OCT 10 1994 55 Sta. 5	ENGINEERING DATA TRANSMITTAL	Page 1 of <u>1</u> 1. EDT 140735
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2. To: (Receiving Organization) Distribution				3. From: (Originating Organization) Program Support				4. Related EDT No.: N/A			
5. Proj./Prog./Dept./Div.: 95-1 Campaign/Waste Management/PS/AS				6. Cog. Engr.: George L. Miller				7. Purchase Order No.: N/A			
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11. Receiver Remarks: For Release.								12. Major Assm. Dwg. No.: N/A			
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								14. Required Response Date: 08/29/94			

15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	Approval Designator	Reason for Transmittal	Originator Disposition	Receiver Disposition
1	WHC-SD-WM-DP-071	N/A	0	95-1 Campaign Evaporator Boildown Results	Q	2	1	

16. KEY					
Approval Designator (F)		Reason for Transmittal (G)		Disposition (H) & (I)	
E, S, Q, D or N/A <small>(see WHC-CM-3-5, Sec.12.7)</small>		1. Approval 2. Release 3. Information		4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	
1. Approved 2. Approved w/comment 3. Disapproved w/comment		4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged			

17. SIGNATURE/DISTRIBUTION (See Approval Designator for required signatures)													
(G)	(H)	(J) Name (K) Signature (L) Date (M) MSIN				(J) Name (K) Signature (L) Date (M) MSIN				(G)	(H)		
Reason	Disp.											Reason	Disp.
2	1	Cog.Eng. G.L. Miller				8/30/94 TL-00							
2	1	Cog. Mgr. J.G. Kristofzski				8-30-94 TL-00							
2	1	QA J.C. Langford				8/30/94 SI-5A							
		Safety											
		Env.											

18. A. E. Young Signature of EDT Originator Date 8-30-94	19. Authorized Representative Date for Receiving Organization	20. J.G. Kristofzski Cognizant Manager Date 8-30-94 (For J&K)	21. DOE APPROVAL (if required) Ctrl. No. ☐ Approved ☐ Approved w/comments ☐ Disapproved w/comments
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RELEASE AUTHORIZATION

Document Number: WHC-SD-WM-DP-071, REV 0

Document Title: 95-1 Campaign Evaporator Boildown Results

Release Date: 10/10/94

* * * * *

This document was reviewed following the
procedures described in WHC-CM-3-4 and is:

APPROVED FOR PUBLIC RELEASE

* * * * *

WHC Information Release Administration Specialist:

K. M. BROZ

(Signature)

(Date)

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SUPPORTING DOCUMENT		1. Total Pages 30
2. Title 95-1 Campaign Evaporator Boildown Results	3. Number WHC-SD-WM-DP-071	4. Rev No. 0
5. Key Words 95-1, 95-1 Campaign, 242-A, Evaporator, Boildown Results APPROVED FOR PUBLIC RELEASE <i>RMB 10/10/94</i>	6. Author Name: G. L. Miller <i>G. L. Miller</i> Signature Organization/Charge Code 8E480/J1240	
7. Abstract N/A		
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9. Impact Level Q

Westinghouse
Hanford Company

P.O. Box 1970 Richland, WA 99352

WHC-SD-WM-DP-071, REV. 0

222-S ANALYTICAL LABORATORY

Project: 242-A EVAPORATOR BOILDOWN RESULTS

Tank: 241-102-AP
241-107-AP
241-108-AP

Date Printed: August 15, 1994

WHC-SD-WM-DP-071, REV. 0

TABLE OF CONTENTS

Correspondence	1
Request for Special Analysis # 1623	16
Analytical Laboratory Procedure (LT-519-183)	17
Analytical Composite Results from Samples	27

This report consists of pages 1 through 27.

WHC-SD-WM-DP-071, REV. 0

CORRESPONDENCE

**Westinghouse
Hanford Company**

WHC-SD-WM-DP-071, REV. 0

**Internal
Memo**

From: Processing and Chemistry Laboratories
Phone: 373-2182 T6-50
Date: March 13, 1992
Subject: EVAPORATOR BOILDOWN RESULTS

12110-PCL92-019

To: J. F. O'Rourke R1-43

cc: B. Griffin II T6-08
P. G. Haigh R1-43
J. R. Jewett T6-50
C. A. Jurgensmeier T6-50
A. L. Olander R1-51
K. J. Patterson T6-50
Process Aids T6-50
MAB File/LB

- References:
- (1) Internal Memo, J. F. O'Rourke to M. A. Beck, "Viscosity Study," dated March 4, 1992.
 - (2) Internal Memo, J. F. O'Rourke to C. A. Jurgensmeier, "Boildown Study End Point," dated February 19, 1992.
 - (3) "242-A Evaporator Feed Characterization Project Statement Of Work For The 222-S Analytical Laboratories," Office of Sample Management, WHC-SOW-91-0002, Revision 1, September, 1991.
 - (4) Internal Memo, C. A. Jurgensmeier to P. G. Haigh, "242-A Evaporator Studies," dated December 19, 1991.
 - (5) Laboratory Notebook, "222-S Laboratory Support," WHC-N-412-1, J.R. Jewett, dated July 16, 1990, pp. 75-89.
 - (6) Robert C. Weast, Ph.D., Handbook of Chemistry and Physics, 58th ed., CRC Press, Cleveland, 1977-1978, p. D-180.

