


JORDAN FOOD AND DRUG ADMINISTRATION	
AMMAN FOOD LABORATORY	Edition: 1 Review: 1 Date: 01-10-2017 Page 1 of 17
OPERATING GAS CHROMATOGRAPHY MASS SPECTROMETER (GC-MS/MS)	No.: AFFsop 9

Content	Page	
1. Objectives	2	
2. Scope	2	
3. Responsibility	2	
4. References	2	
5. Definitions	2	
6. Instruments	2	
7. Procedure for operating the GC-MS/MS	3	
1. Start up	3	
2. Daily Check	6	
3. Data Acquisition	7	
4. Data Analysis	15	
5. Shutdown	17	
Prepared by: Ghaith R. Algharibeh	Review by:	Approved by:
Date: 1-10-2017	Date:	Head of Head of Quality Division

OPERATING GAS CHROMATOGRAPHY MASS SPECTROMETER (GC-MS/MS)

1. OBJECTIVE

The following document describes the procedure to operate the TSQ™ Duo Triple Quadrupole GC-MS/MS (ThermoFisher Scientific - USA)

2. SCOPE

This procedure applies to the TSQ™ Duo set up in the Contaminants Monitoring Division (**CMD**)

3. RESPONSIBILITIES

It is the responsibility of the CMD staff to follow the instructions & ensure adherence to this procedure

4. REFERENCES

1. TraceFinder Acquisition Quick Reference Guide (Revision A XCALI-97725-2015)
2. TraceFinder Analysis Quick Reference Guide (Revision A XCALI-97726-2015)
3. TraceFinder User Guide Software Version 3.3 (XCALI-97728 Revision A May 2015)

5. DEFINITIONS

1. MS: Mass spectrometer
2. GC: Gas chromatography

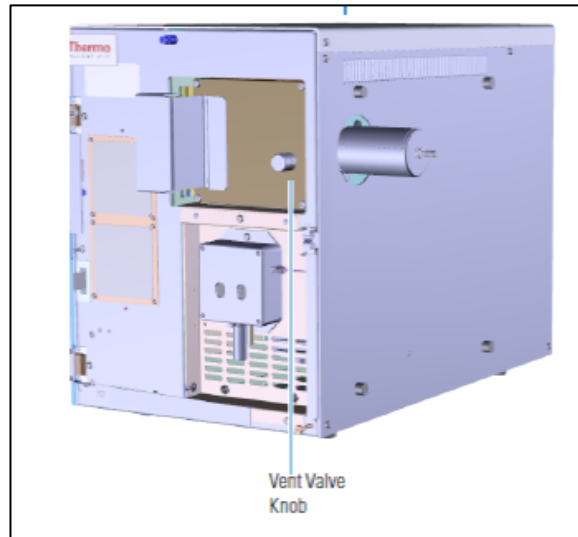
6. INSTRUMENTS

1. TSQ™ Duo Triple Quadrupole Mass Spectrometer (ThermoFisher Scientific - USA)
2. Trace 1310 Gas Chromatograph (ThermoFisher Scientific - USA)
3. TriPlus RSH Auto sampler (ThermoFisher Scientific - USA)

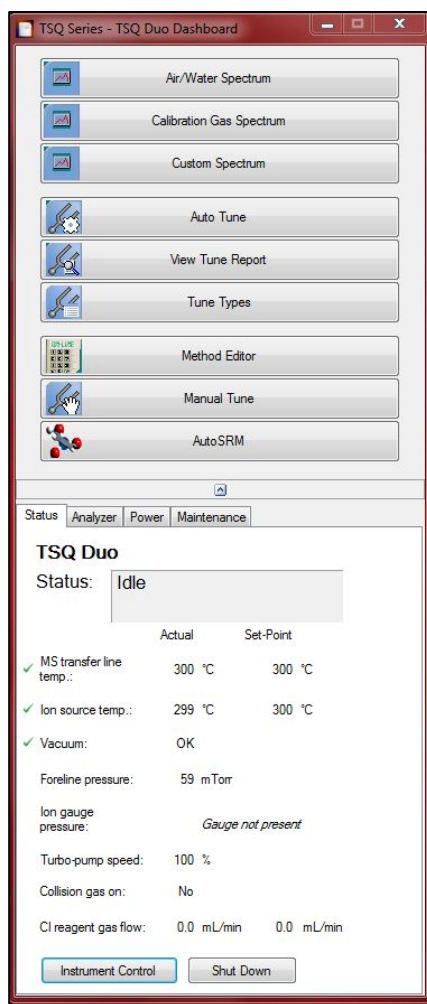
7. PROCEDURE FOR OPERATING THE GC-MS/MS

1. Start Up

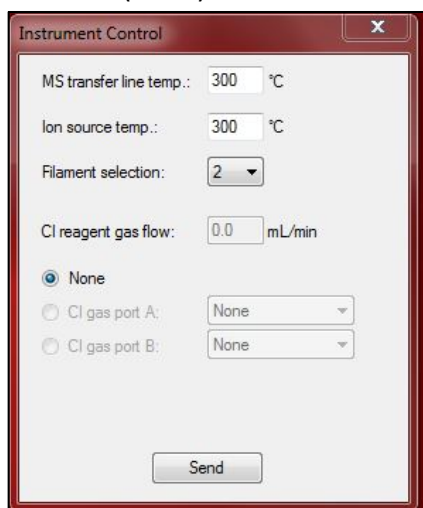
- 1.1. Please note that the system should always be turned **ON**
- 1.2. Switch the (**TriPlus RSH**) Auto sampler power **ON** (main switch is on the black transformer box next to the GC)
- 1.3. Open the valves on the gases supply bottles (Helium and Argon)
- 1.4. Make sure the Helium pressure is about (**5 Bar**) and Argon pressure is exactly (**4.1 Bar**)
- 1.5. Switch the (**Trace 1310**) GC power **ON** (main switch is on the back panel)
- 1.6. Check if the (**TSQ Duo**) vent valve is closed (Located behind TSQ Duo front cover) and switch **ON** power supply (main switch is on the left panel)



