



# Paragon Analytics

## Radiochemistry Case Narrative

### Tritium

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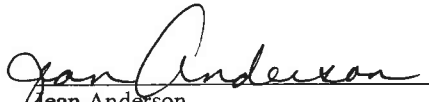
#### Cordilleran Compliance Services, Inc.

##### Rulison Area Well Monitoring

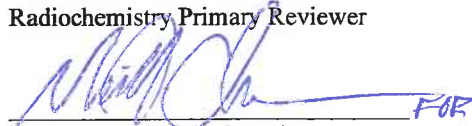
PA WO 0811110

1. This report consists of the analytical results and supporting documentation for two water samples received by Paragon on 11/14/2008.
2. These samples were prepared according to procedure PA SOP700R10.
3. The samples were analyzed for the presence of tritium according to procedure PA SOP704R9. The analyses were completed on 11/23/2008.
4. The analysis results for these samples are reported in units of pCi/L. The samples were not filtered prior to analysis.
5. In the analysis of the raw data, "Window 2" is monitored for high-energy beta contamination. Sample 0811110-1 had a "Window 2" count rate slightly above the upper control limit of 41.58 cpm established from calibration on 11/22/2008 through 11/23/2008, at 42.01 cpm. The sample had observed activity at a level below the achieved MDC. Thus, the data quality is believed to be unaffected and the results are submitted without further qualification.
6. No further anomalous situations were encountered during the preparation or analysis of these samples. All remaining quality control criteria were met.

The data contained in the following report have been reviewed and approved by the personnel listed below. In addition, Paragon Analytics certifies that the analyses reported herein are true, complete and correct within the limits of the methods employed.

  
Jean Anderson  
Radiochemistry Primary Reviewer

12/9/08  
Date

  
Radiochemistry Final Reviewer  
FOR  
EMILY  
BITLER

12/09/08  
Date

PARAGON ANALYTICS  
Radiochemistry Data Package

Section 1

**CHAIN OF CUSTODY**

# ALS Paragon

## Sample Number(s) Cross-Reference Table

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**Paragon OrderNum:** 0811110

**Client Name:** Cordilleran Compliance Services, Inc.

**Client Project Name:** Rulison Area Well monitoring

**Client Project Number:**

**Client PO Number:**

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Client Sample Number	Lab Sample Number	COC Number	Matrix	Date Collected	Time Collected
A11-15D	0811110-1		WATER	13-Nov-08	8:40
A11-15B	0811110-2		WATER	13-Nov-08	8:30



## CONDITION OF SAMPLE UPON RECEIPT FORM

Paragon Analytics

Client: CondillieranWorkorder No: 0811110Project Manager: LSInitials: oo Date: 11-14-08

1. Does this project require any special handling in addition to standard Paragon procedures?		YES	<u>NO</u>
2. Are custody seals on shipping containers intact?	NONE	<u>YES</u>	NO
3. Are Custody seals on sample containers intact?	NONE	<u>YES</u>	NO
4. Is there a COC (Chain-of-Custody) present or other representative documents?		<u>YES</u>	NO
5. Are the COC and bottle labels complete and legible?		<u>YES</u>	NO
6. Is the COC in agreement with samples received? (IDs, dates, times, no. of samples, no. of containers, matrix, requested analyses, etc.)		<u>YES</u>	NO
7. Were airbills / shipping documents present and/or removable?	DROP OFF	<u>YES</u>	NO
8. Are all aqueous samples requiring preservation preserved correctly? (excluding volatiles)	N/A	<u>YES</u>	<u>NO</u>
9. Are all aqueous non-preserved samples pH 4-9?	N/A	<u>YES</u>	NO
10. Is there sufficient sample for the requested analyses?		<u>YES</u>	NO
11. Were all samples placed in the proper containers for the requested analyses?		<u>YES</u>	NO
12. Are all samples within holding times for the requested analyses?		<u>YES</u>	NO
13. Were all sample containers received intact? (not broken or leaking, etc.)		<u>YES</u>	NO
14. Are all samples requiring no headspace (VOC, GRO, RSK/MEE, Rx CN/S, radon) headspace free? Size of bubble: <u>✓</u> < green pea <u>      </u> > green pea	N/A	YES	<u>NO</u>
15. Do perchlorate LCMS-MS samples have headspace? (at least 1/3 of container required)	<u>N/A</u>	YES	NO
16. Were samples checked for and free from the presence of residual chlorine? (Applicable when PM has indicated samples are from a chlorinated water source; note if field preservation with sodium thiosulfate was not observed.)	<u>N/A</u>	YES	NO
17. Were the samples shipped on ice?		<u>YES</u>	NO
18. Were cooler temperatures measured at 0.1-6.0°C?	IR gun used*: <u>#2</u> <u>#4</u>	RAD ONLY	<u>YES</u> NO
Cooler #: <u>1</u>			
Temperature (°C): <u>3.4</u>			
No. of custody seals on cooler: <u>1</u>			
DOT Survey/ Acceptance Information	External µR/hr reading: <u>14</u>		
	Background µR/hr reading: <u>13</u>		
Were external µR/hr readings ≤ two times background and within DOT acceptance criteria? <u>YES</u> NO / NA (If no. see Form 008.)			

Additional Information: PROVIDE DETAILS BELOW FOR A NO RESPONSE TO ANY QUESTION ABOVE, EXCEPT #1 AND #16.

Headspace Bottle # (1) 1, 2, 3, 6, 7, 9  
# 2 - 1, 2

Slime layer in 1-15 & 1-16 (Organic?)

If applicable, was the client contacted? YES / NO / NA Contact: J. Hix Date/Time: 11/12/08Project Manager Signature / Date: [Signature] 11/12/08

\*IR Gun #2: Oakton. SN 29922500201-0066

\*IR Gun #4: Oakton. SN 2372220101-0002

## CONDITION OF SAMPLE UPON RECEIPT FORM

Paragon Analytics

Client: CondolivanWorkorder No: 081110Project Manager: LSInitials: as Date: 11-14-08

## Additional Information:

Was the laboratory directed to proceed with the analysis of any samples yielding the presence of residual chlorine? YES / NO / NA

**NOTE:**

No pH adjustments shall be made without prior consent of Project Manager. After pH adjustments, hold metals and radchem samples  $\geq 24$  hrs. before analysis.

Was the pH of any sample adjusted by the laboratory? YES (See Table below) / NO

**pH Excursion:**

Paragon Sample ID	Client Sample ID	Initial pH	Final pH	Reagent Used	Volume Added (mL)	Lot No. of Reagent	Requested Analysis	Initials / Date / Time
-1-12		7	1.6	conc HNO <sub>3</sub>	1 mL			as 11/14/08 10:30
-1-15		↓	↓	↓	↓			↓
-1-16		↓	↓	↓	↓			↓
-1-17		↓	↓	↓	↓			↓
-2-12		↓	↓	↓	↓			↓
-2-15		↓	↓	↓	↓			↓
-2-16		↓	↓	↓	↓			↓
-2-17		↓	↓	↓	↓			↓

If applicable, was the client contacted? YES / NO / NA Contact: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Project Manager Signature / Date: 11/17/08

ORIGIN ID: GJTA (970) 270-2986  
TIM DOBRANSKY  
CORDILLERAN COMPLIANCE SERVICES, IN  
826 21 1/2 ROAD

GRAND JUNCTION, CO 81505  
UNITED STATES US

Ship Date: 13NOV08  
ActWgt: 20.0 LB MAN  
System#: 390082/CAFE2358  
Account: S 235727234

TO

PARAGON ANALYTICS  
225 COMMERCE DRIVE

FORT COLLINS, CO 80524

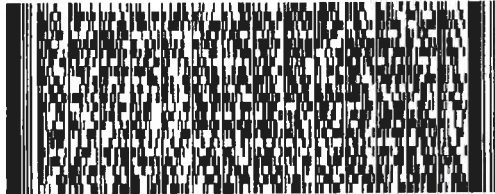
(800) 443-1511

**FedEx**  
Express



CLS050107/22/23

Ref: 8360



Delivery Address  
Barcode

BILL SENDER

PRIORITY OVERNIGHT

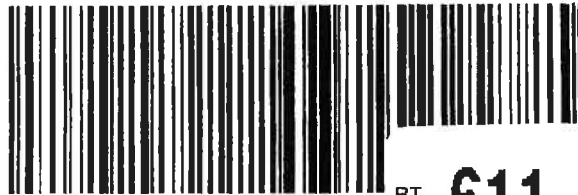
TRK# 9660 0451 2332 Form 0201

80524 -CO-US

72 FTCA

**FRI**  
Deliver By:  
14NOV08

DEN AA



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611 A

FZ

2332  
11.14

PARAGON ANALYTICS  
Radiochemistry Data Package

Section 2

**SAMPLE RESULTS  
SUMMARY**



# Tritium Analysis By Liquid Scintillation Sample Results Summary

Client Name: Cordilleran Compliance Services, Inc.  
Client Project Name: Rulison Area Well monitoring  
Client Project Number:

Laboratory Name: ALS Paragon  
PAI Work Order: 0811110

Page: 1 of 1  
Reported on: Friday, December 05, 2008  
12:18:18 PM

Lab Sample ID	Client Sample ID	Sample Type	Nuclide	Result +/- 2 s TPU	MDC	Units	Matrix	Prep Batch	Date Analyzed	Flags
0811110-1	A11-15D	Sample	H-3	-50 +/- 180	300	pCi/l	WATER	3H081120-1	11/23/2008	U
0811110-2	A11-15B	Sample	H-3	30 +/- 180	300	pCi/l	WATER	3H081120-1	11/22/2008	U

## Comments:

Data Package ID: H30811110-1

### Qualifiers/Flags:

U - Result is less than the sample specific MDC.  
LT - Result is less than Requested MDC, greater than sample specific MDC.  
Y1 - Chemical Yield is in control at 100-110%. Quantitative Yield is assumed.  
Y2 - Chemical Yield outside default limits.  
M - The requested MDC was not met.  
M3 - The requested MDC was not met, but the reported activity is greater than the reported MDC.

### Abbreviations:

TPU - Total Propagated Uncertainty (see PAI SOP 743)  
MDC - Minimum Detectable Concentration (see PAI SOP 709)  
BDL - Below Detection Limit

Date Printed: Friday, December 05, 2008

ALS Paragon  
LIMS Version: 6.213A

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PARAGON ANALYTICS  
Radiochemistry Data Package

Section 3

**QC RESULTS  
SUMMARY**

# Tritium Analysis By Liquid Scintillation

PAI 704 Rev 9

## Method Blank Results

Lab Name: ALS Paragon

Work Order Number: 0811110

Client Name: Cordilleran Compliance Services, Inc.

ClientProject ID: Rulison Area Well monitoring

Lab ID: 3H081120-1MB

Sample Matrix: WATER

Prep SOP: PAI 700 Rev 10

Date Collected: 20-Nov-08

Date Prepared: 20-Nov-08

Date Analyzed: 22-Nov-08

Prep Batch: 3H081120-1

QCBatchID: 3H081120-1-3

Run ID: 3h081120-1x

Count Time: 120 minutes

Final Aliquot: 10.0 ml

Result Units: pCi/l

File Name: Manual Entry

CASNO	Target Nuclide	Result +/- 2 s TPU	MDC	Requested MDC	Lab Qualifier
10028-17-8	H-3	-130 +/- 180	300	400	U

### Comments:

#### Qualifiers/Flags:

U - Result is less than the sample specific MDC.

Y1 - Chemical Yield is in control at 100-110%. Quantitative Yield is assumed.

Y2 - Chemical Yield outside default limits.

LT - Result is less than Requested MDC, greater than sample specific MDC.

#### Abbreviations:

TPU - Total Propagated Uncertainty (see PAI SOP 743)

MDC - Minimum Detectable Concentration (see PAI SOP 709)

BDL - Below Detection Limit

M - Requested MDC not met.

B - Analyte concentration greater than MDC.

B3 - Analyte concentration greater than MDC but less than Requested MDC.

Data Package ID: H30811110-1

# Tritium Analysis By Liquid Scintillation

PAI 704 Rev 9

## Laboratory Control Sample(s)

Lab Name: ALS Paragon

Work Order Number: 0811110

Client Name: Cordilleran Compliance Services, Inc.

ClientProject ID: Rulison Area Well monitoring

Lab ID: 3H081120-1LCS

Sample Matrix: WATER

Prep SOP: PAI 700 Rev 10

Date Collected: 20-Nov-08

Date Prepared: 20-Nov-08

Date Analyzed: 22-Nov-08

Prep Batch: 3H081120-1

QCBatchID: 3H081120-1-3

Run ID: 3h081120-1x

Count Time: 120 minutes

Final Aliquot: 9.90 ml

Result Units: pCi/l

File Name: Manual Entry

CASNO	Target Nuclide	Results +/- 2s TPU	MDC	Spike Added	% Rec	Control Limits	Lab Qualifier
10028-17-8	H-3	12400 +/- 1900	300	13000	94.8	85 - 115	P

### Comments:

#### Qualifiers/Flags:

U - Result is less than the sample specific MDC.

LT - Result is less than Requested MDC, greater than sample specific MDC.

Y1 - Chemical Yield is in control at 100-110%. Quantitative Yield is assumed.

Y2 - Chemical Yield outside default limits.

L - LCS Recovery below lower control limit.

H - LCS Recovery above upper control limit.

P - LCS Recovery within control limits.

M - The requested MDC was not met.

M3 - The requested MDC was not met, but thereported activity is greater than the reported MDC.