INTRODUCTION

The Process Chemistry Laboratories were requested to support the 242-A Evaporator restart as part of the overall 222-S laboratory effort (References 3 and 4). An initial test plan is described in Reference 4 with working level detail laid out in Reference 5 (pp. 81 and 82). A modification to the test plans was requested and documented in Reference 1.

The net purpose of these studies is to determine the characteristics of double-shell tank materials as they are processed in the evaporator.

The results for the boildown study (which includes pressure and temperature versus %WVR and density of final boildown residue) supporting the 242-A Evaporator restart are reported below.

MIXING AND COMPATIBILITY STUDY

The samples were added to the mixing vessel in the order: TK-103-AP, TK-102-AW, TK-106-AW. The percentage of TK-103-AP is 44.4%, that of TK-102A-AW is 40.4% and that of TK-106 is 15.2%. The samples were mixed together with no apparent changes in color, temperature, clarity, or any visually determinable characteristic. The Differential Scanning Calorimetry (DSC) results indicate that a very minor exotherm exists above 230 °C. As this exotherm is near the limit of detection, spread over a temperature range of 120 °C, and is far above the temperature of interest for Evaporator purposes, it can be disregarded.

BOILDOWN PRESSURE, TEMPERATURE STUDY

The boildown was performed in a vacuum distillation apparatus with an adjustable vacuum limiting manometer and an isolatable collection graduated cylinder. The boildown was conducted over a seven hour period. The evaporation was done at 60 torr (to avoid excessive foaming and bumping of solution) for approximately half of the boildown, the pressure then being reduced to 40 torr when the reduction in solution volume allowed this to be done. Percent Waste Volume Reduction (%WVR) was measured by observing the amount of condensate collected in a graduated cylinder. As the graduated cylinder became full, it was isolated from the rest of the system and the condensate removed. Pressure was set using an electronic manometer with a low pressure limiter set at the desired level. Temperature was measured using a J-type thermocouple. The apparatus was calibrated by observing the pressure versus temperature response of pure water, and comparing the values thus obtained to published values.

WHC-SD-WM-DP-071, REV. 0

Table 1. Boildown Pressure Temperature Data

%WVR	WVR (mL)	Pressure (torr)	Temperature °C	Comments
0%	0mL	80	48.2	solution clear, colored light yellow
		60	41.9	
		40	34.9	severe foaming
10%	20mL	80	48.4	
		60	42.8	
		40	36.2	
20%	42mL	80	47.6	
		60	42.3	
		40	35.7	
30%	60mL	80	47.5	
		60	42.3	
		40	35.4	
40%	80mL	80	47.0	first condensate sampling
		60	42.0	
		40	35.5	
50%	100mL	80	47.7	
		60	42.6	
		40	34.7	
60%	120mL	80	47.9	
		60	42.8	
		40	34.8	
70%	140mL	80	48.3	
		260	42.3	
		40	34.7	
80%	160mL	80	49.4	
		60	43.4	
		40	36.4	
90%	180mL	80	57.8	heavy appearing slush developed
		60	49.4	
		40	41.2	
	185mL	no measurement		solution drying out on walls, boildown stopped

WHC-SD-WM-DP-071, REV. 0

The attachment shows the above data in graphical form. The data graphed as "water at 40, 60, 80 torr" are published value from reference 6 that should be independent of percent boildown. The blank run that was done showed no temperature change as %WVR increased, despite the fact the the thermocouple was not in the solution for high values of %WVR.

The density measurement yielded a density of 1.367 g/mL. This density was taken of a slurry that very roughly approximates 90% WVR. There are three sources of error in the %WVR approximations:

- The amount of liquid in the boiling flask is probably somewhat less than the condensate collected, as a few milliliters often are caught in the condenser or other parts of the boildown apparatus. This would cause an underestimation if the %WVR and a high bias to the density.
- Approximately 5 mL of water was added to the boiling flask, to prevent the slurry from hardening during shutdown.
- While some of the slurry was scraped off the sides of the boiling flask, not all was recovered, which would lead to a "lower" apparent %WVR than was actually the case. This would cause a low bias to the density measurement.

The viscosity study was deleted at the request of the customer (Reference 1).

This completes the current efforts of the Process Chemistry Laboratories on behalf of the Evaporator Start-up. If you have any questions, please feel free to call me at 373-2182.

Mark A. Beck, Scientist
Process Chemistry Laboratories

tth
Attachment (1)

**Westinghouse
Hanford Company**

WHC-SD-WM-DP-071, REV. 0

**Internal
Memo**

From: Analytical Customer Interface
Phone: 372-2537 H4-23
Date: July 22, 1993
Subject: RESULTS OF BOILDOWN FOR 242-A FEED TANK SAMPLES

12560-93-010

To: G. L. Miller T6-06

cc: J. G. Kristofzski T6-06
C. M. Loll R1-51
K. N. Pool H4-23
J. F. Orourke R1-43
JHT File/LB

The results obtained from the boildown, as outlined in the Evaporator Statement of Work, planned for the samples taken from the three feed tanks (101-AP, 107-AP and 108-AP) may be summarized in an internal letter. The Evaporator Program Group has deemed this sufficient documentation for reporting the outcome of the scheduled boildown. Please provide this letter to the Evaporator Program in a timely manner after completion of the boildown process.

If you have any questions, please call R. C. Smith on 372-2537.

