













































## Chemical Hazards

The hazardous chemicals used in the process are  $\text{Cr}_2\text{O}_3$  and  $\text{Co}_3\text{O}_4$ , which are used in the oxidation reaction tube, and  $\text{V}_2\text{O}_5$ , which is used to boost combustion. Sometimes the reaction tube cracks, and the packing material leaks out. When this happens, protective gloves and a full-face mask are required during the cleanup process. All the materials, including gloves and any cleaning towels, need to be collected in a plastic bag and disposed of as hazardous waste. When handling  $\text{V}_2\text{O}_5$ , protective gloves and a full-face mask also are required. All samples must be carefully inspected upon receipt for obvious indications of hazards.

The  $\text{SO}_2$  reference gas is colorless and has a suffocating odor. Exposure to  $\text{SO}_2$  can cause respiratory tract burns. Therefore, an appropriate ventilation unit must be installed in the ConFlo unit. A leak test on the tank of gas and the line carrying  $\text{SO}_2$  to the ConFlo must be performed routinely.

## Gas Cylinder Handling

Compressed gas cylinders must be handled and stored according to the Safety and Environmental Health Handbook (U.S. Geological Survey, 2002). Each cylinder must be (1) carefully inspected when received; (2) securely fastened at all times with an approved chain assembly or belt; (3) capped at all times when not in use; (4) capped when transported; (5) transported only by a properly designed vehicle (hand truck); and (6) stored separately with other full, empty, flammable, or oxidizing tanks of gas, as appropriate.

## Specific Waste-Disposal Requirements

Used reaction tubes containing  $\text{Cr}_2\text{O}_3$ ,  $\text{CO}_3\text{O}_4$ , and  $\text{V}_2\text{O}_5$  must be collected in a closed container and must be given to the safety, health, and environment officer for disposal.

## Revision History

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Summary of Product Components

Component	Description	Last Revised In Publication Version	Date of Last Revision

List of Revisions (latest version first):

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## Appendix A. Step-by-Step Procedure to Log-In Samples to LIMS-LSI

1. For samples submitted through NWQL:
  - a. Match up information on sample bottles with information on ASR forms by spreading out ASR forms and placing each bottle on its respective ASR form. Check for broken bottles.
  - b. Assign NWQL #s based on the Julian date of the Monday following the submission date. Check note on bulletin board for “Next NWQL #.” Write NWQL #s on ASR forms, then update “Next NWQL #” on bulletin board.
  - c. Use “New Project Log-In” in LIMS-LSI to assign S-#s; field IDs are the Station IDs. Submission date is the date samples are logged in.
  - d. Enter all ASR information into LIMS-LSI, sample by sample.
  - e. Print out two Project Reports, Labels (two for each sample), and Sample Range forms.
  - f. Put a label on a sample bottle and on the corresponding ASR form; crosscheck Field IDs between bottles and ASR forms.
  - g. Make one photocopy of each ASR form.
  - h. Enter S-# range into the appropriate template.
  - i. Proofread information on Sample Range forms against ASR forms (recruit help if necessary). Make corrections in LIMS-LSI, if necessary and print out corrected Sample Range Forms. Initial and date both Project Reports to indicate that proofreading is complete.
  - j. In LIMS-LSI, save NWQL Headings to a diskette.
  - k. In ISOTOPEs e-mail on the common-use PC, copy headings into new message from diskette by opening headings-file in Notepad and copying/pasting to new message. Print copy of headings and e-mail to each project chief for information review and NWQL [Attn: Jeanne Hatcher (jhatcher@usgs.gov)].
  - l. Mail copies (not originals) of ASR forms and corrected Sample Range forms to NWQL [National Water Quality Laboratory, Attn: Jeanne Hatcher, U.S. Geological Survey, P.O. Box 25708, MS 407, Building 95, Denver Federal Center, Denver, CO 80225-0608 (Tel: 303-236-3481)]. Envelope should be mailed every Friday.
  - m. Punch holes in original ASRs and put in “Central Lab” binder along with project reports and date on piece of tape. Punch holes in NWQL headings and other project report and place in “Samples-in-Progress” binder.
  - n. Add samples to the “Table of Samples to Be Analyzed” in LIMS-LSI.
    - i. Click the “Projects” button of LIMS-LSI.
    - ii. Select the project that was logged in.
    - iii. Click the “Templates” button.
    - iv. Select “EA for S.”
    - v. Click “Add” and close LIMS-LSI.
  - o. Put samples in cabinets for storage until analyzed.
2. For samples submitted directly to RSIL:
  - a. For sample submitter:
    - i. Download “RSIL Excel Worksheet” from RSIL website at <http://isotopes.usgs.gov/>.
    - ii. Fill out the requested sample information.
    - iii. Send diskette and a hard copy along with the samples.
  - b. For RSIL personnel:
    - i. Match up information on sample bottles with submitted “Excel Worksheet.”
    - ii. Enter all Excel Worksheet information into LIMS-LSI by loading the diskette. Submission date is the date samples are logged in.
    - iii. Use “New Project Log-In” in LIMS-LSI to assign S-#s; field IDs are the Station IDs.
    - iv. Print out one Project Reports and container labels (one for each sample).

