METHOD VALIDATION REPORT

Secondary (Lab) Standard Validation for the Analysis of δ^{18} O in Water Samples Using the GasBench and IRMS

Date: December 18, 2009

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SUMMARY

International Standards (IAEA Reference Material)	SLAP2 – Standard Light Antarctic Precipitation 2 GISP – Greenland Ice Sheet Precipitation VSMOW2– Vienna Standard Mean Ocean Water 2						
International Standard (Primary Standard)	Primary Standard		$\delta^{18}O_{VSMOW/SLAP \%}$				
Given Values	SLAP2			-55.5			
	GISP VSMOW2		-24.8				
			0.00				
Primary Standard Experimental Values	Primary Standard	$\delta^{18}O_{VSMOW/SLAP \%}$	<u>S.D.</u>	<u>%CV</u>	<u>%Acc</u>	<u>n</u>	
and Statistics	SLAP2	-55.6	0.0781	0.14	100.11	9	
	GISP	-24.8	0.101	0.41	100.11	9	
	VSMOW2	-0.042	0.0385	91.7*	*	9	
	* Value skewed due to zero being the target value.						
(Secondary) Standards	 Vostok: Originally obtained as an ice core from Vostok Ice Core Team (member G. Domack) which subsequently melted due to freezer malfunction (depth of core given below) Sylvan Beach Tap: B. Wegter (employee Hamilton College) home Bottle Distilled: Fisher, Optima LCMS Grade, Lot: 086933 Well: D. Tewksbury (employee Hamilton College) home Science Center Tap: Hamilton College, Rm. 1036 Deuterium Prepared Lab Standard (see preparation section) Millipore RO: Science Center Rm. 2093 			ction			
Lab (Secondary)	Secondary Standard).	%CV	<u>n</u>	
Standard	Science Center RO	-10.2	0.04	73	0.46	27	
Experimentally Determined δ^{18} O	Vostok	-53.4	0.05	96	0.11	30	
Values and Statistics	Sylvan Beach Tap	-6.60	0.04	86	0.74	30	
values and Statistics	Bottle Distilled	-7.26	0.04		0.65	30	
	Well	-11.4	0.05	50	0.48	30	
	Science Center Tap	-9.65	0.06		0.62	30	
	Lab Standard	-10.3	0.05		0.55	30	
	Millipore RO	-10.2	0.05	40	0.53	27	
Sample Analysis	200 µL						
Volume							

SIGNATURE PAGE

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Written by:

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Date

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1. INTRODUCTION

This report describes the qualification/validation process for Water δ^{18} O Secondary (Lab) Standards using the automated CO₂ equilibration Gas Bench Isotope Ratio Mass Spectrometry technique. Various water samples were analyzed to be evaluated as possible Secondary (Lab) standards. Three international (primary) standards were included in the analyses, they are GISP, SLAP2 and VSMOW2. The goal of the analysis was to identify the laboratory standards which provided acceptable experimental precision and encompassed the δ^{18} O ranges expected for samples submitted for analysis. The Lab Standards identified in the Summary section of this report fulfilled these requirements.

2. EXPERIMENTAL

2.1. CHEMICALS AND MATERIALS

Eight water samples were chosen for this secondary (Lab) standard determination validation, as well as the three international (or primary) standards. The eight laboratory standard candidates were as follows:

- 1. Science Center RO
- 2. Vostok

Bottle # (arbitrarily assigned)	Depth (meters)
Dottie # (ai biti ai iiy assigned)	· · · · · ·
1	$3553.085 \rightarrow 3553.185$
2	$3553.325 \rightarrow 3553.425$
3	3560.635 → 3560.685
4	3556.38 → 3556.48
5	3548.77 → 3548.82
6	3548.77 → 3548.82
7	3548.77 → 3548.82
8	$3548.77 \rightarrow 3548.82$

(Core Bottles denoting depth)

- 3. Sylvan Beach Tap
- 4. Bottle Distilled
- 5. Well
- 6. Science Center Tap
- 7. Prepared Deuterium Laboratory Standard (50 ppm D₂O)
- 8. Millipore RO

Note: The 50 ppm (v/v) D_2O laboratory standard was prepared as follows:

- ~100 mL of Science Center RO water were first placed into a 1000 mL volumetric flask
- Using a pipette, exactly 50 μ L of D₂O (Acros D₂O 100.0 Atom% D, Lot A020127801) were then placed into the volumetric flask
- Science Center RO water was then added to the flask to the mark
- A stir bar was inserted and the solution mixed for ~ 1 day

Note: This standard was actually prepared for the Deuterium validation but it was decided to analyze this sample knowing that it should give a value exactly as the Science Center RO water that was also analyzed.

The three international standards were as follows:

- 1. SLAP2
- 2. GISP
- 3. VSMOW2

Other than the prepared lab standard, all waters were used neat "as received".

A 0.3% CO_2 in Helium was used as the equilibration gas which allowed for oxygen atom incorporation from the water sample into the CO_2 gas introduced to each sample's headspace.

