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Department of Forensic Science

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DISCOVIR INSTRUCTIONS

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Tips

1) Do not process any data or open additional windows while running (New Run Workbook, Data Workup, Utilities Workbook, and Print Workbook are all associated with the DiscovIR and only ONE can be opened at a time).

2) Always run a blank before each sample (standard, blank, sample, blank, sample, blank, etc.)

3) Unless otherwise specified, the background used is collected at the beginning of the run

Daily Start-up

1) Open Utilities workbook (icon on desktop).

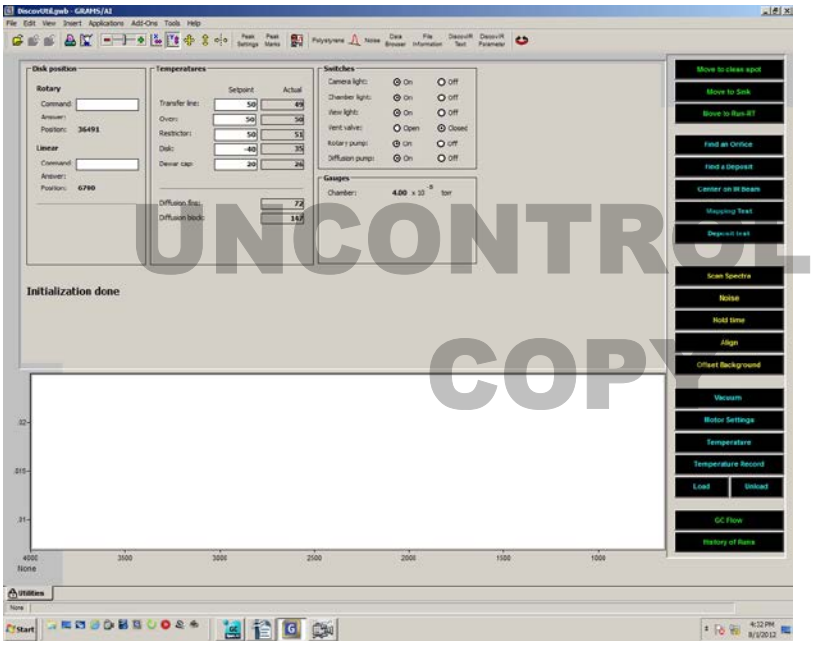
2) Check vacuum pressure under Chamber Gauges (if only rotary pump is on, the pressure should be at least 4 x 10^{-2} torr or less, if both the rotary pump and diffusion pump are on, pressure should be at least 1 x 10^{-3} torr or less).

3) If diffusion pump is not on, turn it on.

4) Turn on camera, chamber and view lights.

5) Fill MCT detector (hole in the top center of the IR) and large dewar (covered by metal cap/holds ~10L) with liquid nitrogen. This can be done at the same time. MCT detector will be 'full' once it starts seeping out the bottom (~1/2 L, lasts ~18 hours). One liter of liquid N_2 is ~1-1.5 hours run time for the large dewar. Allow instrument to cool for ~10 minutes before starting QA process.

6) In Utilities workbook–



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a) click “Scan Spectra” (right hand bar), several boxes will appear and disappear before the final box appears (just a few seconds) → Under new name base (halfway down box) - type today’s date (ex: 061912) → change Path to C:\discovir\data\Month\dailyqa folder (ex: C:\discovir\data\1206June\dailyqa) → Be sure the Background path/name field is empty → click background → “Background complete” message will appear

b) click “Align” (right hand bar) → Under base of file name - type date and quant (ex: 061912quant) → Ensure Path and Background fields contain correct daily QA information (ex: C:\discover\data\1206June\dailyqa\ and C:\discovir\data\1206June\dailyQA\ 061912.Background.spc) → select Spectrum type as Interferogram → click OK → record max voltage once stable (seen as “Maximum so far”) on QA log pages (must be between 4-7.5 volts) → Esc to exit

c) click “Noise” (right hand bar) → change file path to date and noise (ex: 061912noise) →Ensure path name is correct (ex: C:\discovir\data\1206June\dailyqa) click 5 → when average noise box appears, record on QA log pages (must be between 0.4-0.7 mAbs) → Click OK

d) click “Polystyrene” (top bar) → Get spectrum → after collection, click OK, minimize GRAMS software, and open Polystyrene shortcut on desktop → check measured values against NIST reference values listed at the top of the polystyrene text file (must be w/in +/- 2); peak height of the 1601 peak must be greater than 400 → check off on QA log pages

e) Click “Move to Sink” on right hand side to reposition disk

f) Change temperatures to the following:

Transfer Line: 325 degrees
Oven: 325 degrees
Restrictor: 325 degrees
Disk: -40 degrees
Dewar Cap: 20 degrees

**Click to another box after each entry to ensure value is set. Wait for all to reach set temps which will take ~30 minutes.

if for some reason the results for this QA are extremely off, be sure to check that the black dot on the disk is not directly below the column deposit

<u>To Set Up Sequence – GC Portion</u>

**To open GC if not already opened → click on the purple (“OpenLAB Control Panel”) icon on the desktop → click “Launch”

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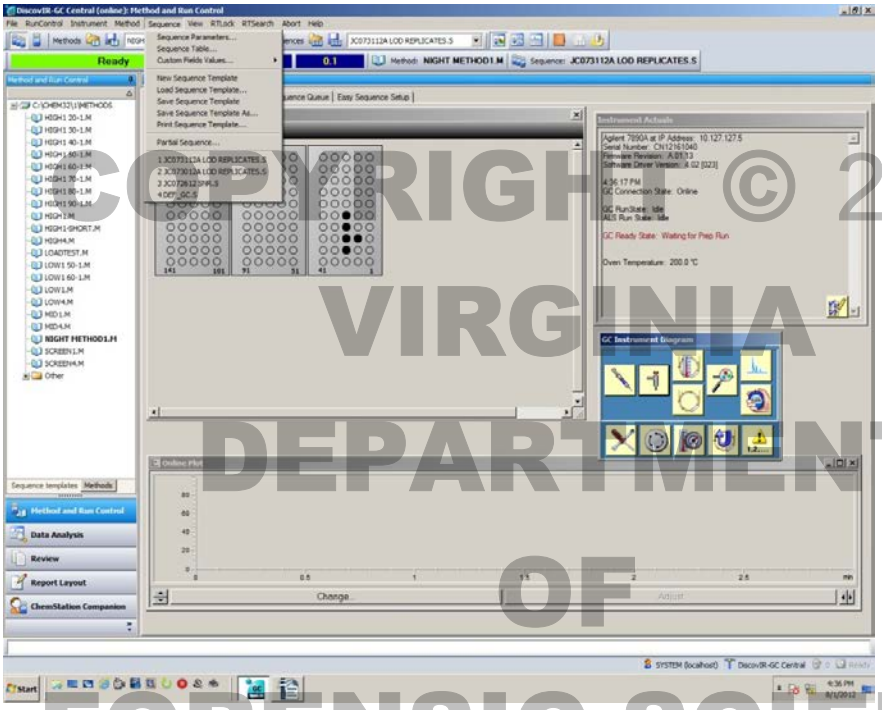
1) Open GC Window via the blue ("GC Open Lab") icon on bottom of screen



