Addendum (Methods for Agilent/ASAP Analytical IRD3 GC-FTIR)

Agilent GC:

Front Column: S/SL Injection Port with recommended HP-1 30 meter column, 0.32 column ID, and 1.0 um film thickness:

*Stored Methods for **AUTOSAMPLE SPLIT INJECTION***

60TO270 Initial 60C degrees hold for 2 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 13.827 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

120TO270 Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 17.01 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

120TO270(20MIN) Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 17.01 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

200TO270 Initial 200C degrees hold for 1 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 21.268 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

200TO270(20MIN) Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow of 6.0 mL/min, Pressure 21.268, Septum purge flow 3mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

ISO270(20MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 24.983 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

ISO270(30MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 24.983 psi, Septum

purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 30.00 minutes.

*Stored Methods for **MANUAL SPLIT INJECTION***

MAN60TO270 Initial 60C degrees hold for 2 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 13.827 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

MAN120TO270 Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 17.01 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

MAN120TO270(20MIN) Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 17.01 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

MAN200TO270 Initial 200C degrees hold for 1 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 21.268 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

MAN200TO270(20MIN) Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0mL/min, Split ratio of 2.0, Split flow of 6.0 mL/min, Pressure 21.268 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total rrun time 20.00 minutes.

MANISO270(20MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 24.983 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

MANISO270(30MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 24.983 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 30.00 minutes.

*Stored Methods for **AUTOSAMPLE SPLITLESS INJECTION***

SL60TO270 Initial 60C degrees hold for 2 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

SL120TO270 Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

SL120TO270(20MIN) Initial 120C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

SL200TO270 Initial 200C degrees hold for 1 minutes ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

SL200TO270(20MIN) Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

SLISO270(20MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

SLISO270(30MIN) 270C degrees isothermal. Constant carrier flow rate of 3.0 mL/min, Splitless, Purge flow to Split Vent 15 mL/min at 0.75 min, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 30.00 minutes.

ASAP Analytical IRD3 (FTIR):

IRD3 Resolution of 8 cm-1 with a Co-Add (Scans) of 2, Background Scans (Collecting Basis Set→Basis Set File 10 times with 6 scans each = 60 Scans) prior to each sample, set at Auto Calculate Gain (typically 1-2), Frequency range 800-4000 cm-1, Apodization: Triangle, Phase Correction: Mertz, Zero Fill: 1, Flow Cell, Transfer Lines A & B at 280C, Smoothing Factor: OFF, Length 100, Symmetry: 2, Offset 60, Source Power at 22, and Laser Wavelength of 0.63299.

SHUTDOWN Resolution of 8 cm-1 with a Co-Add (Scans) of 2, Background Scans (Collecting Basis Set→Basis Set File 10 times with 6 scans each = 60 Scans) prior to each sample, set at Auto Calculate Gain (typically 1-2), Frequency range 800-4000 cm-1, Apodization: Triangle, Phase Correction: Mertz, Zero Fill: 1, Flow Cell at 280C, Transfer Lines A & B at 230C, Smoothing Factor: OFF, Length 100, Symmetry: 2, Offset 60, Source Power Off, and Laser Wavelength of 0.63299.

For Resolution and Co-Add (Scans) Study ONLY:

8 Resolution 4 Scans Resolution of 8 cm-1 with a Co-Add (Scans) of 4, Background Scans (Collecting Basis Set→Basis Set File 10 times with 6 scans each = 60 Scans) prior to each sample, set at Auto Calculate Gain (typically 1-2), Frequency range 800-4000 cm-1, Apodization: Triangle, Phase Correction: Mertz, Zero Fill: 1, Flow Cell, Transfer Lines A & B at 280C, Smoothing Factor: OFF, Length 100, Symmetry: 2, Offset 60, Source Power at 22, and Laser Wavelength of 0.63299.

Always verify that the temperature for the GC interface (flow cell) is at least 10 degrees higher than the maximum temperature of the GC oven. The nitrogen purge for the IRD3 should be set at around 20 psi and the nitrogen make-gas for the IRD flow controller's auxiliary lines A & B should be set at around 5 psi.