Other materials were as follows:

Capillary Column – Varian PN: CP7551, PLOT Fused Silica, CP-PoraPLOT Q, length - 27.5 meter (including 2.5 m particle trap), (0.32 mm I.D., 0.45 mm O.D., 10 mm film thickness) held at 70°C. (Push "P" button on oven controller once and then up or down arrow to set temperature.)

Exetainer Vials – 12 mL Borosilicate, obtained from LabConco with vial caps and disposable septa.

Valco Sample Loop in GasBench - 100 µL

GasBench Sample Block – set at 30°C. (Push "P" button on oven controller twice and then up or down arrow to set temperature.)

He Gas - Grade 5.0 (50 psi tank gauge, 13-14 psi GasBench gauge)

0.3% CO₂ Gas (Grade 4.5) in Helium - Grade 4.6, P/N 105-MIXZW300C. (50 psi tank gauge (quick-connect to yellow w/green stripe plumbed line), adjust to give ~ 125 ml/min flush fill rate, check at vent of FlushFill needle during the FlushFill event.)

 CO_2 Reference Gas - Grade 4.5 (35 psi tank gauge, 30 – 35 psi GasBench gauge, adjust pressure at GB gauge to give ~ 7 – 8 volts m/z 44 signal, cup 2)

Pipettor – Finnpipette 40 – 200 µL maximum range, S/N J57232 (Calibrated – 12/07)

Pipettor Tips – Eppendorf – "Yellow", capacity up to 200 µL (Fisher # 02-707-500)

2.2. INSTRUMENTATION (IRMS, GASBENCH AND PAL)

The IRMS instrument is a Thermo Scientific Delta V Advantage along with a ThermoFinnigan GasBench III and CTC Analytics PAL autosampler system. (The GasBench unit is equipped with a self-contained continuous flow interface.)

IRMS Data Acquisition System: Isodat 2.5 Gas Isotope Ratio MS Software

Acquisition - Used for running the analysis (acquiring data).

Workspace – Used for analysis setup, methods and sequence development, and data review.

Instrument Control – Used to monitor and control various aspects of the instrument.

2.3. ANALYSIS PROCEDURE, SAMPLE PREPARATION AND INSTRUMENT CONDITIONS

Analysis Procedure

Four analysis days (three Primary standard to Secondary standard evaluations and one Secondary to Primary standard evaluation) were performed during the course of the validation. Three of the days consisted of 96 samples and the Secondary to Primary standard evaluation consisted of 83 samples. (It should be noted that ³H sample analysis can be performed on samples that have previously been evaluated for ¹⁸O but not vice versa.)

Nine peaks (consisting of ion current for m/z 44, m/z 45 and m/z 46) of decreasing signal are obtained for each sample (in addition to four reference pulses). The first peak is omitted (due to potential detector saturation) and the statistics (average, S.D., % accuracy, etc.) are generated on the $\delta^{18}O_{00}$ values given by the Isodat software on the remaining eight peaks. The final $\delta^{18}O_{00}$ values and associated statistical parameters given for each water sample were calculated two ways: using the average $\delta^{18}O_{00}$ value of the eight peaks for each sample (intra) and using each $\delta^{18}O_{00}$ value for every peak in each sample (inter). This latter method provided a much bigger population of experimental results (eight values per individual sample) than just using one value (average of eight values) per sample. Both statistical treatments of data yielded essentially identical results for each water sample given in the Summary.

Sample Preparation

The exetainer sample tubes were cleaned by washing in a soap bath and followed by multiple Science Center RO water rinses. Next, the vials were placed in a RO water bath to soak (as a final rinse) at least overnight. Each vial was then removed from the bath and given an acetone rinse. The vials were then placed into an oven to be baked out. The oven was set at ~ 150° C, and the vials were left in at least overnight. After baking, the vials were wrapped in new, clean aluminum foil for storage.

The sample preparation was as follows:

- Into a clean, dry and labeled exetainer vial, 200 µL of water sample were placed using a pipette. (Sample blanks did not contain the water.)
- A cap with septa was then placed on the exetainer tube to seal it.
- Vials were placed into the GasBench sample block (maintained at ~ 30°C) and the cover was secured.
- Each sample vial was then flush-filled with 0.3% CO₂ in Helium gas before the analysis.
 - Attach the two flush-fill needles to the PAL autosampler. Ensure that the needles will pierce the septa and not hit the plastic cap by manually checking alignment (Menu, Utilities, Tray (Select 02), Move to 001).
 - Check each needle's point to verify that they are not bent-over and straighten if necessary.
 - Turn the T-valve so it points away from the GasBench (towards the ConFlo).

