

**GC ANALYSIS OF DMS AND DMSO**

December 11, 2014

**1.0 PURPOSE**

This is a gas chromatographic method to obtain a purity value for dimethyl sulfide (DMS) and dimethyl sulfoxide (DMSO).

**2.0 SAFETY/HEALTH****2.1 PPE**

Wear standard safety equipment consisting of safety glasses or goggles, flame retardant clothing and laboratory nitrile gloves when using these materials.

**2.2 SAFETY SYSTEMS**

N/A

**2.3 HEALTH HAZARDS**

N/A

**3.0 PROCESS DESCRIPTION****3.1 PROCESS OVERVIEW**

N/A

**3.2 SCOPE/BOUNDARIES**

N/A

**3.3 EQUIPMENT INVOLVED**

Agilent 6890 Gas Chromatograph equipped with Chemstation computer and software. Chromatograph is configured with a flame ionization detector and with a QUADREX 007 series bonded phase fused silica capillary column (CAT #07-1-15W-3.0F). Column specifications are: 15 meter, 0.32 mm ID, and 3.0 micron film thickness. Similar columns may be used.

**3.4 INSTRUMENT SET-UP**

- The Agilent 6890 GC has all instrumental parameters stored in "Method" and "Sequence" files for each individual analysis. Usually the GC is always on. If the GC is not on, depress the Power button located just below the oven door. The GC should start up without any further assistance. Allow about 15 minutes for gas flows to come up and FID to ignite. After 15 minutes, if the signal reading on the GC display panel is reading <5.0, contact the Analytical Laboratory Manager for assistance. If necessary, power up associated computer and monitor. If GC and computer are already on go to section B.
- For GC analysis of DMS and DMSO click on the **START** icon and move selector up to **Programs Files**, then over to **Chemstations**, and then click **Instrument 1 on-line**. This will load up the Chemstation software.
- When the Chemstation screen loads up click on **METHOD** in the toolbar. In the dropdown select **LOAD METHOD**. Scroll down through the methods and "double click" on the Customer Name (example: **EXXON**) or if performing the Daily analyses, the **DAILY** method. (Each customer is listed in the METHOD files). This will load the proper analytical method.
- Now click on **SEQUENCE** in the toolbar. In the dropdown select **LOAD SEQUENCE**. Scroll down through the sequences and "double click" on the Customer Name (example: **EXXON**) or if performing the Daily analyses, the **DAILY** sequence. (Each customer is listed in the SEQUENCE files). This will load the proper analytical sequence.

**NOTE: If there are any questions about the actual instrumental parameters or sequence parameters see Quality Manager or QC Chemist, Click on METHOD, then EDIT METHOD and then start with the INJECTORS icon and work your way to the right filling in the required information as listed in attachment C. After completing this, click on METHOD, and then SAVE METHOD. The parameters that were put into the instrument are now saved to the proper method.**

### 3.5 Analysis

- Fill a 2.0 mL auto sampler vial to the neck with the product to be analyzed using the disposable Pasteur Pipettes. Place in position 1 of the auto sampler turntable (Up to 100 samples can be analyzed automatically as listed in the sequence table.) Make sure that Solvent vials in the auto sampler are filled and the rinse vials are empty. (Solvent A is methanol, and Solvent B is water.)
- Click on **SEQUENCE**, and then **SEQUENCE PARAMETERS**. At the **SEQUENCE PARAMETERS** screen type your name in the "Operator's Name" space. Use the mouse pointer to select "Prefix/Counter" and click. Then, under "Signal 2" column write the days date in YYMMDD format. Under the Counter column write 0001 (if necessary.) Now use the mouse pointer to select the "Subdirectory". If no subdirectory is listed type in the Customer name or if performing the Daily analyses, type in **DAILY**. (If the computer prompts that there is no subdirectory of this name and asks to create it, select **YES**.) Click **OK** at bottom of screen.
- Click on **SEQUENCE**, and then **SEQUENCE TABLE**. At the **SEQUENCE TABLE** screen type in the date and sample name in the SAMPLE INFO section. If the sample is for a customer shipment also type in the Order Number and the Lot Number below the date and sample name. Below the SAMPLE INFO section there is a list of the sequence particulars for each sample. It should appear as follows:

Line	Location	Sample Name	Method Name	Inj/Location	Sample Type
1	Vial 1	Product Name	Customer Name	1	Sample

If the particulars are not listed above use mouse to select each and correct as per the example and then click **OK**.

- Click on green START icon on Chemstation page. Instrument will inject sample and then generate a report on printer.

### 4.0 DOCUMENTATION

Data is automatically saved to the file path listed in the method parameters. The data is logged in either the Operator's lab notebook or the Analyst's personal lab notebook. The printed results chromatograms are saved for quick reference. (The actual data is on file in the computer if a report is required at a later time).

### 5.0 GLOSSARY

None associated with this procedure.

### 6.0 REFERENCES

- Attachment A Sample DMS Chromatogram
- Attachment B Sample DMSO Chromatogram

### 7.0 TEST SPECIFICATIONS

- DMS purity 99.5% minimum (Sample chromatogram attached)
- DMSO (Industrial Grade)purity 99.7% (Sample chromatogram attached)

### 8.0 REVISION LOG

DATE	SECTION/PAR	CHANGE DESCRIPTION
8/21/2007	All	Last Revision Before Issued In Web-based QMS
1/8/2009	3.4, 6.0, 7.0, Additional attachments	Deleted "attachment C" and inserted "Quality Manager in 3.4 NOTE. Deleted "attachment C" in 6.0 7.0 Changed 99.2% to 99.5% minimum, Changed Greg Ashe to Quality Manager Added attachments "A" and "B"
1/12/2012	3.3, 3.5	3.3 – deleted (GC-5) in first paragraph. 3.5 – deleted labeled GC5 printer in 4 <sup>th</sup> bullet to reflect Tuscaloosa Operations.
12/11/2014	3.4	Changed GC display panel reading from 1.0 to 5.0 and added QC Chemist to NOTE.