#### Abbreviations:

TPU - Total Propagated Uncertainty (see PAI SOP 743)

MDC - Minimum Detectable Concentration (see PAI SOP 709)

Data Package ID: H30811110-1

Date Printed: Friday, December 05, 2008

ALS Paragon

LIMS Version: 6.213A

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# Tritium Analysis By Liquid Scintillation

PAI 704 Rev 9

## Matrix Spike Results

Lab Name: ALS Paragon  
Work Order Number: 0811110  
Client Name: Cordilleran Compliance Services, Inc.  
ClientProject ID: Rulison Area Well monitoring

Field ID: A11-15D	Sample Matrix: WATER	Prep Batch: 3H081120-1	Final Aliquot: 9.90 ml
Lab ID: 0811110-1MS	Prep SOP: PAI 700	QCBatchID: 3H081120-1-3	Prep Basis: Unfiltered
	Date Collected: 13-Nov-08	Run ID: 3h081120-1x	Moisture(%): 100.000
	Date Prepared: 20-Nov-08	Count Time: 120 minutes	Result Units: pCi/l
	Date Analyzed: 23-Nov-08	Report Basis: Unfiltered	File Name: Manual Entry

CASNO	Target Nuclide	Matrix Spike	Sample Results	MDC	Spike Added	% Rec	Control Limits	Lab Qualifier
10028-17-8	H-3	12200	-50	300	13000	94.2	85 - 115	P

### Comments:

#### Qualifiers/Flags:

U - Result is less than the sample specific MDC.  
LT - Result is less than Requested MDC, greater than sample specific MDC.  
Y1 - Chemical Yield in control at 100-110%. Quantitative yield is assumed.  
Y2 - Chemical Yield outside default limits.  
N - Matrix Spike Recovery outside control limits  
P - Matrix Spike Recovery within control limits  
M - The requested MDC was not met.  
M3 - The requested MDC was not met, but thereported activity is greater than the reported MDC.

#### Abbreviations:

MDC - Minimum Detectable Concentration (see PAI SOP 709)

Data Package ID: H30811110-1

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ALS Paragon

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Section 4

**INDIVIDUAL  
SAMPLE RESULTS**

# Tritium Analysis By Liquid Scintillation

PAI 704 Rev 9

## Sample Results

Lab Name: ALS Paragon

Work Order Number: 0811110

Client Name: Cordilleran Compliance Services, Inc.

ClientProject ID: Rulison Area Well monitoring

Field ID: A11-15D	Sample Matrix: WATER	Prep Batch: 3H081120-1	Final Aliquot: 10.0 ml
Lab ID: 0811110-1	Prep SOP: PAI 700 Rev 10	QCBatchID: 3H081120-1-3	Prep Basis: Unfiltered
	Date Collected: 13-Nov-08	Run ID: 3h081120-1x	Moisture(%): 100.000
	Date Prepared: 20-Nov-08	Count Time: 120 minutes	Result Units: pCi/l
	Date Analyzed: 23-Nov-08	Report Basis: Unfiltered	File Name: Manual Entry

CASNO	Target Nuclide	Result +/- 2 s TPU	MDC	Requested MDC	Lab Qualifier
10028-17-8	H-3	-50 +/- 180	300	400	U

### Comments:

#### Qualifiers/Flags:

U - Result is less than the sample specific MDC.

Y1 - Chemical Yield is in control at 100-110%. Quantitative Yield is assumed.

Y2 - Chemical Yield outside default limits.

LT - Result is less than Requested MDC, greater than sample specific MDC.

M3 - The requested MDC was not met, but the reported activity is greater than the reported MDC.

M - The requested MDC was not met.

#### Abbreviations:

TPU - Total Propagated Uncertainty (see PAI SOP 743)

MDC - Minimum Detectable Concentration (see PAI SOP 709)

BDL - Below Detection Limit

**Data Package ID: H30811110-1**

Date Printed: Friday, December 05, 2008

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# Tritium Analysis By Liquid Scintillation

## PAI 704 Rev 9 Sample Results

Lab Name: ALS Paragon  
Work Order Number: 0811110  
Client Name: Cordilleran Compliance Services, Inc.  
ClientProject ID: Rulison Area Well monitoring

Field ID: A11-15B	Sample Matrix: WATER	Prep Batch: 3H081120-1	Final Aliquot: 10.0 ml
Lab ID: 0811110-2	Prep SOP: PAI 700 Rev 10	QCBatchID: 3H081120-1-3	Prep Basis: Unfiltered
	Date Collected: 13-Nov-08	Run ID: 3h081120-1x	Moisture(%): 100.000
	Date Prepared: 20-Nov-08	Count Time: 120 minutes	Result Units: pCi/l
	Date Analyzed: 22-Nov-08	Report Basis: Unfiltered	File Name: Manual Entry

CASNO	Target Nuclide	Result +/- 2 s TPU	MDC	Requested MDC	Lab Qualifier
10028-17-8	H-3	30 +/- 180	300	400	U

### Comments:

#### Qualifiers/Flags:

U - Result is less than the sample specific MDC.  
Y1 - Chemical Yield is in control at 100-110%. Quantitative Yield is assumed.  
Y2 - Chemical Yield outside default limits.  
LT - Result is less than Requested MDC, greater than sample specific MDC.  
M3 - The requested MDC was not met, but the reported activity is greater than the reported MDC.  
M - The requested MDC was not met.

#### Abbreviations:

TPU - Total Propagated Uncertainty (see PAI SOP 743)  
MDC - Minimum Detectable Concentration (see PAI SOP 709)  
BDL - Below Detection Limit

Data Package ID: H30811110-1



PARAGON ANALYTICS  
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Section 5

**RAW DATA**

# Tritium Analysis By Liquid Scintillation Raw Data Report

Laboratory Name: ALS Paragon  
PAI Work Order: 0811110

Prep SOP: PAI 700  
Analytical SOP: PAI 704

Reported on: Tuesday, November 25, 2008  
1:39:01 PM

Sample ID QC Type	Nuclide Type	Sample Date/Time	Prep Batch QC Batch ID	Ingrowth Date /Time	Quench Factor %Lum	Matrix %Moist.	Samp Aliq Analy Aliq	Inst ID Det ID	AnRunID File Name	Count Date/Time	GrossCPM BkgCPM	BaseEff ProgEff	CntDur(min) Yield	Activity +/- 2 s TPU	MDC Declav	ReportUnits ReportBasis	DER #Spk. Recov RPD	Flags
0811110-1	H-3	11/13/2008	3H081120-1	NA	144	WATER	50 ml	1e6500	3h081120-1x	11/23/2008	9,320	20.05%	120	-50	300	pCi/l	NA	U
	Trg. Analyte	8:40:00 AM	3H081120-1-3	NA	0.02	100	10 ml	33-11	Manual Entry	3:58 AM	9,540	NA	NA	180	NA	Unfiltered	NA	U
0811110-1	H-3	11/13/2008	3H081120-1	NA	146.8	WATER	50 ml	1e6500	3h081120-1x	11/23/2008	63,410	20.05%	120	12200	300	pCi/l	NA	94.2
	Trg. Analyte	8:40:00 AM	3H081120-1-3	NA	0.01	100	9.9 ml	33-12	Manual Entry	6:00 AM	9,540	NA	NA	1900	NA	Unfiltered	NA	P
0811110-2	H-3	11/13/2008	3H081120-1	NA	137.8	WATER	50 ml	1e6500	3h081120-1x	11/22/2008	9,680	20.05%	120	30	300	pCi/l	NA	U
	Trg. Analyte	8:30:00 AM	3H081120-1-3	NA	0.02	100	10 ml	37-01	Manual Entry	8:47 AM	9,540	NA	NA	180	NA	Unfiltered	NA	U
3H081120-1	H-3	11/20/2008	3H081120-1	NA	147.5	WATER	50 ml	1e6500	3h081120-1x	11/22/2008	8,980	20.05%	120	-130	300	pCi/l	NA	U
	Trg. Analyte	10:57:16 AM	3H081120-1-3	NA	0.02	NA	10 ml	37-02	Manual Entry	10:48 AM	9,540	NA	NA	180	NA	Unfiltered	NA	U
3H081120-1	H-3	11/20/2008	3H081120-1	NA	146.9	WATER	50 ml	1e6500	3h081120-1x	11/22/2008	63,980	20.05%	120	12400	300	pCi/l	NA	94.8
	Trg. Analyte	10:57:16 AM	3H081120-1-3	NA	0.01	NA	9.9 ml	37-03	Manual Entry	12:49 PM	9,540	NA	NA	1900	NA	Unfiltered	NA	P

Comments:

Data Package ID: H30811110-1

## Qualifiers/Flags:

U - Result is less than the sample specific MDC.  
Y1 - Chemical Yield is in control at 100-110%. Quantitative yield is assumed.  
Y2 - Chemical Yield outside default limits.  
W - DER is greater than Warning Limit of 1.42  
D - DER is greater than Control Limit of 2.13  
+ - Duplicate RPD not within limits.  
L.T. - Result is less than Request MDC, greater than sample specific MDC  
\* - Aliquot Basis is 'As Received' while the Report Basis is 'Dry Weight'.  
# - Aliquot Basis is 'Dry Weight' while the Report Basis is 'As Received'.

M - Requested MDC not met.

M3 - The requested MDC was not met, but the reported activity is greater than the reported MDC.

L - LCS Recovery below lower control limit.

H - LCS Recovery above upper control limit.

P - LCS, Matrix Spike Recovery within control limits.

N - Matrix Spike Recovery outside control limits

NC - Not Calculated for duplicate results less than 5 times MDC

B - Analyte concentration greater than MDC.

B3 - Analyte concentration greater than MDC but less than Requested MDC.

## Notes:

- The Tracer results are not yield corrected (i.e. activity measured not activity added).
- Where sample time is not available, 12:00 PM (Mountain) is used for decay correction.

## Abbreviations:

TR - Tracer  
TA - Target Analyte  
TPU - Total Propagated Uncertainty (see PAI SOP 743)  
MDC - Minimum Detectable Concentration (see PAI SOP 709)  
DER - Duplicate Error Ratio  
BDL - Below Detection Limit

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22 NOV 2008 08:47

ID: H3- 10 ML GEO.  
 USER: 13 COMMENT: LS6500  
 PRESET TIME : 120.00  
 DATA CALC : CPM H# : YES SAMPLE REPEATS: 1  
 COUNT BLANK : NO IC# : NO REPLICATES : 1  
 TWO PHASE : NO AQC : NO CYCLE REPEATS : 1  
 SCINTILLATOR: LIQUID LUMEX: NO LOW SAMPLE REJ: 0  
 LOW LEVEL : YES HALF LIFE CORRECTION DATE: none

PRINTER : STD  
 RS232 : OFF  
 DISK : OFF

CHAN: 100.0 - 320.0 %ERROR: 1.75 FACTOR: 1.000000 BKG. SUB: 0  
 CHAN: 380.0 - 990.0 %ERROR: 20.00 FACTOR: 1.000000 BKG. SUB: 0

SAM NO	POS.	TIME MIN	HM	WIND1		WIND2		LUMEX %	ELAPSED TIME
				CPM	%ERROR	CPM	%ERROR		
1	33-1	120.00	147.6	9.50	5.92	39.67	2.90	0.03	121.45
2	33-2	120.00	147.0	9.41	5.95	39.62	2.90	0.04	242.99
3	33-3	120.00	147.6	43.36	2.77	41.03	2.85	0.02	364.53
4	33-4	120.00	149.0	10.11	5.74	40.70	2.86	0.02	486.06
5	33-5	120.00	148.1	34.37	3.11	40.56	2.87	0.02	607.59
6	33-6	120.00	148.6	35.47	3.07	41.72X	2.83	0.02	729.13
7	33-7	120.00	147.8	11.20	5.46	41.09	2.85	0.02	850.66
8	33-8	120.00	147.7	9.67	5.87	40.46	2.87	0.02	972.18
9	33-9	56.80	148.0	229.96	1.75	40.97	4.15	0.00	1030.04
10	33-10	120.00	147.9	91.42	1.91	41.12	2.85	0.01	1151.57
11	33-11	120.00	144.0	9.32	5.98	42.01X	2.82	0.02	1273.10
12	33-12	120.00	146.8	63.41	2.29	39.97	2.89	0.01	1394.62

X ABOVE UCL OF 41.58.

MC  
11/25/08

OK  
MC  
11/23/08

ID: H3- 10 ML GEO.  
 USER: 13 COMMENT: LS6500

23 NOV 2008 08:57

PRESET TIME : 120.00  
 DATA CALC : CPM H# : YES SAMPLE REPEATS: 1 PRINTER : STD  
 COUNT BLANK : NO IC# : NO REPLICATES : 1 RS232 : OFF  
 TWO PHASE : NO AQC : NO CYCLE REPEATS : 1 DISK : OFF  
 SCINTILLATOR: LIQUID LUMEX: NO LOW SAMPLE REJ: 0  
 LOW-LEVEL : YES HALF LIFE CORRECTION DATE: none

CHAN: 100.0 - 320.0 %ERROR: 1.75 FACTOR: 1.000000 BKG. SUB: 0  
 CHAN: 380.0 - 990.0 %ERROR: 20.00 FACTOR: 1.000000 BKG. SUB: 0

SAM NO	POS	TIME MIN	HM	WIND1		WIND2		LUMEX %	ELAPSED TIME
				CPM	%ERROR	CPM	%ERROR		
1	37-1	120.00	137.8	9.68	5.87	38.31	2.95	0.02	121.43
2	37-2	120.00	147.5	8.98	6.09	39.61	2.90	0.02	242.96
3	37-3	120.00	146.9	63.98	2.28	39.58	2.90	0.01	364.49
4	37-4	120.00	147.7	9.46	5.94	39.41	2.91	0.03	486.02

MC  
 11/24/08

# LSC Run Log

Instrument ID:

LS 6500

364874

Date	Sample ID	CountTime (min.)	Rack & Position	Test	User #	Batch ID	Position Check	Initials	Comments
11/20/08	31081103-1MB	180	19 - 7	1B-10ML	5	31081103-1	MC	MC	NA
	-1LCS		1 - 8						
	-1LCSB		1 - 9						
	-1CB3		1 - 10						
11/21/08	DAILY Q.C.	10	1 - 14				MC		
11/21/08	31081112-1CB1	90	17 - 1	1B-10ML	5	31081112-1	MC	MC	RE-COUNT; HANG NOT MET
	0810245-4	2.3							
	274-1	2.55							
	2	1.65							
	3	2.55							
	0811034-6	70							
	6D								
	31081112-1CB2								
	0811034-14								
	14MS								
	053-6								
	8								
	11								
	14								
	31081112-1MB								
	-1LCS								
	-1CB3								
11-22-08	31081120-1CB1	120	33 - 1	1B-10ML	13	31081120-1	MC	MC	NA
	0811010-15								
	093-4								
	7								
	9								
	9D								
	11								

Reviewed by / Date

MC 11/25/08

FORM 7525XLS (12/18/2006)

364875

FORM 762r5.XLS (12/18/2006)

89/52/03

PARAGON ANALYTICS  
Radiochemistry Data Package

Section 6

**QUALITY ASSURANCE  
SUMMARY REPORTS**



No *NON-CONFORMANCE REPORTS* or  
*QUALITY ASSURANCE SUMMARY SHEETS*  
are included in this data package.



