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ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-10 CALIBRATION, USE AND MAINTENANCE OF THE VARIAN SATURN 2100T GC/MS Revision 1



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1.0 Purpose and Scope

This procedure prescribes operation guidelines for Varian Saturn 2100T GC/MS. This SP will be used for identification and quantization of chemical compounds and will support the research activities of the Waste Isolation Pilot Plant (WIPP) project.

This document is not meant to substitute for manufacturer's instruction manuals. The user is responsible for reading and understanding the appropriate manuals.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

2.0 Implementation Actions

2.1 Safety and Training

The activities described in this SP shall conform with SNL Environmental Safety and Health programs (ES&H). The ES&H Standard Operating Procedure *Activities in the Sandia National Laboratories/ Carlsbad Program Group laboratory* details the handling of specific hazards in the Carlsbad Laboratory.

2.2 Responsibility

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The technical staff or designee is responsible for performing the measurements, following the requirements of this procedure, documenting the results, and assuring that the use of the latest revision of this document is followed.

2.3 Standards

Standards may be prepared from primary standard materials or purchased as certified reagents. The primary standard materials or certified reagents must be NIST traceable.

2.4 Initial Calibration and Calibration Verification

A minimum of 3 standards bracketing the entire concentration range of interest should be analyzed to generate the initial calibration curve. According to the Varian Software Reference Manual, the manufacturer recommends that three reference standard concentration levels should be prepared for each factor of 10 in the chosen concentration range. For example, if the expected glycerin concentration is $0.8\text{--}6 \times 10^{-3}$ mg/ml in the sample, then 3 standard solutions could be prepared with glycerin concentration of 0.7×10^{-3} mg/ml, 3.5×10^{-3} mg/ml, 7.0×10^{-3} mg/ml. If standards are prepared from primary standard materials, the initial calibration curve should be generated from freshly prepared standards. Certified reagents can be used anytime before the expiration dates.

After the initial calibration curve is generated it is verified by analyzing one standard. Calibration verification is performed daily before the sample analyses. The measured value of the verification standard should fall within 75% to 125% of the certified (or theoretical) value. If the measured value falls out of the 75% to 125% range, the standard shall be reanalyzed. If the reanalyzed result is still out of control limit, the operator should find out the root cause and correct the problem. Otherwise, a new initial calibration curve should be generated. It is acceptable to reprocess the data with new calibration curves as long as the data is collected within 9 hours of the calibration curve and there is no modification of the instrument. If solvent has been used to dilute the analyte, the solvent should be analyzed as an analytical blank. Analytical blank will be analyzed daily before the sample analyses.

2.5 Tuning and Acquiring a 2000 MS Data File

1. To set up the GC/MS for acquiring a sample, you should first enter **System Control** and tune the mass spectrometer. Click on the **System Control** button on the work station toolbar and a **system control-configuration** window is opened. You should see 3900/44 and 2000/40 in the **Configuration** window. If not, double click on **Instrument** button and select **Varian GC/MS #1**.
2. Click **Manual control** button in the **2000.40** window. Then click **Adjustment** button and select **Adjust RF Tuning**. You will obtain a screen which will help you to determine whether the RF voltage is in tune throughout the entire mass range of the Saturn GC/MS. The text just to the left of the RF button will indicate if an adjustment is necessary. If the RF Ramp is not passing the test requirements, open the MS front door and adjust RF adjustment screw. Click the **Save Result** button when RF tuning is complete.
3. Click **Adjust Calibration Gas**, this will adjust calibration gas (FC-43, perfluorotributylamine, $C_{12}F_{27}N$) pressure. The text just to the left of RF button will show if an adjustment is necessary. The acceptable ion time range is 300-1000 μsec . If the ion time out of range, open the MS front door and use the Calibration Gas needle valve to bring Cal Gas status to OK.
4. We are not using Chemical ionization. **Do not adjust CI reagent.**
5. Run auto tune. Click on **Auto Tune** and select **Air / Water Check** and **FC-43 Mass Calibration**. Click **Start Auto Tune**. Wait until the "Auto Tune completed" appears in the message window. Consult Saturn GC/MS Hardware Maintenance Manual for troubleshooting when it is necessary.
6. The **Current Module Attributes** (section window on the left on **Hide Keypad** button) shows the current settings and tune status of the instrument. You can verify before a run that the instrument tune is valid.