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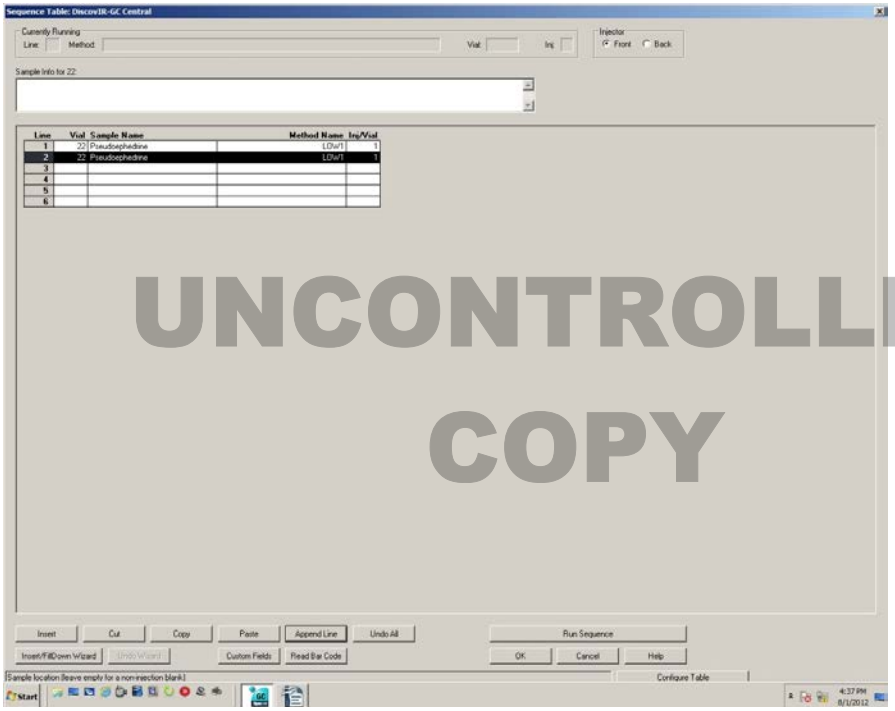
2) Under "Sequence" on top bar → Load "Sequence Template" → "Default" sequence
*always start sequences beginning with the default sequence because there are certain parameters selected for the sequence that will be lost if you start with a "new" sequence

3) To open sequence table → Under "Sequence" on top bar → "Sequence Table"



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4) Enter Data for sequence → "OK" when finished

- **be sure to add blank before EACH sample**
- "Append Line" adds lines to the sequence
- Inj/Vial column can have "1" in it for entire sequence
- Method Options include (4 signifies a 4 microliter injection)

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Low1, Low4: 90-190 degrees C @ 30 degrees per minute, hold for 3 minutes
Mid1, Mid4: 170-270 degrees C @ 30 degrees per minute, hold for 3 minutes
High1, High4: 225-315 degrees C @ 30 degrees per minute, hold for 10 minutes
Screen1, Screen4: 105-315 degrees C @ 40 degrees per minute, hold 14.65 minutes
Night

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5) Add Blank with Night Method at the end of your sequence

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6) Under Sequence → "Save Sequence Template As" (ex: KH061912)

OF

<u>To Set Up Sequence – IR Portion</u>

1) Open "New Run Workbook" (Green Arrow Icon on Desktop)

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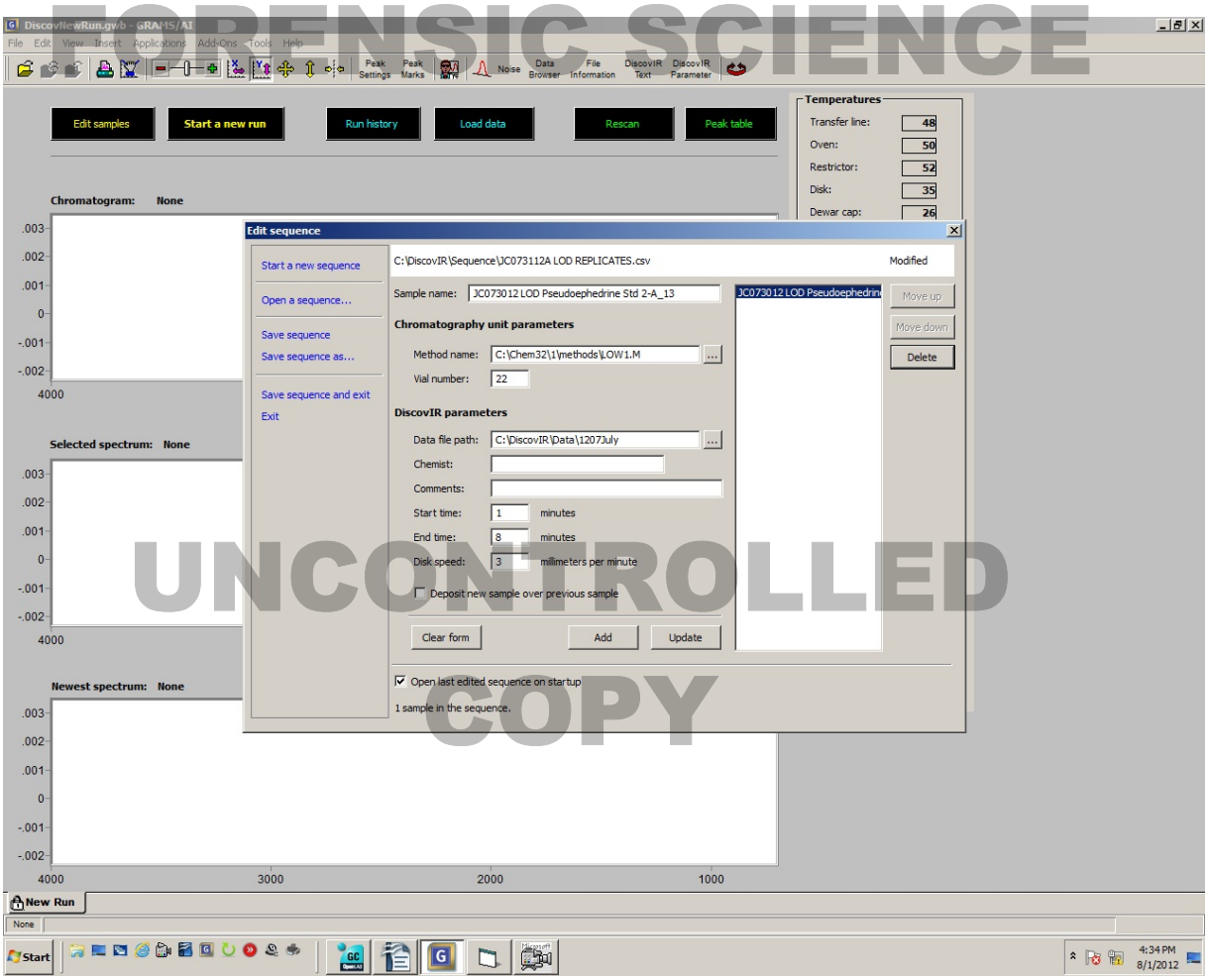