- v. Put a label on a sample bottle and crosscheck Field ID's between bottles and Excel Worksheet forms.
- vi. Enter S-# range into the appropriate template.
- vii. Punch holes in original Excel Worksheet and all the information for the project and put in "Samples-in-Progress" binder.
- viii. Add samples to the "Table of Samples to Be Analyzed" in LIMS-LSI.
  - 1. Click the "Projects" button of LIMS-LSI.
  - 2. Select the project that was logged in.
  - 3. Click the "Templates" button.
  - 4. Select "EA for S."
  - 5. Click "Add" and close LIMS-LSI.
- ix. Put samples in cabinets for storage until analyzed.

## Appendix B. Step-by-Step Procedure to Generate an Excel Sample Workbook or to Print a Template and a Samples-to-Be-Analyzed List

### Excel Sample Workbook

1. Use default worksheet-weighing template and add samples to be analyzed.
2. Fill the appropriate amount in the cells of the rows labeled “Set weight mg.”
3. Print first worksheet in workbook.
4. Write the tray ID and the date on both the diskette and paper template.
5. Put them near the balance.

### Template

1. Use “Print Template” in LIMS-LSI.
2. Select “appropriate template” for EA and “Delta Plus” for mass spectrometer.
3. Select “New Template” (dialog box informs you how many samples are waiting to be analyzed).
4. Click “OK.”
5. Click “Print.”
6. Insert diskette to receive sample headings.
7. Click “OK.”
8. Write the day of the week that these samples should be analyzed on both the diskette and paper template.
9. Put them near the balance.
10. Exit LIMS-LSI.

**Worksheet 1. Weighing template.**

[International reference materials are in bold font.]

		<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>
	Our Lab ID	<b>S-97</b>	<b>S-97</b>	<b>S-97</b>	<b>S-97</b>	<b>S-1301</b>	<b>S-1302</b>	S-9031	S-9031	S-9032	S-9032	S-9033	S-9033
A	Set weight mg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	Actual weight, mg	0.3	0.29	0.31	0.31	0.304	0.298	0.299	0.302	0.305	0.303	0.305	0.303
	Comment												
	Our Lab ID	S-9034	S-9034	S-9035	S-9035	<b>S-97</b>	<b>S-1301</b>	<b>S-1302</b>	S-9037	S-9037	S-9038	S-9038	S-9039
B	Set weight mg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	Actual weight, mg	0.301	0.302	0.295	0.303	0.299	0.295	0.301	0.299	0.293	0.304	0.301	0.304
	Comment												
	Our Lab ID	S-9039	S-9040	S-9040	S-9041	S-9041	<b>S-97</b>	<b>S-1301</b>	<b>S-1302</b>	S-9043	S-9043	S-9044	S-9044
C	Set weight mg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	Actual weight, mg	0.301	0.301	0.295	0.306	0.297	0.302	0.3	0.305	0.304	0.297	0.304	0.301
	Comment												
	Our Lab ID	S-9045	S-9045	S-9046	S-9046	S-9047	S-9047	<b>S-97</b>	<b>S-1301</b>	<b>S-1302</b>	S-9049	S-9049	S-9050
D	Set weight mg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	Actual weight, mg	0.295	0.303	0.32	0.299	0.298	0.296	0.303	0.305	0.305	0.297	0.302	0.296
	Comment												
	Our Lab ID	S-9050	S-9051	S-9051	S-9052	S-9052	S-9053	S-9053	<b>S-97</b>	<b>S-1301</b>	<b>S-1302</b>		
E	Set weight mg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3		
	Actual weight, mg	296	0.296	0.295	0.299	0.305	0.305	0.299	0.302	0.297	0.304		
	Comment												

Worksheet 2. Samples to be analyzed.

