles were 10°-20° lower than the measurements collected at 25°C and room temperature. Data is presented on p.50.

In addition to collecting contact angle data, the autodispenser was reset and the lines should be rinsed before the next session.

The block was cleaned according to SN 831/4 after it was allowed to cool for approximately one hour.

8/14/07

New calibration:
~~9~~ NM 8/14/07 vertical pixel: 9.178 µm
horizontal pixel: 9.176 µm
aspect ratio: 0.9999

Rinsed the lines of the autodispenser 6 times to remove any trapped air and replace the DI water in the lines with fresh DI water.

Five drops of CaCl and neutral brine solution was observed at 50°C to measure the contact angles. The CaCl contact angles were about the same as the 25°C measurements but are approximately 10° less than the room temperature measurements. The neutral brine solution had contact angles approximately 5° less than the 25°C and room temperature

Recorded by: Nancy Martin | Date 8/14/07 | Verified by: | Date

Book No. ____________ TITLE ____________

measurements.

The block was cleaned according to SN 83114 after it was allowed to cool for approximately 2 hours.

8/15/07

New calibration:
vertical pixel: 9.070
horizontal pixel: 9.070
aspect ratio: 1.0000

Measured the contact angle of five drops of alkaline brine solution at 50°C. Temperature controller box had to be set at 75°C to maintain the surface temperature because the room was cold.

The contact angles of the alkaline brine solution at 50°C were similar to the data collect at 25°C but less than the contact angles collected at room temperature.

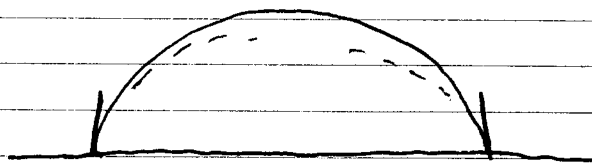
8/20/07

New calibration:
vertical pixel: 9.124 μm
horizontal pixel: 9.121 μm
aspect ratio: 0.9998

Measured the contact angle of five drops of DI water at 60°C.

The higher temperature created bubbles in the drop distorting the overall shape of the contact angle. The figure on the proceeding ~~pro~~ NH 8/20/07 page shows the drop (black) and where the measurements were recorded on the drop. (red).

Recorded by: Nancy Harlin | Date 8/20/07 | Verified by: | Date



The dotted line is usually flush against the curve of the drop. In this case, however, the measurement is taken towards the inside of the drop instead of around the edge.

When comparing the 60°C data with the other measurements, the 60°C contact angles resembled the ~~50°C~~ NH 8/20/07 25°C data rather than the 50°C data. This may be due to the addition of bubbles at the higher temperature.

Five drops of neutral brine and alkaline brine solution were measured at 60°C. No bubbles were generated in the drop like the DI water.

Overall, the contact angles at a higher temperature appears to be less than the measurements taken at the other temperatures for both solutions.

8/21/07

Cleaned the block according to SN 831/4 after it was allowed to cool over night.

New calibration:

vertical pixel: 9.328 μm
horizontal pixel: 9.325 μm
aspect ratio: 0.9997

Measured the contact angle of five drops of CaCl brine solution at 60°C.

Recorded by: Nancy Hanlon | Date 8/21/07 | Verified by: | Date

Overall, the contact angle at 60°C was less than the other observed measurements.

8/22/07

Presented on the proceeding pages is the data collected from the contact angle experiments discussed on pages 30-48 of this notebook.

The standard deviations were determined from the 10 measurements collected for each drop by the software.

CONTACT ANGLE MEASUREMENTS OF DI WATER, CaCl BRINE, NEUTRAL BRINE, AND ALKALINE BRINE AT ROOM TEMPERATURE, 25C, 50C, and 60C

DI Water

Room Temperature

	Theta R	Theta L	Mean
Drop 1	83.89 ± 0.13	81.76 ± 0.14	82.82 ± 0.08
Drop 2	88.78 ± 0.04	85.71 ± 0.02	87.25 ± 0.02
Drop 3	90.23 ± 0.07	89.58 ± 0.22	89.91 ± 0.11
Drop 4	88.77 ± 0.05	86.68 ± 0.04	87.72 ± 0.02
Drop 5	93.11 ± 0.04	86.41 ± 0.02	89.76 ± 0.02
Average	88.956	86.028	87.492