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2) Under "Edit Samples" key → Fill in information for sequence

Reminder: Add a blank before each sample; click the box that says "Deposit a new sample over previous sample for each SAMPLE (unclick for each blank)

a) Sample Name

****DO NOT use "#" keys or other character symbols (".", "/", etc.)****
(ex: KH Cocaine Standard_01)
(ex: KH Methanol Blank_02)
(ex: KH C12-12345_03)

b) Method Name

Low1, Low4
Mid1, Mid4
High1, Mid4
Screen1, Screen4
Night

c) Vial Number

d) Data file path – be sure to put data in the correct folder (ex: C://DiscovIR/Data/1206June)

e) Start Time and End Time

Low1, 4: 1min start – 8 min stop
Mid1, 4: 1 min start – 8 min stop
High1, 4: 1 min start – 14 min stop
Screen 1, 4: 1 min start – 20 min stop
Night: 0 min start – 1 min stop

f) Disk speed 3 mm/min

→ "Add" key will add entry as a new sample to run in the box on right hand side
→ If changes need to be made to a specific entry, make the changes and click "Update"; this feature will not add another line, it will overwrite the current line (if you don't click update, any changes will not be saved)

g) Add blank to the end of sequence using the "Night" method

3) On left hand column, "Save sequence As" (ex: KH061912)

4) Click "Exit" on left hand column

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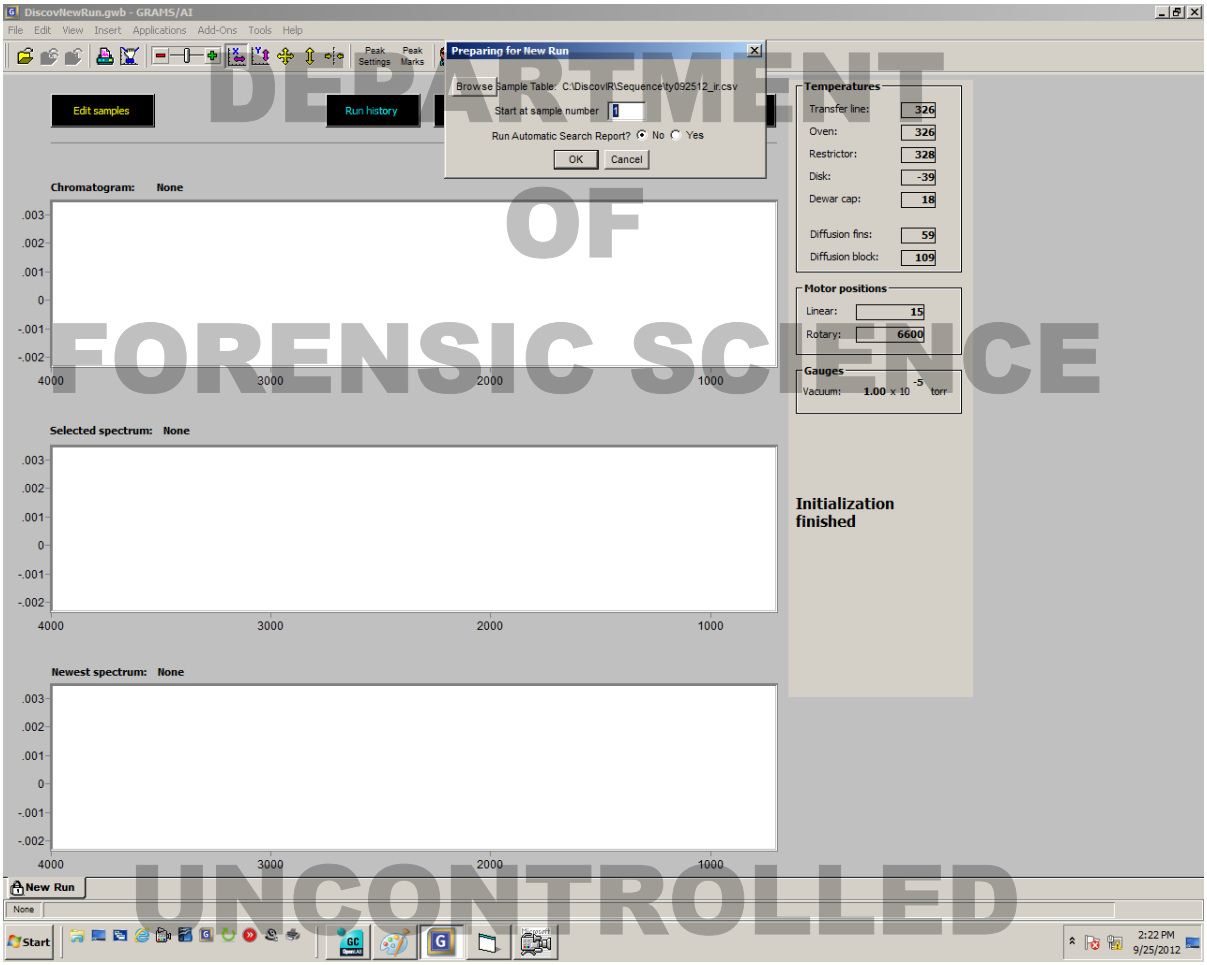
To Run Sequence

1) If GC sequence is set up and ready to go, → Click "Start a New Run" on the IR window → Wait until the message box "Preparing for a New Run" appears: Be sure the start at sample number box is correct, select "No" for "Run Automatic Search Report", and if available, select "Yes" for Enter standby mode (this feature will turn off diffusion pump and set transfer line, oven, and restrictor temperatures to 100 degrees after run is complete) → Click OK

   ** DiscovIR MUST BE STARTED FIRST in order to collect a background before the GC starts…if the GC sequence table is incomplete, finish that before starting a new run on the DiscovIR**



2) After the DiscovIR is started, reopen GC Window via the blue ("Open Lab GC") icon on the bottom task bar

