

Pyrolysis GCMS

Instrument Operation



—
TrACEES Platform

Trace Analysis for
Chemical, Earth and
Environmental Sciences

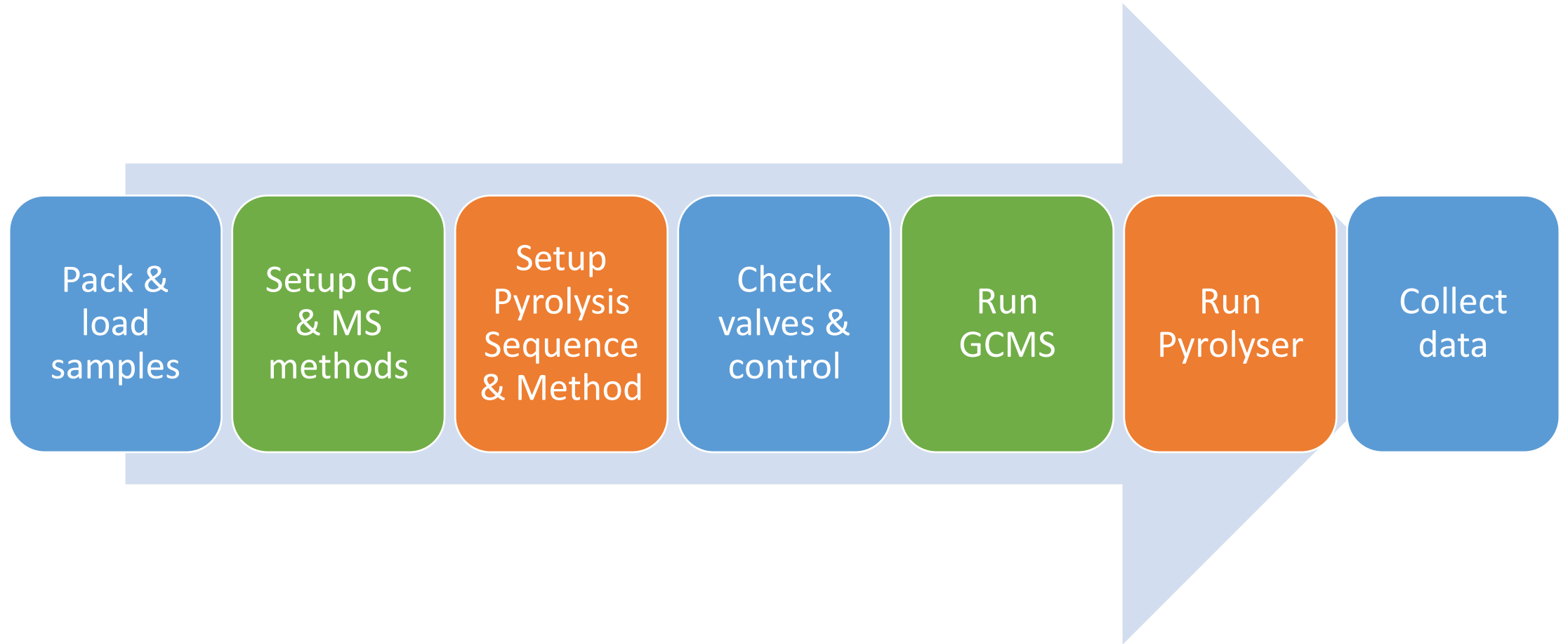
Doc No. WT-PyGC-v1.0

Created by: Alex Duan

Version No. 1.0

August 2018

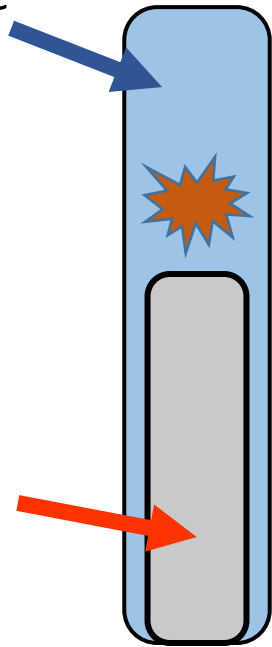
Work flow