J. H. Tillman
Manager

nlj

7

From: Waste Treatment Systems Engineering
Phone: 373-4214 R1-43
Date: July 7, 1994
Subject: 242-A EVAPORATOR CAMPAIGN 2 STUDIES

7CF40-94-012

To: M. A. Beck T6-09

cc: M. D. Guthrie R1-43
J. R. Jewett T6-09
E. Q. Le R1-43
G. L. Miller T6-06
R. J. Nicklas R1-43
B. H. VonBargen R1-43
RJN:EQL File/LB

- References:
- (1) R. C. Smith, "242-A Evaporator Project Analytical Services Statement Of Work," WHC-SOW-93-0006, Revision 1, May 1993.
 - (2) M. A. Beck, "Determination Of Boiling Pressures And Temperatures As A Function Of % WVR (Boildown)," LT-519-183, Revision A-0, June 1994.

The intent of this letter is to provide instructions for the 222-S Process Chemistry Laboratory (PCL) to proceed the 242-A Evaporator Campaign 2 studies. PCL shall conduct the studies on a composite sample which will consist of 35 percent TK-101-AP, 36 percent TK-107-AP and 29 percent TK-108-AP. The activities to be performed by PCL will be divided in three parts: a mixing/compatibility study, a boildown study, and a specific gravity study.

MIXING/COMPATIBILITY STUDY:

Mixing/compatibility study will be a Differential Scanning Calorimetry of the composite sample. The exotherm and endotherm values will be measured from laboratory thermal analysis conducted up to 500 degree Celsius. Upon mixing, observations of chemical reactions such as gas evolution, heat generation, precipitation, dissolution of solids, color change, clarity, and any visually determinable characteristic shall be recorded.

BOILDOWN PRESSURE AND TEMPERATURE STUDY:

The boildown will be performed per LT-519-183, "Determination Of Boiling Pressures And Temperatures As A Function Of WVR (Boildown)". The temperature will be measured at 40, 60, and 80 torr at waste volume reduction (WVR) intervals of 10 percent. During the boildown, observations of chemical reactions and physical changes such as color, foaming, appearance, precipitation, phase separation (solids settle or remain suspended) shall be recorded.

At the time of nucleation, the pressure, temperature, and WVR shall be recorded. PREDICT calculations indicate the 30% volume solids limit will be reached after a WVR of 98.2% has been attained at 40 torr. It is also predicted that the 8 molar caustic limit will be reached after a WVR of 98.9% has been attained at 60 and 80 torr. These WVRs should be used as the end points (final boildown products) for the boildown study.

SPECIFIC GRAVITY STUDY:

A specific gravity study shall be performed on boildown product of 80%, 90%, WVR, and final boildown products at 40, 60, and 80 torr. Visual states (water-like or gel-like) of boildown solution at 80%, 90% WVR, and final products shall be observed and recorded.

All data and observations shall be recorded in controlled laboratory notebooks for future retrieval. If you need further clarification or additional information regarding the 242-A Evaporator Campaign Studies, please contact me at 373-4214.

E. Q. Le, engineer
Waste Treatment System Engineering

jrt

From: Processing and Chemistry Laboratories
Phone: 373-2182 T6-09
Date: July 28, 1994
Subject: EVAPORATOR BOILDOWN RESULTS

8E110-PCL94-058

To: E. Q. Le R1-43

cc: B. H. Von Bargaen R1-43
R. J. Nicklas R1-43
J. R. Jewett T6-50
G. L. Miller T6-06
Process Aids T6-50
MAB File/LB

- References: (1) Internal Memo, E. Q. Le to M. A. Beck, et al.,
"242-A Evaporator Campaign 2 Studies," dated
July 7, 1994.
- (2) R. C. Smith, "242-A Evaporator Project Analytical
Services Statement of Work," WHC-SOW-93-0006, Revision
1, May 1993.
- (3) M. A. Beck, "Determination of Boiling Pressures and
Temperatures as a Function of % WVR (Boildown), "LT-519-
183, Revision A-0, June 1994.
- (4) Beck, M. A., 1990, "222-S Laboratory Support,"
Controlled Laboratory Notebook WHC-N-421-1, pp. 129-142.
- (5) R. C. Weast, Ph.D., Handbook of Chemistry and Physics,
58th ed., CRC Press, Cleveland, 1977-1978, p. D-180.

INTRODUCTION

The Process Chemistry Laboratories were requested to support the 242-A Evaporator restart as part of the overall 222-S laboratory effort (References 1 and 2). The boildown was conducted by following procedure LT-519-183 Rev A-0 (Reference 3), and was recorded in a controlled laboratory notebook (Reference 4).

The net purpose of these studies is to determine the characteristics of double-shell tank materials as they are processed in the evaporator.

The results for the boildown study (which includes pressure and temperature versus %WVR and density of final boildown residue) supporting the 242-A Evaporator restart are reported below.

WHC-SD-WM-DP-071, REV. 0

MIXING AND COMPATIBILITY STUDY

Aliquots from the following tanks were composited: 241-102-AP, 241-107-AP and 241-108-AP. The percentage of 241-101-AP in the aliquot is 36%, that of 241-107-AP is 35% and that of 241-108-AP is 29%. The samples were mixed together with no apparent changes in color, temperature, clarity, or any other visually determinable characteristic. The Differential Scanning Calorimetry (DSC) results indicate that only the endotherm associated with water evaporation/boiling is seen. No exotherms of any kind are evident in the DSC plots.