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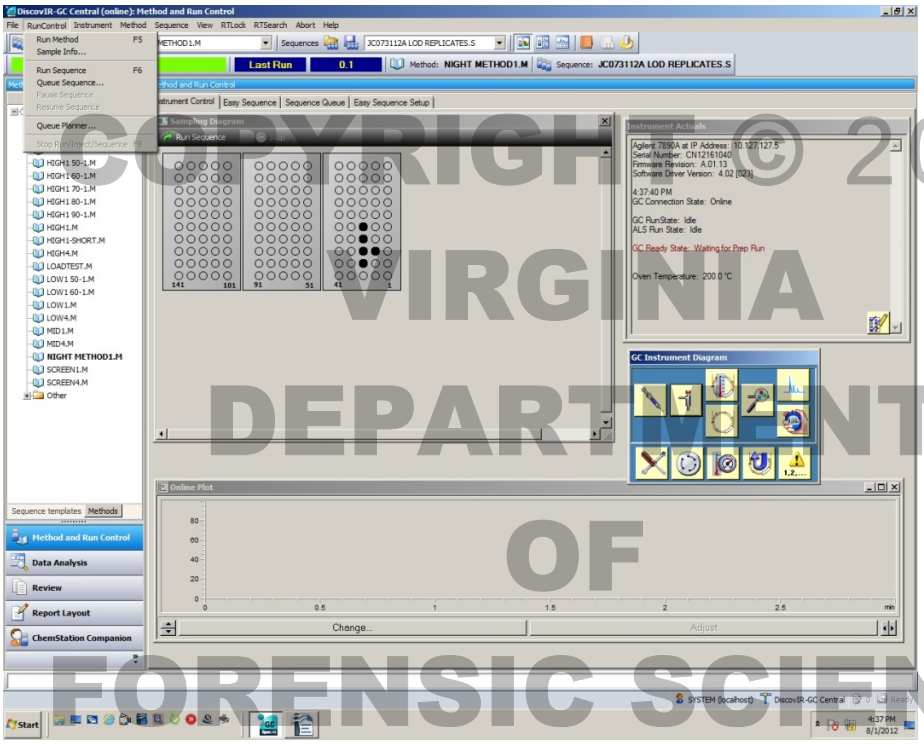
3) Under “Run Control” → “Run Sequence” → Wait a few seconds to be sure first injection is made



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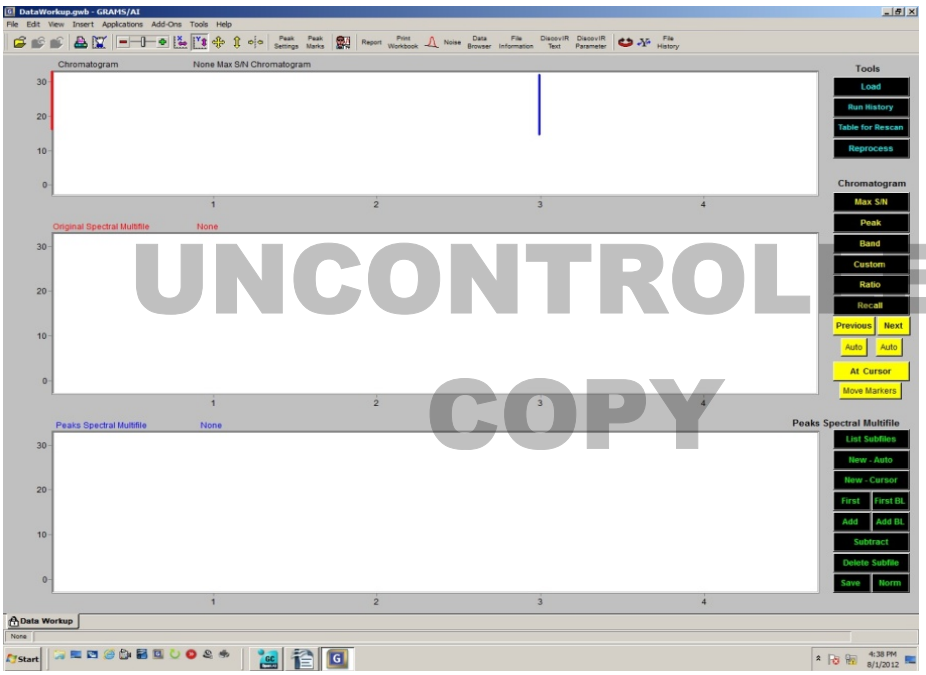
Data Processing

1) Go to Data Workup (red button with 2 arrows on desktop) → “Load” on top right hand side of program buttons bar



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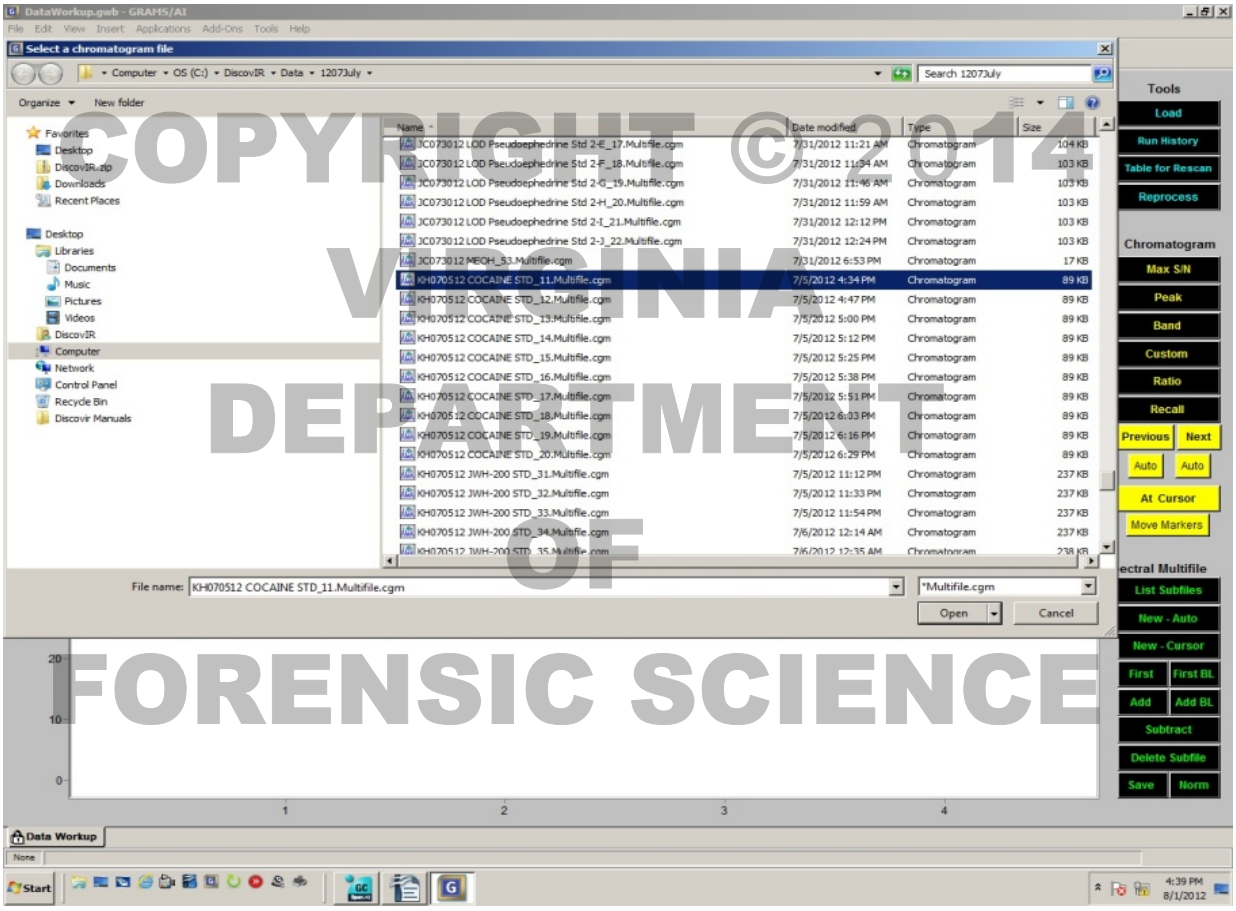
2) Find data file by name (multifile.cgm extension) (ex: KH Cocaine Standard_01.multifile.cgm) and open



3) FOR SAMPLE DATA:

Left click on the center of a peak and be sure that the horizontal line (threshold) for the cursor captures all of the peaks in the spectra (be careful not to include baseline) → Click "New-Cursor" on the right hand side (message box saying "# peak found" will appear and disappear) → Click "Save" on the bottom right hand side bar (message box saying "# spectra saved" will appear and disappear).