7. **Note on required frequency of tuning:** on daily basis, the only tuning checks that need to be made are the air / water check and FC-43 mass calibration. When the trap temperature is changed the RF voltage ramp should be checked and adjusted. To optimize the mass axis stability, mass calibration should be run with the GC column oven at or near the maximum temperature for planned GC/MS runs. However, this precaution is not required if the GC is run in constant flow mode. The Varian field engineer recommends not to check the electron multiplier voltage biweekly even though it is suggested in the manual. He recommends examining the electron multiplier voltage tuning by applying “daily checks.mth”.
8. Perform “daily checks.mth” segment 1 (air and water check) by turning on RF, filament, and multiplier. When the GC is directly connected to the MS the ion counts should be around 500-900. However, when the TGA is connected to GC/MS through the 2-way valve the ion counts may as high as 5000. Try to achieve the least possible air-water count by leak checking (see step 12 below) and tightening the nuts. **Note:** The 2-way valve is delicate. Do not touch the 2-way valve unless you are trained to use TGA.
9. Perform “daily checks.mth” segment 2 (dust check) by turning on RF, and multiplier. The HMN/Background should be around 2000.
10. Perform “daily checks.mth” segment 3 (Cal Gas check) by turning on Cal Gas, RF, filament, and multiplier. Check to make sure a peak at m/z 131 is present.
11. Perform “daily checks.mth” segment 4 (peak 131 check) by turning on Cal Gas, RF, filament, and multiplier. The peak valley between peak at m/z 131 and peak at m/z 132 should be lower or equal to half peak height of peak at m/z 132. In other words the two peaks at 131 and 132 should be clearly discernable.
12. Performing “daily checks.mth” segment 5 (leak check) by turning on RF, filament, multiplier, and turning off Cal Gas. Carefully spray Air Duster (1,1,1, and 2-tetrafluoroethane) near the GC column, injection port and around MS then monitor the spectrum for m/z around 69. If the system is relatively leak free then there will not be a sudden increase of peak size on the spectrum.

2.6 Qualitative and Quantitative Analysis of GC/MS

1. The operating conditions depend on the sample. It is beyond the scope of this SP to give instruction on how to develop a proper operating condition. The operator is advised to consult with manufacture and reference books. Guideline on how to prepare a GC/MS acquisition method is located in the Tutorial Manual on pages 46-55 and the Saturn 2000 MS Manual-Operation Manual, pages 148-228, pages 316-361.
2. There are different methods to identify chemical compounds using GC/MS. The operator is instructed to read the Tutorial Manual to learn all of them. This SP will only give one commonly used method which identify compounds through a compound list.
3. Go to **File>Activate Method** to display the file selection dialog. Highlight the method (file type = mth) and click **Open**. When **System Control –Varian Saturn #1 Ready** appears, Click on **Inject Single Sample** option. Fill in information about the sample run in the **Inject Single Sample** dialog. You may also specify the **Data** files and **Recalc list**. Click **Inject**. When **System control –Varian Saturn #1 waiting** appears, inject sample at a constant speed. Raw data from the sample run is saved under “c:\VarianWS\data\the sample name”. The file type is sms.
4. Open the MS data review application by clicking the **MS Data Review** button on the MS workstation toolbar. The **Plot Chromatograms and Spectra** window will open. Select **File>Select File/Chromatogram** and highlight the data file of interest. At this time the chromatography of interest is displayed in **MS Data Review Window**.
5. In the **MS Data Review Window** select **Spectrum List>Create New Spectrum List**. This command opens the **Create Spectrum List** dialog. Type a name and choose a location in the **Create New Spectrum List**. Click **Save**. A dialog window will ask “Make this file the