PARAGON ANALYTICS  
Radiochemistry Data Package

Section 7

**LABORATORY  
BENCH SHEETS**



# Radiochemistry Instrument Worksheet

Paragon Analytics

Prep Batch: 3H0811201

Prep Procedure: H3

10-ml

Analytical QASS (NCR) / N 11026

Prep Num	LabID	QC Type	Init Aliq	Fin Aliq	Units	Report Units	Cnt 1 Filed/Inst	Cnt 1 Rack-Pos	Cnt 1 Pos Chk By	Cnt 2 Filed/Inst	Cnt 2 Rack-Pos	Cnt 2 Pos Chk By	Cnt 3 Filed/Inst	Cnt 3 Rack-Pos	Cnt 3 Pos Chk By	Notes
11/27/10	0811010-15	SMP	50	10	ml	pCi/l	45-650	33-Z	MC							
1	0811093-4	SMP	50	10	ml	pCi/l	3									
1	0811093-7	SMP	50	10	ml	pCi/l	4									
1	0811093-8	SMP	50	10	ml	pCi/l	5									
1	0811093-9	DUP	50	10	ml	pCi/l	6									
1	0811093-11	SMP	50	10	ml	pCi/l	7									
1	0811093-13	SMP	50	10	ml	pCi/l	9									
1	0811093-15	SMP	50	10	ml	pCi/l	10									
1	0811110-1	SMP	50	10	ml	pCi/l	11									
1	0811110-1	MS	50	9.901	ml	pCi/l	12									
1	0811110-2	SMP	50	10	ml	pCi/l	37-1									
1	3H081120-1CB1	MB	50	10	ml	pCi/l	33-1									
1	3H081120-1CB2	MB	50	10	ml	pCi/l	1-8									
1	3H081120-1CB3	MB	50	10	ml	pCi/l	37-4									
1	3H081120-1	MB	50	10	ml	pCi/l	37-Z									
1	3H081120-1	LCS	50	9.901	ml	pCi/l	1-3									

11/25/08

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	648.3610.05	2,894.841	DFM/ml	11/20/08	0.5	ml	ST-002

# Radiochemistry Instrument Worksheet




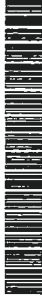




















Paragon Analytics

Prep Batch: 6H08112051

## Reporting Units

LabID:	TstGrpName:	RptUnits:
081110-1	H3	pCi/l
081110-2	H3	pCi/l
0811093-4	H3_BJC_STD	pCi/l
0811093-7	H3_BJC_STD	pCi/l
0811093-9	H3_BJC_STD	pCi/l
0811093-11	H3_BJC_STD	pCi/l
0811093-13	H3_BJC_STD	pCi/l
0811093-15	H3_BJC_STD	pCi/l
0811010-15	H3_ITLV	pCi/l

## Sample Barcodes

0811010-15 3H081120-1PS1			0811093-4 3H081120-1PS2	
0811093-7 3H081120-1PS3			0811093-9 3H081120-1PS4	
0811093-9DUP 3H081120-1PS5			0811093-11 3H081120-1PS6	
0811093-13 3H081120-1PS7			0811093-15 3H081120-1PS8	
0811110-1 3H081120-1PS9			0811110-1MS 3H081120-1PS10	
0811110-2 3H081120-1PS11			3H081120-1CB1MB 3H081120-1PS12	
3H081120-1CB2MB 3H081120-1PS13			3H081120-1CB3MB 3H081120-1PS14	
3H081120-1MB 3H081120-1PS15			3H081120-1LCS 3H081120-1PS16	

2

# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Procedure: H3

Reviewed By: gdw *gdw* Review Date: 11/20/2008

Non-Routine Pre-Treatment? Y 1 N Batch: N/A Re-Prep? Y 1 N Prep Analyst: Gabriel D. Wagner *gdw*  
 Prep SOP: PAI 700 Rev: 10 Prep Date: 11/20/2008  
 Prep SOP: NONE Matrix Class: liquid Prep Dept: RS

Cocktail: UG LLT  
 Cocktail Pipet: T-002  
 Aliquot Pipet: RS-101

Sample Num	LabID	QC Type	Dish No.	Init Aliq ml	Fin Aliq ml	Prep Basis	Water Added(ml)	Moisture(%)	Analysis Vol.(ml)	Standards	Prep Notes
1	0811010-16	SMP		50	10	Unfiltered	0	100	10		
2	0811093-4	SMP		50	10	Unfiltered	0	100	10		
3	0811093-7	SMP		50	10	Unfiltered	0	100	10		
4	0811093-9	SMP		50	10	Unfiltered	0	100	10		
5	0811093-9	DUP		50	10	Unfiltered	0	100	10		
6	0811093-11	SMP		50	10	Unfiltered	0	100	10		
7	0811093-13	SMP		50	10	Unfiltered	0	100	10		
8	0811093-15	SMP		50	10	Unfiltered	0	100	10		
9	0811110-1	SMP		50	10	Unfiltered	0	100	10		
10	0811110-1	MS		50	9.500990	Unfiltered	0	100	10	S1	
11	0811110-2	SMP		50	10	Unfiltered	0	100	10		
12	3H081120-1CB1	MB		50	10	Unfiltered	0	100	10		
13	3H081120-1CB2	MB		50	10	Unfiltered	0	100	10		
14	3H081120-1CB3	MB		50	10	Unfiltered	0	100	10		
15	3H081120-1	MB		50	10	Unfiltered	0	100	10		
16	3H081120-1	LCS		50	9.500990	Unfiltered	0	100	10	S1	

Comments

UG LLT #97-080701

Spiked By: Gabriel D. Wagner *gdw* Date: 11/20/2008

Witnessed By: Jeff Kujawa Date: 11/20/2008

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot Units	Pipet ID
S1	H-3	648.3610.05	2,894.841	DPM/ml	11/20/08	0.5	ST-002

# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Batch: 3H0811201

Prep Procedure: H3

**Prep Batch Not Validated!!!**

Reviewed By: Review Date:

Non-Routine Pre-Treatment? Y / N Batch:

Re-Prep? Y / N Batch:

Prep QASS / NCR? Y / N

Prep SOP: PAI 700 Rev: 10

Prep Analyst: Gabriel D. Wagner *GDW*

Balance:

Cocktail: UG LLT

Prep SOP: NONE

Prep Date: 11/20/2008

Balance:

Cocktail Pipet: T-002

Matrix Class: liquid

Prep Dept: RS

Allquot Pipet: RS-101

Sample Num	Prep Num	LabID	QC Type	Dish No.	Init Alq ml	Fin Alq ml	Prep Basis	Water Added (ml)	Moisture (%)	Analysis Vol. (ml)	Standards	Prep Notes
1	1	0811010-15	SMP		50	10	Unfiltered	0	100	10		
2	1	0811093-4	SMP		50	10	Unfiltered	0	100	10		
3	1	0811093-7	SMP		50	10	Unfiltered	0	100	10		
4	1	0811093-9	SMP		60	10	Unfiltered	0	100	10		
5	1	0811093-9	DUP		50	10	Unfiltered	0	100	10		
6	1	0811093-11	SMP		50	10	Unfiltered	0	100	10		
7	1	0811093-13	SMP		50	10	Unfiltered	0	100	10		
8	1	0811093-15	SMP		50	10	Unfiltered	0	100	10		
9	1	0811110-1	SMP		50	10	Unfiltered	0	100	10		
10	1	0811110-1	MS		50	9,900,990	Unfiltered	0	100	10	S1	
11	1	0811110-2	SMP		50	10	Unfiltered	0	100	10		
12	1	3H081120-1	MB		50	10	Unfiltered	0	100	10		
13	1	3H081120-1	LCS		50	9,900,990	Unfiltered	0	100	10	S1	

Comments

UG LLT #97-080701

Spiked By: Gabriel D. Wagner *GDW* Date: 11-20-2008

Witnessed By: *GR* Date: 11/20/08

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	648.3610.05	2,894,841	DPM/ml	11/20/08	0.5	ml	ST-002

*Exp. 4/30/09*

# **SAMPLE CONDITION FORM (LIQUID)**

ANALYST: *GDW*

ANALYSIS DATE: *11-20-08*

METHOD: *3H*

WORK ORDER	SAMPLE ID	SAMPLE CONDITION		
		pH	Color	Remarks
<i>0811010</i>	<i>15</i>	<i>5</i>	<i>Lt. Brown</i>	<i>Lt. sediment</i>
<i>0811093</i>	<i>4</i>	<i>6</i>	<i>clear</i>	<i>none</i>
<i>↓</i>	<i>7</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>↓</i>	<i>9</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>↓</i>	<i>11</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>↓</i>	<i>13</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>↓</i>	<i>15</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>0811110</i>	<i>1</i>	<i>↓</i>	<i>orangish brown</i>	<i>strong fuel odor moderate suspended sediment</i>
<i>↓</i>	<i>2</i>	<i>7</i>	<i>↓</i>	<i>↓</i>
<div style="position: relative; height: 100px;"> <div style="position: absolute; top: 0; right: 0; transform: rotate(-45deg);"> <i>GDW</i> <i>11-20-08</i> </div> </div>				

## TRITIUM RUN LOG

358352

Logbook/Page No.

SOP 700 Rev 10

Form 1306r1.doc (3/30/2003)

Date	Workorder/ Sample Number	Column ID	Flask ID	Run No.	Comments	Technician's Initials
11-12-08	0811034-14	A	T14	2	3H081112-1	GDW
	↓ -14ms	B	T29			
	0811053-11	C	T20			
	↓ -14	D	T16			
	3H081112-1 MB	E	T30			
	↓ -1LCS	F	T17			
	0811053-6	A	T23	3		
	↓ -8	B	T27	↓		
11-20-08	0811010-15	A	T15	1	3H081120-1	GDW
	0811093-4	B	T17			
	↓ -7	C	T14			
	↓ -9	D	T21			
	↓ -9D	E	T30			
	↓ -11	F	T20			
	↓ -13	A	T28	2		
	↓ -15	B	T10			
	0811110-2	C	T23			
	3H081120-1 MB	D	T24			
	0811110-1	A	T16	3		
	↓ -1ms	B	T27			
	3H081120-1LCS	C	T16			
11-20-08	3H081119-1 MB <del>3H081120-1 MB</del> 11-20-08	N/A	N/A	1	3H081119-1 SOP 754r5	GDW
	↓ -1LCS					
	0810010-1					

Reviewed by / Date ghe 11/20/08

PARAGON ANALYTICS  
Radiochemistry Data Package

Section 8

**STANDARDS  
TRACEABILITY  
DOCUMENTS**





Prepare a working dilution of ~ 4,000 dpm/ml of  $^3\text{H}$ ,  
648.2382.75.

1) Determine density of DI water

Mass of 100 ml vol. flask

68.2975g

12

Mass of flask and 100 ml DI  $\text{H}_2\text{O}$

168.0291g

1

Net Mass of water

99.7316g

$\rho = .9973 \text{ g/ml}$

2) Transfer Std.

Mass of empty Amber jar (No lid)

257.75g

26

Mass of Jar & Std transferred

264.58g

1

\* Net Mass of Std. Transferred

6.8506g

12

from below. JDO 4/28/08

4) Dilute to Final Vol. w/ DI water

Mass of Std.  $\text{H}_2\text{O}$ , Jar

750.9g

26

Mass of Jar from above

257.75g

Net Mass of new dilution.

493.15g

\* Standard was transferred by difference from a plastic cup

Mass of Cup and ~ 10g of Standard

23.6533g

12

Mass of Cup and Std. [Not] Transferred

16.8027g

1

Final Activity Calculation

$$\left(369,530.45 \frac{\text{dpm}}{\text{ml}}\right) \left(6.8506\text{g}\right) \left(.9973 \frac{\text{g}}{\text{ml}}\right) = 5141.07 \frac{\text{dpm}}{\text{ml}}$$

$$\left(.9958 \frac{\text{g}}{\text{ml}}\right) \left(493.15\text{g}\right)$$

Std ID: 648.3610.05

Description: H-3

Expiration: 4/30/2009

Activity: 5141.07 dpm/mL

NC  
7/13/08

2s Uncertainty: 37.02 dpm/mL

Ref. Date: 9/3/1998

Ref Time: N/A

Prep Date: 4/30/2008 Prep by: JD

Matrix/Comp. DI WATER

Half Life (y): 1.23E+01

#### Reverification Log

Analysis Date	Initials	Expiration Date

NC  
7/13/08

Page



Signed

4/28/08  
Date

NC  
7/13/08



Signed

7/13/08  
Date

Prepare a ~~working level~~ <sup>10 dilution</sup> dilution (at approximately 250 DPM/mL) tritium standard using RSD #648 and diluting with DI water.