FOR BLANK DATA:

Open blank file as described above (multifile extension) (ex: KH Methanol Blank_02.multifile.cgm) → Click "First" on the right hand side bar (follow instructions on screen pop-up box to select a section of the spectra) → "Save"

If Necessary:

a) To add additional peaks that were not selected with the "New-Cursor" threshold → Click on the center of the additional peak → Click "Add" on the right hand side bar (follow instructions on screen pop-up box) → Click "Save"

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b) To clean up spectra (rid of water, noise, or CO_2 peaks) by specifying the baseline spectra around the peak to be used as the background → Click on "First BL" on right hand side → Follow instructions in the "Define a new Peak Subfile" window to box in the peak of interest (right mouse moves box to left or right of peak, left mouse click 'saves' the box) → If more than one peak is in spectra, click on "Add BL" for all subsequent peaks → Click "Save"

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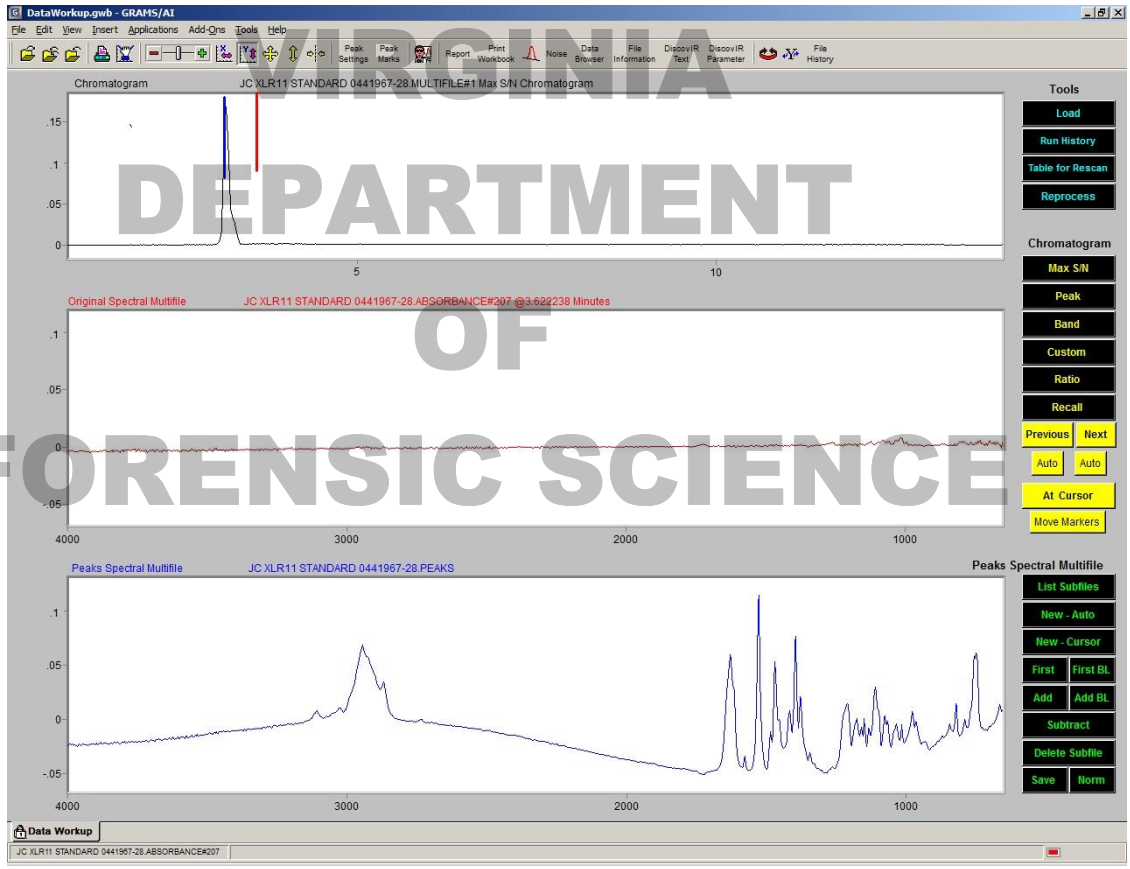
c) Click on "List Subfiles" on the right hand side to see selected peaks



Top box: Chromatogram multifile (multifile.cgm)
Middle box: spectral multifile of every single scan (absorbance.spc)
Bottom box: spectral multifile of peaks at a specific retention time (peaks.spc)

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Printing

Option 1 - Report in Data Workup (red circle with arrows icon)

1) Click “Report” icon on top bar of the screen (when scrolled over, says “Searches libraries and generates PDF report”). The following screen will appear. You can scroll through the list by left clicking on each hit. The top spectra is the sample (in red) and the lower spectra is the library hit (in blue) The lower the quality number, the better the match.