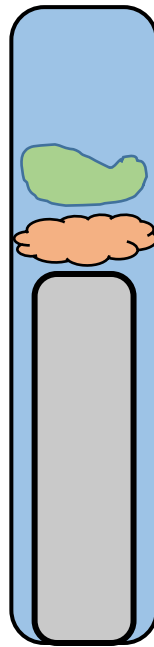
Sample & Packing

- Ideal 10-50ug

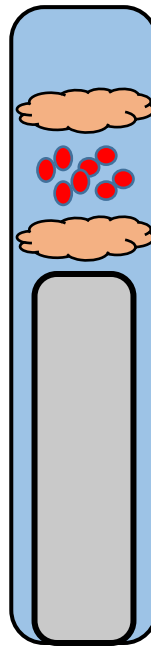
Quartz tube



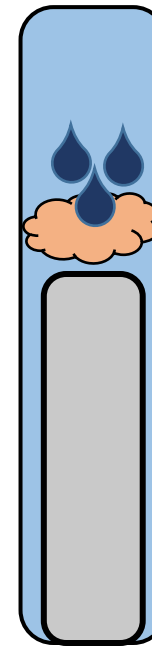
Solid



Easily melting
e.g. wax



Fine powder
e.g. coal



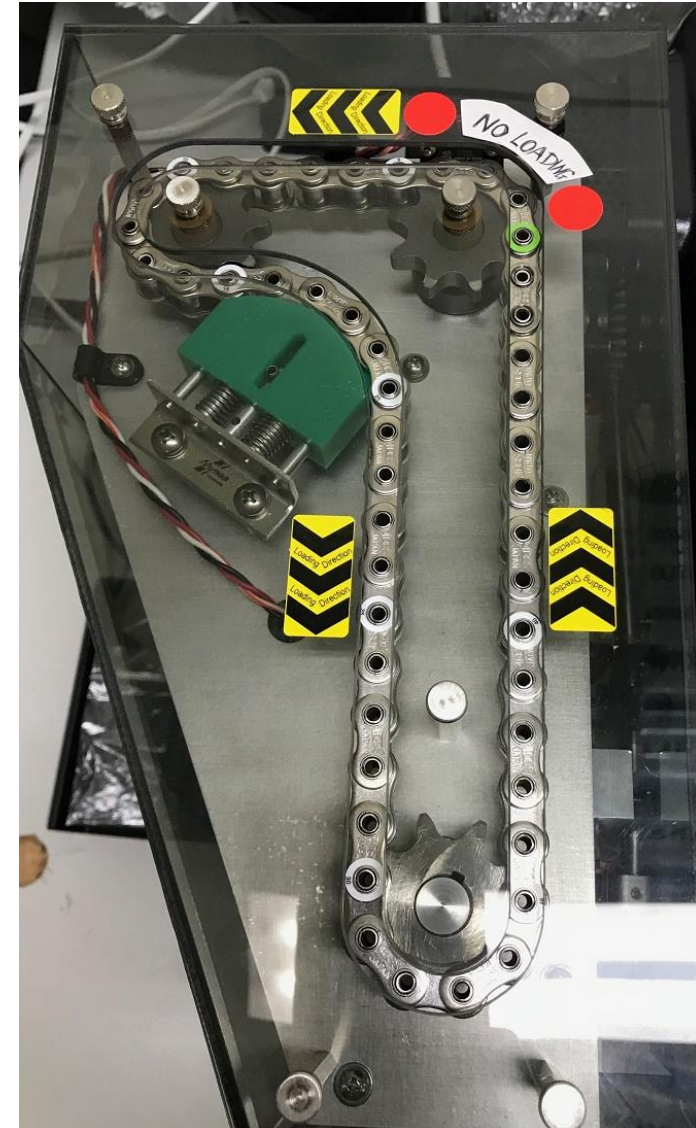
Liquid