[International reference materials are in bold font]

Line	Identifier	Port	Comment	Amount	Amt Unit
1	<b>S-97</b>	A1		0.3	mg
2	<b>S-97</b>	A2		0.29	mg
3	<b>S-97</b>	A3		0.31	mg
4	<b>S-97</b>	A4		0.31	mg
5	<b>S-1301</b>	A5		0.304	mg
6	<b>S-1302</b>	A6		0.298	mg
7	S-9031	A7		0.299	mg
8	S-9031	A8		0.302	mg
9	S-9032	A9		0.305	mg
10	S-9032	A10		0.303	mg
11	S-9033	A11		0.305	mg
12	S-9033	A12		0.303	mg
13	S-9034	B1		0.301	mg
14	S-9034	B2		0.302	mg
15	S-9035	B3		0.295	mg
16	S-9035	B4		0.303	mg
17	<b>S-97</b>	B5		0.299	mg
18	<b>S-1301</b>	B6		0.295	mg
19	<b>S-1302</b>	B7		0.301	mg
20	S-9037	B8		0.299	mg
21	S-9037	B9		0.293	mg
22	S-9038	B10		0.304	mg
23	S-9038	B11		0.301	mg
24	S-9039	B12		0.304	mg
25	S-9039	C1		0.301	mg
26	S-9040	C2		0.301	mg
27	S-9040	C3		0.295	mg
28	S-9041	C4		0.306	mg
29	S-9041	C5		0.297	mg
30	<b>S-97</b>	C6		0.302	mg
31	<b>S-1301</b>	C7		0.3	mg
32	<b>S-1302</b>	C8		0.305	mg
33	S-9043	C9		0.304	mg
34	S-9043	C10		0.297	mg
35	S-9044	C11		0.304	mg
36	S-9044	C12		0.301	mg
37	S-9045	D1		0.395	mg
38	S-9045	D2		0.303	mg
39	S-9046	D3		0.32	mg
40	S-9046	D4		0.299	mg
41	S-9047	D5		0.298	mg

Line	Identifier	Port	Comment	Amount	Amt Unit
42	S-9047	D6		0.296	mg
43	<b>S-97</b>	D7		0.303	mg
44	<b>S-1301</b>	D8		0.305	mg
45	<b>S-1302</b>	D9		0.305	mg
46	S-9049	D10		0.297	mg
47	S-9049	D11		0.302	mg
48	S-9050	D12		0.296	mg
49	S-9050	E1		0.296	mg
50	S-9051	E2		0.296	mg
51	S-9051	E3		0.295	mg
52	S-9052	E4		0.299	mg
53	S-9052	E5		0.305	mg
54	S-9053	E6		0.305	mg
55	S-9053	E7		0.299	mg
56	<b>S-97</b>	E8		0.302	mg
57	<b>S-1301</b>	E9		0.297	mg
58	<b>S-1302</b>	E10		0.304	mg

## Appendix C. Step-by-Step Procedure for Weighing and Storing Samples

1. Samples should be homogenized and dried.
2. Insert Template diskette in the computer connected to the microbalance.
3. Condition balance (this step should be done once a day):
  - a. Place empty capsule on balance and close door. Wait for reading to stabilize (the “mg” on the display appears). Tare the balance.
  - b. Remove and replace capsule and make sure the stabilized weight is 0.000 mg.
  - c. Repeat these steps until the balance is stable.
4. Remove capsule from balance, add sample and weigh the filled capsule. Repeat until you have sufficient mass to yield 40  $\mu\text{g}$  of sulfur.
5. Enter sample weight on the template.
6. Add 600  $\mu\text{g} \pm 100 \mu\text{g}$  of  $\text{V}_2\text{O}_5$  to sample.
7. Fold cup; secure sample in it.
8. Repeat steps 4 to 7 for every sample.
9. Note:
  - a. Do not cross-contaminate samples.
  - b. Make sure the spatula and sample area are cleaned using Kimwipes between each sample.
  - c. Always allow balance to stabilize before removing capsule.

