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GC/FID Standard Operating Procedure

Procedure:

Department: Bioeconomy Institute

Building/ Room Number: BRL 1115

Supervisor: Jacquelyn Baughman

Procedure Overview:

Health and safety information for materials used: Proper PPE needs to be worn while handling chemicals that are tested. Compressed gas cylinders are used.

Hazard Control Measures:

- Safety glasses and lab coat
- Latex or nitrile gloves (for handling samples)

Waste Disposal Procedures: Biodiesel can be placed into appropriate waste containers.

Decontamination Procedures: None

Spill Containment and clean up procedures: None

Using substances requiring special procedures: No

Written By: Pedro Ortiz

Date: 4/25/2008

Revised By: Zach Bartlett

Date: 6/10/2011

Approved By:

Date:

Detailed procedures, operation instructions, maintenance, and emergency contact information list is attached.

Biomass Particle Size Reduction/Grinding using Retsch Type SM2000 Heavy-Duty Cutting Mill

Equipment Description:

The Varian 430-GC is a single-channel chromatography instrument that is used to analyze gas phases of liquid or headspace samples. It is equipped with a flame ionization detector (FID), and a water-stable wax chromatography column from Restek, although the columns on this instrument are interchangeable. UHP/zero helium gas is used as a carrier (mobile phase), while UHP/zero hydrogen and breathing-grade air are used to generate a flame inside the detector. The GC is controlled from a computer with a software package called GALAXIE Chromatography Data System. This software is used to control the GC and for data acquisition.

Potential Hazards:

- There are no specific hazards related with this equipment but some caution needs to be taken with the surrounding equipment (e.g. gas cylinders, hazardous chemicals, electrical outlets, etc.)
- Compressed cylinders used are: helium, hydrogen, and air
- The auto-sampler system contains moving parts that are controlled by the automation. Keep extremities away from the needle and the moving parts.

Start-Up Checklist:

- Make sure that the carrier gas cylinders (helium, hydrogen, and air) are open and set to 80, 40, and 40 psi respectively. If the total pressure of any of the cylinders is at 500 psi or below, notify the supervisor so that a replacement cylinder can be timely ordered.
- Should be able to see the green lights on the front face of the instrument showing it is ON and READY. If a red light shows, the common practice is to restart the instrument by turning the power switch OFF and ON.
- Make sure the solvent vials on the auto-sampler contain enough of the corresponding solvent for the tests to be performed. Vial I should be filled with HPLC grade methanol and vial III filled with deionized water.

Instrument Operation:

1. Start Up

- a. To start up the GALAXIE software, double click on the GALAXIE icon on your desktop.
- b. Enter your assigned username in the space identified as *User Identification*, and click ok. If a GALAXIE window is already open, but not running on a sequence, click **Session>New Login**.
- c. The program will initialize and show a blank *Data* screen.
- d. Start the communication with the Varian 430-GC by checking the box as identified **Varian 430-GC-R** box located on the systems tab.
- e. Then the default screen will show the activity of the detector and the *Status overview*. Here you can check the status of the system. The system needs to be *Ready* in order to run samples.

Note: Browse through the different windows in the program by using the Data, Systems, and Calibration tabs at the bottom left part of your screen.

Sampling:

1. Starting a new sequence
 - a. If you already have a saved sequence, go to **Opening an existing sequence**.

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- b. Go to the top taskbar in the GALAXIE interface and click on **File>New>New Sequence**.
- c. The *New Sequence Wizard* will open and will ask to select the system, number of samples (you can always add or delete lines later), and a file name.
- d. Fill the required spaces and click the *Next* to go to the next screen and then click *OK*. The *Sequence* will appear on the *Data* tab, showing the status and the options.
- e. New lines can be added with the plus sign button and deleted from bottom up using the minus sign button. Each line represents a sample to be taken and analyzed.

Opening an existing sequence:

- a. A sequence is a list of samples to be analyzed including the information on the file names and analytical methods to be used. Setting up a new sequence is not always necessary; if a sequence was previously saved it can simply be loaded to analyze a similar set of data. Make sure you include a short description about the application of the particular sequence.
- b. Go to the top taskbar in the GALAXIE interface and click on **File>Open>Open Sequence**.
- c. Select the previously saved sequence you want to use from the data directory.