- In Isodat Acquisition, verify instrument configuration is set for GasBench+PAL, click the mouse on the gasbench flush-fill button in the GasBench area, this will purge the 0.3% CO₂ in Helium gas flush-fill line. Note: If Instrument Control is open, always close it prior to using Isodat Acquisition.
- \circ Allow the 0.3% CO₂ in Helium gas line to purge for ~ 15 minutes.
- Use the *FlushFill_6min_180.seq* as a template (in Workspace), create a flush-fill sequence for the appropriate number of samples.
- Ensure the sequence contains the correct method, e.g., *Vial_Flush_6min.met*.
- Ensure the use of an appropriate AS Method, **Internal No 1**, (A200S-1) 6 injections of 61 seconds each (see Figure 2).
- $\circ~$ On CTC gameboy, ensure 10 μL syringe is selected (Menu, Change syringe).
- In Acquisition, start the flush-fill sequence just created. Identify the folder for the data with the date and type of analysis. Note: To minimize potential computer issues, it is recommended to reset the computer before starting any extended analysis sequence.
- \circ Once started, verify the flush-fill flow rate by placing a flow meter onto the vent tube of the flush-fill needle (check this on both needles!), the flow rate should be ~ 125 mL/min.
- When the Helium flush-fill has been completed, turn the T-valve back 90° to point to the back wall and shut off the 0.3% CO₂ gas in Helium at the cylinder.
- Remove both flush-fill needles from the PAL autosampler.
- Let samples remain in heated sample block for a minimum of 18 hours for the ¹⁸O incorporation/equilibration to occur.
- Attach the sampling needle to the left position on the PAL autosampler syringe holder. Ensure black magic marker on sampling needle nut faces the user to ensure correct needle alignment and check manually (also check needle tip point and straighten if warranted).
- Verify correct Tune file is in place and ensure it was passed to gas configuration.
- Open Instrument Control software, check and record the MS pressure.
- Open the GasBench inlet valve on the IRMS.
- Wait a few minutes for the pressure to stabilize, and record the pressure.
- Turn on the filament.
- Monitor m/z 18 (H₂O) on cup 3. (The m/z 18 signal should drop below 1000 mV within 1-2 hours of turning on the filament.)
- With the m/z 18 signal below 1000 mV, perform on-off (CO2_On-Off.met) and linearity (CO2_On-Off.met) system suitability using CO₂ as the reference gas. (δ¹⁸O On-off: std.dev. < 0.08‰, δ¹⁸O Linearity: regression slope std. dev. < 0.08‰ with increasing CO₂ pressure (see Figures 8 and 9).
- Adjust the CO₂ reference gas to give a reference peak (m/z 44, cup 2) signal of between 7000 and 8000 mV (m/z 45 ~ 8500 mV, m/z 46 ~ 10,000 mV).
- Create, identify, and save a new Analysis sequence using the file *180_H20_96_Samples.seq* as a template (see Figure 7).
- Use *180_H20_100uL_Loop.met* as the analysis method (see Figures 3 6).
- Ensure the correct autosampler method is entered in the sequence, **Internal No. 9** (A200S-9) 11 injections of 59 seconds each (See Figure 2).

- Verify that Isodat Acquisition, and Isodat Workspace programs are open (and Instrument Control is closed). Note: To minimize potential computer issues, it is recommended to reset the computer before starting any extended analysis sequence.
- In Acquisition, check and record mass spectrometer pressure, the CO₂, N₂, H₂, m/z 18 (cup 3), m/z 32 (cup 3), and m/z 40 (cup 3) intensities.
- Verify system readiness for analysis, e.g., Helium tank pressures, capillary column temperature, T-valve position, alignment of syringes, vial location and identification, etc.
- Verify that the correct sequence has been selected and double check the information.
- When all is correct, click "Start".
- Identify the folder in which the data files are to be stored (typically use 180 followed by an underscore and then the analysis date).
- Next choose how to identify the data files.
- Un-check the "Auto Enum" button.
- Start the analysis by checking the "OK". (Depending on the number of samples, the sequence can continue for more than 20 hours.)
- Completed files can be reviewed in Isodat Workspace...\Results\filename. (see Figures 10 12 for example chromatograms of a blank, a Primary standard, and a Lab standard).
- When the analysis is complete, review the files in Workspace to verify all samples were properly acquired and analyzed. (It is useful to record any anomalous findings or notes on the analysis worksheet.)
- Print the data files in Workspace.
- Re-process the data files using the export file *GB_180_Export.wke*, this will put the data into EXCEL format (see Figure 13).
- Transfer the re-processed data via an appropriate technique to another computer for statistical analysis.
 - First copy the data into a new worksheet.
 - Clean up the spreadsheet, set significant figures, alignments, headings, etc, to make the spreadsheet easier to handle and interpret.
 - Sort on "Peak No." to separate out the reference peaks.
 - Cut and paste the reference peak data into a new worksheet.
 - \circ $\,$ After the reference peaks have been removed, sort on the sample ID.
 - Create a calibration curve for δ^{18} O‰ using the primary standards, plot the known values vs. the IRMS determined values.
 - Plot the trend line, the equation of the trend line is the regression formula used to determine the corrected $\delta^{18}O\%$ values.
 - \circ Perform statistical analysis (mean, standard deviation, accuracy, and %CV) on all average δ^{18} O‰ values determined for each sample. This is the intra-statistical analysis.
 - Next, perform the same statistical analysis on all the individual peaks of each sample. This is the inter-statistical analysis.