1) Determine the density of DI water:

Mass of empty 100 mL class A volumetric flask

67.1522 g Bal #13

Mass of flask + water

166.7351 g

Net mass of 100 mL H<sub>2</sub>O

99.5829 g

$\rho = .9958 \text{ g/mL}$

2) Transfer contents of Ampoule SRM 4927F to a

~~40 mL VOA vial~~ 500 mL glass amber bottle

mass of ~~40 mL VOA vial~~ w/o lid

255.04 g Bal #26

mass of open ampoule + 50 mL beaker

40.9075 g Bal #12

mass of empty ampoule + 50 mL beaker

36.0148 g

Net mass of STD.

4.8927 g

3) Dilute STD. with DI water.

mass of bottle w/o lid

255.04 g Bal #26

mass of bottle, STD, + DI water

757.1 g

Net mass of STD + DI water

502.1 g

4. Final Activity Calculation.

$$(634.7 \text{ KBq/g})(1000 \text{ Bq/KBq})(60 \text{ DPM/Bq})(4.8927 \text{ g})(.9958 \text{ g/mL})$$

502.1 g

$= 369,530.45 \frac{\text{DPM}}{\text{mL}}$

U.S. Department of Commerce  
National Institute of Standards  
and Technology  
SRM 4927F  
Hydrogen-3

<4 MBq in distilled water

CAUTION  
RADIOACTIVE



Read and Understood By

Continued on Page

*Chad W. [Signature]*  
Signed

3/27/03  
Date

*Renee Hall [Signature]*  
Signed

3/27/03  
Date



# National Institute of Standards & Technology

## Certificate

PA ID 0648  
recd 12-04-02

### Standard Reference Material 4927F

### Hydrogen-3 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive hydrogen-3, as water, in 5 mL of distilled water. The solution is contained in a flame-sealed NIST borosilicate-glass ampoule. The SRM is intended for the calibration of beta-particle counting instruments and for the monitoring of radiochemical procedures.

#### Radiological Hazard

The SRM ampoule contains hydrogen-3 with a total activity of approximately 3.2 MBq. Hydrogen-3 decays by beta-particle emission. None of the beta particles escape from the SRM ampoule. During the decay process no photons are emitted. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]\*. There is no detectable external radiation. The SRM should be used only by persons qualified to handle radioactive material.

#### Chemical Hazard

The SRM ampoule contains only distilled water. There is no chemical hazard. If the ampoule is to be opened to transfer the solution, the recommended procedure is given on page 2.

#### Storage and Handling

The SRM should be stored and used at a temperature between 5 and 65 °C. The solution in an unopened ampoule should remain stable and homogeneous until at least September 2008.

The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material. If the ampoule is transported it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. The solution in the ampoule is a dangerous good (hazardous material) because of the radioactivity.

#### Preparation

This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, L.R. Karam, Group Leader. The overall technical direction and physical measurements leading to certification were provided by L.L. Lucas and M.P. Unterweger of the Radioactivity Group.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.W.L. Thomas.

Bert M. Coursey, Chief  
Ionizing Radiation Division

Gaithersburg, Maryland 20899  
June 1999  
Half-life and text revised October 2000

Nancy M. Trahey, Chief  
Standard Reference Materials Program

#### **Recommended Procedure for Opening the SRM Ampoule**

- 1) If the SRM solution is to be diluted, it is recommended that the diluting solution have a composition comparable to that of the SRM solution.
- 2) Wear eye protection, gloves, and protective clothing and work over a tray with absorbent paper in it. Work in a fume hood.
- 3) Shake the ampoule to wet all of the inside surface of the ampoule. Return the ampoule to the upright position.
- 4) Check that all of the liquid has drained out of the neck of the ampoule. If necessary, gently tap the neck to speed the process.
- 5) Holding the ampoule upright, score the narrowest part of the neck with a scribe or diamond pencil.
- 6) Lightly wet the scored line. This reduces the crack propagation velocity and makes for a cleaner break.
- 7) Hold the ampoule upright with a paper towel, a wiper, or a support jig. Position the scored line away from you. Using a paper towel or wiper to avoid contamination, snap off the top of the ampoule by pressing the narrowest part of the neck away from you while pulling the tip of the ampoule towards you.
- 8) Transfer the solution from the ampoule using a pycnometer or a pipet with dispenser handle. NEVER PIPETTE BY MOUTH.
- 9) Seal any unused SRM solution in a flame-sealed glass ampoule, if possible, to minimize the evaporation loss.

See also reference [4]\*.

# PROPERTIES OF SRM 4927F

## Certified values

Solution density	$(0.998 \pm 0.002) \text{ g} \cdot \text{mL}^{-1}$ at 20.0 °C [b]*
Radionuclide	Hydrogen-3
Reference time	1200 EST, 3-September-1998
Massic activity of the solution [c]	$634.7 \text{ kBq} \cdot \text{g}^{-1}$
Relative expanded uncertainty ( $k=2$ )	0.72% [d] [e]

## Uncertified values

Physical Properties:			
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule		
Ampoule specifications	Body outside diameter	(16.5 ± 0.5) mm	
	Wall Thickness	(0.60 ± 0.04) mm	
	Barium content	Less than 2.5%	
	Lead-oxide content	Less than 0.02%	
	Other heavy elements	Trace quantities	
Solution mass	Approximately 5.0 g		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol·L <sup>-1</sup> )	Mass Fraction (g·g <sup>-1</sup> )
	H <sub>2</sub> O HHO	55 6 × 10 <sup>-7</sup>	100 1 × 10 <sup>-8</sup>
Radiological Properties:			
Radionuclidic impurities	None detected [f]		
Half lives used	Hydrogen-3: (4500 ± 8) d [g]		
Calibration method and measuring instrument(s)	4πB gas counting of SRM 4927E using the NIST length-compensated internal gas proportional counters and intercomparison of SRMs 4927E/4927F using two 4πB liquid-scintillation counting systems [h]		

**EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [d]\***

Input Quantity $x_i$ , the source of uncertainty (and individual uncertainty components where appropriate)	Method Used To Evaluate $u(x_i)$ , the standard uncertainty of $x_i$ (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$ , (%) [i]	Relative Sensitivity Factor, $ \partial y/\partial x_i  \cdot$ $(x_i/y)$ [j]	Relative Uncertainty Of Output Quantity, $u_i(y)/y$ , (%) [k]
Massic count rate of SRM 4927E, corrected for background and decay [h]	Standard deviation of the mean for 23 sets of gas counting measurements (A)	0.18	1.0	0.18
Gram-mole measurements	Estimated (B)	0.20	1.0	0.20
Live-time [p]	Estimated (B)	0.10	1.0	0.10
Extrapolation of count-rate-versus-energy to zero energy	Estimated (B)	0.20	1.0	0.20
Half life of H-3	Standard uncertainty of the half life (A)	0.18 [m]	0.009 [n]	0.002
Liquid-scintillation intercomparison of SRM 4927F and SRM-4927E	Standard deviation of the mean for 7 sets of liquid-scintillation measurements (A)	0.06	1.0	0.06
Radionuclidic impurities	Limit of detection (B) [q]	100.	0.0005	0.05
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)				0.36
Coverage Factor, $k$				<u>x 2</u>
Relative Expanded Uncertainty of the Output Quantity, $U/y$ , (%)				0.72



## NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One  $\mu\text{Sv}$  is equal to 0.1 mrem.  
 Distance from Ampoule (cm): 1 30 100  
 Approximate Dose Rate ( $\mu\text{Sv/h}$ ): <0.1 (Not detectable)
- [b] The stated uncertainty is two times the standard uncertainty.
- [c] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [d] The reported value,  $y$ , of massic activity (activity per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as  $y = f(x_1, x_2, x_3, \dots, x_n)$ , where  $f$  is a mathematical function derived from the assumed model of the measurement process.
- The value,  $x_i$ , used for each input quantity  $i$  has a **standard uncertainty**,  $u(x_i)$ , that generates a corresponding uncertainty in  $y$ ,  $u_i(y) = |\partial y / \partial x_i| \cdot u(x_i)$ , called a **component of combined standard uncertainty** of  $y$ .
- The **combined standard uncertainty** of  $y$ ,  $u_c(y)$ , is the positive square root of the sum of the squares of the components of combined standard uncertainty.
- The combined standard uncertainty is multiplied by a **coverage factor** of  $k = 2$  to obtain  $U$ , the **expanded uncertainty** of  $y$ .
- Since it can be assumed that the possible estimated values of the massic activity are approximately normally distributed with approximate standard deviation  $u_c(y)$ , the unknown value of the massic activity is believed to lie in the interval  $y \pm U$  with a level of confidence of approximately 95 percent.
- For further information on the expression of uncertainties, see references [2] and [3].
- [e] The value of each standard uncertainty component, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval  $U/2$  to  $2U$  (i.e., within a factor of 2 of the estimated value).
- [f] The estimated limit of detection for radionuclidic impurities is  $300 \text{ Bq} \cdot \text{g}^{-1}$ .
- [g] The stated uncertainty is the standard uncertainty. See reference [5].
- [h] Extensive gas-counting measurements were made on the SRM 4927E solution during 1998 and 1999. The SRM 4927F solution was intercompared with the SRM 4927E solution using liquid-scintillation counting.
- [i] Relative standard uncertainty of the input quantity  $x_i$ .

- [j] The relative change in the output quantity  $y$  divided by the relative change in the input quantity  $x_i$ . If  $|\partial y / \partial x_i| \cdot (x_i / y) = 1.0$ , then a 1% change in  $x_i$  results in a 1% change in  $y$ . If  $|\partial y / \partial x_i| \cdot (x_i / y) = 0.05$ , then a 1% change in  $x_i$  results in a 0.05% change in  $y$ .
- [k] Relative component of combined standard uncertainty of output quantity  $y$ , rounded to two significant figures or less. The relative component of combined standard uncertainty of  $y$  is given by  $u_c(y)/y \equiv |\partial y / \partial x_i| \cdot u(x_i)/y = |\partial y / \partial x_i| \cdot (x_i / y) \cdot u(x_i)/x_i$ . The numerical values of  $u(x_i)/x_i$ ,  $|\partial y / \partial x_i| \cdot (x_i / y)$ , and  $u_c(y)/y$ , all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.
- [m] The relative standard uncertainty of  $\lambda \cdot t$  is determined by the relative standard uncertainty of  $\lambda$  (i.e., of the half life). The relative standard uncertainty of  $t$  is negligible.
- [n]  $|\partial y / \partial x_i| \cdot (x_i / y) = |\lambda \cdot t|$
- [p] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.
- [q] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e.  $u(x_i)/x_i = 100\%$ .  $|\partial y / \partial x_i| \cdot (x_i / y) = \{(\text{response per Bq of impurity}) / (\text{response per Bq of H-3})\} \cdot \{(\text{Bq of impurity}) / (\text{Bq of H-3})\}$ . Thus  $u_c(y)/y$  is the relative change in  $y$  if the impurity were present with a massic activity equal to the estimated limit of detection.

#### REFERENCES

- [1] International Organization for Standardization (ISO), *ISO Standards Handbook - Quantities and Units*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900.
- [2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900. (Listed under ISO miscellaneous publications as "ISO Guide to the Expression 1993".)
- [3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.
- [4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814 U.S.A.
- [5] L.L. Lucas and M.P. Unterwieser, *Comprehensive Review and Critical Evaluation of the Half-Life of Tritium*, J. Res. Natl. Inst. Stand. Technol. **105**, 541-549 (2000).



PARAGON ANALYTICS  
Radiochemistry Data Package

Section 9

**ADDITIONAL  
SUPPORTING  
DOCUMENTATION**



Liquid Scintillation Counter

Instrumentation Calibration

Initial Efficiency Calibration  
Standards Traceability

# LS6500 10mL geometry Tritium Background Determination

Interim control limits are established from the initial calibration for the geometry of interest. Limits are +/- 3 standard deviations from the initial unquenched calibration blank data. Once enough historical data is acquired, new historical limits are set as follows:  
Control limits for reagent blanks are established from 30 individual historical data points (10 batches). Limits are +/- 3 standard deviations from 30 individual historical data points. Individual reagent blanks and the average of reagent blanks from each batch are in control if the Count Rate (CPM) is within the established control limits.

## CURRENTLY UNDER HISTORICAL LIMITS

Updated on 9/2/08 LNT												
COUNT DATE		#	Sample ID	Count Duration (m)	Count Rate (CPM)	Total Cts.	Mean	Individual Reagent Blanks			Average of Reagent Blanks	
								LCL	UCL	Pass ?	LCL	UCL
11/22/2008		37	3H081120-1CB1	120	9.50	1140.00		8.23	10.77	PASS		
11/22/2008		38	3H081120-1CB2	120	9.67	1160.40		8.23	10.77	PASS		
11/23/2008		39	3H081120-1CB3	120	9.46	1135.20	9.54	8.23	10.77	PASS	8.23	10.77
												PASS

## Tritium "Window 2" Control Limits (LS 6500)

The background count rate is determined from the average of the reagent blanks for the batch. Window 2 control limits are established using the average count rate from the three reagent blanks associated with each prep batch +/- 3X the estimated poisson uncertainty.