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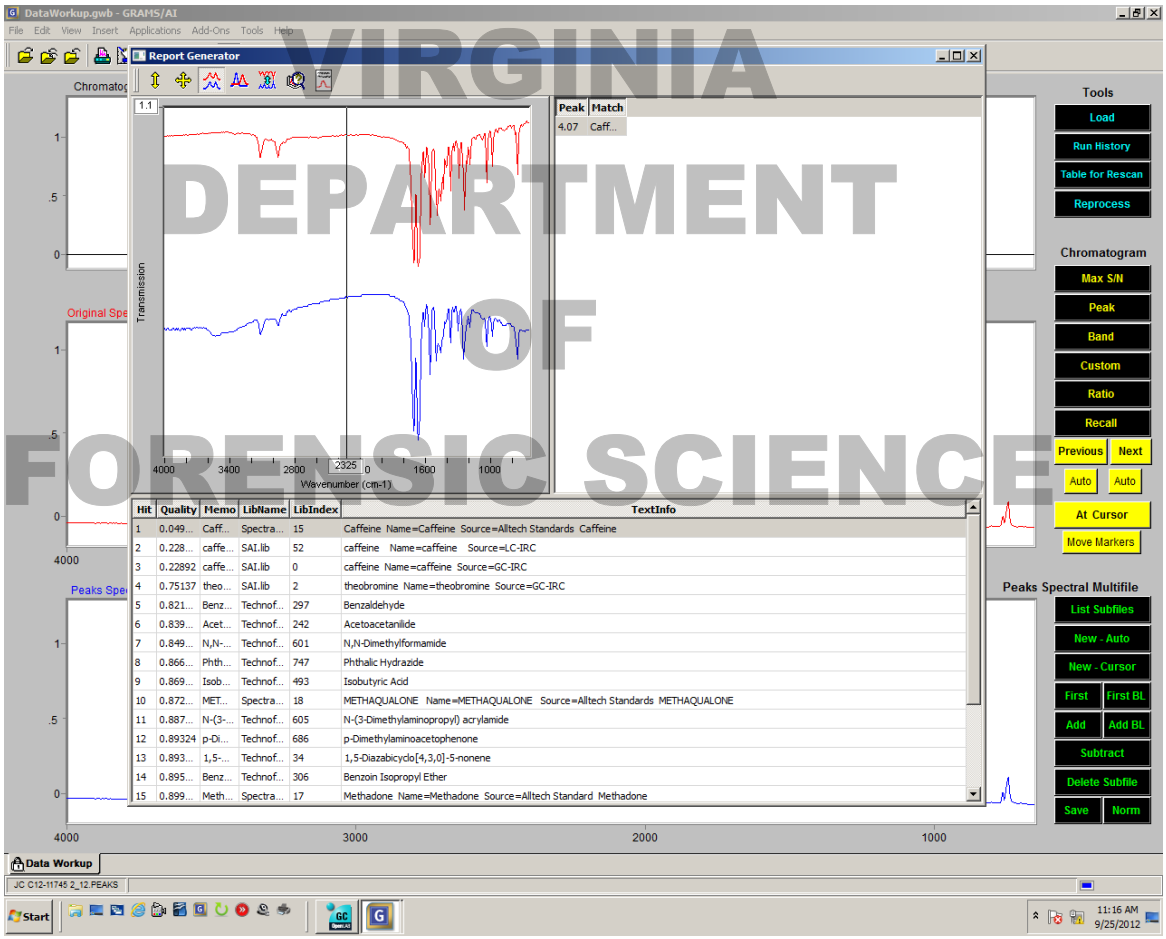

Additional useful red/blue spectra buttons across top bar

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When scrolled over, says “Autoscale Y axis” (up and down arrow): scales only the y axis
When scrolled over, says “Autoscale” (4 pointed arrow): scales both x and y axis to display the entire graph
When scrolled over, says “Stacked trace mode”: Sample on top in red, library hit on bottom in blue (seen in image above)
When scrolled over, says “Overlaid trace mode”: Sample and library hit stacked on top of one another
When scrolled over, says “Toggle between Absorbance and Transmission”: Toggles between absorbance/transmission

2) Click on the paper/report icon on the top bar of the Report Generator screen (when scrolled over, says “generate report”) → a box saying “Failed to parse the Chemstation method file. Associated files will be left blank” may appear. → click “OK” → Print

<u>Option 2 – Manual Printout (Options – baseline correction, zoom, alternate views)</u>

1) Go to Print Workbook (button with printer on desktop) → Click on folder icon in the top left hand corner tool bar

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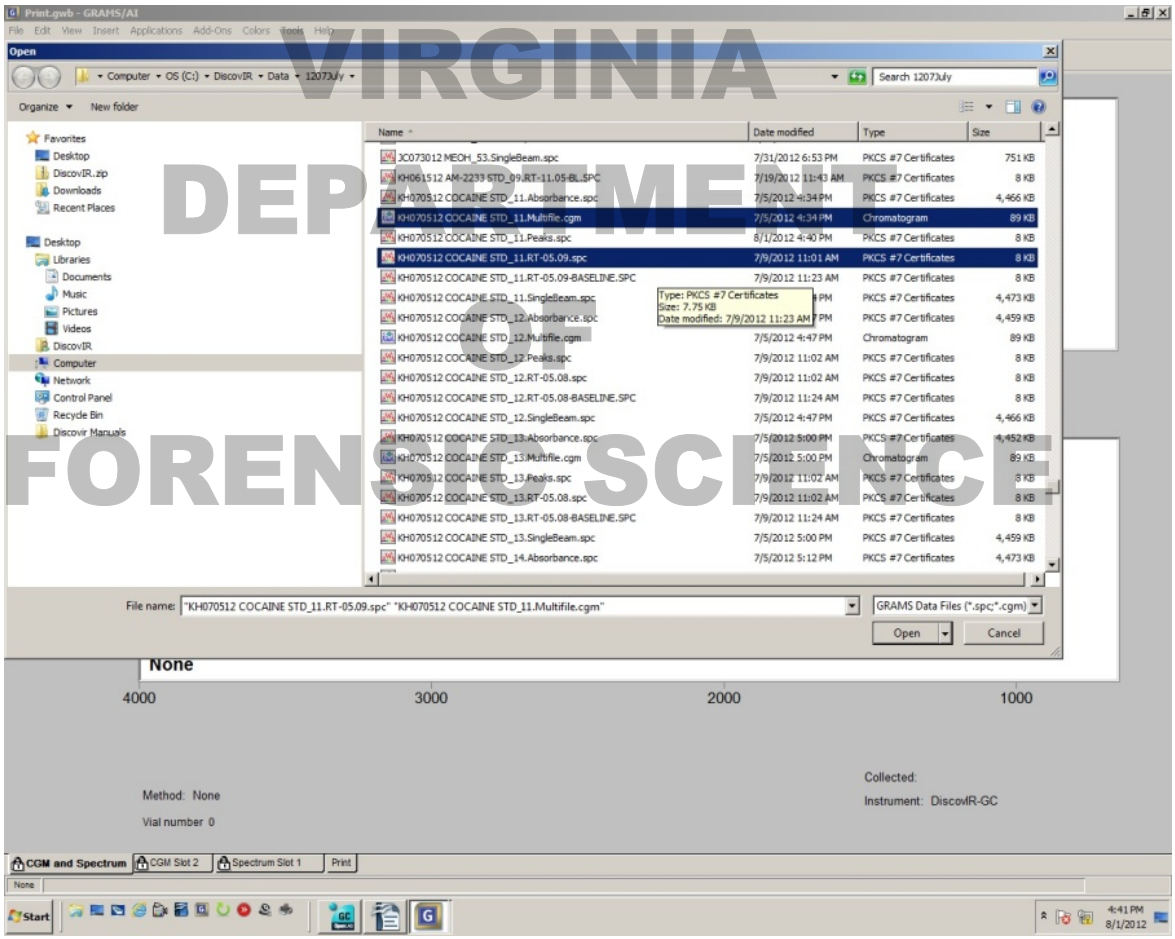

2) Find the sample file with the “Multifile.cgm” extension (ex: KH Cocaine Standard_01.multifile.cgm) → While holding the “ctrl” button, select all files for that sample that have an “RT-0#.##.spc” extension (ex: KH Cocaine Standard_01.RT-1.35.spc) There should be one RT.spc file for each saved peak in your sample. → click “Open”