Instrument Conditions

GasBench

- Capillary Column Temperature 70°C
- Capillary Column Flow Rate 1.0 ml/min 1.5 ml/min
- Sample Block Temperature 30°C
- Flush-Fill Flow rate ~125mL/min
- He Pressure (at Tank) 50 psi
- He pressure (at GasBench) 13 14 psi (flow rate ~ 0.8 ml/min)
- 0.3% CO₂ in He pressure (at Tank) ~45 psi (adjust to give ~125ml/min FlushFill rate)
- CO₂ pressure at Tank 35 psi at GasBench – adjust to 7 – 8 volts m/z 44 signal in cup 2

PAL

- Syringe Configuration 10 µL
- FlushFill method Internal 1
- Analysis method Internal 9

IRMS

- Electron Energy 124 eV
- Tune File e.g.: autofocus_CO2_GB_(Date of last tune)
- ~ High Vacuum (Valve open) ~5.5e-7mB
- ~High Vacuum (Valve closed) ~9.5e-8mB
- Instrument configuration GasBench+PAL
- CO2 reference peak intensity (m/z 44 cup 2) ~8000 mV
- Method FlushFill Vial_Flush_6min.met Analysis – 180_H2O_100uL_Loop.met

2.4. WATER STANDARD VALIDATION DATA

The Excel files used for this validation can be found on the Hamilton College network, the path is Campus on ESS

P:\Instrumentation\Geosciences\Data\Thermo_IRMS\GasBench\Water\Oxygen_18\(file names). The file names and contents are listed below:

- 1. 081309_18O_Val_1.xlxs Validation day 1 results
- 2. 081809_18O_Val_2.xlxs Validation day 2 results
- 3. 082109_18O_Val_3.xlxs Validation day 3 results
- 4. 18O_082709_Sec_to_Primary.xlxs Experimentally determined values for Secondary standards used to determine Primary standard values

5. 18O_Validation_Summary.xlxs – Accuracy and precision analysis for all analyses performed during validation

Table 1:

Validation Day 1 Statistics (Primary Standards)

File Name: 081309_180_Val_1.xlxs Primary Standards Statistics	
SLAP	δ ¹⁸ Ο ‰
average	-55.560
Std. Deviation	0.118
%CV	0.21
%Acc	100.11
n	3
Known	-55.5
δ ¹⁸ O _{VSMOW/SLAP}	
VSMOW2	δ ¹⁸ O ‰
average	0.00367
Std. Deviation	0.0560
%CV	1528.3*
%Acc	*
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	0.00
GISP	δ ¹⁸ Ο ‰
average	-24.852
Std. Deviation	0.110
%CV	0.44
%Acc	100.21
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	-24.8

File Name: 081309_18O_Val_1.xlxs

Note: %CV = Coefficient of Variation

% Acc = Accuracy

* Value skewed due to zero being the target value

Table 2:

Secondary Standards Statistics	
Well	δ ¹⁸ Ο ‰
average	-11.38496
Std. Deviation	0.0782
%CV	0.69
n	10
Prepared Lab Standard	δ ¹⁸ Ο ‰
average	-10.33679
Std. Deviation	0.0584
%CV	0.56
n	10
Millipore RO	δ ¹⁸ Ο ‰
average	-10.22694
Std. Deviation	0.0600
%CV	0.59
n	9
Science Center RO	δ ¹⁸ Ο ‰
average	-10.20802
Std. Deviation	0.0527
%CV	0.52
n	9

Validation Day 1 Statistics (Secondary Standards)

Note: %CV = Coefficient of Variation

%Acc = Accuracy

Table 2: (cont'd.)

Secondary Standards Statistics	
Science Center Tap	δ ¹⁸ Ο ‰
<u>Science Center Tap</u>	0 0 700
average	-9.64723
Std. Deviation	0.0675
%CV	0.70
n	10
Bottled Distilled	δ ¹⁸ Ο ‰
average	-7.25881
Std. Deviation	0.0598
%CV	0.82
n	10
Vostok	δ ¹⁸ Ο ‰
average	-53.47425
Std. Deviation	0.0593
%CV	0.11
n	10
Sylvan Beach Tap	δ ¹⁸ Ο ‰
average	-6.58259
Std. Deviation	0.0569
%CV	0.86
n	10

Validation Day 1 Statistics (Secondary Standards)

Note: %CV = Coefficient of Variation

% Acc = Accuracy

Validation Day 2 Statistics (Primary Standards)

Primary Standards Statistics	
SLAP	δ ¹⁸ Ο ‰
average	-55.526
Std. Deviation	0.0672
%CV	0.12
%Acc	100.05
n	3
Known	-55.5
δ ¹⁸ Ovsmow/slap	
VSMOW2	δ ¹⁸ Ο ‰
average	-0.0551
Std. Deviation	0.0618
%CV	112.09*
%Acc	*
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	0.00
GISP	δ ¹⁸ Ο ‰
average	-24.826
Std. Deviation	0.0565
%CV	0.23
%Acc	100.11
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	-24.8

File Name: 081809_18O_Val_2.xlxs

Note: %CV = Coefficient of Variation

% Acc = Accuracy

* Value skewed due to zero being the target value

Table 4:

Validation Day 2 Statistics (Secondary Standards)

File Name: 081809_18O_Val_2.xlxs

Secondary Standards Statistics	
Well	δ ¹⁸ Ο ‰
average	-11.35209
Std. Deviation	0.0392
%CV	0.35
n	10
Prepared Lab Standard	δ ¹⁸ Ο ‰
average	-10.35583
Std. Deviation	0.0586
%CV	0.57
n	10
Millipore RO	δ ¹⁸ O ‰
average	-10.23311
Std. Deviation	0.0413
%CV	0.40
n	9
Science Center RO	δ ¹⁸ Ο ‰
average	-10.21444
Std. Deviation	0.0564
%CV	0.55
n	9

Note: %CV = Coefficient of Variation

%Acc = Accuracy

Table 4: (cont'd.)