Quartz wool

Load sample tubes

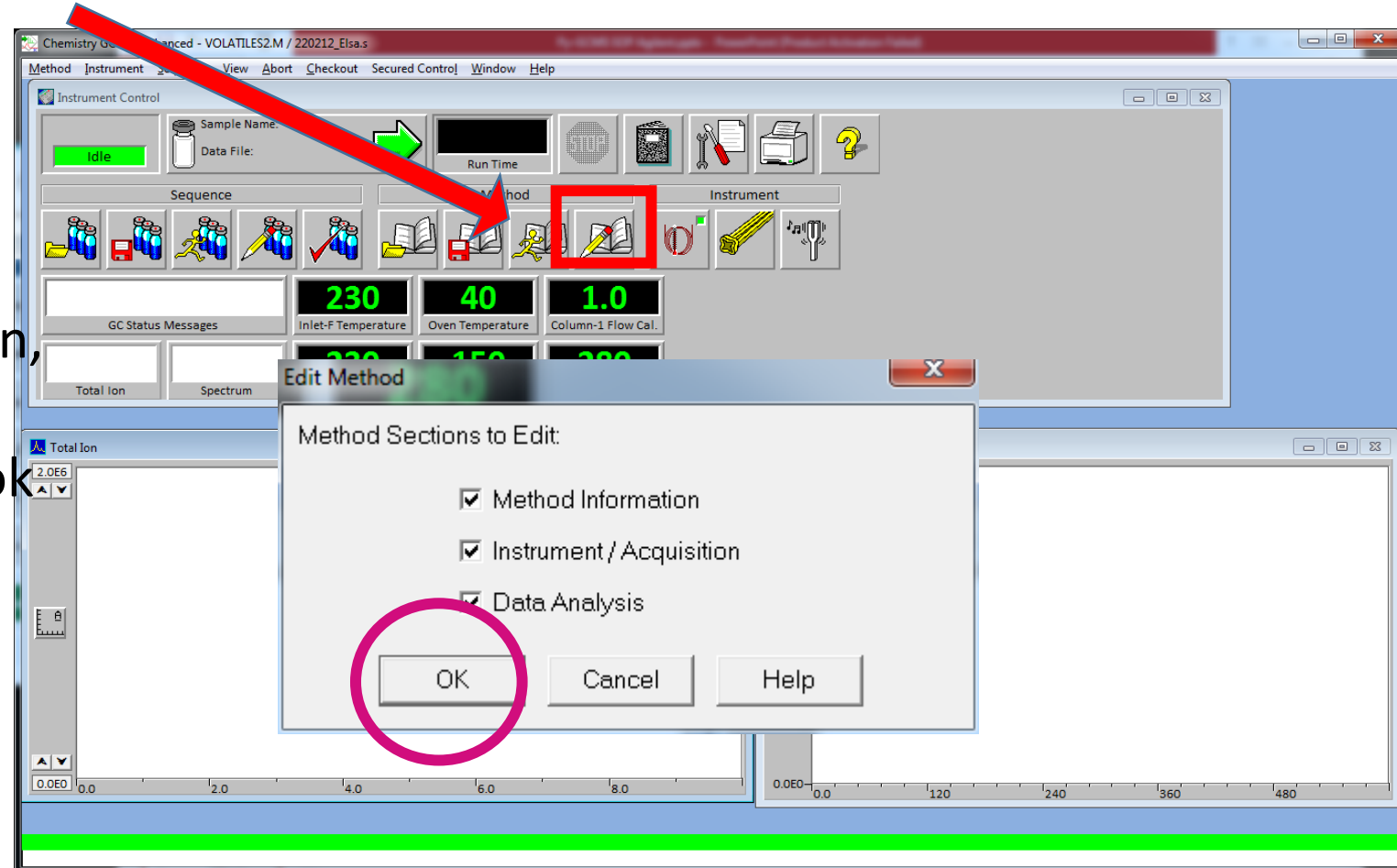
- The chain is the sample holder
- Load sample tubes in holes
- **NOT between the Red Dots**
- The black arrows on yellow label indicates the direction of sample increment (**anti-clockwise**, #1, #2, #3, ...)