CaCl Brine

Room Temperature

	Theta R	Theta L	Mean
Drop 1	91.63 ± 0.03	86.07 ± 0.02	88.85 ± 0.02
Drop 2	84.51 ± 0.04	88.85 ± 0.05	86.68 ± 0.03
Drop 3	90.08 ± 0.05	91.36 ± 0.03	90.72 ± 0.03
Drop 4	82.47 ± 0.05	80.21 ± 0.14	81.34 ± 0.08
Drop 5	78.85 ± 0.05	86.39 ± 0.04	82.62 ± 0.04
Average	85.508	86.576	86.042

Neutral Brine

Room Temperature

	Theta R	Theta L	Mean
Drop 1	76.93 ± 0.03	78.58 ± 0.02	77.76 ± 0.01
Drop 2	74.48 ± 0.07	73.83 ± 0.01	74.15 ± 0.04
Drop 3	82.98 ± 0.01	80.49 ± 0.02	81.73 ± 0.01
Drop 4	71.64 ± 0.03	80.97 ± 0.02	76.31 ± 0.01
Drop 5	81.77 ± 0.02	88.03 ± 0.02	84.90 ± 0.01
Average	77.56	80.38	78.97

Recorded by: Nancy Maclin

Date: 8/22/07

Alkaline Brine
Room Temperature

	Theta R	Theta L	Mean
Drop 1	78.25 ± 0.01	83.78 ± 0.02	81.02 ± 0.01
Drop 2	79.47 ± 0.02	73.57 ± 0.01	76.52 ± 0.01
Drop 3	77.57 ± 0.01	78.29 ± 0.02	77.93 ± 0.01
Drop 4	75.87 ± 0.01	80.61 ± 0.01	78.24 ± 0.01
Drop 5	79.02 ± 0.02	79.99 ± 0.02	79.51 ± 0.01
Average	78.036	79.248	78.644

DI Water
25C

	Theta R	Theta L	Mean
Drop 1	69.95 ± 0.04	64.54 ± 0.02	67.24 ± 0.03
Drop 2	69.06 ± 0.03	62.61 ± 0.02	65.84 ± 0.01
Drop 3	70.21 ± 0.06	67.82 ± 0.03	69.02 + 0.02
Drop 4	67.92 ± 0.03	73.10 ± 0.02	70.51 + 0.02
Drop 5	66.73 ± 0.03	63.92 ± 0.02	65.32 + 0.01
Average	68.774	66.398	67.586

CaCl Brine
25C

	Theta R	Theta L	Mean
Drop 1	78.42 ± 0.05	74.76 ± 0.19	76.59 ± 0.10
Drop 2	64.25 ± 0.14	68.08 ± 0.05	66.17 ± 0.08
Drop 3	73.30 ± 0.03	74.05 ± 0.03	73.68 ± .0.02
Drop 4	77.48 ± 0.03	80.03 ± 0.03	78.75 ± 0.02
Drop 5	72.93 ± 0.03	65.36 ± 0.03	69.14 ± 0.02
Average	73.276	72.456	72.866

Neutral Brine
25C

	Theta R	Theta L	Mean
Drop 1	69.87 ± 0.02	75.47 ± 0.01	72.67 ± 0.01
Drop 2	74.88 ± 0.02	79.13 ± 0.02	77.00 ± 0.01
Drop 3	83.66 ± 0.02	80.95 ± 0.02	82.31 ± 0.01
Drop 4	80.27 ± 0.02	74.03 ± 0.02	77.15 ± 0.01
Drop 5	69.03 ± 0.06	72.64 ± 0.03	70.83 ± 0.02
Average	75.542	76.444	75.992

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Project No. ____________

Book No. ____________ TITLE ____________

Alkaline Brine
25C

	Theta R	Theta L	Mean
Drop 1	69.72 ± 0.03	80.46 ± 0.20	75.09 ± 0.10
Drop 2	75.60 ± 0.03	69.80 ± 0.03	72.70 ± 0.02
Drop 3	60.63 ± 0.02	62.01 ± 0.01	61.32 ± 0.02
Drop 4	75.82 ± 0.03	68.41 ± 0.01	72.11 ± 0.01
Drop 5	75.35 ± 0.28	72.66 ± 0.28	74.01 ± 0.20
Average	71.424	70.668	71.046

DI Water
50C

	Theta R	Theta L	Mean
Drop 1	54.76 ± 0.03	55.36 ± 0.04	55.06 ± 0.03
Drop 2	54.60 ± 0.01	56.80 ± 0.03	55.70 ± 0.02
Drop 3	67.76 ± 0.07	69.53 ± 0.08	68.64 ± 0.05
Drop 4	58.57 ± 0.04	64.59 ± 0.06	61.58 ± 0.03
Drop 5	59.00 ± 0.04	60.34 ± 0.04	59.67 ± 0.03
Average	58.938	61.324	60.13