Secondary Standards Statistics	
Science Center Tap	δ ¹⁸ Ο ‰
average	-9.66312
Std. Deviation	0.0531
%CV	0.55
n	10
Bottled Distilled	δ ¹⁸ Ο ‰
average	-7.24647
Std. Deviation	0.0457
%CV	0.63
n	10
Vostok	δ ¹⁸ Ο ‰
average	-53.40472
Std. Deviation	0.0735
%CV	0.14
n	10
	10
<u>Sylvan Beach Tap</u>	δ ¹⁸ Ο ‰
average	-6.62288
Std. Deviation	0.0546
%CV	0.82
n	10

Validation Day 2 Statistics (Secondary Standards)

Note: %CV = Coefficient of Variation

% Acc = Accuracy

Validation Day 3 Statistics (Primary Standards)

Primary Standards Statistics	
<u>SLAP</u>	δ ¹⁸ O ‰
average	-55.591
Std. Deviation	0.0515
%CV	0.09
%Acc	100.17
n	3
Known	-55.5
$\delta^{18}O_{VSMOW/SLAP}$	
VSMOW2	δ ¹⁸ Ο ‰
average	-0.0753
Std. Deviation	0.0320
%CV	42.55*
%Acc	*
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	0.00
GISP	δ ¹⁸ O ‰
average	-24.802
Std. Deviation	0.105
%CV	0.42
%Acc	100.01
n	3
Known δ ¹⁸ O _{VSMOW/SLAP}	-24.8

File Name: 082109_18O_Val_3.xlxs

Note: %CV = Coefficient of Variation

% Acc = Accuracy

* Value skewed due to zero being the target value

Table 6:

Validation Day 3 Statistics (Secondary Standards)

File Name: 082109_18O_Val_3.xlxs

Secondary Standards Statistics	
Well	δ ¹⁸ Ο ‰
average	-11.36173
Std. Deviation	0.0476
%CV	0.42
n	10
Prepared Lab Standard	δ ¹⁸ Ο ‰
average	-10.33867
Std. Deviation	0.0542
%CV	0.52
n	10
Millipore RO	δ ¹⁸ Ο ‰
average	-10.20939
Std. Deviation	0.0606
%CV	0.59
n	9
Science Center RO	δ ¹⁸ Ο ‰
average	-10.22948
Std. Deviation	0.0330
%CV	0.32
n	9

Note: %CV = Coefficient of Variation

%Acc = Accuracy

Table 6: (cont'd.)

Secondary Standards Statistics	
	2180.44
Science Center Tap	δ ¹⁸ O ‰
average	-9.64643
Std. Deviation	0.0593
%CV	0.61
n	10
	10
Bottled Distilled	δ ¹⁸ O ‰
average	-7.26981
Std. Deviation	0.0355
%CV	0.49
n	10
Vostok	δ ¹⁸ Ο ‰
average	-53.46616
Std. Deviation	0.0459
%CV	0.09
n	10
<u>Sylvan Beach Tap</u>	δ ¹⁸ O ‰
average	-6.60138
Std. Deviation	0.0344
%CV	0.52
n	10

Validation Day 3 Statistics (Secondary Standards)

Note: %CV = Coefficient of Variation

% Acc = Accuracy

Table 7:

Validation Day 4 (Secondary-to-Primary) Statistics (Primary Standards)

Primary Standards Statistics		
SLAP	δ ¹⁸ Ο ‰	
	-55.620	
average		
Std. Deviation	0.0643	
%CV	0.28	
%Acc	100.16	
n	7	
Known	-55.5	
δ ¹⁸ O _{VSMOW/SLAP}		
VSMOW2	δ ¹⁸ Ο ‰	
average	-0.0818	
Std. Deviation	0.0570	
%CV	69.73*	
%Acc	*	
n	7	
Known	0.00	
δ ¹⁸ Ovsmow/slap		
GISP	δ ¹⁸ Ο ‰	
average	-24.839	
Std. Deviation	0.0698	
%CV	0.28	
%Acc	100.16	
n	7	
Known	-24.8	
δ ¹⁸ O _{VSMOW/SLAP}		

File Name: 18O_082709_Sec_to_Primary.xlxs

Note: %CV = Coefficient of Variation

% Acc = Accuracy

* Value skewed due to zero being the target value

Table 8:

Validation Day 4 (Secondary-to-Primary) Statistics (Secondary Standards) File Name: 180_082709_Sec_to_Primary.xlxs