Updated 9/2/08 LNT

COUNT DATE	Sample ID	Count	Average count	Count Rate (CPM)	Batch Average Reagent Blank	Lower Control Limit	Upper Control Limit	PASS/ FAIL
		Duration (min.)	Duration (min.)					
11/22/2008	3H081120-1CB1	120	120	39.67	39.85	38.12	41.58	PASS
11/22/2008	3H081120-1CB2	120		40.46				
11/23/2008	3H081120-1CB3	120		39.41				
	0811093-13	57		40.97		37.33	42.36	

# 10-mL Tritium Efficiency Calibration LS6500

10 mL sample + 10 mL Ultima Gold LLT

8/27/2008

LS6500

Standard used: 699.2613.19

94000.71 dpm/ml as of 9/3/1998

1/2 life = 1.23E+01 yrs.

current activity = 53558.31 dpm/ml

volume = 0.1961 ml

Average Spike Activity = 10501.61 dpm

<u>Sample ID</u>	<u>WIND1 cpm</u>	<u>WIND2 cpm</u>	<u>%LUMEX</u>	<u>H #</u>	
0723514-10LCS	2156.23	44.10	0.00	143.8	
0723514-11LCS	2096.00	47.36	0.00	144.8	
0723514-12LCS	2093.28	39.52	0.00	144.6	
0723514-07MB	9.43	42.44	0.02	144.8	
0723514-08MB	9.77	40.97	0.01	143.8	
0723514-09MB	9.43	41.48	0.01	144.2	
average LCS=	2115.17	41.63	0.01	144.3	averages
average bkg=	9.54				
net cpm=	2105.63	<u>WIND2 cpm</u>	<u>%LUMEX</u>	<u>H #</u>	
/known dpm=	10501.61	44.13	5.00	159.3	UCL
		39.13	0.00	129.3	LCL
efficiency=	0.2005	See Tech. Mgr.	See Tech. Mgr.	Std. Addition	Corrective Action

Instrument Technician:

Kimberly Teaman 8/29/08  
Signature & Date

Supervisory Review:

Gene Hall 9/3/08  
Signature & Date

### <sup>3</sup>H Efficiency Calibration Verification / Method Blank Verification 8/27/08

#### Calibration Source Check

LS 6500

Analysis Date: 8/27/2008

Nuclide: <sup>3</sup>H

Half Life: 1.23E+01 yrs

#### Calibration Check Source:

Spike Standard: 648.3020.69

Reference Date: 9/3/1998

Spiked DPM/mL: 4011.21 dpm/mL

Spike Volume: 0.5 mL

Spiked into: 50.0 mL

Current Spk. Act.: 10.74 pCi/mL

CONTROL LIMITS: 83%-115%

#### Calibration Check Source Count

Sample ID	Position	Prep Date	Cut. Dur.	Anal. Vol.	GrsCPM	BlkgCPM	Efficiency	Activity	Units	Chem. Yield	LCS Recovery:	Pass/Fail
0723516-10LCS	57-10	11/28/2007	75	9.90099	54.75	9.54	0.2005	10.70	pCi/mL	100%	99.6%	PASS
0723516-11LCS	57-11	11/28/2007	75	9.90099	54.53	9.54	0.2005	10.65	pCi/mL	100%	99.2%	PASS
0723516-12LCS	57-12	11/28/2007	75	9.90099	54.84	9.54	0.2005	10.72	pCi/mL	100%	99.8%	PASS

#### Method Blank Check Count

Sample ID	Rack	Prep Date	Cut. Dur.	Anal. Vol.	GrsCPM	BlkgCPM	Efficiency	Chem. Yield	k (denom.)	activity	MDC	Pass/Fail	Units	1 $\sigma$ IU			1 $\sigma$ PU		
														0.056	2 $\sigma$ IU	2 $\sigma$ PU	0.051	2 $\sigma$ PU	2 $\sigma$ TPU
0723516-07MB	57-7	11/28/2007	75	10	9.17	9.54	0.2005	100%	4.268	-0.0875	0.39	PASS	pCi/mL	-0.00980	-0.00892	-0.00892	-0.00892	0.234	0.234
0723516-08MB	57-8	11/28/2007	75	10	9.44	9.54	0.2005	100%	4.268	-0.0242	0.40	PASS	pCi/mL	-0.00271	-0.00247	-0.00247	-0.00247	0.236	0.236
0723516-09MB	57-9	11/28/2007	75	10	10.01	9.54	0.2005	100%	4.268	0.1094	0.41	PASS	pCi/mL	0.01225	0.01225	0.01225	0.01225	0.239	0.240

ID#H3- 10 ML GEO.  
 USER:13 COMMENT:LS6500

27 AUG 2008 11:59

PRESET TIME : 75.00  
 DATA CALC : CPM H# : YES SAMPLE REPEATS: 1 PRINTER : STD  
 COUNT BLANK : NO IC# : NO REPLICATES : 1 RS232 : OFF  
 TWO PHASE : NO AQC : NO CYCLE REPEATS : 1 DISK : OFF  
 SCINTILLATOR: LIQUID LUMEX: NO LOW SAMPLE REJ: 0  
 LOW LEVEL : YES HALF LIFE CORRECTION DATE: none

CHAN: 100.0 - 320.0 %ERROR: 1.75 FACTOR: 1.000000 BKG. SUB: 0  
 CHAN: 380.0 - 990.0 %ERROR:20.00 FACTOR: 1.000000 BKG. SUB: 0

SAM NO	POS	TIME MIN	H#	WIND1		WIND2		LUMEX %	ELAPSED TIME
				CPM	%ERROR	CPM	%ERROR		
1	57-1	75.00	144.8	9.43	7.52	42.44	3.54	0.02	76.08
2	57-2	75.00	143.8	9.77	7.39	40.97	3.61	0.01	152.26
3	57-3	75.00	144.2	9.43	7.52	41.48	3.59	0.01	228.42
4	57-4	6.10	143.8	2156.23	1.74	44.10	12.19	0.00	235.17
5	57-5	6.25	144.8	2096.00	1.75	47.36	11.62	0.00	242.11
6	57-6	6.25	144.6	2093.28	1.75	39.52	12.73	0.00	249.02
7	57-7	75.00	144.7	9.17	7.62	41.17	3.60	0.02	325.20
8	57-8	75.00	144.6	9.44	7.52	41.17	3.60	0.01	401.38
9	57-9	75.00	145.0	10.01	7.30	41.83	3.57	0.01	477.56
10	57-10	75.00	145.6	54.75	3.12	41.84	3.57	0.01	553.74
11	57-11	75.00	145.0	54.53	3.13	40.64	3.62	0.01	629.94
12	57-12	75.00	144.2	54.84	3.12	41.89	3.57	0.01	706.12

OK 8/28/08

# LSC Run Log

Instrument ID:

LS 6500

364841

Date	Sample ID	CountTime (min.)	Rack & Position	Test	User #	Batch ID	Position Check	Initials	Comments
8/27/08	0723514-07MB	75	57-1	H3-10mL	13	3H071126-1	AS	AS	H-310mL S.P. Calib.
	-08MB		-2						
	-09MB		-3						
	-10ICS	6.10	-4						
	-11ICS	6.25	-5						
	-12ICS		-6						
	0723516-07MB	75	-7			3H071128-1			
	-08MB		-8						
	-09MB		-9						
	-10ICS		-10						
	-11ICS		-11						
	-12ICS		-12						
8/28/08	3H080817-10B1	40	30-1	H3-10mL	11	3H080817-1	AS	AS	N/A
	0808081-1		-2						
	-2		-3						
	-3		-4						
	-4		-5						
	-5		-6						
	-6		-7						
	-7		-8						
	-8		-9						
	-9		-10						
	-10		-11						
	-10D		-12						
	-10M5		31-1						
	3H080817-10B2		-2						
	0808081-11		-3						
	-12		-4						
	-13		-5						
	-14		-6						

Reviewed by / Date

AS 8/29/08

FORM 76215-XLS (12/18/2006)



# Radiochemistry Instrument Worksheet

Paragon Analytics

Prep Batch: 34071126

Prep Procedure: H3 - 5mL (10mL) Single Point Calibration

Analytical QASS / NCR? Y N N/A

Prep Num	LabID	QC Type	Init Aliq	Fin Aliq	Units	Cnt 1 File/Inst	Cnt 1 Rack-Pos	Cnt 1 Chk By	Cnt 2 File/Inst	Cnt 2 Rack-Pos	Cnt 2 Chk By	Cnt 3 File/Inst	Cnt 3 Rack-Pos	Cnt 3 Chk By	Notes
1	0723514-01	MB	50	5	ml										
1	0723514-02	MB	50	5	ml										
1	0723514-03	MB	50	5	ml										
1	0723514-04	LCS	50	4.901961	ml										
1	0723514-05	LCS	50	4.901961	ml										
1	0723514-06	LCS	50	4.901961	ml										
1	0723514-07	MB	50	10	ml										
1	0723514-08	MB	50	10	ml										
1	0723514-09	MB	50	10	ml										
1	0723514-10	LCS	50	9.803922	ml										
1	0723514-11	LCS	50	9.803922	ml										
1	0723514-12	LCS	50	9.803922	ml										

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	689.2813.19	55,943.597	DPM/ml	11/28/07	1	ml	RS-006

## Sample Barcodes

0723514-01MB 3H071126-1PS1	0723514-02MB 3H071126-1PS2
0723514-03MB 3H071126-1PS3	0723514-04LCS 3H071126-1PS4
0723514-05LCS 3H071126-1PS5	0723514-06LCS 3H071126-1PS6
0723514-07MB 3H071126-1PS7	0723514-08MB 3H071126-1PS8
0723514-09MB 3H071126-1PS9	0723514-10LCS 3H071126-1PS10
0723514-11LCS 3H071126-1PS11	0723514-12LCS 3H071126-1PS12

# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Batch: 310711261

Prep Procedure: H3

Reviewed By: JRK *[Signature]* Review Date: 11/28/2007

Non-Routine Pre-Treatment? ☒ Y / ☒ N Batch: 11 Re-Prep? ☒ Y / ☒ N Prep QASS / NCR? ☒ Y / ☒ N

Prep Analyst: Jeff Kujawa  
Prep Date: 11/28/2007  
Prep Dept: RS

Balance: 11  
Cocktail: UG LLT  
Cocktail Pipet: T-002  
Aliquot Pipet:

Sample Prep Num	LabID	QC Type	Dish No.	Init Aliq ml	Fin Aliq ml	Prep Basis	Water Added (ml)	Moisture (%)	Analysis Vol (ml)	Standards	Prep Notes
1	0723514-01	MB	50	5	5	Unfiltered	0	100	5		
2	0723514-02	MB	50	5	5	Unfiltered	0	100	5		
3	0723514-03	MB	50	5	5	Unfiltered	0	100	5		
4	0723514-04	LCS	50	4.901961	5	Unfiltered	0	100	5	S1	
5	0723514-05	LCS	50	4.901961	5	Unfiltered	0	100	5	S1	
6	0723514-06	LCS	50	4.901961	5	Unfiltered	0	100	5	S1	
7	0723514-07	MB	50	10	10	Unfiltered	0	100	10		
8	0723514-08	MB	50	10	10	Unfiltered	0	100	10		
9	0723514-09	MB	50	10	10	Unfiltered	0	100	10		
10	0723514-10	LCS	50	9.803922	10	Unfiltered	0	100	10	S1	
11	0723514-11	LCS	50	9.803922	10	Unfiltered	0	100	10	S1	
12	0723514-12	LCS	50	9.803922	10	Unfiltered	0	100	10	S1	

Spiked By: Jeff Kujawa Date: 11/28/2007

Witnessed By: Derek B. Caduff Date: 11/28/2007

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	600.2613.19	55,943.587	DPM/ml	11/28/07	1	ml	RS-008

Comments: UG LLT LOT# 97-070701.

# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Batch: 34071126

Prep Procedure: H3

Prep Batch Not Validated!!!

Review Date:

Reviewed By:

Re-Prep? Y / N

Batch:

Batch:

Pre-Treatment? Y / N

Prep SOP: PAI 700

Rev: 10

Prep SOP: NONE

Matrix Class: liquid

Prep Analyst: Jeff Kujawa

Prep Date: 11/26/2007

Prep Dept: RS

Balance:

Balance:

Cocktail: UG LLT

Cocktail Pipet: T-002

Aliquot Pipet:

Sampl Num	Prep Num	LabID	QC Type	Dish No.	Init Aliq ml	Fin Aliq ml	Prep Basis	Water Added(ml)	Moisture(%)	Analysis Vol.(ml)	Standards	Prep Notes
1	1	0723514-01	MB		50	5	Unfiltered	0	100	5		
2	1	0723514-02	MB		50	5	Unfiltered	0	100	5		
3	1	0723514-03	MB		50	5	Unfiltered	0	100	5		
4	1	0723514-04	LCS		50	4.901961	Unfiltered	0	100	5	S1	
5	1	0723514-05	LCS		50	4.901961	Unfiltered	0	100	5	S1	
6	1	0723514-06	LCS		50	4.901961	Unfiltered	0	100	5	S1	
7	1	0723514-07	MB		50	10	Unfiltered	0	100	10		
8	1	0723514-08	MB		50	10	Unfiltered	0	100	10		
9	1	0723514-09	MB		50	10	Unfiltered	0	100	10		
10	1	0723514-10	LCS		50	9.803922	Unfiltered	0	100	10	S1	
11	1	0723514-11	LCS		50	9.803922	Unfiltered	0	100	10	S1	
12	1	0723514-12	LCS		50	9.803922	Unfiltered	0	100	10	S1	

Spiked By:

Date:

Witnessed By:

Date:

Soln #	Nuclide	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	699.2613.19	55,943.587	DPM/ml	11/26/07	1	ml
							RS-006

exp: 8/17/08

Comments

UG LLT LOT# 97-070701.