Setup GCMS

GC

- Open GC method editor
- Ok ->
 - Documentation Description, Ok
 - Instrument Notes, blank, ok
 - Data acquisition, ok
 - Instrument Control



GC

- Ok
- Ok ->

Method Information

Method Comments:
This is the default method

☐ Save Copy of Method with Data

Method Sections to Run

☐ Pre-Run Macros/Commands:
Instrument Control: ...
Data Analysis: ...

☒ Data Acquisition
☒ Data Analysis
☐ Post-Run Macros/Commands:
Instrument Control: ...
Data Analysis: ...

OK Cancel Help

Inlet and Injection Parameters

Sample Inlet: GC
Injection Source: Manual
☒ Use MS

Inlet Location:
☒ Front ☐ Rear ☐ Dual

MS Connected to:
☒ Front Inlet ☐ Rear Inlet

OK Cancel Help

GC

- Inlet
 - Heater temperature
 - Split ratio
- Column
 - Flow rate (typically 1.0 mL/min)

Instrument | Edit | Inlets: (6890)

■ Oven Temp

Temperature (°C)

Plot...

0 2 4 6 8 10 Time (min)

Injector Valves **Inlets** Columns Oven Detectors Signals Aux Runtime Options

Front: EPC Split-Splitless Inlet

Mode: Split Gas: He

On	Actual	Setpoint
☒ Heater, °C	230	300
☒ Pressure, psi	7.02	7.03
☒ Total Flow, mL/min	14.3	54.2

Split Ratio: 50.0 : 1 Split Flow: 149.8 mL/min

☒ GasSaver 15.0 mL/min @ 2.00 min

Front

Apply

OK

Cancel

Help

Instrument | Edit | Columns: (6890)

■ Oven Temp

Temperature (°C)

Plot...

0 2 4 6 8 10 Time (min)

Injector Valves Inlets **Columns** Oven Detectors Signals Aux Runtime Options

Column Mode: Const Flow Inlet: Front Detector: MSD Outlet psi: Vacuum

Installed Column Inventory#: ZB-5ms (not calibrated)

Manufacturer's Specifications Model No: ??? ZB-5ms 325°C Max Capillary 30.0 m x 250 µm nominal

He Flow

Setpoint	Actual	Unit
Pressure: 7.03	7.03	psi
Flow: 1.0	1.0	mL/min
Average Velocity: 36		cm/sec

Flow	ml/min ²	ml/min	Hold min	Run Time
Initial		1.0	0.00	12.00
Ramp 1	0.00	0.0	0.00	
Ramp 2	0.00	0.0	0.00	
Ramp 3	0.00	0.0	0.00	
Post Run			0.00	12.00

Change...