## Appendix D. Step-by-Step Procedure of Zero Blank Autosampler Operation

1. Before opening the lid, make sure the isolation valve (between sample chamber and reaction tube) is closed (arrow towards you) and make sure the He purge isolation valve (on the left) is closed.
2. Open the purge vent on the top of the autosampler to vent the sample chamber.
3. Release the three fittings holding the lid close and open the lid.
4. Place your samples and close the lid. Load 49 samples for a 50-position carousel, leave the first hole empty.
5. Secure the lid with three bolts. Start all three bolts, and then lightly tighten two at a time, moving around the lid until they are all completely tight.
6. Open the He purge isolation valve (screw down), purge the sample chamber for 5 min at He flow rate of 298 mL/min. Make sure the purge vent valve (on top) is open. Press your finger on top of the vent valve for 2 s, then release; you should hear the sound of a pressure release.
7. Close the He purge vent valve (screw down), wait for 2 min for gas pressure at sample chamber and He flow rate to stabilize. Close He purge isolation valve.
8. Open the isolation valve (arrow toward up). Wait for 2 min for stabilized baseline. Normally when the emission current set at 0.5 mA/0.48 mA, with He carrier flow rate of 90 mL/min, one should see  $m/z$  28 on cup 1 at about 50 mV,  $m/z$  29 on cup 2 at about 38 to 50 mV and  $m/z$  30 on cup 3 at about 600 mV to 1 V (Appendix J).

## Appendix E. Step-by-Step Procedure to Add Sample Information to Sequence Table

1. Insert diskette with “Weighing Template” on it.
2. Open “Sequence File” under “EA configuration.”
3. Open “Weighing Template” in Excel.
4. “Copy” and “Paste” the list of Sample ID, sample weight from template to sequence table.
5. Select appropriate “method” for each sample.
6. Define reference line and blank line.
7. Start.
8. Give Folder Name: Comment
  - a. Select pre: “Date.”
9. Give File Name:
  - a. Select pre: “Analyzes #.”
  - b. Select post: “Identifier 1.”
  - c. Select: “Print Result.”
10. Click OK.
11. Wait for first sample to be analyzed.

## Appendix F. Step-by-Step Procedure to Retrieve Data from ISODAT 2.0 for LIMS-LSI and for Data Back-Up

1. For LIMS-LSI:
  - a. Select “Result” in ISODAT 2.0.
  - b. Select the result folder you wish to export data from.
  - c. Select all the individual analyses you want to retrieve by right clicking the selected file.
  - d. Select “Reprocess.”
  - e. Give a file name.
  - f. Add export template previously designed.
  - g. Open.
  - h. Click OK. (Reprocessing takes 1–2 min.)
  - i. Open Excel.
  - j. Find the file you reprocessed (E-drive, Finnigan, ISODAT 2.0, Global, User, CONFLO II, Interface, Result: File name).
  - k. Open it in Excel.
    1. Save as: “A” drive. Keep in Lotus format.
  - m. Define all reference peaks by adding #1 under column “Is Ref.?”
  - n. Now the file can be imported to LIMS-LSI.
2. For Data Back Up Computer:
  - a. Go to Windows explorer in ISODAT 2.0 computer.
  - b. Find the drive where the data are (D).
  - c. Choose: “Finnigan.”
  - d. Choose: “User.”
  - e. Choose the inlet system where you have data (Gas Bench or EA).
  - f. Choose “Result folder.”
  - g. Transfer data to Zip disk or “memory stick.”

## Appendix G. Step-by-Step Procedure to Transfer Data to LIMS-LSI, to Transfer Data to Back-Up Computer, and to Reevaluate Old Data

1. Transfer data to LIMS-LSI:
  - a. Start LIMS.
  - b. Choose "Import Analysis."
  - c. Choose mass spectrometer "Delta Plus."
  - d. Click "Import."
  - e. Select the file that will be imported.
  - f. Select columns containing  $\delta^{34}\text{S}$  data. Check the boxes to import these two columns.
  - g. Click Import. Note: Sample ID, sample weight, Peak Area, Analyses #s are automatically imported to LIMS-LSI.
2. Transfer data to Data Back Up computer:
  - a. Go to Windows Explorer in the Data Back Up computer.
  - b. Find the drive your back ups are stored.
  - c. Choose: "RSIL."
  - d. Choose: "Mass. Spec. Analysis Back Up."
  - e. Choose: "P." (Stands for Delta<sup>Plus</sup> MS).
  - f. Under that folder create a new folder. The name of the folder should be the date range when those analyses were done that you want to back up.
  - g. Transfer data to that folder.
  - h. Make back ups every 2 weeks or so, as required.
3. Reevaluate old data:
  - a. Find your samples in the Data Back Up computer.
  - b. Transfer data by a Zip disk to the computer where a virtual version of ISODAT 2.0 is installed.
  - c. Reevaluate your data.