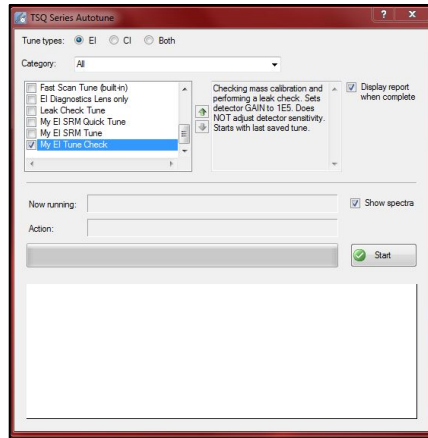
- 1.7. Switch **ON** the PC
- 1.8. Open TSQ series dashboard



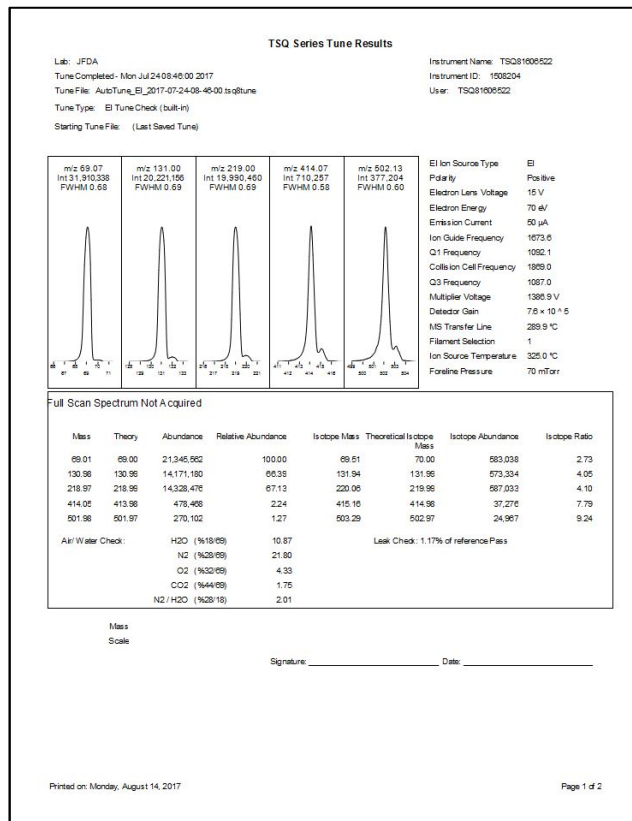
- 1.9. Wait until (**Turbo-pump speed**) goes from 0% to 100%
- 1.10. Click on (**Instrument Control**) button & set the (**MS transfer line temp.**) and (**Ion Source temp.**) to the required temperatures and click (**Send**)



- 1.11. Wait until the (**Foreline pressure**) goes down below 85 mTorr (takes a few hours, the best is to leave the system to condition overnight)
- 1.12. Go to Auto Tune and start (**My EI Tune Check**)

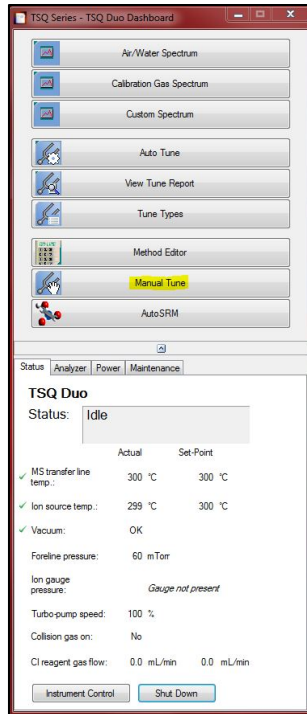


- 1.13. Confirm the leak value is passing
- 1.14. Confirm the signal intensity, mass calibration and air leak are **OK** and proceed with sample analysis