BOILDOWN PRESSURE, TEMPERATURE STUDY

The boildown was performed in a vacuum distillation apparatus with an adjustable vacuum-limiting manometer and an isolatable collection graduated cylinder. The boildown was conducted over a seven hour period. The evaporation was done at 80 torr (to avoid excessive foaming and bumping of solution). Percent Waste Volume Reduction (%WVR) was measured by observing the amount of condensate collected in a graduated cylinder. As the graduated cylinder became full, it was isolated from the rest of the system and the condensate was removed. Temperature in the boiling liquid was measured using a J-type thermocouple. The indicated values of temperature were converted to actual temperature. The thermocouple was calibrated in reference to a National Institute of Standards and Technology (NIST) traceable thermometer. The temperature readings of both the thermocouple and the standard thermometer were taken every 5 °C between 20 °C and 70 °C. Readings were also taken at 80, 90 and 99 °C (since the pressure in the lab is too low to allow for boiling at 100 °C). Pressure was set using an electronic manometer with a low pressure limiter set at the desired level. The pressure measurement was calibrated by observing the pressure versus temperature response of pure water, and comparing the values thus obtained to the corresponding literature (Reference 5) values for pure water. The blank run that was done showed no temperature change as %WVR increased, despite the fact the thermocouple was not in the solution for high values of %WVR.

DENSITY MEASUREMENT

The density was measured at approximately 25 °C at atmospheric pressure. The liquid was transferred from the boildown flask to a pre-weighed volumetric flask. The liquid was stirred, and every attempt made to ensure that suspended solids were included in the sample. The measurement of the liquid at several different temperatures and pressures, as specified in Reference 1, was not possible since the equipment, and the sample size did not permit such measurement.

RESULTS

The attachment shows the tabulated data (next page) in graphical form. The data graphed as straight lines labeled "42, 60, 79 torr" are published values of pure water at those pressures (from Reference 5) that are independent of percent boildown.

WHC-SD-WM-DP-071, REV. 0

%WVR	Volume Collected (mL)	Indicated Pressure (torr)	Corrected Pressure (torr)	Indicated Temperature (°C)	Corrected Temp (°C)	Visual Observations and Remarks
0	-	745	713	20.2	20.6	Ambient Air prior to hot plate startup
0	0	75	79	47.0	47.2	
0	0	55	60	41.6	41.8	Prone to bumping as pressure is reduced
0	0	35	41	34.4	34.8	This low pressure value is highly variable, Temperature varies ± 0.2 °C
10	12	75	79	47.2	47.4	
10	12	55	60	42.2	42.4	
10	12	36	42	36.0	36.4	
20	24	75	79	47.4	47.6	
20	24	55	60	42.0	42.2	Temp measured after a big bump
20	24	36	42	35.4	35.8	
30	36	75	79	47.0	47.2	
30	36	55	60	42.2	42.4	Condenser temperature about 7 °C during test
30	36	36	42	35.2	35.6	
40	48	75	79	47.2	47.4	
40	48	55	60	42.2	42.4	
40	48	35	41	35.4	35.8	
50	60	75	79	46.8	47	
50	60	55	60	44.7	44.9	Temp value possibly incorrectly read or recorded
50	60	35	41	34.8	35.2	
60	72	75	79	47.0	47.2	No unusual foaming no cloudiness at all
60	72	55	60	41.4	41.6	
60	72	35	41	35.8	36.2	
70	84	75	79	47.4	47.6	@ 92 mL liquid collected large bubble (1-2 cm diameter) foam
70	84	55	60	41.6	41.8	foam volume up to 6 times the volume of liquid (150 mL vs 25 mL)
70	84	35	41	35.2	35.6	no cloudiness
80	96	75	79	47.0	47.2	1st cloudiness at 80 torr and 46.8 °C, light floc type solids
80	96	55	60	41.4	41.6	
80	96	35	41	34.6	35	Specific gravity 1.03 g/mL
90	108	75	79	48.2	48.4	granular specks on flask wall above bubble burst line
90	108	55	60	42.4	42.6	dilute Tang(TM) texture (yellow green color)
90	108	35	41	35.4	35.8	Specific Gravity 1.12 g/mL
	~109					Specific gravity 1.3 g/mL Note that only 1.5 ml liquid remained in the flask

WHC-SD-WM-DP-071, REV. 0

The density measurement yielded a densities of 1.03 g/mL at 80 %WVR, 1.12 at 90 % WVR and 1.3 g/mL at slightly above 90 % WVR.

The %WVR values should be considered approximations with low bias. The amount of liquid in the boiling flask is somewhat less than the condensate collected, as a few milliliters often are caught in the condenser or other parts of the boildown apparatus. This would cause an underestimation if the %WVR and a high bias to the density. Some water is also lost to the vacuum system and is not collected in the receiving flask which would also lead to a "lower" apparent %WVR than is actually the case.

CONCLUSIONS

The composite material is largely water, and has no unusual characteristics. The foaming seen was of very light-walled bubbles, and should not present a problem.

The customer should realize that the values of % WVR are biased low (the actual values of %WVR are higher than those reported). This bias is especially prominent at high values of %WVR.

The amount of solids in solution is quite low compared to the usual amount of solids in Hanford waste tanks.

This completes the current efforts of the Process Chemistry Laboratories on behalf of the second Evaporator campaign. If you have any questions, please feel free to call me at 373-2182.