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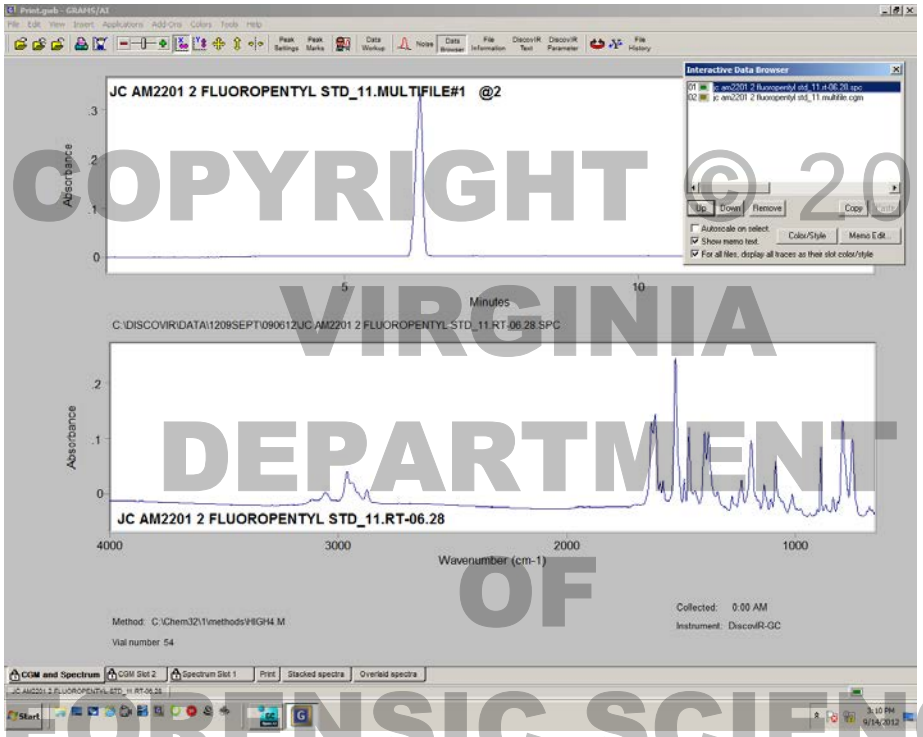
3) On the top toolbar, click on “Data Browser”. This will bring up a box that can be positioned anywhere on the screen and should list the files that were just opened. You can use the “Up” or “Down” command keys to change the position of the spectra seen on the screen. Only the files listed in positions 1 and 2 of the Data Browser are seen on the screen at one time. The “multifile” is the chromatogram (should be in top box when printing, but is listed as the second item in box) and the “RT” files are the spectra. Can delete the RT files when finished by clicking “Remove” key.

4) File → Print (be sure the change orientation to Landscape under the "setup…" key)



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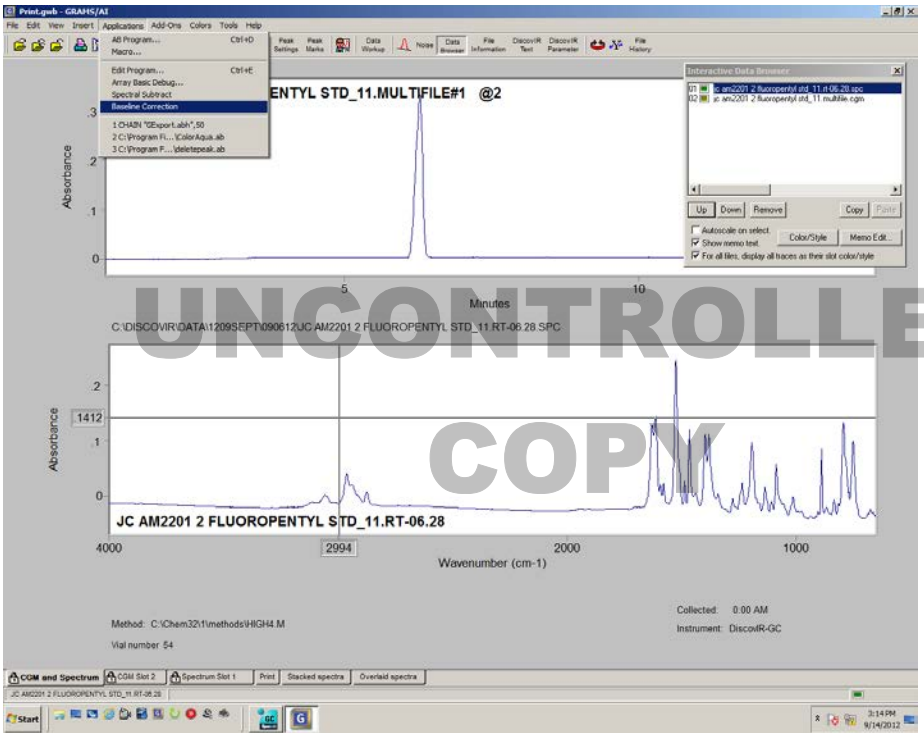
Baseline Correction

1) Be sure the cursor is in the spectra box (bottom) and click "Applications" on the top menu bar → "Baseline Correction".



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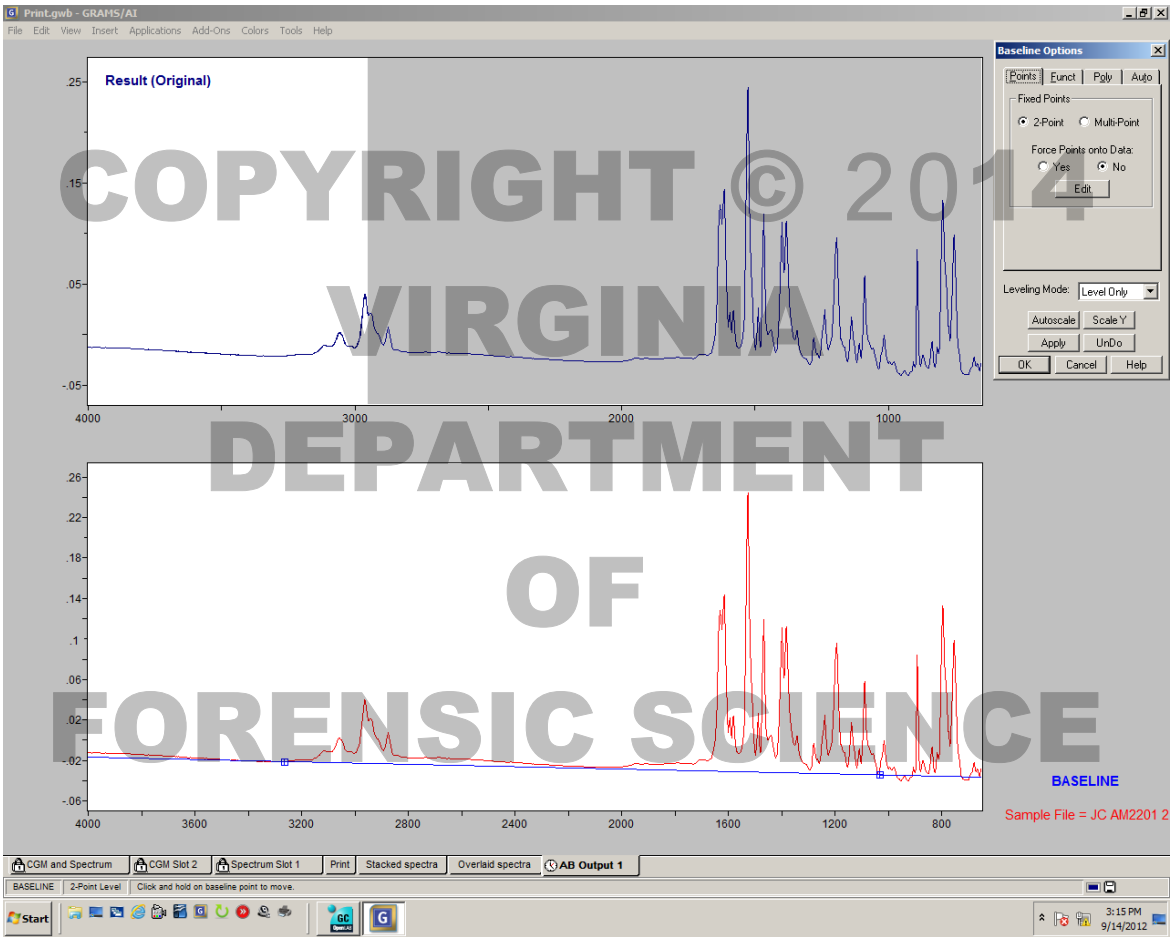
The following box will appear:



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2) In the "Baseline Options" box on the right hand side, select "Multi-point" and "Level + Zero" in the Leveling Mode drop-down menu

   - On the red spectrum, left click and drag the blue squares to the desired positions on the baseline
   - Right clicking allows you to add additional points (blue squares) along the baseline → Click "Apply" and "OK" → A "Baseline" box with message "Keep result of Calculation on "Data File Name" appears → Click "Replace"

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3) "File" → "Save Trace As" → Make sure the correct folder is selected (ex: c:/DiscovIR/data/1206June) → Add –BASELINE at end of file name (ex: KH C12-12345_03_RT-03.28-BASELINE.spc → "Save"

4) "File" → "Print" (be sure to change Orientation to Landscape under the "Setup…" key)

5) To print instrument parameters (method, temperatures, etc.), select file with the "Multifile.cgm" extension in the Data Browser → Click on "DiscovIR text" button on the top menu bar (text file in a new window will appear) → Print

Library Search in Data Workup Workbook or Print Workbook

1) Under the “Add Ons” tab on the top menu bar, select “Spectral ID”

   **Although the spectrum appears in the box, click on the “Open Folder” icon on the top bar and open the data file (this will allow the data file name to appear on the library search printout).

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2) Click on the first magnifying glass icon on the top bar (with the spectrum in it) that when scrolled over, says “Spectrum search”. This will open the library matches for the sample. You can scroll through the list by left clicking on each hit. The top spectra is the sample (in red) and the lower spectra is the library hit (in blue) The lower the quality number, the better the match.

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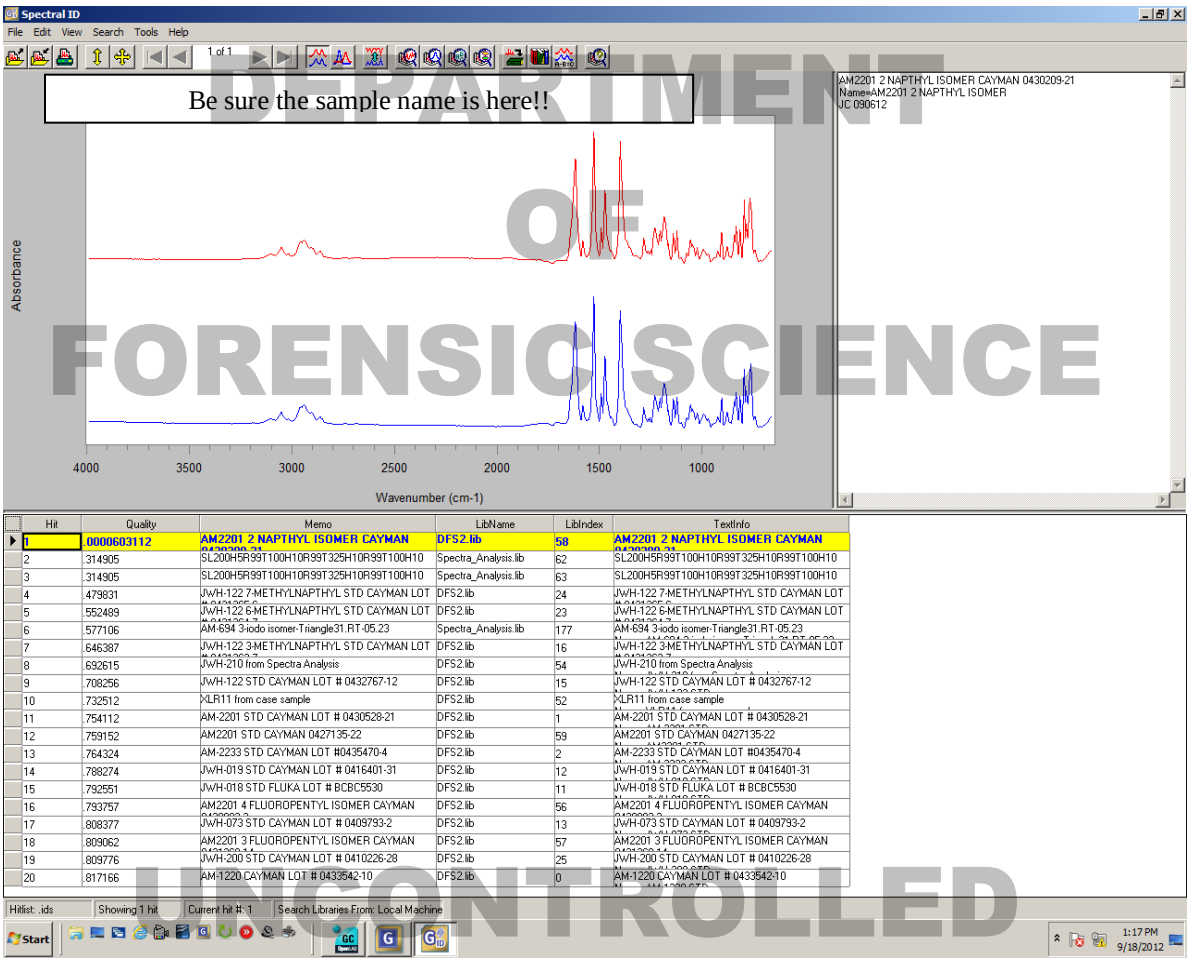

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When scrolled over, says “Overlaid trace mode”: Sample and library hit stacked on top of one another

When scrolled over, says “Toggle between Absorbance and Transmission”: Toggles between absorbance/transmission

3) File, select Print

Daily Shut Down

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1) Close New Run Workbook if open, open Utilities workbook

2) Turn off camera, chamber, and view lights

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3) Ensure that the transfer line, oven and restrictor temps are at 100 degrees.

4) Ensure GC is on “Night” method

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