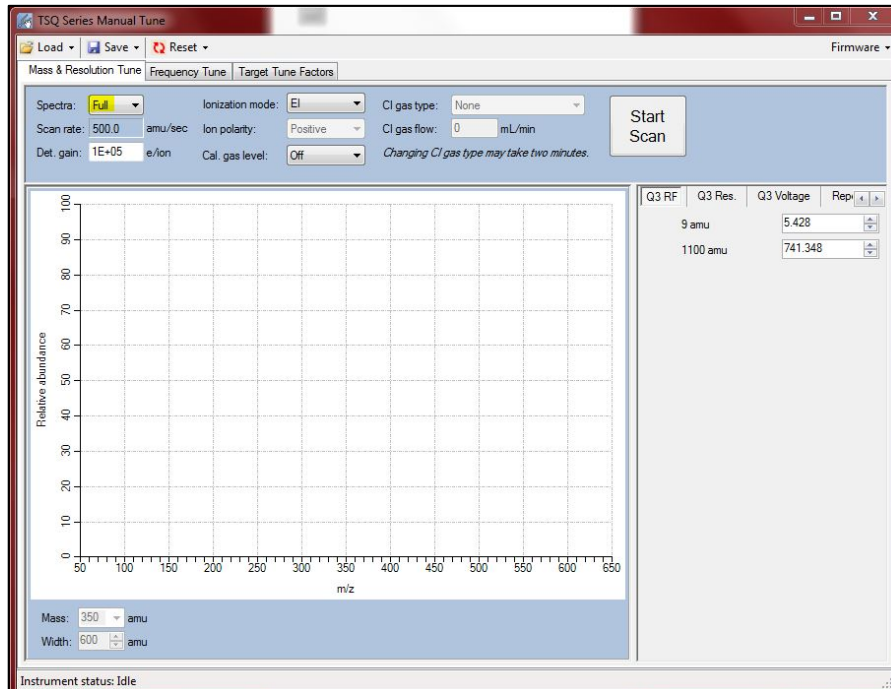


2. Daily Check

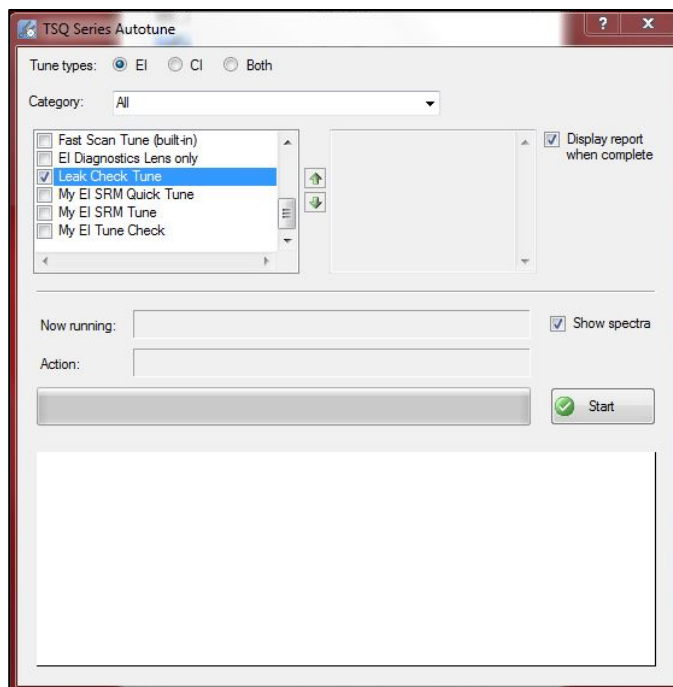
- 2.1. Open TSQ series dashboard
- 2.2. Click on **(Manual Tune)**



2.3. (TSQ Series Manual Tune) window will pop up



- 2.4. Choose **(Full)** spectra, keep **(Cal. Gas level) (Off)** and click on **(Start Scan)**
- 2.5. Clean instrument will show peaks intensity in the range of **(1E+04)**
- 2.6. If peaks intensity are in the range of **(1E+05)** or **(1E+06)** then this might be a sign of contamination
- 2.7. Stop scan, set spectra to **(5)** switch cal. Gas level to **(EI)** and click on **(Start Scan)** and check the intensity of the following masses:
 - 2.7.1. Mass **(219)** intensity should be a round **(20000000)** count **(2E+07)**
 - 2.7.2. Mass **(414)** intensity should be in the middle of **(1E+05)** or higher
 - 2.7.3. Mass **(502)** intensity should be in the middle of **(1E+05)** or higher
- 2.8. Go to Auto Tune and start **(Leak Check tune)**



- 2.9. Confirm the leak value is below 6%

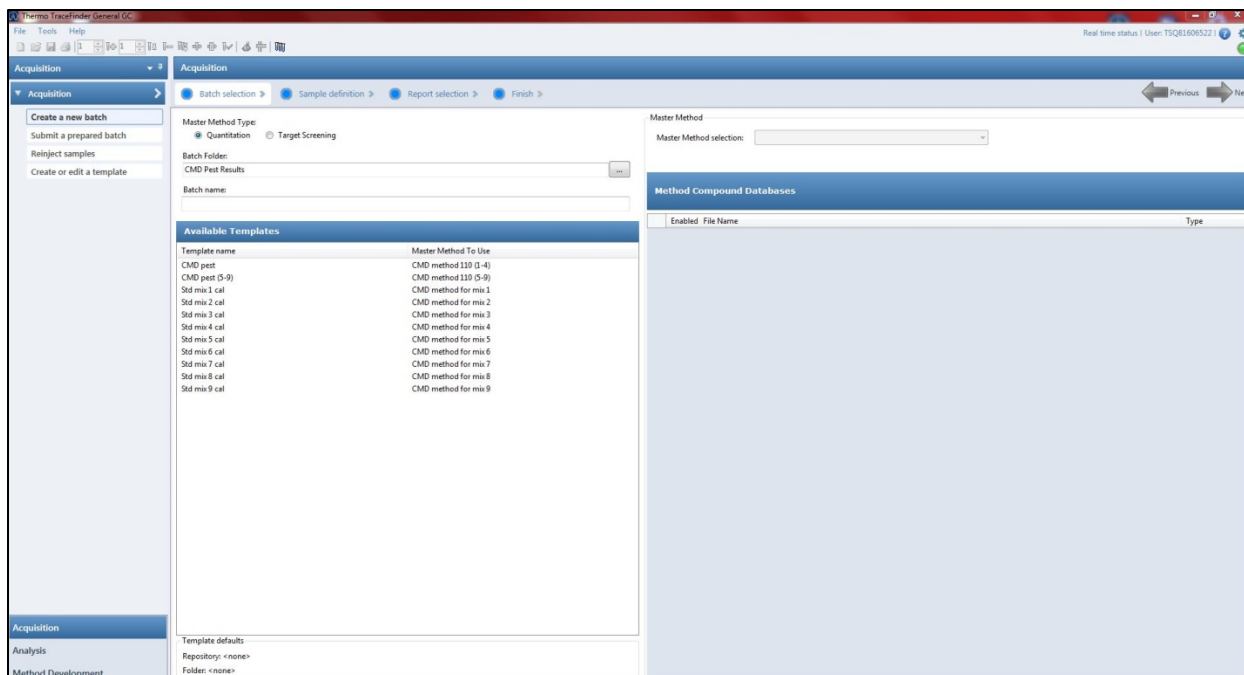
3. Data Acquisition

There are two ways of acquiring data, the first is by **(Batch Acquisition)** (**sequential analysis of multiple samples**) & the second by **(Quick Acquisition)** usually to quickly acquire one sample