File Name. 180_082709_Sec_to_Filmary.xixs	
Secondary Standards Statistics	
Well	δ ¹⁸ Ο ‰
<u>•••••</u>	0 0 /00
average	-11.415
Std. Deviation	0.0457
%CV	0.40
%Acc	100.43
n	6
Experimentally Determined	-11.366
$\delta^{18}O_{\rm VSMOW/SLAP}$	11000
Prepared Lab Standard	δ ¹⁸ Ο ‰
· · · · · · · · · · · · · · · ·	
average	-10.346
Std. Deviation	0.0814
%CV	0.79
%Acc	100.01
n	7
Experimentally Determined	-10.344
$\delta^{18}O_{\rm VSMOW/SLAP}$	10.011
Millipore RO	δ ¹⁸ Ο ‰
average	-10.290
Std. Deviation	0.0441
%CV	0.43
%Acc	100.66
n	7
Experimentally Determined	-10.223
$\delta^{18}O_{VSMOW/SLAP}$	
Science Center RO	δ ¹⁸ Ο ‰
average	-10.310
Std. Deviation	0.0478
%CV	0.46
%Acc	100.90
n	7
Experimentally Determined	-10.217
$\delta^{18}O_{VSMOW/SLAP}$	
Notes 0/CV Coefficient of Variation	

Note: %CV = Coefficient of Variation

%Acc = Accuracy

Table 8: (cont'd.)

Validation Day 4 (Secondary-to-Primary) Statistics (Secondary Standards)

Secondary Standards Statistics		
Science Center Tap	δ ¹⁸ Ο ‰	
average	-9.636	
Std. Deviation	0.0758	
%CV	0.79	
%Acc	99.83	
n	7	
Experimentally Determined	-9.652	
$\delta^{18}O_{VSMOW/SLAP}$		
Bottled Distilled	δ ¹⁸ Ο ‰	
average	-7.282	
Std. Deviation	0.0659	
%CV	0.91	
%Acc	100.33	
n	7	
Experimentally Determined	-7.258	
$\delta^{18}O_{VSMOW/SLAP}$		
Vostok	δ ¹⁸ Ο ‰	
average	-53.557	
Std. Deviation	0.0456	
%CV	0.09	
%Acc	100.01	
n	6	
Experimentally Determined	-53.448	
$\delta^{18}O_{VSMOW/SLAP}$		
Sylvan Beach Tap	δ ¹⁸ Ο ‰	
average	-60642	
Std. Deviation	0.0501	
%CV	0.75	
%Acc	100.60	
n	6	
Experimentally Determined	-6.602	
$\delta^{18}O_{VSMOW/SLAP}$		
Note: %CV = Coefficient of Variation		

Note: %CV = Coefficient of Variation

%Acc = Accuracy

Table 9:

Analysis Date	Validation Day	Regression Line	\mathbf{R}^2
08/13/2009	Day 1	y=0.9978x-1.0040	1.00
08/18/2009	Day2	y=0.9964x-1.1421	1.00
08/21/2009	Day3	y=0.9969x-1.1180	1.00
08/27/2009	Day4	y=0.9983x-1.1356	1.00

Regression line equations used to correct $\delta^{18}O$ % instrument values

3. COMMENTS

Three standards, in duplicate (one at the beginning of the analysis and one at the end) were used to generate the regression line.

The Primary Standards that were used in the regression line generation were not used in the calculations of the experimentally determined δ^{18} O‰ read-back values or the statistics generated for them. Only the additional Primary Standards (n=3) analyzed in each run were used for this purpose.

An analysis of the $\delta^{18}O_{\infty}^{\infty}$ value determined for each sample was plotted versus acquisition time. It was determined that there was no temporal bias and as such no drift corrections of determined $\delta^{18}O_{\infty}^{\infty}$ values were made.

 δ^{18} O‰ values given in the above Tables originate from the "intra" values determined in the Excel spreadsheets since the "intra" and "inter" values were essentially identical.

Day 4 Validation (Secondary to Primary Standard experiment) was performed only to evaluate the integrity of the Lab (Secondary) Standards for regression line generation and subsequent sample read-backs. This data was not used in any statistical calculations. (Sylvan Beach Tap, Vostok and the well water sample were used to generate the regression line.)

Reported values in the various Tables are not corrected to the appropriate significant figures. Only in the Conclusions and Summary are they corrected to the appropriate number of significant figures.

%Accuracy = Experimental Value/Known (Established) Value X 100

4. DATA RETRIEVAL

The raw data files are stored on the Thermo IRMS instrument computer in the GeoSciences laboratory in the following location:

C:\Thermo\Isodat NT\Global\User\Gas Bench\Results\O18_Analysis Folder\

- 18O_Val_081309\filename.dxf
- 18O_Val_081809\filename.dxf
- 18O_Val_082109\filename.dxf
- 18O_082709_Sec_Primary\filename.dxf

The Excel Worksheets are stored on the Hamilton College network in the following location: Campus on "ESS"(P:)\Instrumentation\Geosciences\Data\Thermo_IRMS\ GasBench\Water\Oxygen_18\Validation Data\filename.xlsx and Campus on "ESS"(P:) \Instrumentation\Geosciences\Data\Thermo_IRMS\GasBench\Water\Oxygen_18\Analysis Worksheets\filename.xlsx.