Apply

OK

Cancel

Help

GC

- Oven

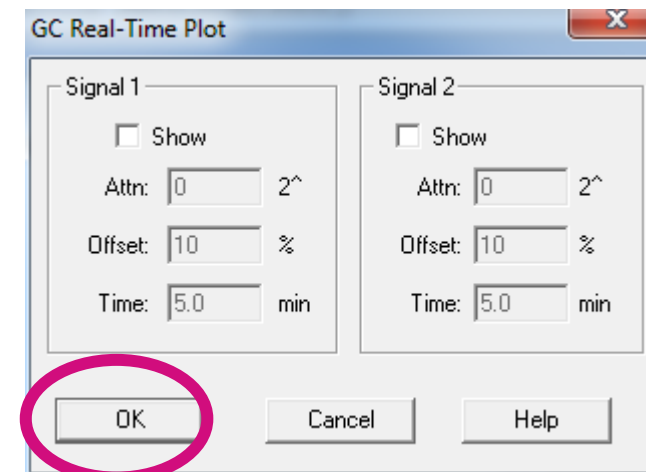
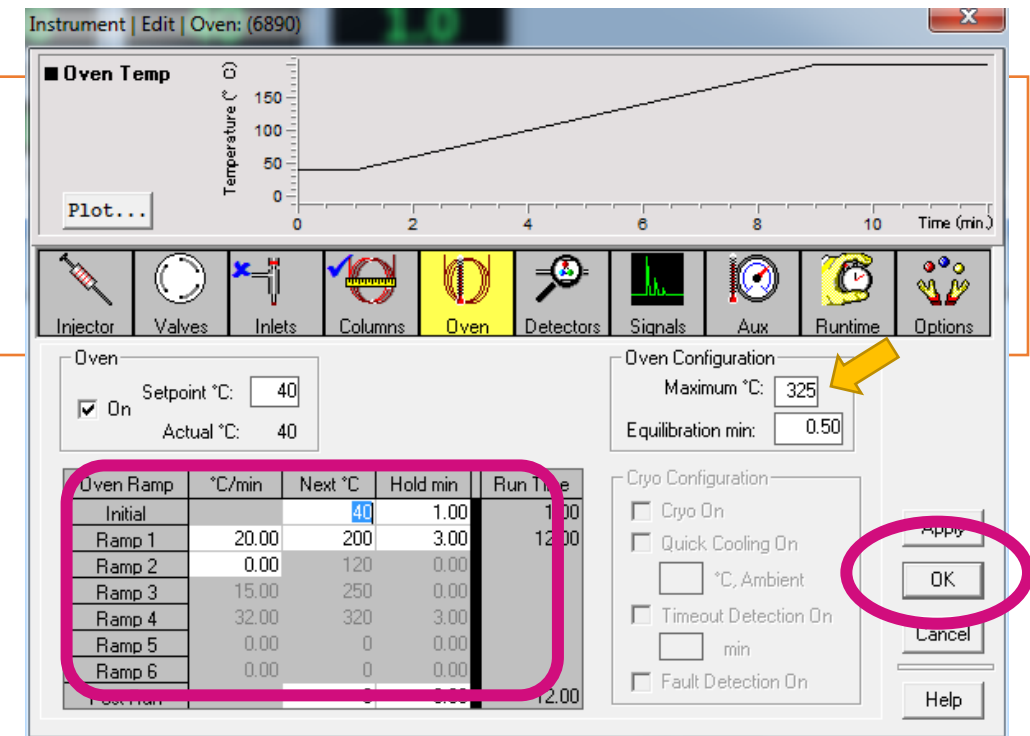
- Set temperature program
- Max temperature for column is displayed in this window