3.1. Batch Acquisition

- 3.1.1. Place the sample in the Autosampler

3.1.2. On the desktop, open Thermo TraceFinder™ 3.3 application

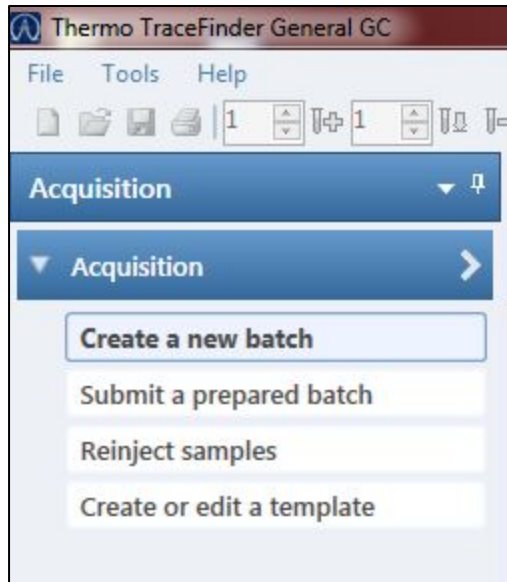


3.1.3. Click **(Acquisition)** in the navigation pane



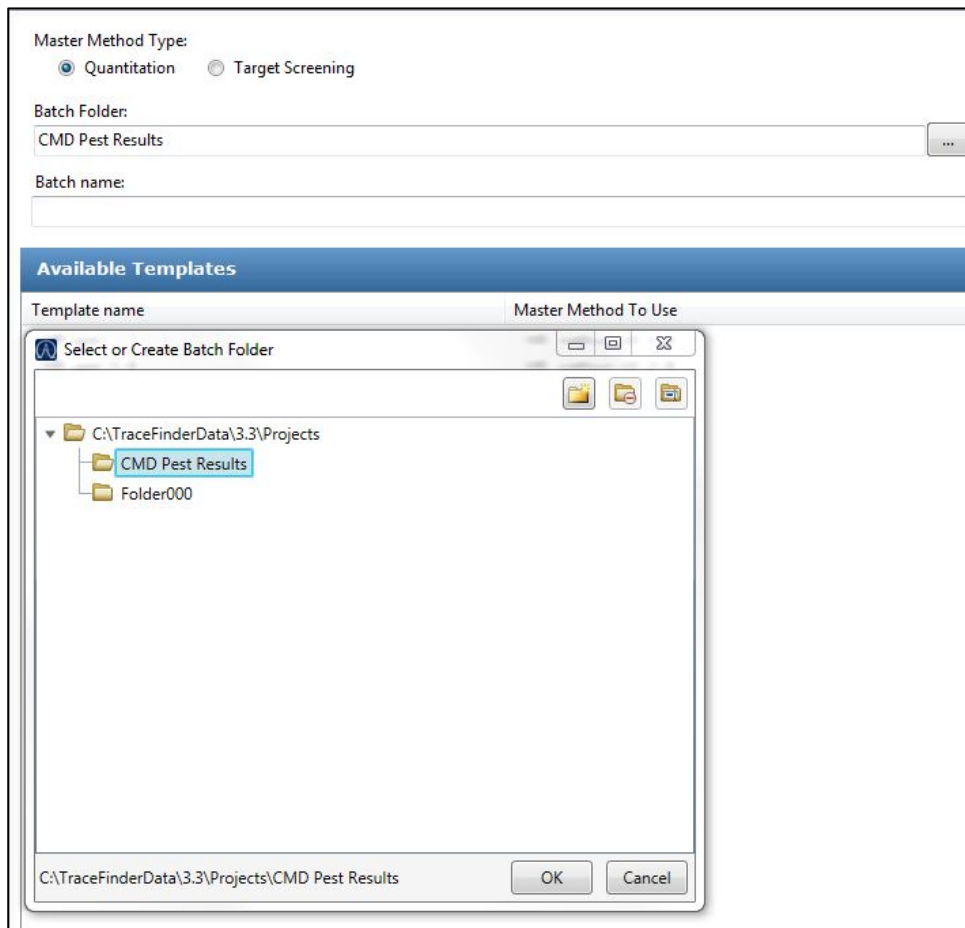
3.1.4. The navigation pane for the Acquisition mode opens

3.1.5. In the **(Acquisition)** navigation pane, click **(Create a New Batch)**



3.1.6. Select the **(Quantitation)** in **(Master Method Type)**

3.1.7. Select the **(CMD Pest Results)** in **(Batch Folder)**



- 3.1.8. Type a unique name for the new batch in the **(Batch Name box)** (e.g. **16-8-2017**)

Master Method Type:
 Quantitation Target Screening

Batch Folder:
CMD Pest Results

Batch name:
16-8-2017

- 3.1.9. In the **(Available Templates)** pane, select the template and method combination that you want to use (e.g. **(Std mix 1 cal) template name with (CMD method for mix 1) master method to use**)