Mark A. Beck, Advanced Scientist
Process Chemistry Laboratories

adm

E. Q. Le
Page 5
July 28, 1994

8E110-PCL94-058

WHC-SD-WM-DP-071, REV. 0

Attachment to 8E110-PCL94-058
Evaporator Boildown Results
Page 1 OF 1

WHC-SD-WM-DP-071, REV. 0

REQUEST FOR SPECIAL ANALYSIS (RSA)					RSA No. <u>1623</u>		
1. Sample Origin <u>AP-BOIL</u>		2. Date Submitted <u>8/2/93</u>		4. Requester Name <u>George L Miller</u>		3. Charge Code/Work Package <u>NY17</u>	
		3. Submitted By <u>George L Miller</u>		5. Requester Phone/MSIN <u>373-4739 / T6-06</u>		7. Date Required	
8. Customer ID <u>AP-BOIL</u>		<div style="display: flex; justify-content: space-between;"> <div>Collected</div> <div>9. Laboratory ID</div> </div> <div style="display: flex; justify-content: space-between;"> <div>Date</div> <div>Time</div> </div> <u>V17</u>		12. Number of Samples <u>1</u>		13. Volume of Samples <u>125 ml</u>	
				14. Protocols ☐ None ☐ RCRA ☐ NQA-1 ☒ Other ☐ CERCLA			
				15. Sample Type ☐ Solid ☐ Soil ☐ Slurry ☐ Gas ☒ Water ☐ Waste ☐ Solution ☐ Sludge ☐ Oil ☐ Other (specify):			
				16. Storage Requirements ☒ None ☐ Specify: <u>WHC-SD-WM-DP-071, REV. 0</u>			
				17. Process Knowledge/Known Listed Wastes ☐ Unknown ☒ Known (Please attach list) <u>Evaporator Feed</u> ☐ Previously submitted for this Project			
10. Determination		11. Expected Range		18. Survey			
<u>Appearance</u>				<div style="display: flex; justify-content: space-between;"> <div><u>Stenderstock</u></div> <div>Dose Rate</div> </div> <div style="display: flex; justify-content: space-between;"> <div>HPT Signature</div> <div><u><.5</u></div> </div>			
<u>DSC</u>				19. Disposition of Waste ☐ Return to Client/Location ☐ Dispose per 222-S Procedures ☒ Other per TPP: <u>WHC-SD-WM-TPP-048</u>			
<u>Sp. Gravity</u>				20. Additional Information			
<u>PT Curve</u>				<u>This sample is to be generated as a Master composite from three individual tank composites:</u> <u>101-AP-COMP,</u> <u>107-AP-COMP,</u> <u>108-AP-COMP</u>			
<u>30% Boil down</u>				21. Chain of Custody			
<u>- Phase Separation</u>				☐ No ☒ Yes Number: <u>Internal Only</u>			
<u>- Viscosity</u>				22. Assigned Custodial Group (select one)			
<u>- Sp. Gravity</u>				☒ PCL ☐ PDSU ☐ ENV ☐ Process			
<u>Comments</u>				23. Sample Received By: <u>Shandra Cobb</u>			
<u>Date: 0802-93</u>		<u>Time: 1535</u>		24. Laboratory Manager (if required)			
				<u>16</u> Date:			

ANALYTICAL LABORATORIES

Issued By: **J. R. Jewett**

Manager,
Analytical Laboratory

M. A. Beck

Title **DETERMINATION OF BOILING
PRESSURES AND TEMPERATURES AS A
FUNCTION OF %WVR (BOILDOWN)**

Impact Level NA

1.0 SUMMARY

WHC-SD-WM-DP-071, REV. 0

Material of interest undergoes vacuum distillation. The temperature and pressure are measured as a function of Waste Volume Reduction (WVR). Percent WVR is the percentage of the original volume of the material that has been removed. Tests on the distillation residue may be requested.

2.0 APPLICATIONS

This method is used to determine pressure/temperature curves as a function of WVR. Samples of the boildown residue may be taken to determine the specific gravity or viscosity, or other nondestructive tests. This method may be used on aqueous samples. A small amount of entrained organic material presents no problem.

3.0 LIMITATIONS

Customer-Provided Information Needed To Start — Prior to starting the boildown, the customer must provide an endpoint in terms of percent Waste Volume Reduction. Evaporator personnel provide the endpoint from a PREDICT computer calculation. The customer also must provide desired pressure(s) and %WVR(s) for the boildown (past Evaporator work has been done at 40, 60, and 80 torr and every 10% WVR). The customer may request specific gravity and/or viscosity measurements at specified %WVR. The customer is encouraged to minimize the tests on the residue done prior to the endpoint, as this entails breaking open the test apparatus to gain access to the residue, and a complete stoppage of the boildown test itself.

Low concentrations of solids lead to difficulties in determining the endpoint, as the endpoint for such solutions is a few mL in a large flask.

Tests on the residue prior to the endpoint must be nondestructive. Sample must be returned to the boildown flask prior to continuing with the test. This requirement is due to the low volumes of residue, especially at high %WVR.

Release Date 06/28/94	Review Date 06/28/96	Document No. LT-519-183	Rev Mod A-0	Page 1 of 10
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4.0 SAFETY

WHC-SD-WM-DP-071, REV. 0

Samples containing more than 10% organic material in the presence of nitrates should be avoided, and will require extra care (explosion awareness, blast shielding and other measures as required after consultation with Safety personnel). A Job Safety Analysis is required prior to work on such samples. Solutions that show an exotherm at temperatures below 110° C should be assumed to have organic/nitrate mixes and should be treated as such.