Page 1 of 1

H3 Bench Sheet

Date Printed: 11/26/2007 16:51

Paragon Analytics

LIMS Version: 6.091A

Supersedes:

11

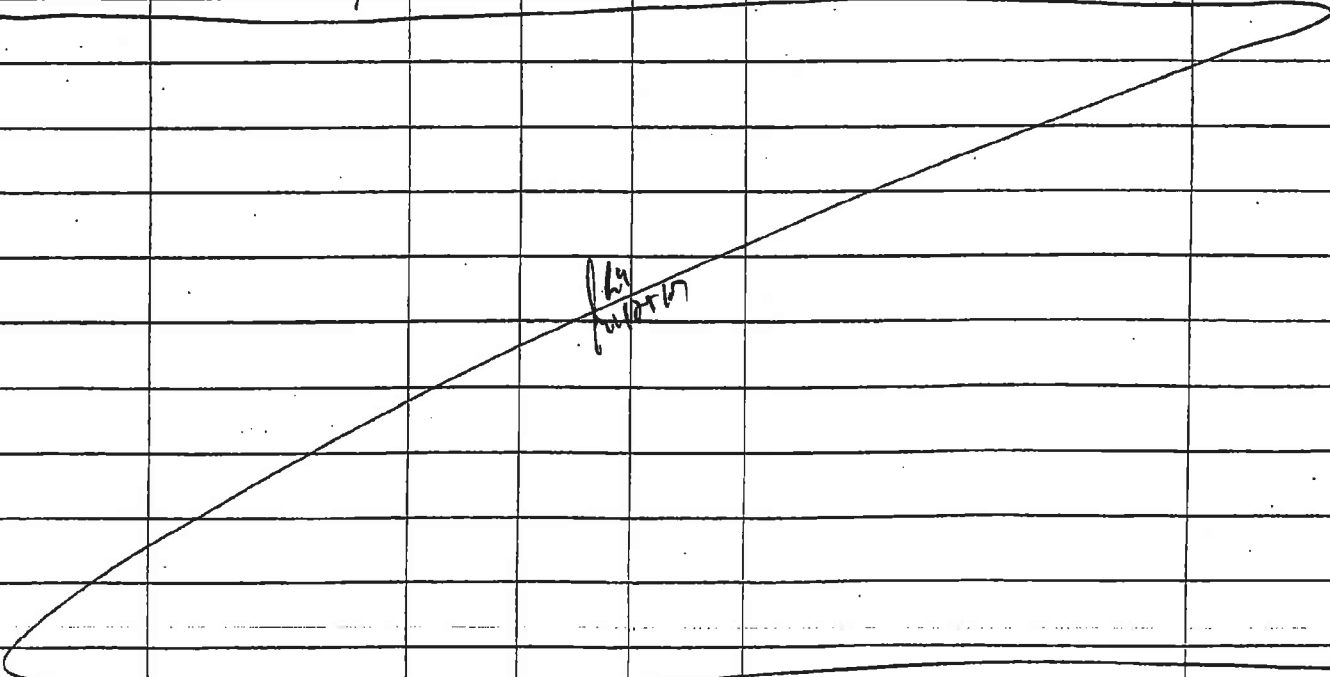
## TRITIUM RUN LOG

319185

Logbook/Page No.

SOP 700 Rev 1.0

Form 1306r1.doc (3/30/2003)

Date	Workorder/ Sample Number	Column ID	Flask ID	Run No.	Comments	Technician's Initials
11/20/07	071002S-D	N/A	N/A	9	Batch 3H071114-2 exp 754 RE	EMF
11/26/07	0723514-1, 7	A	T2	2	Batch 3H071126-1	JLH
	-2, 8	A	T7	1		
	-3, 9	B	T8			
	-4, 10	C	T11			
	-5, 11	D	102			
	-6, 12	F	600			
11/25/07	0723516-1, 7	A	102	1	Batch 3H071128-1	JLH
	-2, 8	B	600			
	-3, 9	C	T2			
	-4, 10	D	T7			
	-5, 11	E	T8			
	-6, 12	F	T11			
						

Reviewed by / Date

JLH 11/25/07

# Radiochemistry Instrument Worksheet

Paragon Analytics

Prep Batch: 3H071128-1

Prep Procedure: H3-ICB; ICI's (5.10 ml 5-P Calibration)

Analytical QASS / NCR? Y / N N/A

Prep Num	LabID	QC Type	Init Alq	Fin Alq	Units	Cnt 1 File/Inst	Cnt 1 Rack-Pos	Cnt 1 Pos	Chk By	Cnt 2 File/Inst	Cnt 2 Rack-Pos	Cnt 2 Pos	Chk By	Cnt 3 File/Inst	Cnt 3 Rack-Pos	Cnt 3 Pos	Chk By	Notes
1	0723516-01	MB	50	5	ml													
1	0723516-02	MB	50	5	ml													
1	0723516-03	MB	50	5	ml													
1	0723516-04	LCS	50	4.950495	ml													
1	0723516-05	LCS	50	4.950495	ml													
1	0723516-06	LCS	50	4.950495	ml													
1	0723516-07	MB	50	10	ml													
1	0723516-08	MB	50	10	ml													
1	0723516-09	MB	50	10	ml													
1	0723516-10	LCS	50	9.900990	ml													
1	0723516-11	LCS	50	9.900990	ml													
1	0723516-12	LCS	50	9.900990	ml													

Soln #	Nuclide	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
*S1	H-3	648.3020.69	2,386.637	11/28/07	0.5	ml	RS-003

## Sample Barcodes

0723516-01MB 3H071128-1PS1	0723516-02MB 3H071128-1PS2	0723516-03MB 3H071128-1PS3	0723516-04LCS 3H071128-1PS4	0723516-05LCS 3H071128-1PS5	0723516-06LCS 3H071128-1PS6	0723516-07MB 3H071128-1PS7	0723516-08MB 3H071128-1PS8	0723516-09MB 3H071128-1PS9	0723516-10LCS 3H071128-1PS10	0723516-11LCS 3H071128-1PS11	0723516-12LCS 3H071128-1PS12
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# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Batch: 3H071128-1

Prep Procedure: H3

Reviewed By: JRK/ks Review Date: 11/29/2007

Non-Routine Pre-Treatment? Y / ☒ Batch: 11/28/2007

Re-Prep? Y / ☒ Batch: 11/28/2007

Balance: 11/28/2007

Prep QASS / NCR? Y / ☒ Batch: 11/28/2007

Prep SOP: PAL 700 Rev: 10

Prep SOP: NONE

Matrix Class: liquid

Prep Analyst: Jeff Kujawa

Prep Date: 11/28/2007

Prep Dept: RS

Cocktail: UG LLT

Cocktail Pipet: T-002

Aliquot Pipet:

Sample Num	Prep Num	LabID	QC Type	Dish No.	Init Aliq ml	Fin Aliq ml	Prep Basis	Water Added (ml)	Moisture (%)	Analysis Vol (ml)	Standards	Prep Notes
1	1	0723516-01	MB	50	5	5	Unfiltered	0	100	5		
2	1	0723516-02	MB	50	5	5	Unfiltered	0	100	5		
3	1	0723516-03	MB	50	5	5	Unfiltered	0	100	5		
4	1	0723516-04	LCS	50	4.950495	4.950495	Unfiltered	0	100	5	S1	
5	1	0723516-05	LCS	50	4.950495	4.950495	Unfiltered	0	100	5	S1	
6	1	0723516-06	LCS	50	4.950495	4.950495	Unfiltered	0	100	5	S1	
7	1	0723516-07	MB	50	50	10	Unfiltered	0	100	10		
8	1	0723516-08	MB	50	50	10	Unfiltered	0	100	10		
9	1	0723516-09	MB	50	50	10	Unfiltered	0	100	10		
10	1	0723516-10	LCS	50	9.900990	9.900990	Unfiltered	0	100	10	S1	
11	1	0723516-11	LCS	50	9.900990	9.900990	Unfiltered	0	100	10	S1	
12	1	0723516-12	LCS	50	9.900990	9.900990	Unfiltered	0	100	10	S1	

Spiked By: Jeff Kujawa Date: 11/28/2007

Witnessed By: Gabriel D. Wagner Date: 11/28/2007

Scin #	Nuclide	ScinID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	648.3020.69	2,386.637	DPN/ml	11/28/07	0.5	ml	RS-003

Comments

UG LLT LOT# 97-070701.

Page 1 of 1 H3 Bench Sheet

Date Printed: 11/28/2007 18:15

Paragon Analytics

LIMS Version: 6.053A

Supersedes: 11/14/07 2:35

# Radiochemistry Prep Worksheet

Paragon Analytics

Prep Batch: 31071128-1

Prep Procedure: H3

**Prep Batch Not Validated!!!**

Review Date:

Reviewed By:

Non-Routine Pre-Treatment? Y / N Batch:

Prep QASS / NCR? Y / N

Prep SOP: PAI 700 Rev: 10

Prep SOP: NONE

Matrix Class: liquid

Prep Analyst: Jeff Kulawa

Prep Date: 11/28/2007

Prep Dept: RS

Balance:

Balance:

Cocktail: UG LLT

Cocktail Pipet: T-002

Allquot Pipet:

Samp Num	Prep Num	LabID	QC Type	Dish No.	Init Aliq ml	Fin Aliq ml	Prep Basis	Water Added(ml)	Moisture(%)	Analysis Vol.(ml)	Standards	Prep Notes
1	1	0723516-01	MB		50	5	Unfiltered	0	100	5		
2	1	0723516-02	MB		50	5	Unfiltered	0	100	5		
3	1	0723516-03	MB		50	5	Unfiltered	0	100	5		
4	1	0723516-04	LCS		50	4.950495	Unfiltered	0	100	5	S1	
5	1	0723516-05	LCS		50	4.950495	Unfiltered	0	100	5	S1	
6	1	0723516-06	LCS		50	4.950495	Unfiltered	0	100	5	S1	
7	1	0723516-07	MB		50	10	Unfiltered	0	100	10		
8	1	0723516-08	MB		50	10	Unfiltered	0	100	10		
9	1	0723516-09	MB		50	10	Unfiltered	0	100	10		
10	1	0723516-10	LCS		50	9.900990	Unfiltered	0	100	10	S1	
11	1	0723516-11	LCS		50	9.900990	Unfiltered	0	100	10	S1	
12	1	0723516-12	LCS		50	9.900990	Unfiltered	0	100	10	S1	

Spiked By: JKh Date: 11/28/07

Witnessed By: GDW Date: 11/28/07

Soln #	Nuclide	SolnID	Prep Conc	Units	Prep Date	Aliquot	Units	Pipet ID
S1	H-3	648.3020.69	2.386.637	DFM/ml	11/28/07	0.5	ml	S1-002

exp. 9/13/08

92%  
PS-003 11/28/07

Comments

UG LLT LOT# 97-070701

Page 1 of 1 H3 Bench Sheet

Date Printed: 11/28/2007 7:35

Paragon Analytics

LIMS Version: 6.093A

Supersedes:

10/1

## TRITIUM RUN LOG

319185

Logbook/Page No.

SOP 700 Rev 1.0

Form 1306r1.doc (3/30/2003)

Date	Workorder/ Sample Number	Column ID	Flask ID	Run No.	Comments	Technician's Initials
11/20/07	0710025-D	N/A	N/A	9	Batch 3H071114-2 800 754 R5	EMF
11/26/07	0723514-1, 7	A	T2	2	Batch 3H071126-1	JH
	-2, 8	A	T7	1		
	-3, 9	B	T8			
	-4, 10	C	T11			
	-5, 11	D	102			
	-6, 12	F	600			
11/29/07	0723516-1, 7	A	102	1	Batch 3H071129-1	JH
	-2, 8	B	600			
	-3, 9	C	T2			
	-4, 10	D	T7			
	-5, 11	E	T8			
	-6, 12	F	T11			
<div style="position: absolute; top: 50%; left: 50%; transform: translate(-50%, -50%);">             11/29/07              11/29/07           </div>						

Reviewed by / Date

JH 11/29/07



PROJECT 699.2613.19

3H

Notebook No. 2613

19

Continued From Page

Prepare a working dilution at approx 75,000 dpm/mL by  
diluting Std 699.2613.19 with DI water

Prepared by  
RG 9/16/05  
Std ID: 699.2613.19  
Description: 3H  
Expiration: 8/11/06  
Activity: 94000.71 dpm/mL  
2s Uncertainty: 676.81 dpm/mL  
Ref. Date: 9/3/98  
Ref Time: N/A  
Prep Date: 7/29/04  
Matrix/Comp: DI Water  
Half Life (y): 1.23E+01  
Requires NCR for ICPT work

1) See Density on page 18 of logbook 2613

2) Transfer Std. 699.2613.19 to a 125 mL Amber glass bottle

Mass of bottle w/o lid  
Mass of bottle + Std  
Net mass of Std:

94.7389g  
96.6785g  
1.9396g

Bal #12

3) Add DI water to final dilution

Reverification Log	Analysis Date	Initials	Expiration Date
9/24/06	RG	9/24/06	
9/24/06	GLS	9/24/06	

Mass of bottle w/o lid from above  
Mass of bottle Std + DI water  
Net mass of Std + DI water

94.7389g  
192.4676g  
97.6687g

Bal #12

4) Final Activity Calculation

$$(634.7 \text{ kBq/g}) (4.815\%) (1000 \text{ Bq/kBq}) (1.9396 \text{ g}) (1.9396 \text{ g})$$

$$(38.5765 \text{ g}) (97.6687 \text{ g})$$

$$= 911,000.71 \text{ DPM/mL}$$

SD 7/31/03

Std ID: 699.2613.19

Description: 3H  
Expiration: 7/28/04  
Activity: 94000.71 dpm/mL

2s Uncertainty: 676.81 dpm/mL

Ref. Date: 9/3/98

Ref Time: N/A

Prep Date: 7/28/03 Prep by: JRK

Matrix/Comp: DI Water

Half Life (y): 1.23E+01

Description: 3H  
Expiration: 7/30/05  
Activity: 94000.71 dpm/mL

2s Uncertainty: 676.81 dpm/mL

Ref. Date: 9/3/98

Ref Time: N/A

Prep Date: 7/29/04 Prep by: JLK

Matrix/Comp: DI Water

Half Life (y): 1.23E+01

*Chad Wagle*  
Signed

SD 9/1/03

7/28/03

Date

*[Signature]*  
Signed

8/1/03  
Date

Prepare a 1<sup>st</sup> dilution of Amptole 699 by diluting with DI water.