Available Templates	
Template name	Master Method To Use
CMD pest	CMD method 110 (1-4)
CMD pest (5-9)	CMD method 110 (5-9)
Std mix 1 cal	CMD method for mix 1
Std mix 2 cal	CMD method for mix 2
Std mix 3 cal	CMD method for mix 3
Std mix 4 cal	CMD method for mix 4
Std mix 5 cal	CMD method for mix 5
Std mix 6 cal	CMD method for mix 6
Std mix 7 cal	CMD method for mix 7
Std mix 8 cal	CMD method for mix 8
Std mix 9 cal	CMD method for mix 9

Master Method

Master Method selection:

- 3.1.10. Click **(Next)** to continue to the next page
- 3.1.11. The **(Sample Definition)** page opens showing the chosen template

Acquisition

Batch selection ▶ Sample definition ▶ Report selection ▶ Finish ▶

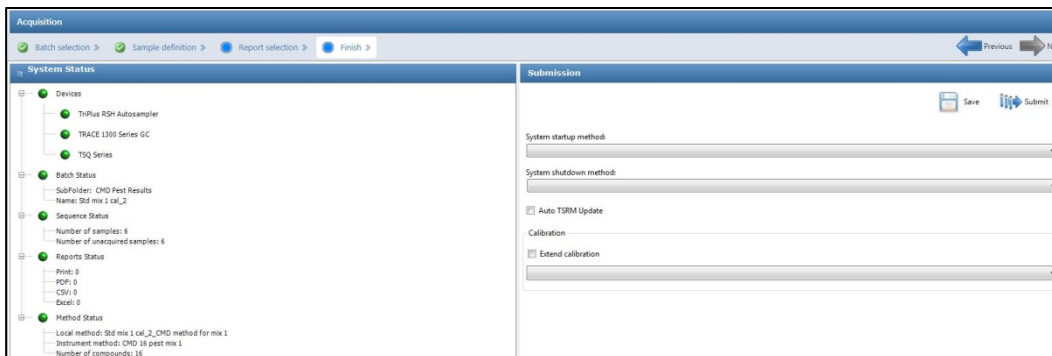
Samples Auto Samples Reference Sample

	Status	Filename	Sample type	Groups	Qual Processing	Level	Sample ID	Sample name	Comment	Vial position	Injection volume	Conversion Factor	Barcode Expected
1	●	STD 1	Cal Std		<input type="checkbox"/>	STD 1	10 ppb	STD		1		1.000	1.000
2	●	STD 2	Cal Std		<input type="checkbox"/>	STD 2	50 ppb	STD		2		1.000	1.000
3	●	STD 3	Cal Std		<input type="checkbox"/>	STD 3	100 ppb	STD		3		1.000	1.000
4	●	STD 4	Cal Std		<input type="checkbox"/>	STD 4	150 ppb	STD		4		1.000	1.000
5	●	STD 5	Cal Std		<input type="checkbox"/>	STD 5	200 ppb	STD		5		1.000	1.000
6	●	666-1	Unknown		<input type="checkbox"/>		666-1	Cucumber		6		1.000	1.000
7	●	667-1	Unknown		<input type="checkbox"/>		667-1	Lettuce		7		1.000	1.000
8	●	668-1	Unknown		<input type="checkbox"/>		668-1	Pepper		8		1.000	1.000

Sample Controls

Add 1 Insert 1 Import

- 3.1.12. Select the number of sample rows to add and then click **(Add)**
- 3.1.13. For each sample, type the lab report number in the **(Filename)** column (e.g. **666-1, 667-1, 668-1**)
- 3.1.14. For each sample, select **(Unknown)** from the Sample Type list
- 3.1.15. For each sample, type the lab report number in the **(Sample ID)** column (e.g. **666-1, 667-1, 668-1**)
- 3.1.16. For each sample, type the sample type in the **(Sample name)** column (e.g. **cucumber, lettuce, pepper**)
- 3.1.17. For each sample, type a vial position in the **(Vial Position)** column
- 3.1.18. For each sample, type (1 µl) in the **(Injection Volume)** column
- 3.1.19. For each sample, type a dilution factor (If present) in the **(Conversion Factor)** column
- 3.1.20. Click **(Next)** to continue to the next page
- 3.1.21. The **(Finish)** page opens