CaCl Brine
50C

	Theta R	Theta L	Mean
Drop 1	66.46 ± 0.04	69.93 ± 0.04	68.20 ± 0.03
Drop 2	67.94 ± 0.05	73.59 ± 0.05	70.76 ± 0.04
Drop 3	73.24 ± 0.04	76.50 ± 0.05	74.87 ± 0.04
Drop 4	76.31 ± 0.06	81.44 ± 0.07	78.87 ± 0.04
Drop 5	83.53 ± 0.03	77.23 ± 0.04	80.38 ± 0.02
Average	73.496	75.738	74.616

Neutral Brine
50C

	Theta R	Theta L	Mean
Drop 1	69.63 ± 0.08	74.28 ± 0.08	71.96 ± 0.07
Drop 2	70.58 ± 0.13	72.56 ± 0.04	71.57 ± 0.08
Drop 3	65.60 ± 0.06	72.22 ± 0.07	68.91 ± 0.05
Drop 4	71.97 ± 0.11	76.87 ± 0.03	74.42 ± 0.06
Drop 5	77.37 ± 0.29	74.51 ± 0.03	75.94 ± 0.15
Average	71.03	74.088	72.56

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Alkaline Brine
50C

	Theta R	Theta L	Mean
Drop 1	68.75 ± 0.04	67.84 ± 0.05	68.3 ± 0.04
Drop 2	70.12 ± 0.02	72.08 ± 0.04	71.10 ± 0.02
Drop 3	73.57 ± 0.04	67.14 ± 0.05	70.36 ± 0.04
Drop 4	76.60 ± 0.06	73.46 ± 0.07	75.03 ± 0.04
Drop 5	76.08 ± 0.07	79.02 ± 0.12	77.55 ± 0.07
Average	73.024	71.908	72.468

DI Water
60C

	Theta R	Theta L	Mean
Drop 1	63.64 ± 0.04	68.26 ± 0.03	65.95 ± 0.03
Drop 2	69.41 ± 0.03	68.75 ± 0.06	69.08 ± 0.03
Drop 3	67.33 ± 0.03	64.28 ± 0.06	65.80 ± 0.04
Drop 4	65.71 ± 0.03	63.53 ± 0.03	64.92 ± 0.03
Drop 5	64.01 ± 0.03	67.65 ± 0.07	65.83 ± 0.04
Average	66.02	66.494	66.316

CaCl Brine
60C

	Theta R	Theta L	Mean
Drop 1	65.51 ± 0.02	62.90 ± 0.05	64.20 ± 0.03
Drop 2	67.84 ± 0.05	72.29 ± 0.05	70.07 ± 0.04
Drop 3	63.14 ± 0.03	62.13 ± 0.07	62.63 ± 0.04
Drop 4	59.66 ± 0.05	57.01 ± 0.05	58.33 ± 0.04
Drop 5	65.18 ± 0.05	67.49 ± 0.08	66.34 ± 0.06
Average	64.266	64.364	64.314

Neutral Brine
60C

	Theta R	Theta L	Mean
Drop 1	65.58 ± 0.08	65.49 ± 0.13	65.53 ± 0.07
Drop 2	67.36 ± 0.07	67.94 ± 0.07	67.65 ± 0.04
Drop 3	65.19 ± 0.04	68.52 ± 0.09	66.85 ± 0.05
Drop 4	61.58 ± 0.04	68.73 ± 0.04	65.16 ± 0.03
Drop 5	67.18 ± 0.07	72.24 ± 0.08	69.71 ± 0.05
Average	65.378	68.584	66.98

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Date 8/23/07

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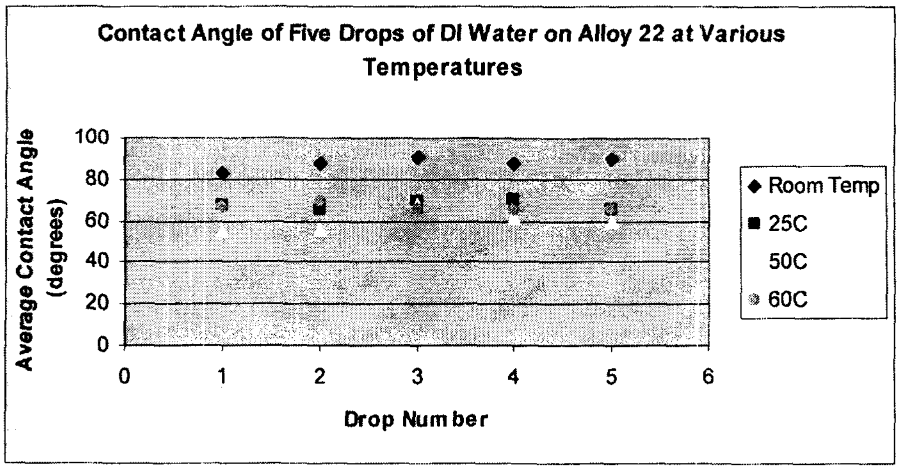
Date