1) Determine the density of DI water

Mass of empty Class A <sup>100 mL</sup> volumetric flask	107.7950 g	Balance
Mass of flask + 100 mL of DI water	167.3755 g	Balance
Net mass of DI water	99.5799 g	

$$\rho = 0.9958 \text{ g/mL}$$

2) Transfer contents of ampoule 699 into a 40 mL amber glass VOA vial

Mass of VOA vial w/o lid	24.3883 g	Balance
Mass of opened ampoule before transfer	40.8432 g	Balance
Mass of opened ampoule after transfer	36.0281 g	Balance
Net mass of Std transferred	4.8151 g	

3) Add DI water to final dilution

Mass of VOA vial <sup>w/ lid</sup> from above	24.3883 g	Balance
Mass of VOA vial, std + DI water	62.9618 g	Balance
Net mass of Std + DI water	38.5735 g	

4) Final Activity Calculation

$$(634.7 \text{ KBq/g}) (4.8151 \text{ g}) \left( \frac{600 \text{ dpm/Bq}}{1000 \text{ Bq}} \right) \left( \frac{1000 \text{ Bq}}{1 \text{ MBq}} \right) (0.9958 \text{ g/mL})$$

$$38.5735 \text{ g}$$

$$= 4,733,412.57 \frac{\text{dpm}}{\text{mL}}$$

U.S. Department of Commerce  
National Institute of Standards  
and Technology  
SRM 4827F  
Hydrogen-3  
<4 MBq in distilled water

CAUTION  
RADIOACTIVE



Continued on Page

*Chad W. [Signature]*  
Signed

7/28/03  
Date

Next Hand Understood By  
*[Signature]*  
Signed

7/31/03  
Date



# National Institute of Standards & Technology

## Certificate

PAF ID 0099  
rec'd 5-09-03

### Standard Reference Material 4927F Hydrogen-3 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive hydrogen-3, as water, in 5 mL of distilled water. The solution is contained in a flame-sealed NIST borosilicate-glass ampoule. The SRM is intended for the calibration of beta-particle counting instruments and for the monitoring of radiochemical procedures.

#### Radiological Hazard

The SRM ampoule contains hydrogen-3 with a total activity of approximately 3.2 MBq. Hydrogen-3 decays by beta-particle emission. None of the beta particles escape from the SRM ampoule. During the decay process no photons are emitted. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]\*. There is no detectable external radiation. The SRM should be used only by persons qualified to handle radioactive material.

#### Chemical Hazard

The SRM ampoule contains only distilled water. There is no chemical hazard. If the ampoule is to be opened to transfer the solution, the recommended procedure is given on page 2.

#### Storage and Handling

The SRM should be stored and used at a temperature between 5 and 65 °C. The solution in an unopened ampoule should remain stable and homogeneous until at least September 2008.

The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material. If the ampoule is transported it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. The solution in the ampoule is a dangerous good (hazardous material) because of the radioactivity.

#### Preparation

This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, L.R. Karam, Group Leader. The overall technical direction and physical measurements leading to certification were provided by L.L. Lucas and M.P. Unterwieser of the Radioactivity Group.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.W.L. Thomas.

Bert M. Coursey, Chief  
Ionizing Radiation Division

Gaithersburg, Maryland 20899  
June 1999  
Half-life and text revised October 2000

Nancy M. Trahey, Chief  
Standard Reference Materials Program

#### Recommended Procedure for Opening the SRM Ampoule

- 1) If the SRM solution is to be diluted, it is recommended that the diluting solution have a composition comparable to that of the SRM solution.
- 2) Wear eye protection, gloves, and protective clothing and work over a tray with absorbent paper in it. Work in a fume hood.
- 3) Shake the ampoule to wet all of the inside surface of the ampoule. Return the ampoule to the upright position.
- 4) Check that all of the liquid has drained out of the neck of the ampoule. If necessary, gently tap the neck to speed the process.
- 5) Holding the ampoule upright, score the narrowest part of the neck with a scribe or diamond pencil.
- 6) Lightly wet the scored line. This reduces the crack propagation velocity and makes for a cleaner break.
- 7) Hold the ampoule upright with a paper towel, a wiper, or a support jig. Position the scored line away from you. Using a paper towel or wiper to avoid contamination, snap off the top of the ampoule by pressing the narrowest part of the neck away from you while pulling the tip of the ampoule towards you.
- 8) Transfer the solution from the ampoule using a pycnometer or a pipet with dispenser handle. NEVER PIPETTE BY MOUTH.
- 9) Seal any unused SRM solution in a flame-sealed glass ampoule, if possible, to minimize the evaporation loss.

See also reference [4]\*.

# PROPERTIES OF SRM 4927F

## Certified values

Solution density	$(0.998 \pm 0.002) \text{ g} \cdot \text{mL}^{-1}$ at 20.0 °C [b]*
Radionuclide	Hydrogen-3
Reference time	1200 EST, 3 September 1998
Massic activity of the solution [c]	$634.7 \text{ kBq} \cdot \text{g}^{-1}$
Relative expanded uncertainty ( $k=2$ )	0.72% [d] [e]

## Uncertified values

Physical Properties:			
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule		
Ampoule specifications	Body outside diameter	$(16.5 \pm 0.5) \text{ mm}$	
	Wall Thickness	$(0.60 \pm 0.04) \text{ mm}$	
	Barium content	Less than 2.5%	
	Lead-oxide content	Less than 0.02%	
	Other heavy elements	Trace quantities	
Solution mass	Approximately 5.0 g		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration ( $\text{mol} \cdot \text{L}^{-1}$ )	Mass Fraction ( $\text{g} \cdot \text{g}^{-1}$ )
	$\text{H}_2\text{O}$ $^3\text{HHO}$	55 $6 \times 10^{-7}$	1.00 $1 \times 10^{-8}$
Radiological Properties:			
Radionuclidic impurities	None detected [f]		
Half lives used	Hydrogen-3: $(4500 \pm 8) \text{ d}$ [g]		
Calibration method and measuring instrument(s)	4 $\pi$ B gas counting of SRM 4927B using the NIST length-compensated internal gas proportional counters and intercomparison of SRMs 4927B/4927F using two 4 $\pi$ B liquid-scintillation counting systems [h]		



EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [d]\*

Input Quantity $x_i$ , the source of uncertainty (and individual uncertainty components where appropriate)	Method Used To Evaluate $u(x_i)$ , the standard uncertainty of $x_i$ (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$ , (%) [f]	Relative Sensitivity Factor, $ \partial y/\partial x_i  \cdot$ $(x_i/y)$ [g]	Relative Uncertainty Of Output Quantity, $u(y)/y$ , (%) [k]
Massic count rate of SRM 4927E, corrected for background and decay [h]	Standard deviation of the mean for 23 sets of gas counting measurements (A)	0.18	1.0	0.18
Gram-mole measurements	Estimated (B)	0.20	1.0	0.20
Live-time [p]	Estimated (B)	0.10	1.0	0.10
Extrapolation of count-rate-versus-energy to zero energy	Estimated (B)	0.20	1.0	0.20
Half life of H-3	Standard uncertainty of the half life (A)	0.18 [m]	0.009 [n]	0.002
Liquid-scintillation intercomparison of SRM 4927F and SRM 4927E	Standard deviation of the mean for 7 sets of liquid-scintillation measurements (A)	0.06	1.0	0.06
Radiochemical impurities	Limit of detection (B) [q]	100.	0.0005	0.05
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)				0.36
Coverage Factor, $k$				<u>2</u>
Relative Expanded Uncertainty of the Output Quantity, $U/y$ , (%)				0.72

## NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One  $\mu\text{Sv}$  is equal to 0.1 mrem.  
 Distance from Ampoule (cm):           1       30     100  
 Approximate Dose Rate ( $\mu\text{Sv/h}$ ):   <0.1 (Not detectable)
- [b] The stated uncertainty is two times the standard uncertainty.
- [c] Massic activity is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [d] The reported value,  $y$ , of massic activity (activity per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as  $y = f(x_1, x_2, x_3, \dots, x_n)$ , where  $f$  is a mathematical function derived from the assumed model of the measurement process.
- The value,  $x_i$ , used for each input quantity  $i$  has a standard uncertainty,  $u(x_i)$ , that generates a corresponding uncertainty in  $y$ ,  $u_i(y) \equiv |\partial y / \partial x_i| \cdot u(x_i)$ , called a component of combined standard uncertainty of  $y$ .
- The combined standard uncertainty of  $y$ ,  $u_c(y)$ , is the positive square root of the sum of the squares of the components of combined standard uncertainty.
- The combined standard uncertainty is multiplied by a coverage factor of  $k = 2$  to obtain  $U$ , the expanded uncertainty of  $y$ .
- Since it can be assumed that the possible estimated values of the massic activity are approximately normally distributed with approximate standard deviation  $u_c(y)$ , the unknown value of the massic activity is believed to lie in the interval  $y \pm U$  with a level of confidence of approximately 95 percent.
- For further information on the expression of uncertainties, see references [2] and [3].
- [e] The value of each standard uncertainty component, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval  $U/2$  to  $2U$  (i.e., within a factor of 2 of the estimated value).
- [f] The estimated limit of detection for radionuclidic impurities is  $300 \text{ Bq} \cdot \text{g}^{-1}$ .
- [g] The stated uncertainty is the standard uncertainty. See reference [5].
- [h] Extensive gas-counting measurements were made on the SRM 4927E solution during 1998 and 1999. The SRM 4927F solution was intercompared with the SRM 4927E solution using liquid-scintillation counting.
- [i] Relative standard uncertainty of the input quantity  $x_i$ .

- [j] The relative change in the output quantity  $y$  divided by the relative change in the input quantity  $x_i$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 1.0$ , then a 1% change in  $x_i$  results in a 1% change in  $y$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 0.05$ , then a 1% change in  $x_i$  results in a 0.05% change in  $y$ .
- [k] Relative component of combined standard uncertainty of output quantity, rounded to two significant figures or less. The relative component of combined standard uncertainty of  $y$  is given by  $u_c(y)/y = |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$ . The numerical values of  $u(x_i)/x_i$ ,  $|\partial y/\partial x_i| \cdot (x_i/y)$ , and  $u_c(y)/y$ , all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.
- [m] The relative standard uncertainty of  $\lambda \cdot t$  is determined by the relative standard uncertainty of  $\lambda$  (i.e., of the half life). The relative standard uncertainty of  $t$  is negligible.
- [n]  $|\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$
- [p] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.
- [q] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e.  $u(x_i)/x_i = 100\%$ .  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of H-3})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of H-3})\}$ . Thus  $u_c(y)/y$  is the relative change in  $y$  if the impurity were present with a massic activity equal to the estimated limit of detection.

#### REFERENCES

- [1] International Organization for Standardization (ISO), *ISO Standards Handbook - Quantities and Units*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900.
- [2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900. (Listed under ISO miscellaneous publications as "ISO Guide to the Expression 1993".)
- [3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.
- [4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814, U.S.A.
- [5] L.L. Lucas and M.P. Unterwiesing, *Comprehensive Review and Critical Evaluation of the Half-Life of Tritium*, J. Res. Natl. Inst. Stand. Technol. 105, 541-549 (2000).



Prepare a working level dilution of approx. 400 dpm/mL from 648.2382.75 diluting with DI water.

① Determine the density of water

Mass of empty 100mL class A volumetric flask: 68.5644g

Mass of flask + 100mL DI water: 168.2311g

Net mass of water: 99.6667g

$$\rho = 0.997 \text{ g/mL}$$

Bal 12



② <sup>Ag-3</sup> ~~Mass~~ Transfer standard to 500mL Amber bottle

3/8/07

mass of standard in 100mL poly cup: 12.8145g

mass of poly cup after transfer: 7.4323g

Net mass of standard transferred: 5.3822g

Bal 12



③ Dilute with DI water

Mass of Amber bottle (500mL): 262.87g

Mass of bottle + standard + DI water: 759.3g

Net mass of standard + DI water: 496.43g

Bal 26



④ Final activity calculation

\* Note: Density of 648.2382.75 is 0.9958 g/mL per 3/8/07

$$\frac{(369.530.45 \text{ dpm/mL})(5.3822 \text{ g})(0.997 \text{ g/mL})}{(0.9958 \text{ g/mL})(496.43 \text{ g})} = 4011.21 \text{ dpm/mL}$$

Std ID: 648.3020.69

RG 3/20/07

Description: H-3

Expiration: 3/12/2008

Activity: 4011.21 dpm/mL

2s Uncertainty: 28.88 dpm/mL

Ref. Date: 9/3/1998

Ref Time: N/A

Prep Date: 3/8/2007 Prep by: AB

Matrix/Comp. DI H2O

Half Life (y): 1.23E+01

Reverification Log		
Analysis Date	Initials	Expiration Date
8/13/07	JLB	8/13/08
2/29/08	NEC	2/25/09

RG 3/20/07

RG 3/20/07

Continued on Page

Read and Understood By

AB Bailey  
Signed

3/8/07  
Date

Renee Haller  
Signed

3/20/07  
Date

PROJECT 048.2382.75 34

Notebook No. 2382

75

Continued From Page

Prepare a working <sup>10 dilution</sup> solution (of approximately 250 ppm) of tritium standard using RSC #6442 and diluting with DI water.