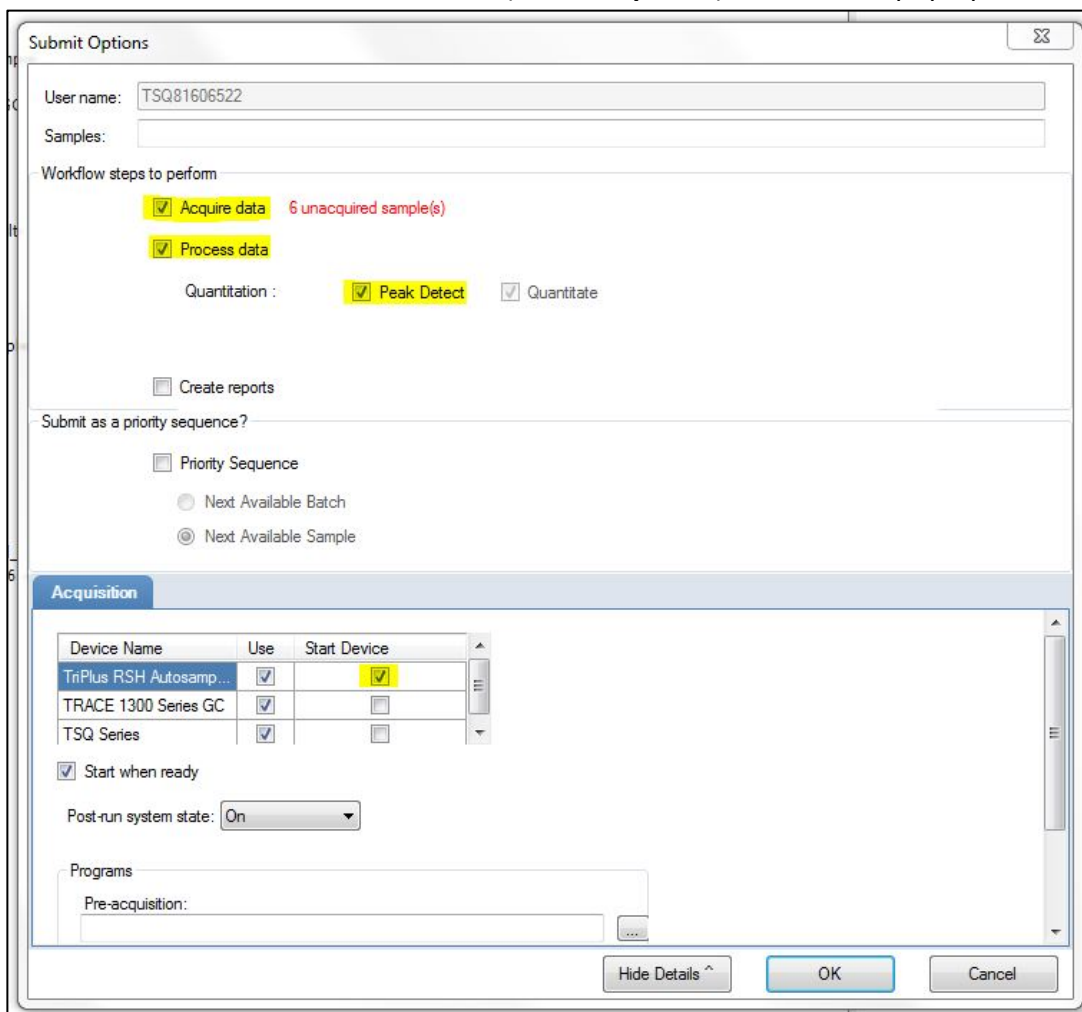


3.1.22. Make sure that Instrument status indicators are green which indicates that all devices are turned on

3.1.23. Click **(Submit)** to submit the batch



3.1.24. **(Submit options)** window will pop up



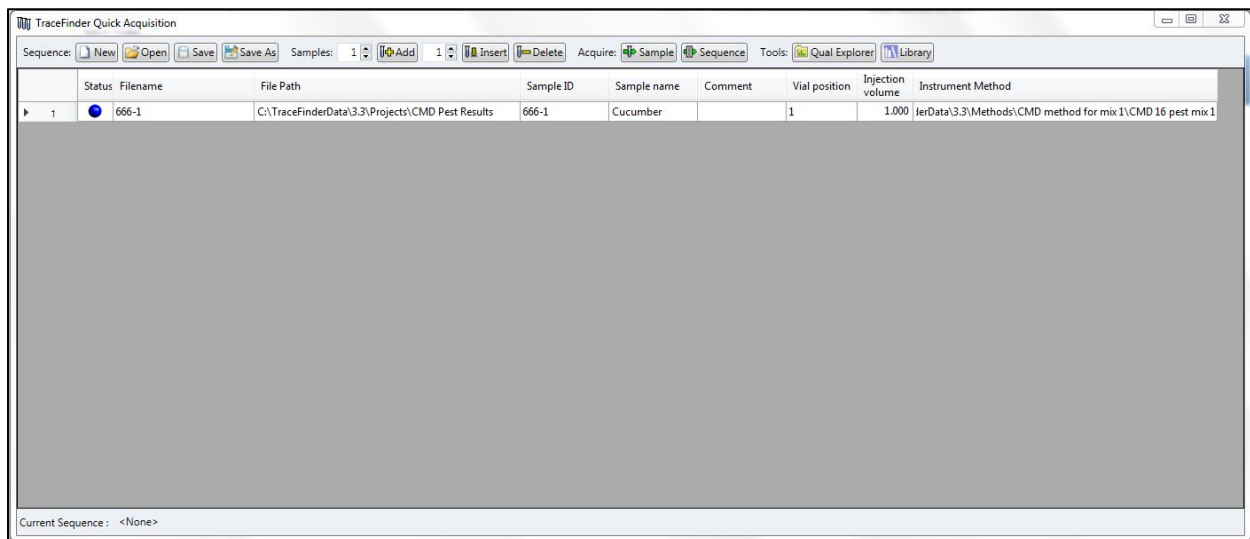
- 3.1.25. In (**Workflow steps to perform**), make sure that (**Acquire Data**), (**Process Data**), (**Peak detect**) and (**Quantitate**) options are all checked
- 3.1.26. In (**Acquisitions**), make sure that all the devices are checked and the (**Start Device**) is (**TriPlus RSH**)
- 3.1.27. Select the (**Start When Ready**) check box
- 3.1.28. Select the system state after it acquires the last batch to (**On**)
- 3.1.29. Click (**OK**) to submit the batch

3.2. Quick Acquisition

- 3.2.1. Place the sample in the Autosampler
- 3.2.2. On the desktop, open Thermo TraceFinder™ 3.3 application
- 3.2.3. Choose (**Tools > Quick Acquire Sample**) from the main menu or click the Quick Acquire Sample icon