5. CONCLUSIONS

This analysis identified water samples which could be used for Lab (Secondary) Standards during unknown $\delta^{18}O_{\infty}$ investigations. This validation also provided $\delta^{18}O_{\infty}$ values for these Lab Standards (to be used for regression line generation) along with statistical evaluations of those values. The following is a summary of the results:

Water Sample	$\delta^{18}O_{VSMOW/SLAP}$ ‰	Std. Dev.	%CV	n
	10.0	0.0450	0.46	07
Science Center RO	-10.2	0.0473	0.46	27
Vostok	-53.4	0.0596	0.11	30
Sylvan Beach Tap	-6.60	0.0486	0.74	30
Distilled	-7.26	0.0470	0.65	30
Well	-11.4	0.0550	0.48	30
Science Center Tap	-9.65	0.0600	0.62	30
Prepared Lab Standard	-10.3	0.0571	0.55	30
Millipore RO	-10.2	0.0540	0.53	27

Table 10: δ^{18} O Values and Statistical Analysis (Secondary Standards)

The experimentally determined values and the statistics for the Primary Standards are given below to assess method accuracy and variability across the 3 days of validation:

Table 11: δ^{18} O Values and Statistical Analysis (Primary Standards)

Primary Standard	$\delta^{18}O_{VSMOW/SLAP}$ %0	Std. Dev.	%CV	% Acc	n
SLAP2	-55.6	0.0781	0.14	100.11	9
GISP	-24.8	0.101	0.41	100.11	9
VSMOW2	-0.042	0.0385	91.7*	*	9

* Value skewed due to zero being the target value

6. **REFERENCES**

Thermo Electron Delta V Advantage Operating Manual Finnigan GasBench II Operating Manual

7. FIGURES

Figure 1:

 δ^{18} O Experimentally Determined Values, Sorted by δ^{18} O (average of three runs)

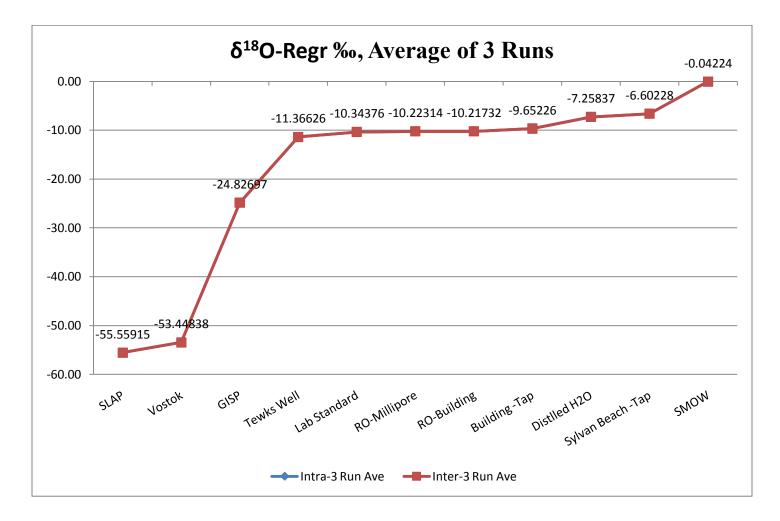


Figure 2:

PAL Autosampler Methods Used for $\delta^{18}O$ Analysis and FlushFill

Internal No. 1 (A200S-1) (Flu	ushFill)	Internal No. 9 (A200S-9) (Analysis)				
Cycle	GC-Inj	Cycle	GC-Inj			
Syringe	10 µL	Syringe	10 µL			
Sample Volume	10.0 µL	Sample Volume	10.0 µL			
Air Volume	0 µL	Air Volume	0 µL			
Pre Cln Slv1	0	Pre Cln Slv1	0			
Pre Cln Slv2	0	Pre Cln Slv2	0			
Pre Cln Spl	0	Pre Cln Spl	0			
Fill Volume	0 nL	Fill Volume	0 nL			
Fill Speed	$5.0 \ \mu L/s$	Fill Speed	5.0 µL /s			
Fill Strokes	6	Fill Strokes	11			
Pullup Del	61	Pullup Del	59 s			
Inject to	Flush	Inject to	Flush			
Inject Speed	50 µL /s	Inject Speed	50 µL /s			
Pre Inj Del	0 ms	Pre Inj Del	0 ms			
Pst Inj Del	0 ms	Pst Inj Del	0 ms			
Pst Cln Slv1	0	Pst Cln Slv1	0			
Pst Cln Slv2	0	Pst Cln Slv2	0			

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Figure 3: Method File – Instrument Screen

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Figure 4: Method File – Time Events Screen

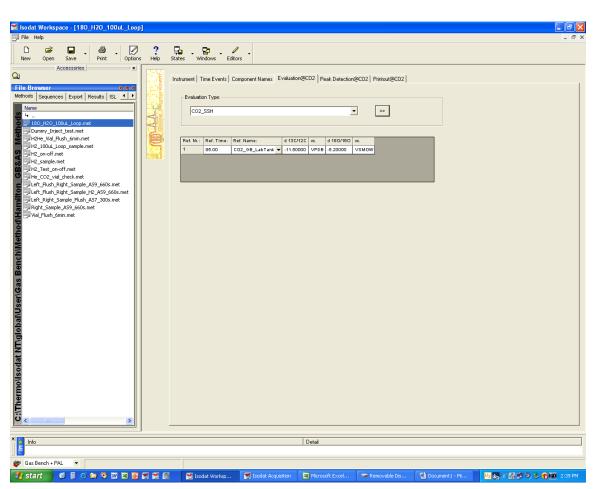


Figure 5: Method File – Evaluation@CO2 Screen

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Figure 6: Method File – Peak Detection@CO2 Screen

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Figure 7: Example of Water δ^{18} O Sequence File.