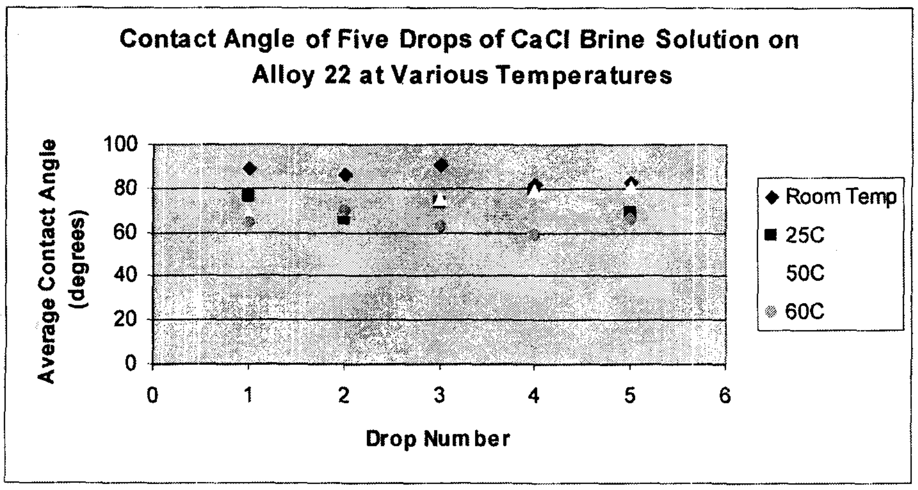
<u>Alkaline Brine</u>

60C

	Theta R	Theta L	Mean
Drop 1	56.68 ± 0.06	64.45 ± 0.06	60.57 ± 0.04
Drop 2	62.03 ± 0.06	61.69 ± 0.05	61.86 ± 0.05
Drop 3	62.45 ± 0.06	68.95 ± 0.06	65.70 ± 0.04
Drop 4	58.20 ± 0.21	58.00 ± 0.05	58.10 ± 0.11
Drop 5	59.92 ± 0.05	64.25 ± 0.04	62.09 ± 0.04
Average	59.856	63.468	61.664

The following graphs are illustrations from the ~~x~~ data represented above. The graphs were generated in excel. nh 8/22/07





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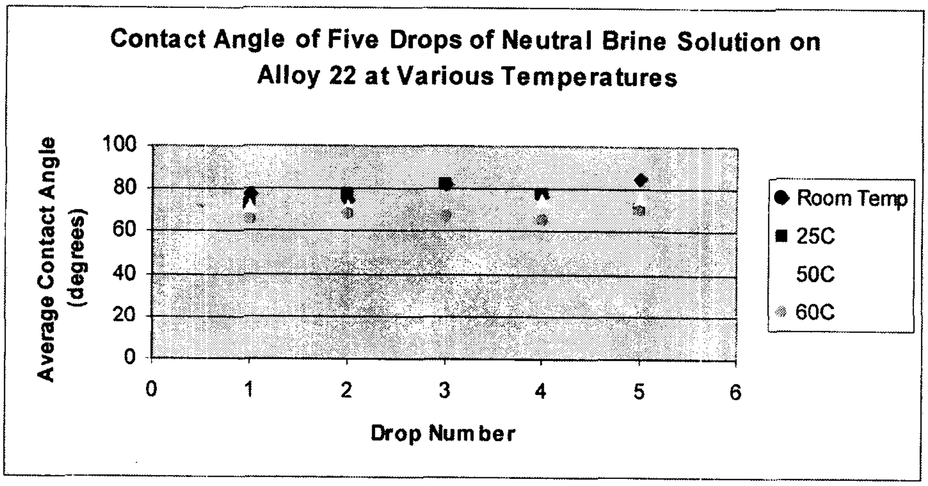