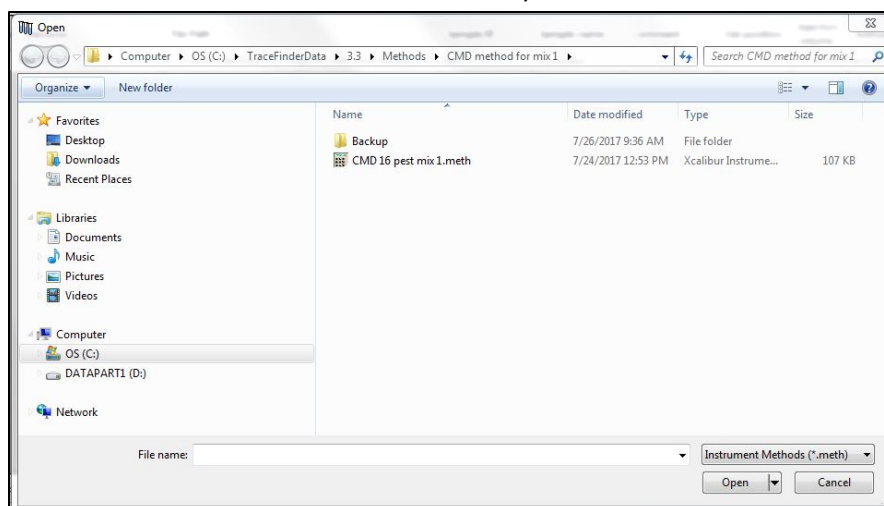


3.2.4. The (TraceFinder Quick Acquisition) window opens

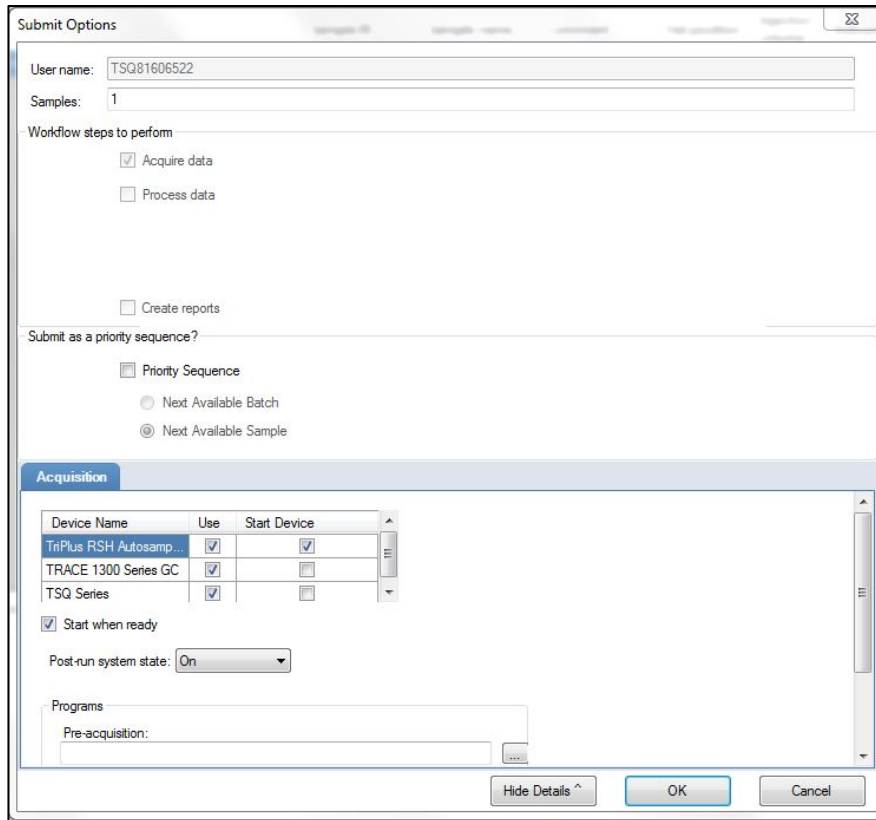


- 3.2.5. Type the lab report number in the (**Filename**) column (e.g. **666-1**)
- 3.2.6. Select (**Unknown**) from the Sample Type list
- 3.2.7. Type the lab report number in the (**Sample ID**) column (e.g. **666-1**)

- 3.2.8. Type the sample type in the (**Sample name**) column
(e.g. **cucumber**)
- 3.2.9. Type a vial position in the (**Vial Position**) column
- 3.2.10. Type (1 μ l) in the (**Injection Volume**) column
- 3.2.11. Double click the space under (**Instrument Method**)
to select an instrument method (e.g. **CMD method
for mix 1**)



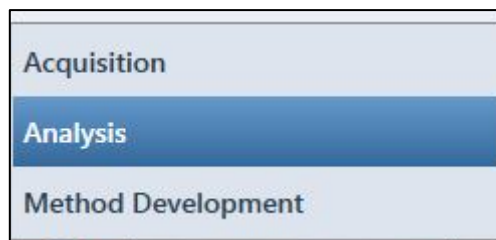
- 3.2.12. Click the (**Acquire Sample**) buttons
- 3.2.13. (**Submit options**) window will pop up



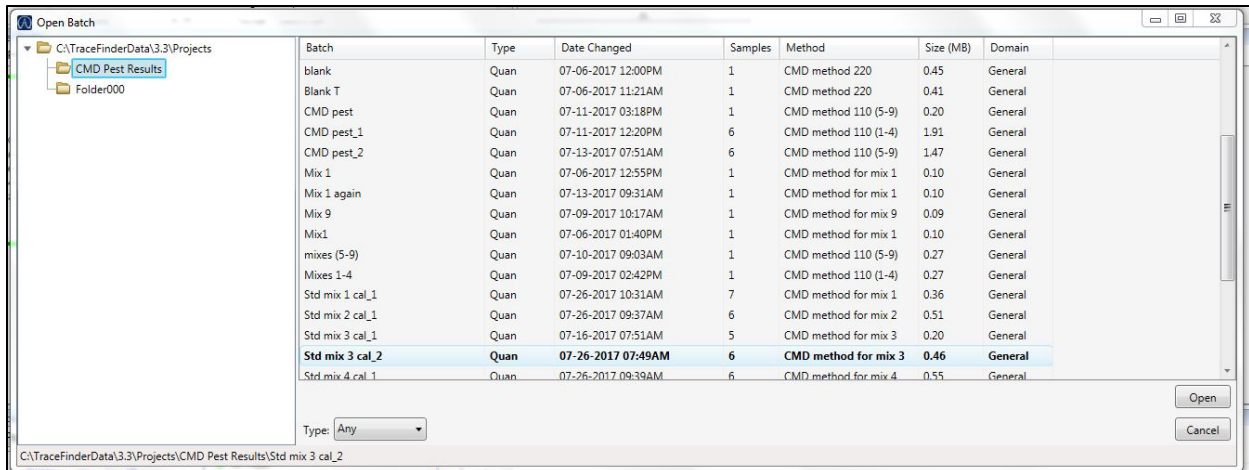
- 3.2.14. In (**Acquisitions**), make sure that all the devices are checked and the (**Start Device**) is (**TriPlus RSH**)
- 3.2.15. Select the (**Start When Ready**) check box
- 3.2.16. Select the system state after it acquires the last batch to (**On**)
- 3.2.17. Click (**OK**) to acquire