- active file?" Click **Yes**. Select **Spectrum list >Build Spectrum List From Active Chromatogram**. The **Edit Integration Parameter Dialog** will open. Use the default value, click **OK**.
6. Click **Library Search Spectrum**, highlight one peak and click **Library search spectrum** to get the "hit list" which is a compound list of possible matches from the library search (If the best match in the hit list is not the compound you used in your standard preparation, highlight the compound name you used in your standard from the "hit list" and click **Replace Match** in **Spectrum List Window**). Repeat this step until all the peaks have been identified. Click **Updates All Searches with Match**. You may print out the chromatogram, spectrum and the spectrum list by click **Print**. Make sure to check **Preview reports** under **File** in **MS Data Review Window** so you can preview the report before it is printed out. The qualitative analysis to identify the compounds of interest is now complete.
 7. If you are performing quantitative analysis on a data file without a data handling method and recalculation list, go to step 10.
 8. Select the **Process Data** button in the **MS Data Review** toolbar. This will open **Process Data View**. Select the proper method file and recalculation file by clicking **Browse** button. Click on **Edit** behind the **Recalc List File** button. The **Recalc List Window** will open. Click **Add**, and then click **Browse**. Select the data file to be analyzed and click **Open File**. Select **All Lines**, **Make a report** and **Preview reports** under **Processing Rules**. Click **Process** button.
 9. Check **Make a Report** and **Preview Reports** under **File** in the **Process Data** window. In the **Process Data** view, click the **Print** tab and select among the listed options, such as calibration curve report, summary report.
 10. To do quantitative analysis we first need to build a compound table. Click **View/edit Methods** button from the Varian star toolbar. Edit a data handling method following the instructions in MS Data handling Users Guide. You can also use the default value in **Calculation Setup** and **Result Treatment**.
 11. Click **Compound Table**, and then click **Select Data File**. Highlight the data file of the midpoint of the calibration standards. Click **Open File**.
 12. Close **MS Data Review Window** and go back to the **Method Builder** window. Click **Build Compound List**. Following step 5 and step 6 to build the compound list. Click **Import Compound List**, and then click **Select**.
 13. If an internal standard has been used, double click on the line in the compound table containing the internal standard name. This will open a new window with **Compound Attributes, Quan Ions, Calculation....**Select **Compound Attributes** and select **Internal Standard** in the **Compound Type** section. Click **Calculation**. In the **Calculation Dialog** enter information about the number of calibration levels and their concentrations. You can enter this information for the first compound in the table, and then use the **Fill down** feature to apply it to the other analytes in the compound table. Follow the instructions in the tutorial manual to examine the quality of the mass spectra for the highest and lowest concentration level of the calibration curve. Follow the instruction from the tutorial manual to confirm that the integration parameter is acceptable at other calibration levels.
 14. Building a recalculation list for calibration: Click on the **Automation File Editor** application button from the Varian workstation toolbar. Right click on a data file and select **RecalcList File > Edit**. In **Automation File Editor** select **File > New > RecalcList**. Save the recalculation list (file type=rcl). When the new **RecalcList** appears, begin by adding a line and selecting **New Calibration Block** as the **Sample type**. In the **Quantization - RecalcList Window**, click **Browse**. The **Open Data File** dialog will appear, enter the file names for the data files at each concentration level and associate them with the calibration levels. Select **File>Save** and **File>Exit**.

15. Open the **Process Data** view in the **MS Data Review** by clicking the **Process Data** icon. Select the proper method file and recalculation file by clicking **Browse**. Select **All Lines** under processing rules and check **Make a Report** and **Preview Reports** in the **Process Data** window. Click **Process** button.
16. Make sure to check **Preview Reports** form **File** in **MS Data Review Window** so you can review the calibration curve report. Review the calibration curve following the tutorial manual. Click **Print** to print out the calibration curve for each compound of interest.

2.7 Start-up and Shutdown

The GC/MS does not need to be shutdown over night or over the weekend. Turning the system off and on creates electrical surges and wide changes in temperature that could shorten the life of certain electronic components. Therefore, it is suggested that the instrument be left on at all times. You can shutdown the system only if GC/MS will not be used for over a month, or if maintenance needs to be performed. Read Hardware Operation Manual (page 75) for more details.

2.8 Maintenance

Please follow the instructions in the Hardware Operation Manual to carry out maintenance tasks. The general GC maintenance, including check and renew gas supplies, leak check, gas purifier replacement and injector maintenance, is located on pages 115-127. The periodic maintenance of MS is located on pages 79-86.

3.0 Records

Analysis printouts and summary reports will be recorded in laboratory notebooks or in project-specific binders. All records will be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records). The following table summarizes the Quality Assurance (QA) requirements:

QA Record

- Analysis Printout
- Summary Report
- Scientific Notebook

4.0 Appendices

No appendices are applicable to this SP.

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