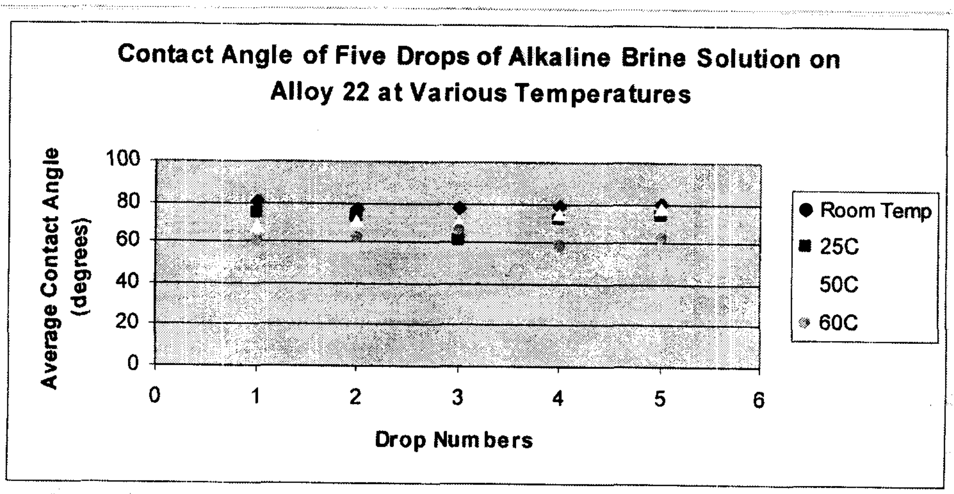

8/28/07

Because the brine solutions cannot be measured at 95°C a solution of simulated concentrated water will be used to observe the contact angles at 60°C, 75°C, and 90°C. The solution was supplied by Brian Derby of Div. 18.

Cleaned the block according to SN 831/4.

New calibration:
vertical pixel: 9.077 μm
horizontal pixel: 9.075 μm
aspect ratio: 0.9998

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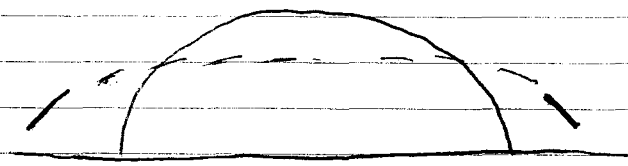
Book No. ____________ TITLE ____________

Tried to measure the contact angle at 60°C with no success. The software would not recognize the angles of the drop. While bubbles would distort the shape of DI and brine solutions, increased salt concentration made the drop unrecognizable as shown below.



Not only is the shape of the drop not recognized, but the point at which the drop comes in contact with the surface is not measured correctly.

One possible explanation for the distorted contact angle is that the reflection of light off of the salt in solution is similiar to that of the atmosphere making the drop unrecognizable.

Cleaned block according to SN 831/4 after it was allowed to cool for approximately 1 hour.

8/29/07

New calibration
vertical pixel: 9.082
horizontal pixel: 9.082
aspect ratio: 1.0001

Tried to measure the contact angle of simulated concentrated water at room temperature. Some drops would spread beyond the observation screen on the PC making me measure each side seperately. Some of the

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TITLE ______ Book No. ______

drops also beaded.

8/30/07

New calibration:
vertical pixel: 9.325
horizontal pixel: 9.321
aspect ratio: 0.9996

Measured the contact angle of simulated concentrated water at 50°C. Some of the drops spread only allowing enough time to measure one side of the drop whereas others beaded up normally.

According to the data below there is not much of a difference between simulated concentrated water contact angles at room temperature vs. 50°C.

Simulated Concentrated Water
Room Temperature

	Theta R	Theta L	Mean
Drop 1	49.50 ± 0.07	49.68 ± 0.07	49.59 ± 0.19
Drop 2	56.97 ± 0.06	59.89 ± 0.03	57.30 ± 0.18
Drop 3	68.06 ± 0.02	69.91 ± 0.02	68.99 ± 0.01
Drop 4	76.63 ± 0.04	83.09 ± 0.03	79.86 ± 0.02
Drop 5	79.00 ± 0.03	74.52 ± 0.03	76.76 ± 0.02
Average	66.032	67.418	66.5

Simulated Concentrated Water
50C

	Theta R	Theta L	Mean
Drop 1	na	37.60 ± 0.11	na
Drop 2	na	44.26 ± 0.07	na
Drop 3	66.32 ± 0.08	75.80 ± 0.09	71.06 ± 0.04
Drop 4	70.99 ± 0.12	68.53 ± 0.19	69.76 ± 0.10
Drop 5	59.25 ± 0.04	58.86 ± 0.07	59.06 ± 0.04
Drop 6	65.25 ± 0.06	60.88 ± 0.07	63.06 ± 0.03
Drop 7	62.78 ± 0.03	67.50 ± 0.05	65.14 ± 0.03
Average	64.92	59.06	65.62

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Project No.

Book No. TITLE

There are no further entries in this notebook.

E. C. Dean
11/4/2008

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