## Appendix H. Step-by-Step Procedure to Determine and Apply Correction Factors and Evaluate Data

1. Open “Correction Factors and Normalization Equations” in LIMS-LSI.
2. Select MS (Delta<sup>Plus</sup>) and element.
3. Select “Query.”
4. Double click on the last sample analyzed on that day.
5. Evaluate data of the reference materials.
6. Choose “Normalize with all References.”
7. Accept “Expansion Correction and Additive Correction factors.”
8. Print out correction factor sheet.
9. Report daily reference values and correction factors along with date and range of analysis number to the “EA” binder.
10. Go back to LIMS-LSI main menu by closing open windows.
11. Choose “Print Samples in Progress,”
  - a. Open “Sample in Progress.”
  - b. Choose appropriate isotope.
  - c. Choose appropriate prefix (W for water, N for nitrogen, S for sulfur).
  - d. Put in sample ID range from “Samples-to-Be-Analyzed” sheet. Click Print.
12. Review results; determine repeats.
13. Put repeats back to Table of Samples to Be Analyzed.
  - a. Go back to LIMS-LSI main menu.
  - b. Open “Print Templates.”
  - c. Select appropriate template name for the mass spectrometer the samples are analyzed.
  - d. Find the sample in the “List of samples.”
  - e. Change “Repeats” from 0 to 1.
  - f. Close Windows and exit LIMS-LSI.

## Appendix I. Step-by-Step Procedure to Check Elemental Analyzer for Leaks

1. Close the VENT carrier output using the proper cap (provided).
2. Adjust the He pressure to 150 kPa (regulator is on the front central panel), wait 3 min to equilibrate gas in the system.
3. Close He inlet valve by turning the above regulator counter clockwise.
4. If the gauge needle does not move, there is no leak.
5. If the gauge needle moves, indicating the lost of pressure, there is a leak in the system. The decreasing pressure rate accounts for the degree of leakage.
6. Locate the leak by separating and testing the system segment by segment, using the He detector.

## Appendix J. Daily Checklist

Analyst in charge: \_\_\_\_\_ Date: \_\_\_\_\_

Weekly:

1. Change working reference gas tank and He gas tank if the pressure is < 500 psi. Order new ones for a spare; reference gas is SO<sub>2</sub> ANHY by Matheson, He carrier is zero grade, O<sub>2</sub> is research grade.
2. Change water and CO<sub>2</sub> trap after approximately 8,000 samples, or when it is necessary.
3. Check and clean autosampler carousel.
4. Change the reaction tube after approximately 200 or 250 samples, or when it is necessary.

THESE ITEMS ARE TO BE CHECKED OFF AS YOU CHECK THEM DAILY!

1. Check He, O<sub>2</sub>, and reference gas flow. [ ]
2. Check EA reaction-tube temperature (1020 °C)
3. Change insertion tube (ash-collector liner) in the reaction tube. [ ]
4. Check background masses. [ ]

At emission Current: 0.34 mA/0.33 mA

Cups	Mass	Intensity (V)	Mass	Intensity (V)	Mass	Intensity (V)	Resistor (Ω)	Capacity (pF)
1	28	0.15	44	0	64	0.002	3 × 10 <sup>8</sup>	680
2	29	0.015	45	0	66	0.002	3.5 × 10 <sup>10</sup>	5
3	30	0.05–0.3	46	0.002			3 × 10 <sup>11</sup>	2

5. Perform Ref on/off method three times to stabilize IRMS. [ ]
6. Check peak center. If it is off default value by more than 50, do mass calibration. [ ]
7. Analyze Ref on/off method 10 times to stabilize IRMS (standard deviation should be better than ±0.1 %). [ ]

Optimal ConFlo Pressure Setting SO <sub>2</sub> (bar)	SO <sub>2</sub> signal (V) Mass 66 on Cup 2
0.50	3.000

8. Check paper in printer. [ ]

## Appendix K. Changing the Insertion Tube

The insertion tube needs to be changed before a template is run.