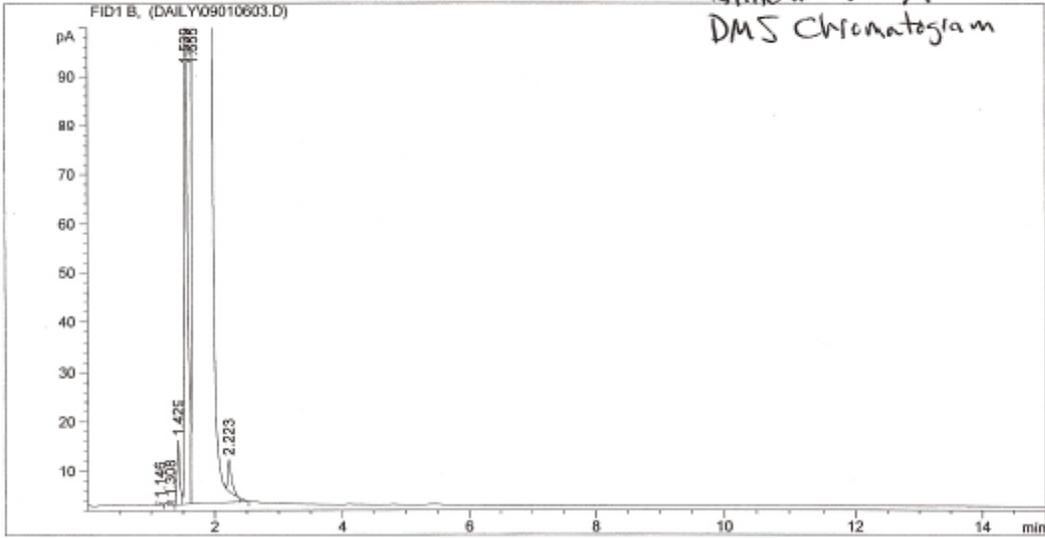
Comments, questions or suggestions, please e-mail

Quality Manager

01-06-09 DMS STG

-----  
Injection Date : 1/6/2009 9:14:57 AM                   Seq. Line : 3  
Sample Name : DMS                                        Location : Vial 23  
Acq. Operator : K.MCCAIN                                Inj : 1  
  Inj Volume : 1 µl  
  
Acq. Method : C:\HPCHEM\1\METHODS\DMS.M  
Last changed : 10/1/2008 8:38:21 PM by k.mccain  
Analysis Method : C:\HPCHEM\1\METHODS\DMSO.M  
Last changed : 9/30/2008 10:01:30 AM by g. ashe  
DMSO ANALYSIS

Attachment "A"  
DMS Chromatogram



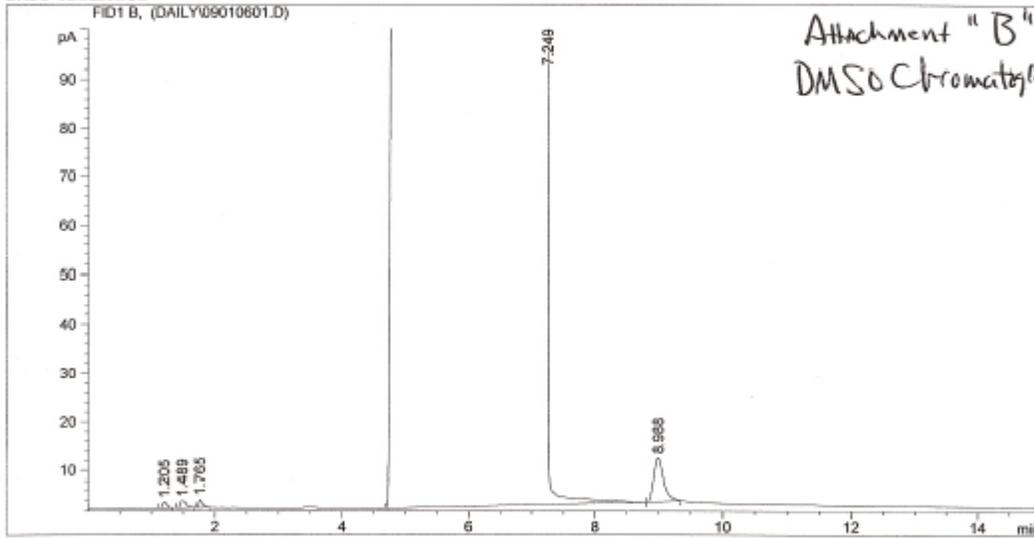
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Area Percent Report  
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Sorted By : Retention Time  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 B,

01-06-09 DMSO STG

=====  
Injection Date : 1/6/2009 8:42:24 AM                   Seq. Line : 1  
Sample Name : DMSO                                        Location : Vial 21  
Acq. Operator : K.MCCAIN                                 Inj : 1  
  Inj Volume : 1 µl  
Method : C:\HPCHEM\1\METHODS\DMSO.M  
Last changed : 9/30/2008 10:01:30 AM by g. ashe  
DMSO ANALYSIS



=====  
Area Percent Report  
=====

Sorted By : Retention Time  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 B,

Peak	RetTime	Sig	Type	Area	Height	Area %
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