High activity samples (greater than 100 mR/hr at the hood face) will require additional shielding. Reducing the amount of sample is not an option in this procedure, if specific gravity or viscosity measurements on the residue are requested. High alpha activity samples require extra care to avoid contamination problems.

5.0 EQUIPMENT

Water, ASTM TYPE II (or better) (MSDS #1848)

6.0 REAGENTS

Controlled laboratory notebook

National Institute of Standards and Technology (NIST), or American Society for Testing and Materials (ASTM) certified, traceable thermometer.

200 mL beaker

Boildown apparatus (see Figure 1)

NOTES:

- Key safety features of the boildown apparatus are:
 - Outlet "Q" must be inside the hood
 - Ballast tank "N" must be a large, thick walled, tape wrapped vessel.
- Because this procedure is rarely run (once a year or so), the apparatus may be in need of reassembly. Portions of this apparatus are sometimes used in other experiments.

Document No.	Rev Mod	Page
LT-519-183	A-0	2 of 10

7.0 PROCEDURE STEPS

WHC-SD-WM-DP-071, REV. 0

7.1 Thermocouple Calibration

- 7.1.1 Place a beaker of ASTM Type II water (with stir bar) on a hot plate/stirrer.
- 7.1.2 Connect the thermocouple to a thermocouple reader of the same type.
- 7.1.3 Place the thermocouple and a certified thermometer in the beaker of room temperature water.
- NOTE: Neither the thermocouple or the certified thermometer should not be touch the sides of the beaker, or each other.
- 7.1.4 Stir the water for about 3 minutes.
- 7.1.5 Measure the temperature.
- 7.1.6 Begin heating the water slowly (about 1° C/ minute).
- 7.1.7 From room temperature to 60° C, measure the temperature of the water every 5° C.
- 7.1.8 From 60° C to boiling, measure the temperature every 10° C.
- 7.1.9 Calculate the correction needed, if any, to convert the thermocouple readings to temperature.

7.2 Calibration — Pressure Gauge and Trial Boildown.

- 7.2.1 Place the verified thermocouple in the boildown apparatus (see Figure 1).
- 7.2.2 Add water (ASTM type II) to the boildown apparatus.
- 7.2.3 Follow the procedure below (Section 7.3, Steps 7.3.2 to 7.3.12) to run a boildown on the water at the customer specified pressures.
- NOTE: Use this trial run to determine if there are any problems with the operation of the apparatus (vacuum leaks, condensers, etc.).
- 7.2.4 Look up the vapor pressure of water at the temperatures measured in the CRC Handbook of Chemistry and Physics.
- 7.2.5 Calculate (using Equation 2) the correction needed to convert the pressure readings to true pressure in the boildown flask.

Document No.

LT-519-183

Rev Mod

A-0

Page

3 of 10

7.3 Boildown

- 7.3.1 Determine that a differential scanning calorimeter/calorimeter test has been run on the boildown material, and that no exotherms are present below 110° C.

WARNING

COMPLETE A JOB SAFETY ANALYSIS IF EXOTHERMS ARE PRESENT BELOW 110° C.

- 7.3.2 Make sure that you know the pressures, temperatures and %WVR for measurement.

NOTE: Figure 1 shows the locations of items referenced in "quotes" in the following Sections.

- 7.3.3 Add a measured (by graduated cylinder) volume of material and a magnetic stir bar to the boildown flask "C".

NOTE: The Appendix shows a Boildown Data Sheet.

- 7.3.4 Record the volume measured in the preceding Step.

7.3.4.1 Record an estimate of the volume based on the scribe marks on the sides of the boildown flask.

7.3.4.2 Record the temperature in the flask as shown on the temperature indicator "T",

- 7.3.5 Set the pressure gauge/controller "P" to 1000 torr.

- 7.3.6 Assemble all apparatus parts.

NOTE: All vents to the atmosphere must be closed, including valve "I," and any other open valves.

- 7.3.7 Begin stirring (stirrer control is on hot plate "B").

NOTE: Stirring needs to be vigorous enough to develop a vortex that extends at least one quarter of the way from the surface.

- 7.3.8 Close collection valve "H" and open reflux valve "G".

- 7.3.9 Start the vacuum pump "O".

- 7.3.10 Slowly lower the pressure using a screwdriver in the set point screw of the pressure controller "P".

NOTE: Always reduce the pressure slowly to avoid sudden boiling (which can manifest itself as violent "bumps").

Document No.

LT-519-183

Rev Mod

A-0

Page

4 of 10

- 7.3.11 Set the pressure to the highest pressure to be used for the boildown.
- 7.3.12 Adjust hot plate "B" and jack "A" as necessary to provide a steady boil.

7.4 Temperature and Pressure Measurement

NOTE: The %WVR equation is shown in the Calculations Section.

- 7.4.1 When the %WVR (as measured on the condensate collection flask) reaches a desired measurement point:

7.4.1.1 Close valve H.

7.4.1.2 Open reflux valve "G".

- 7.4.2 Record the temperature ("T") and pressure ("P") and any visual observations about the behavior of the sample.

NOTE: Observations of foaming, precipitation, and any salient event are key parts of this procedure. Every noteworthy event must be recorded in the notebook.

- 7.4.3 Slowly lower the pressure using the screwdriver in the set point screw of the pressure controller "P" to the next lower boildown pressure.

- 7.4.4 Repeat Steps 7.3.1 through 7.3.12 until the lowest pressure to be measured has been reached.