->OK

- GC Real-Time Plot

- No need to make changes here

->OK



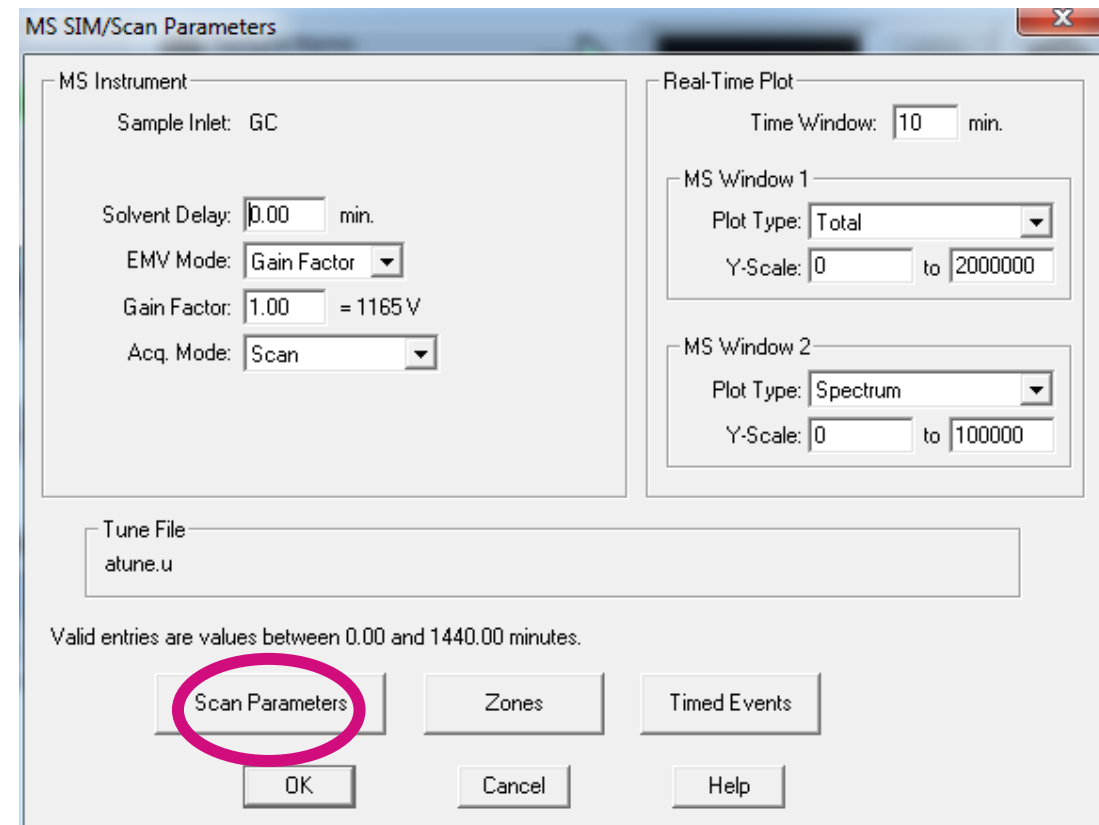
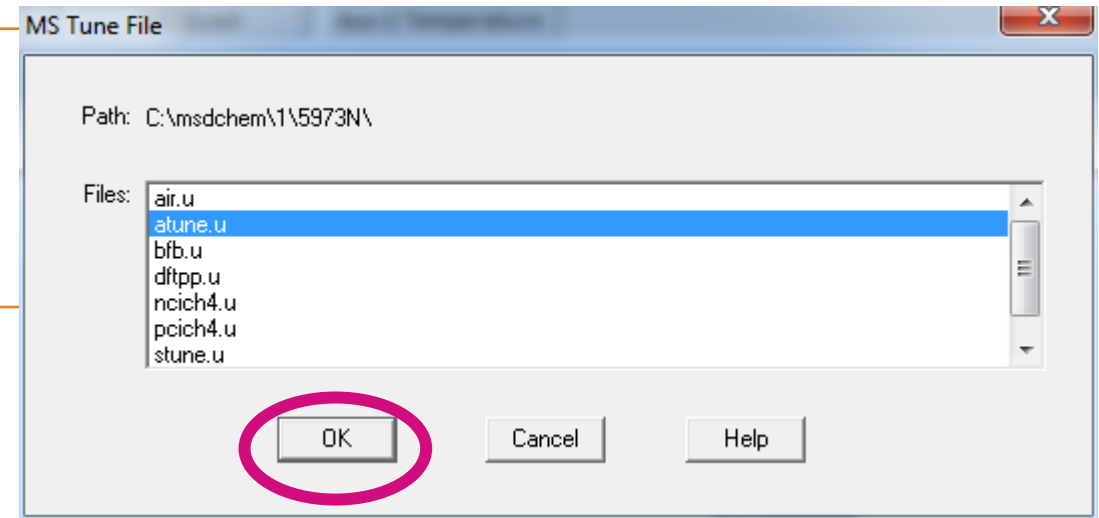
MS

- MS Tune File
 - Select atune.u

->OK

- MS SIM/Scan Parameters

->Scan Parameters



MS

- Edit Scan Parameters
 - Set MS scan range

-> Close

- MS SIM/Scan Parameters

->Scan Parameters

Scanning Mass Range | Threshold and Sampling Rates | Plotting

Start Time (minutes): 0.00

Start at Mass... (amu): 30.00

End at Mass... (amu): 550.00

Scan Group 1 ☒

Scan Group 2 ☐

Scan Group 3 ☐

Summary of Settings

Group	Start Time	Low Mass	High Mass	Threshold	Samples	S
1	0.00	30.00	550.00	150	2	2.