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Figure 8: ¹⁸O On-Off Check (Using CO₂)

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Figure 9: ¹⁸O Linearity Check (Using CO₂)

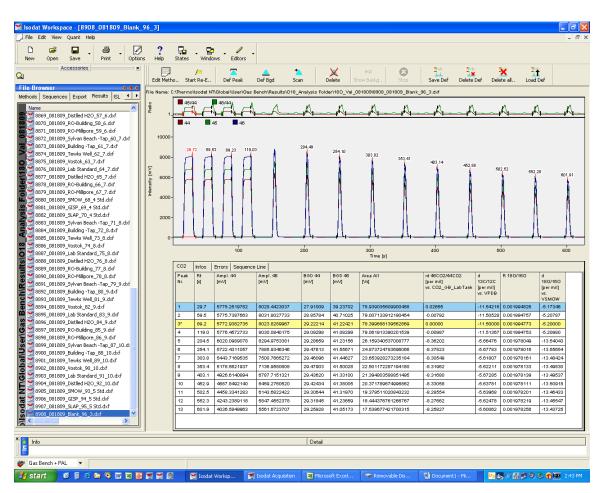


Figure 10: ¹⁸O Data Acquisition File - Blank

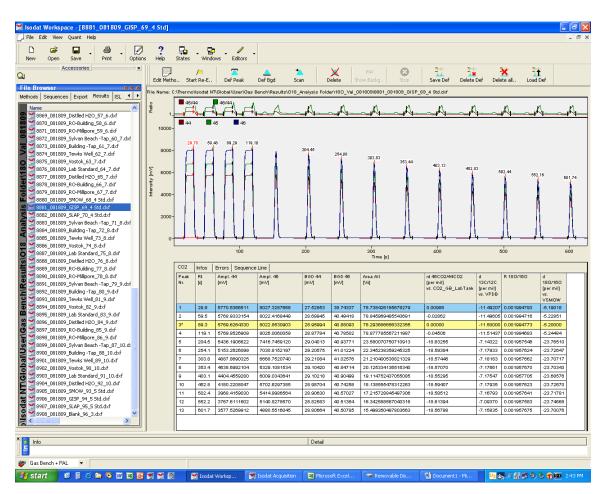


Figure 11: ¹⁸O Data Acquisition File – Primary Standard (GISP)

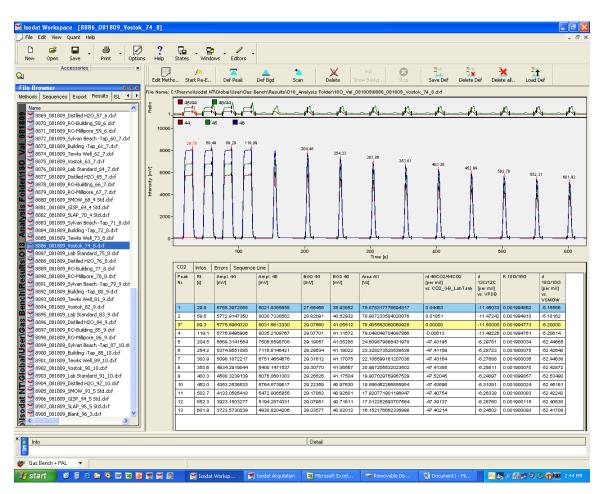


Figure 12: ¹⁸O Data Acquisition File – Sample (Vostok Water)

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	Err Script	Error Grid	BGD 46	Result Data
	Can Reference Refil	Sequence Part - Reference Refill	Area All	Result Data
	Multiport Ext Inlet Peak Center	Sequence Part - MultiPort Sequence Part - MS	d 46C02/44C02 IR 46C02/44C02	Result Data Result Data
	Pre-Process	Sequence Part - MS Sequence Part - Equilibration Unit	iii d 13C/12C	Result Data
	🔷 EQ Unit - Port	Sequence Part - Equilibration Unit	d 180/160	Result Data
	EQ Unit - Bank	Sequence Part - Equilibration Unit	333 d 170/160	Result Data
	Pressadjust	Sequence Part - Dual Inlet Device Sequence Part - Dual Inlet Device		
	Port	Sequence Part - Conflo		
	Type	Sequence Part - Conflo		
	Amount	Sequence Part - Conflo		
	Preview			npl. 44 Ampl. 45 Ampl. 48 BGD 44 BGD 45 BGD 46
	> Time Code FileHeader: Filenam	e Analysis Method Comment Identifier 1 Identifi	er 2 Preparation Is Ref.?? Peak Nr. Start Rt An	1p1. 44 Ampl. 46 Ampl. 48 BGD 44 BGD 46 BGD 48
		101		9
Info		Detail		

Figure 13: ¹⁸O Export File – GB_18O_Export