1) Determine the density of DI water:

Mass of empty 100 mL Class A volumetric flask

67.1522 g Bal #13

Mass of flask + water

116.7351 g

Net mass of 100 mL H<sub>2</sub>O

49.5829 g

 $\rho = .9958 \text{ g/mL}$ 

2) Transfer contents of Ampoule SRM 4427F to a

500 mL glass amber bottle

mass of bottle w/o lid

255.04 g Bal #26

mass of open ampoule + 50 mL beaker

40.9075 g Bal #26

mass of empty ampoule + 50 mL beaker

36.0145 g

Net mass of SRM

4.8927 g

3) Dilute SRM with DI water

mass of bottle w/o lid

255.04 g Bal #26

mass of bottle, SRM, + DI water

757.1 g

Net mass of SRM + DI water

502.1 g

4. Final Activity Calculation:

$$(634.7 \text{ kBq/g}) (1000 \text{ g/L}) (60 \text{ DPM/g}) (4.8927 \text{ g}) (.9958 \text{ g/mL})$$

502.1 g

 $= 369,530.45 \frac{\text{DPM}}{\text{mL}}$ 

U.S. Department of Commerce  
National Institute of Standards  
and Technology  
SRM 4427F  
Hydrogen-3

~ 55 MBq in distilled water

CAUTION  
RADIOACTIVE

Read and Understood By

Continued on Page

*Chad [Signature]*  
Signed

3/27/03  
Date

*Renee Hallberg*  
Signed

3/27/03  
Date



# National Institute of Standards & Technology

## Certificate

PAID 0648  
1000 17-04-00

### Standard Reference Material 4927F Hydrogen-3 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive hydrogen-3, as water, in 5 mL of distilled water. The solution is contained in a flame-sealed NIST borosilicate-glass ampoule. The SRM is intended for the calibration of beta-particle counting instruments and for the monitoring of radiochemical procedures.

#### Radiological Hazard

The SRM ampoule contains hydrogen-3 with a total activity of approximately 3.2 MBq. Hydrogen-3 decays by beta-particle emission. None of the beta particles escape from the SRM ampoule. During the decay process no photons are emitted. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]\*. There is no detectable external radiation. The SRM should be used only by persons qualified to handle radioactive material.

#### Chemical Hazard

The SRM ampoule contains only distilled water. There is no chemical hazard. If the ampoule is to be opened to transfer the solution, the recommended procedure is given on page 2.

#### Storage and Handling

The SRM should be stored and used at a temperature between 5 and 65 °C. The solution in an unopened ampoule should remain stable and homogeneous until at least September 2008.

The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material. If the ampoule is transported it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. The solution in the ampoule is a dangerous good (hazardous material) because of the radioactivity.

#### Preparation

This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, L.R. Karam, Group Leader. The overall technical direction and physical measurements leading to certification were provided by L.L. Lucas and M.P. Unterwieser of the Radioactivity Group.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.W.L. Thomas.

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Gaithersburg, Maryland 20899  
June 1999  
Half-life and text revised October 2000

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Standard Reference Materials Program

#### Recommended Procedure for Opening the SRM Ampoule

- 1) If the SRM solution is to be diluted, it is recommended that the diluting solution have a composition comparable to that of the SRM solution.
- 2) Wear eye protection, gloves, and protective clothing and work over a tray with absorbent paper in it. Work in a fume hood.
- 3) Shake the ampoule to wet all of the inside surface of the ampoule. Return the ampoule to the upright position.
- 4) Check that all of the liquid has drained out of the neck of the ampoule. If necessary, gently tap the neck to speed the process.
- 5) Holding the ampoule upright, score the narrowest part of the neck with a scribe or diamond pencil.
- 6) Lightly wet the scored line. This reduces the crack propagation velocity and makes for a cleaner break.
- 7) Hold the ampoule upright with a paper towel, a wiper, or a support jig. Position the scored line away from you. Using a paper towel or wiper to avoid contamination, snap off the top of the ampoule by pressing the narrowest part of the neck away from you while pulling the tip of the ampoule towards you.
- 8) Transfer the solution from the ampoule using a pycnometer or a pipet with dispenser handle. NEVER PIPETTE BY MOUTH.
- 9) Seal any unused SRM solution in a flame-sealed glass ampoule, if possible, to minimize the evaporation loss.

See also reference [4]\*.

# PROPERTIES OF SRM 4927F

## Certified values

Solution density	$(0.998 \pm 0.002) \text{ g} \cdot \text{mL}^{-1}$ at 20.0 °C [b]*
Radionuclide	Hydrogen-3
Reference time	1200 EST, 3 September 1998
Massic activity of the solution [c]	$634.7 \text{ kBq} \cdot \text{g}^{-1}$
Relative expanded uncertainty ( $k=2$ )	0.72% [d] [e]

## Uncertified values

Physical Properties:			
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule		
Ampoule specifications	Body outside diameter	$(16.5 \pm 0.5) \text{ mm}$	
	Wall Thickness	$(0.60 \pm 0.04) \text{ mm}$	
	Barium content	Less than 2.5%	
	Lead-oxide content	Less than 0.02%	
	Other heavy elements	Trace quantities	
Solution mass	Approximately 5.0 g		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration ( $\text{mol} \cdot \text{L}^{-1}$ )	Mass Fraction ( $\text{g} \cdot \text{g}^{-1}$ )
	$\text{H}_2\text{O}$ $^3\text{HHO}$	55 $6 \times 10^{-7}$	1.00 $1 \times 10^{-8}$
Radiological Properties:			
Radiomclidic impurities	None detected [f]		
Half lives used	Hydrogen-3: $(4500 \pm 8) \text{ d}$ [g]		
Calibration method and measuring instrument(s)	4 $\pi\beta$ gas counting of SRM 4927E using the NIST length-compensated internal gas proportional counters and intercomparison of SRMs 4927E/4927F using two 4 $\pi\beta$ liquid-scintillation counting systems [h]		

EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [d]\*

Input Quantity $x_i$ , the source of uncertainty  (and individual uncertainty components where appropriate)	Method Used To Evaluate $u(x_i)$ , the standard uncertainty of $x_i$ (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$ , (%) [i]	Relative Sensitivity Factor, $ \partial y/\partial x_i  \cdot$ $(x_i/y)$ [j]	Relative Uncertainty Of Output Quantity, $u(y)/y$ , (%) [k]
Massic count rate of SRM 4927E, corrected for background and decay [h]	Standard deviation of the mean for 23 sets of gas counting measurements (A)	0.18	1.0	0.18
Gram-mole measurements	Estimated (B)	0.20	1.0	0.20
Live-time [p]	Estimated (B)	0.10	1.0	0.10
Extrapolation of count-rate-versus-energy to zero energy	Estimated (B)	0.20	1.0	0.20
Half life of H-3	Standard uncertainty of the half life (A)	0.18 [m]	0.009 [n]	0.002
Liquid-scintillation intercomparison of SRM 4927F and SRM 4927E	Standard deviation of the mean for 7 sets of liquid-scintillation measurements (A)	0.06	1.0	0.06
Radiometric impurities	Limit of detection (B) [q]	100	0.0005	0.05
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)				0.36
Coverage Factor, $k$				<u>2</u>
Relative Expanded Uncertainty of the Output Quantity, $U(y)/y$ , (%)				0.72



## NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One  $\mu\text{Sv}$  is equal to 0.1 mrem.  
 Distance from Amponle (cm): 1 30 100  
 Approximate Dose Rate ( $\mu\text{Sv/h}$ ): <0.1 (Not detectable)
- [b] The stated uncertainty is two times the standard uncertainty.
- [c] Massic activity is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [d] The reported value,  $y$ , of massic activity (activity per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as  $y = f(x_1, x_2, x_3, \dots, x_n)$ , where  $f$  is a mathematical function derived from the assumed model of the measurement process.
- The value,  $x_i$ , used for each input quantity  $i$  has a standard uncertainty,  $u(x_i)$ , that generates a corresponding uncertainty in  $y$ ,  $u_i(y) = |\partial y / \partial x_i| \cdot u(x_i)$ , called a component of combined standard uncertainty of  $y$ .
- The combined standard uncertainty of  $y$ ,  $u_c(y)$ , is the positive square root of the sum of the squares of the components of combined standard uncertainty.
- The combined standard uncertainty is multiplied by a coverage factor of  $k = 2$  to obtain  $U$ , the expanded uncertainty of  $y$ .
- Since it can be assumed that the possible estimated values of the massic activity are approximately normally distributed with approximate standard deviation  $u_c(y)$ , the unknown value of the massic activity is believed to lie in the interval  $y \pm U$  with a level of confidence of approximately 95 percent.
- For further information on the expression of uncertainties, see references [2] and [3].
- [e] The value of each standard uncertainty component, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval  $U/2$  to  $2U$  (i.e., within a factor of 2 of the estimated value).
- [f] The estimated limit of detection for radionuclides is  $300 \text{ Bq} \cdot \text{g}^{-1}$ .
- [g] The stated uncertainty is the standard uncertainty. See reference [5].
- [h] Extensive gas-counting measurements were made on the SRM 4927B solution during 1998 and 1999. The SRM 4927F solution was intercompared with the SRM 4927B solution using liquid-scintillation counting.
- [i] Relative standard uncertainty of the input quantity  $x_i$ .

- [j] The relative change in the output quantity  $y$  divided by the relative change in the input quantity  $x_i$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 1.0$ , then a 1% change in  $x_i$  results in a 1% change in  $y$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 0.05$ , then a 1% change in  $x_i$  results in a 0.05% change in  $y$ .
- [k] Relative component of combined standard uncertainty of output quantity  $y$ , rounded to two significant figures or less. The relative component of combined standard uncertainty of  $y$  is given by  $u_i(y)/y = |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$ . The numerical values of  $u(x_i)/x_i$ ,  $|\partial y/\partial x_i| \cdot (x_i/y)$ , and  $u_i(y)/y$ , all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.
- [m] The relative standard uncertainty of  $\lambda \cdot t$  is determined by the relative standard uncertainty of  $\lambda$  (i.e., of the half life). The relative standard uncertainty of  $t$  is negligible.
- [n]  $|\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$
- [p] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.
- [q] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e.  $u(x_i)/x_i = 100\%$ .  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of H-3})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of H-3})\}$ . Thus  $u_i(y)/y$  is the relative change in  $y$  if the impurity were present with a massic activity equal to the estimated limit of detection.

#### REFERENCES

- [1] International Organization for Standardization (ISO), *ISO Standards Handbook - Quantities and Units*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900.
- [2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900. (Listed under ISO miscellaneous publications as "ISO Guide to the Expression 1993".)
- [3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.
- [4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814 U.S.A.
- [5] L.L. Lucas and M.P. Unterwieser, *Comprehensive Review and Critical Evaluation of the Half-Life of Tritium*, J. Res. Natl. Inst. Stand. Technol. 105, 541-549 (2000).



DAILY INSTRUMENT PERFORMANCE CHECKS - LS6500				
	Daily IPCs consist of the following standards;			
	Efficiency Check			
	Beckman Tritium Standard	Beckman C-14 Standard	PAI Reagent Blank (10-mL DI Water + 10 mL UG LL1	
	Lot HNP0509	Lot CNP3311	7/24/2008	
	105800 dpm	101000 dpm		
	10/29/2003 REF	10/29/2003 REF		
	10/29/2008 EXP	10/29/2008 EXP		
Interim Control Limits				8/19/2008
	<u>Decay Corrected Tritium</u>	<u>Carbon-14</u>	<u>Reagent Blank</u>	<u>Quench Factor</u>
UCL	49552.49	67094.79	19.79	149.5
Mean Value	47192.85	63899.80	17.00	142.4
LCL	44833.20	60704.81	12.21	135.3

Obs	Date	Decay Corrected			Reagent			Quench	
		<u>H-3 CPM</u>	<u>H-CPM</u>		<u>C-14 CPM</u>	<u>H-3 Bkg CPM</u>		<u>H#</u>	
11	8/27/2008	35487.0	46608.01	OK	65240.7	OK	15.20	OK	144.5
12	8/28/2008	36390.3	47801.78	OK	65118.3	OK	13.80	OK	146.3

# Liquid Scintillation Counter

## Quality Control Data

### Daily Instrument Performance Checks

DAILY INSTRUMENT PERFORMANCE CHECKS - LS6500					
Daily IPCs consist of the following standards;  Efficiency Check  Beckman Tritium Standard Lot HPE0410  104700 dpm  3/19/2008 REF  3/19/2013 EXP	Beckman C-14 Standard				
	Lot CPE2711		PAI Reagent Blank (10-mL DI Water + 10 mL UG LLT)		
	100,100 dpm		7/24/2008 ref		
	3/19/2008 REF		7/24/2009 exp		
	3/19/2013 EXP				
Interim Control Limits					10/20/2008
	<u>Decay Corrected Tritium</u>	<u>Carbon-14</u>	<u>Reagent Blank</u>	<u>Quench Factor</u>	
UCL	55138.40	69099.80	19.79	153.0	
Mean Value	52512.76	65809.34	16.00	145.7	
LCL	49887.12	62518.87	12.21	138.4	

Obs	Date	Decay Corrected			Reagent			Quench		
		H-3 CPM	H-CPM		C-14 CPM		H-3 Bkg CPM	H#		
35	11/21/08	50292.1	52248.91	OK	64131	OK	13.80	OK	149.9	OK
36	11/23/08	48996.3	50918.43	OK	63184.2	OK	15.40	OK	151	OK