Low to High mass range must be in ascending order from 1.60 - 800.00.

Close Help

MS SIM/Scan Parameters

MS Instrument

Sample Inlet: GC

Solvent Delay: 0.00 min.

EMV Mode: Gain Factor

Gain Factor: 1.00 = 1165 V

Acq. Mode: Scan

Real-Time Plot

Time Window: 10 min.

MS Window 1

Plot Type: Total

Y-Scale: 0 to 2000000

MS Window 2

Plot Type: Spectrum

Y-Scale: 0 to 100000

Tune File: atune.u

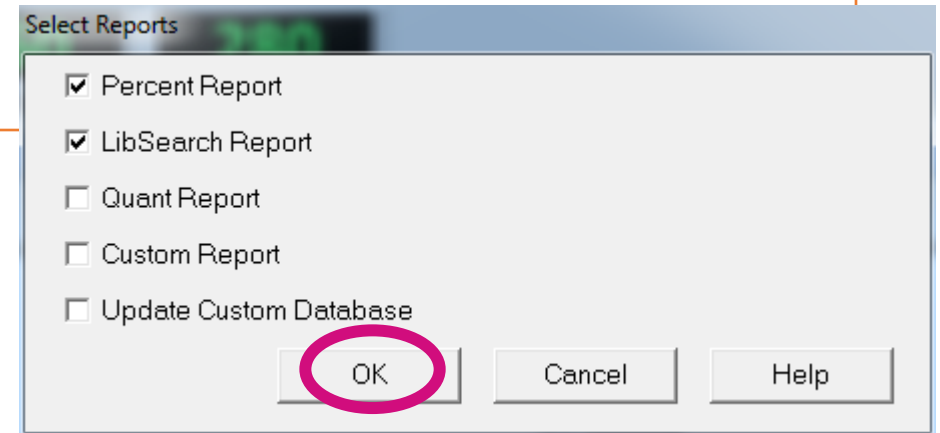
Valid entries are values between 0.00 and 1440.00 minutes.

Scan Parameters Zones Timed Events

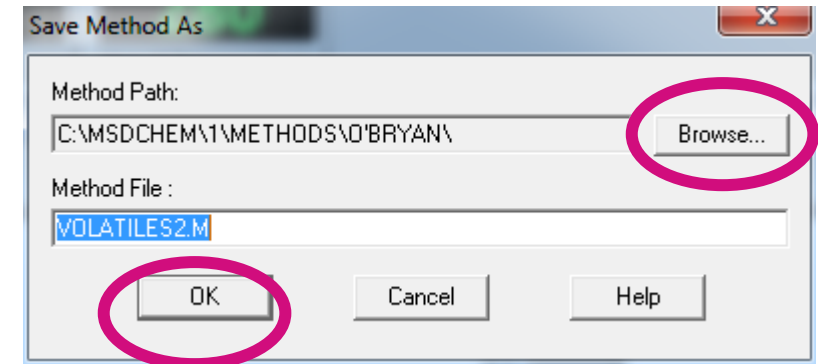
OK Cancel Help

GCMS

- Select Reports, other report details
 - > Keep clicking OK until “Save Method File” window appears



- Save Method As
 - > Browse
 - Select your method folder
 - Name your method
 - > OK



Setup Pyrolyser

Setting up Pyrolysis Sequence & Method

CDS 6250 Autosampler - Blank Test Sequence

File Tools Configuration Communications View Help

Overview

Pyroprobe

Interface

Sequence

Iso Zones

Trap

Cryofocuser

CDS 6250 Autosampler

Interface **300°C**

Probe **OFF**

Transfer Line **325°C**

Valve Oven **324°C**

Pressure **PSI** Flow Rate **mL/min**

Inlet	System	
80.3	0.2	---

Run Sequence 0%

GC is Not Ready GC Not Rdy

DO NOT change Temperature setting:

- Interface
- Transfer line
- Valve

Sequence (FIRST)