1. Manually close needle valve near the ion source.
2. On the menu of the Model 2500 EA under "Sp. Fun.," choose "Std By" and press "enter." The O<sub>2</sub> and He should be turned off.
3. Unscrew the metal seals under the sample carousel to access the reaction tube.
4. Using the heat protective glove and the wire tool, remove the insertion tube from the reaction tube and place in the metal can. Be aware that the tube will be very hot!
5. Insert clean tube that has 0.5 cm of quartz wool packed at the bottom, and tap it down with the wire tool.
6. Replace the carousel and tighten the metal seals with a wrench.
7. Turn the O<sub>2</sub> and He back on by pressing "enter" and return the screen to the temperature readout by pressing "Sp. Fun."
8. Open the needle valve.

## Appendix L. Changing the Water Trap

Change the water trap after every 200 samples.

1. Retrieve quartz turnings, quartz wool, magnesium perchlorate, and a clean water trap from Delta<sup>Plus</sup> supplies.
2. Under a hood, pack one end of the clean water trap with 1 cm of quartz wool.
3. On a piece of wax paper, create a mix of approximately 70 % magnesium perchlorate and 30 % quartz turnings, pack the mix into the water trap tube, leaving 1 cm of empty space.
4. Pack the open end of the water trap with quartz wool.
5. Unscrew the red plastic ends from the used water trap and replace it with the clean water trap; be sure to replace the rubber O-rings on the ends of the clean water trap and tighten the red ends.
6. Insert the plug into the vent and watch for any decrease in pressure to check for a leak in the system. If everything looks acceptable after 5 min, replace the vent line.
7. Under the “Sp. Fun.” menu of the model 2500 EA, choose “CNT” (counter) and zero “D,” to set back the water-trap counter.
8. Clean the water trap by pushing all the mixture out of the tube into the trash can and use a clean cotton swab to wipe out the inside of the tube. Rinse the tube with DIW, and dry it in the oven. Return this tube to the water trap bag in the drawer.

## Appendix M. Changing the Reaction Tube

The reaction tube should be changed approximately every 200 to 250 samples.

1. Manually close the needle valve near the ion source.
2. Turn the Model 2500 EA to “Std By” with both gases off.
3. Lower the reaction tube temperature to less than 900 °C.
4. Retrieve the reaction tube from the cabinet.
5. Insert the insertion tube into the top of the reaction tube; there will already be a plastic tube in the reaction tube that should be removed. The bottom of the reaction tube has copper in it.
6. Place a black O-ring on the top of the reaction tube. Make sure that all O-ring connections are clean and free of any dust.
7. Loosen the top metal seal and remove the bottom metal seal. Go back and completely remove the top seal. It will be very hot! Use oven gloves!
8. Remove quartz reaction tube and place in metal can, place clean reaction tube in column. Make sure the O-ring is clean; then replace the top.
9. On the bottom, put on the metal piece with the metal O-ring first and then add the clean rubber O-ring. Raise the bottom portion and screw together. Then, tighten the connection with wrenches, and put the metal support back in place.
10. Turn the gases back on; then, go to “CNT” in the menu under “Sp. Fun.” and zero “B,” the oxidation reaction tube as well as “A” and “C,” to set back the counter.
11. Insert the plug into the vent and watch for any decrease in the pressure to check for a leak in the system. If everything looks okay after 3 min, replace the vent line.
12. Open the needle valve.

## Appendix N. Step-by-Step Procedure to Report Data

1. Open “Store Samples in Progress” in LIMS-LSI.
2. Choose the appropriate isotope.
3. Choose sample ID range from “Sample in Progress” print out.
4. Store data.
5. Go back to the LIMS-LSI main menu.
6. Open “Project” and find the appropriate project in the list.
7. Select “Print Report” and check whether the project report contains all the results. If not, search for the missing results in the database.
8. Select “Results,” transfer data in Excel format or (and) text format to a diskette, and report data to customer through e-mail.
9. Click “Print Report” to print a project report and put it in the “Correspondence” binder along with all other documents in the “Samples In Progress” binder that are related to this project.
10. If the samples were submitted through the NWQL, click “NWQL Export” and transfer data in NWQL format to a disc; report these data to the NWQL by e-mail. Print a copy of the NWQL export file and save in the “Correspondence” binder with the project report printed in the step above.