Setting up a sequence and sample analysis:

- a. Select an analytical method from the *Method* drop down menu arrow of the first line. After selecting the method, all the settings needed show their method default values on this line.
- b. Here you can select a prefix, ID, and suffix for the filenames of the chromatograms. The preferred values are the default ones-the sample ID associated with the vial. If it is absolutely necessary to change the filename extensions like the prefix, click on the three dot button on the first line of the *RunName(prefix)* column. This will open the *Token run name* window, where you can select the parameter to be used as filename prefix by double clicking on the name.
- c. You can also choose to use any custom name by writing it down on the *Define run name formula* block. For example: if you choose the date and time of the run as your filename double click where it says *StartRunTime* and the *ok* button.
- d. Make the *RunID(suffix)* column blank for all samples.
- e. Select the number of injections you would like to be performed. Three is standard.
- f. Make sure the vial number matches up with the number on the auto-sampler tray.
This Step is Critical.
- g. It is recommended that the last line in the sequence is the *Standby* method. The reason for this is to stop the consumption of air and H₂.
- h. Now you can select the method for each line manually or you can copy the method selected on the first line for other lines.
- i. To start analyzing samples simply click on the green *Play* button located at the top of the sequence list.
- j. If the temperature set-points on the sequence do not match with current temperatures the sequence will not start immediately. If the actual temperatures are the *Standby* or *Bakeout* temperatures the GC will take approximately 5 minutes to stabilize.
- k. To stop the automation click on the **navy Stop** button, if you want to close the sequence you will need to then click on the **red Stop** button that appears once you stop the automation.

Reading individual results:

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- a. On the Galaxie software each chromatogram can be opened individually to read the results.
- b. On the top taskbar of the GALAXIE window click **File>Open>Open Chromatogram...**
- c. Then go to the folder where the data was saved and look for the particular chromatogram you want to look at. The data is organized in folders identified by all three: username, month and year of test.
- d. Once the desired chromatogram is open, click on *results*. The chromatograms and the table of results will be located on the right side of *Data* tab. On the results table the quantity (v/v) column corresponds to the concentration of the gas identified on that line and the *Time* (Min) corresponds to the retention time. For any unknowns note the retention time since this might be useful to later determine what these signals mean.

Calibration verification

- a. Use a single or a set of standard solutions to check the calibration on the desired method.
- b. Set up a line on your sequence where you analyze using the analytical method and test the standard as an unknown and indicate the appropriate vial number on the tray. Typically the standard for verification is injected 3-4 times.
- c. To see the results follow the Reading results procedure below. The final results should show the correct peak identification and concentrations (*Quantity v/v*) on the standards as prepared. If the results do not match take note of your findings and notify a supervisor.

Shutdown

- a. Make sure you keep the work area clean at all times.
- b. The 430-GC should remain turned on at all times but the computer may be shut down if instrument is left unused for long periods of time (e.g. weekends).
- c. Make sure the instrument is left on *Standby* method keeping the temperatures low, the injector heaters off, and the flame and detector turned off.
- d. Close the main valves of the H₂ and air cylinders. This will help saving these gases while the instrument is on *Standby*.

Maintenance

- a. Sometimes the software could show some errors related with the set-up of a sequence. Follow the onscreen instructions to fix the problem.
- b. If the computer loses communication with the instrument it will show an error both on the screen and on the front panel LED. If this is the case try restarting the computer. If this does not fix the problem contact the supervisor.
- c. Proper care of the equipment is necessary to prevent any malfunction and unscheduled maintenance.
- d. Internal components of the 430-GC are non-serviceable. Maintenance work for this equipment is to be performed by a Varian Inc technician.

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Emergency Contacts

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Approved Trainers:

Dustin Dalluge, Zach Bartlett, Adrian Ramirez

Training Sign-Off

<u>Trainee</u>	<u>Date</u>	<u>Trainer</u>
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