- 7.4.5 Set pressure controller "P" to the highest measurement setting.

- 7.4.6 Close reflux valve "G" and open collection valve "H".

- 7.4.7 Allow the solution to boil until the next %WVR value to be measured is reached.

7.5 Condensate Collection

- 7.5.1 When the condensate collection vessel "K" becomes full (or close to full), close valves "I" and "H," thereby connecting "K" to the rest of the boildown vessel.

- 7.5.2 Open reflux valve "G," to allow condensate recirculation.

- 7.5.3 Open the collection vessel "K" to the air (move valve "I" to position "J").

- 7.5.4 Remove the collection vessel "K" from the boildown apparatus.

Document No.	Rev Mod	Page
LT-519-183	A-0	5 of 10

PROCEDURE STEPS (Continued)**WHC-SD-WM-DP-071, REV. 0**

- 7.5.5 Remove the water from the collection vessel "K".
- 7.5.6 Reattach the collection vessel "K" to the boildown apparatus.
- 7.5.7 Switch the collection vessel air valve "I" to connect the vacuum to the collection vessel. (Switch it away from position "J".)
- 7.5.8 Open "H" that connects the collection vessel to the rest of the boildown apparatus.
- 7.5.9 Close reflux valve "G," so condensate reaches the collection vessel "K".

7.6 Residue Sampling

- 7.6.1 When the %WVR reaches requested values, a sample of residue should be removed from the residue pot "C".
- 7.6.2 Verify that reflux valve "G" and collection valve "H" are closed.
- 7.6.3 Turn the vacuum pump "O" to OFF.
- 7.6.4 Open vent valve "I" to position "J".
- 7.6.5 Turn heater "B" to OFF.
- 7.6.6 When boiling has stopped, open pot vent valve "F".
- 7.6.7 Turn stirrer "B" to OFF.
- 7.6.8 Disconnect residue pot "C" from the rest of the apparatus by disassembling clamp "E".
- 7.6.9 Lower jack "A," heater/stirrer "B," and residue pot "C".
- 7.6.10 Withdraw sample for testing.
- 7.6.11 Upon completion of the test, return the sample to the residue pot "C".
- 7.6.12 Raise the residue pot "C" to contact the rest of the apparatus.
- 7.6.13 Assemble the clamp "E," ensuring a tight seal between the pot "C" and the rest of the apparatus.
- 7.6.14 Close any open valves, including pot vent valve "F".
- 7.6.15 Restart the boildown by following Steps 7.3.5 through 7.3.12.
- 7.6.16 Repeat Steps 7.4.1 through 7.6.13 as needed until the endpoint is reached.

Document No.

LT-519-183

Rev Mod

A-0

Page

6 of 10

7.6.17 When the end point is reached, do Steps 7.6.1 through 7.6.8.

NOTE: At very high %WVR, the solution will probably become a slurry (a thick suspension of solids in a small amount of water). Make sure that the slurry does not dry out. The test may be stopped prior to the pre-established end point if the slurry is about to dry out.

8.0 CALCULATIONS

$$\text{Actual temperature } (^{\circ}\text{C}) = m * \text{temperature indicated } (^{\circ}\text{C}) + b$$

$$\text{True pressure (torr@T}_{\text{actual}}) = n * (\text{Indicated pressure (torr@T}_{\text{actual}})) + C$$

Where:

n, m, b, c = Regression coefficients from calibrations

$$\% \text{WVR} = \frac{\text{Cumulative VOLUME collected (mL)} * 100}{\text{Original VOLUME of boildown sample (mL)}}$$

9.0 DISCUSSION

Requests for this service usually have their origin with the 242-A Evaporator. This procedure is written with such requests in mind. Other customers may request this service, and have different requirements for visual observation, condensate collection, or specific temperatures and/or pressures. This procedure should accommodate a wide range of requests for boildown services.

10.0 REFERENCES

Weast, R. C., 1989, *CRC Handbook of Chemistry and Physics*, pg D-195, CRC Press, Boca Raton, Florida.

Bechtold, D. B., 1993, *T-101 Boildown*, WHC-N-341-1, Controlled Laboratory Notebook, pg 59-67, Westinghouse Hanford Company, Richland, Washington.

Beck, M. A. to J. F. O'Rourke *Evaporator Boildown Results*, Internal Memo 12110-PCL-92-019, March 13, 1992, Westinghouse Hanford Company, Richland, Washington.

Document No.

LT-519-183

Rev Mod

A-0

Page

7 of 10

Figure 1. Schematic Drawing of Boildown Apparatus

WHC-SD-WM-DP-071, REV. 0

A. Jack	F. Pot vent	K. Receiver
B. Heater/stirrer	G. Reflux valve	L. Coolant in
C. Pot	H. Collection valve	M. Coolant out
D. Thermocouple	I. Receiver vent/vacuum valve	N. Ballast
E. O-ring joint	J. Receiver vent	O. Vacuum Pump

P. Pressure indicator/controller
Q. Vacuum pump exhaust
R. Hood Boundary
T. Temperature indicator

DBB 2-24-93

Document No.

LT-519-183

Rev Mod

A-0

Page

8 of 10

Figure 2. Instructions for adjustment of the seal on valve "H"

WHC-SD-WM-DP-071, REV. 0

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INSTRUCTIONS FOR USE

All valves are adjusted for use before leaving the factory. However if it is required to use them under conditions of high vacuum or high pressure they should be re-adjusted according to the instructions below.