- Create Sequence
- Click Sequence Tab
- Click File then New Sequence
- Add Row or Repeat Last Row for multiple samples

The screenshot displays the 'CDS 6250 Autosampler - Blank Test Sequence' window. The 'File' menu is open, showing options like 'New Sequence', 'Open a Sequence...', 'Save Current Sequence', 'Save Current Sequence As...', 'Print Current Sequence', and 'Exit'. The 'Sequence' tab is selected in the left sidebar. The main area shows a table with columns: Method Name, Run Leak Check, Use GC Ready, Issue GC Start, Trap, Dry, Clean, and Notes. The table contains four rows: 'Blank Test' (Run Leak Check: checked, Use GC Ready: checked, Issue GC Start: checked, Trap: checked, Dry: checked, Clean: checked, Notes: Blank (Empty)), 'Blank Test' (Run Leak Check: checked, Use GC Ready: checked, Issue GC Start: checked, Trap: checked, Dry: checked, Clean: checked, Notes: Blank (Empty)), 'Ivan Test' (Run Leak Check: checked, Use GC Ready: checked, Issue GC Start: checked, Trap: checked, Dry: checked, Clean: checked, Notes: Kraton), and 'Ivan Test' (Run Leak Check: checked, Use GC Ready: checked, Issue GC Start: checked, Trap: checked, Dry: checked, Clean: checked, Notes: Polyethelene). Below the table, there are buttons for 'Add Blank Row to End' and 'Repeat Last Row...' (both highlighted with red boxes), 'Delete Current Row', and 'Insert Row Below Current'. At the bottom, there is a 'Run Sequence' button and a progress bar showing 0%. The status bar at the very bottom indicates 'GC is Not Ready' and 'GC Not Rdy'.

Method Name	Run Leak Check	Use GC Ready	Issue GC Start	Trap	Dry	Clean	Notes
Blank Test	✓	✓	✓	✓	✓	✓	Blank (Empty)
Blank Test	✓	✓	✓	✓	✓	✓	Blank (Empty)
Ivan Test	✓	✓	✓	✓	✓	✓	Kraton
Ivan Test	✓	✓	✓	✓	✓	✓	Polyethelene

Method

- Create Method
- Click Pyroprobe Tab
- Click File then New Method
- Pay attention to Units
- Then update Method in Sequence

CDS 6250 Autosampler - Blank Test Sequence (Modified)

File Tools Configuration Communications View Help

New Method
Open a Method...
Save Current Method
Save Current Method As...
Print Current Method
Exit

Pyroprobe Run Time:
16.010 Seconds (0.267 Minutes)

	INITIAL	RAMP	FINAL
Time	700	5.00	750
Units	☒ Second(s) ☐ Minute(s)	☒ °/mSecond ☐ °/Second ☐ °/Minute	☒ Second(s) ☐ Minute(s)

Sequence
Iso Zones
Trap
Cryofocuser

Clean
°C Seconds
800 10.00

Dry
°C Seconds
1000 5.00

Actual °C
OFF

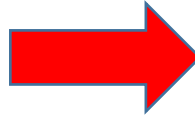
Run Sequence 0%

GC is Not Ready GC Not Rdy

Valves and control unit

Checks

- Pyrolyzer carrier gas valve is in **correct** position



- Check GCMS and Pyrolyer status

Run the measurement

- Start GCMS Run
 - Enter Data path, Data file name, Sample Name, Misc Info (if any)
- > OK and Run Method
- Start Pyrolyser
- > Run Sequence

Start Run

Basic | Advanced

Current Method Injection Style:

Inlet Location: ☐ Front ☐ Rear ☐ Dual

MS Connected to: ☐ Front Inlet ☐ Rear Inlet

Operator Name:

Data Path:

Sample

Data File Name:

Sample Name:

Misc Info:

Expected Barcode:

Sample Amount:

Multiplier:

Vial Number:

Tray Name:

Select Injection Volume:

☐ Current Method µL

☐ Override using µL

Method Sections to Run

☒ Data Acquisition

☒ Data Analysis