Adjustment Instructions

1 Turn the cap to screw the PTFE valve stem onto the tapered glass seating. The chamfered end of the PTFE valve stem should make a continuous seal which is clearly visible as a white ring (1) Fig 1.

2 Adjust the gland seal (2) Fig 1 as indicated below. When correctly adjusted the PTFE valve stem should appear to wet the inner glass wall of the valve body over a 3-4mm band.

3 Insert a narrow bladed screw driver through the hollow screw in the cap until it engages the smaller inner screw within the valve stem (Fig 2). For taps up to 6mm bore size a screwdriver with a blade width of 3mm (1/8 in) should be used. For larger bores with a blade width of 5mm (7/16 in) is necessary.

4 Tightening this inner screw then increases the pressure on a resilient insert. This pressure is transmitted radially and improves the gland seal against the valve body to give better performance under high vacuum/pressure conditions. Loosening this screw will achieve the reverse effect.

5 Always slacken off the seal before removing the valve stem from the barrel.

The following special points should be noted:

1 Avoid over tightening when screwing down the valve stem or when adjusting the gland seal. Excessive pressure can damage the valve.

2 Cleanliness of PTFE valve stem and glass body is essential for maximum performance. A surfactant detergent applied in accordance with the manufacturer's instruction is recommended.

3 Valves incorporated in high vacuum systems may be degassed *in situ* by warming with a hot air gun or by gentle flaming. Care should be taken to avoid overheating the cap as it is made of polypropylene which softens at temperatures above 130°C.

4 Take care when using a spark coil for leak testing. Continual use of this equipment in the vicinity of the stopcock can cause damage to the PTFE valve stem.

In following a policy of continuous improvement we reserve the right to change specifications without notice.

Fig 1

Fig 2

MODE D'EMPLOI

Tous les robinets sont réglés pour une utilisation normale lorsqu'ils quittent nos ateliers. Si néanmoins on doit les utiliser sous vide poussé ou à pression élevée leur réglage devra être modifié selon les instructions suivantes.

Réglage

1 Tourner le capuchon afin de visser le pointeau jusqu'à contact avec le siège conique du corps en verre. L'extrémité chanfreinée du pointeau doit faire un joint continu qui apparaît nettement comme un anneau blanc.

2 Régler le joint du pointeau en suivant les instructions des paragraphes 3, 4 et 5 lorsqu'il est correctement réglé le pointeau colle à la surface intérieure du verre sur une bande de 3 à 4 mm.

3 Introduire un tournevis par le trou situé au sommet du capuchon et enfoncer la lame jusqu'au contact avec la petite vis qui se trouve à l'intérieur du pointeau. Pour les robinets jusqu'à voie de 6 mm utiliser un tournevis à lame de 3 mm pour les robinets à voie plus importante un tournevis à lame de 5 mm est nécessaire.

4 En serrant cette vis on augmente la pression appliquée à un joint élastique. Cette pression est transmise radialement et accroît le serrage du joint de pointeau contre le corps du robinet ce qui améliore les performances sous vide poussé ou à pression élevée. En desserrant cette vis on obtient l'effet inverse.

5 Toujours décompresser le joint de pointeau avant d'enlever le pointeau du corps du robinet.

Remarques importantes

1 Éviter un serrage excessif du joint de pointeau ainsi que du pointeau lui-même sur son siège pour ne pas détériorer le robinet.

2 S'assurer de la propreté du pointeau PTFE et du corps en verre. Une parfaite propreté est nécessaire pour atteindre les performances maximum.

3 Des robinets incorporés à des ensembles fonctionnant sous vide poussé peuvent être dégazés par chauffage à l'air chaud ou avec une flamme douce. Veiller à ne pas chauffer le capuchon de polypropylène au-dessus de 130°C.

4 Faire attention à l'utilisation de détecteur de vide à arc électrique. Une utilisation prolongée de ce type de détecteur peut endommager le pointeau de PTFE.

Notre politique d'amélioration constante de nos fabrications peut nous amener à modifier les caractéristiques de nos produits.

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Document No.

LT-519-183

Rev Mod

A-0

Page

10 of 10

WHC-SD-WM-071, REV. 0

Serial No	Sample Point	Date	Time Issued	Priority			
V 17.-5000	AP-BOIL	8- 2-93	14:16	26			
Determination	Method Standard	Result Units	Charge Code	Reruns			
COMMENT	XXXXXX	NONE	N1A7F	0			
Sample Size	Customer ID						
1 boil down - complete mgt	AP-BOIL						
Remarks Calculations Results							
GENERAL INFORMATION FOR THIS SAMPLE							
<p><i>This sample is a composite of the following samples</i></p> <p>101-AP-COMP (V-15) 45 mls</p> <p>107-AP-COMP (V-34) 43.75 mls</p> <p>108-AP-COMP (V-77) 36.25 mls</p> <p>Total Volume of composite SAMPLE = 125 mls</p>							
Analyst 1	Analyst 2	Analyst 3	Analyst 4	Analyst 5			
<i>2/1/94 - Mark</i>	<i>2/1/94 - Mark</i>	<i>2/1/94 - Mark</i>	<i>2/1/94 - Mark</i>	<i>2/1/94 - Mark</i>			
Hrs	Hrs	Hrs	Hrs	Hrs			
Date	Time Completed	Lab Unit Mgr					
4-6-94	1100	Keith Fuller	<i>[Signature]</i>				

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