4. Data Analysis

- 4.1. Click (**Analysis**) in the navigation pane

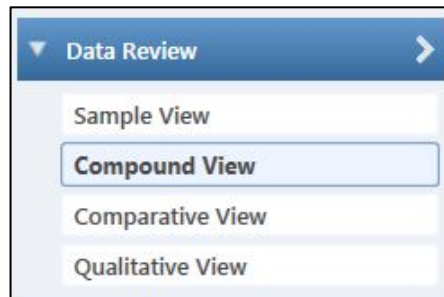


- 4.2. The navigation pane for the Analysis mode opens
- 4.3. In the (**Analysis**) navigation pane, click (**File > open > Batch...**)

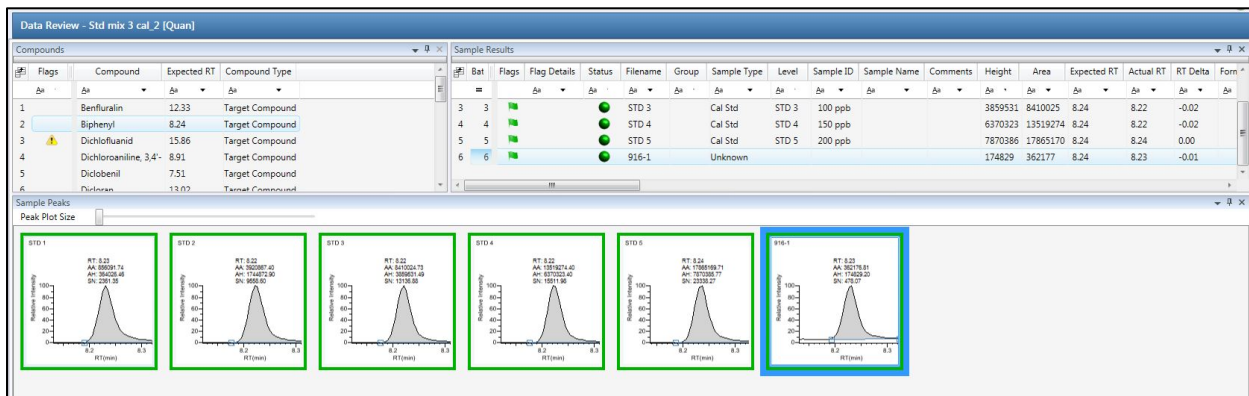


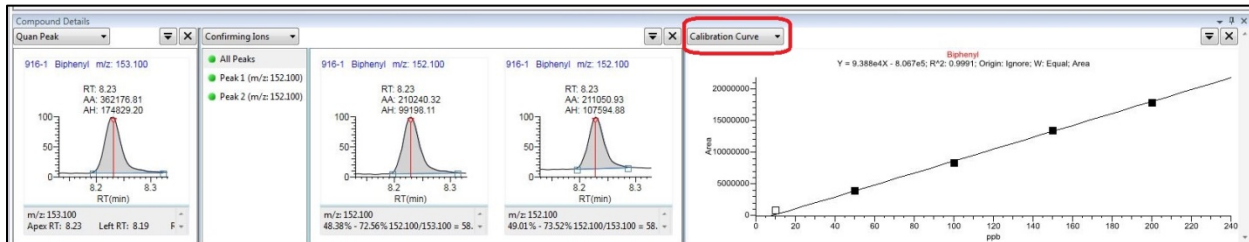
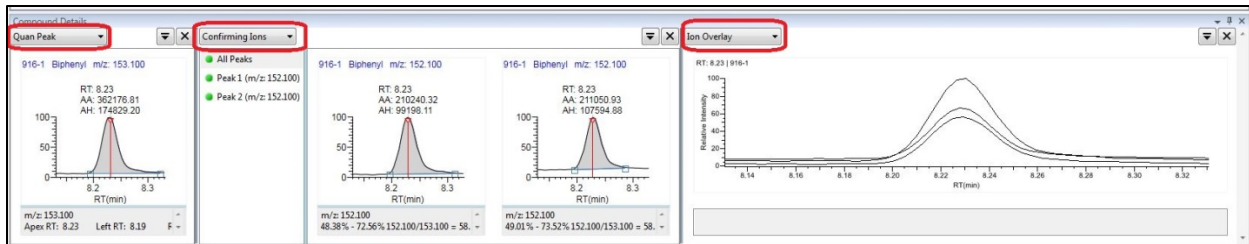
4.4. Open the Acquired batch

4.5. In the **(Data Review)** pane, click **(Compound View)**



4.6. In **(Compound Details)**, check each compound for **(Quan Peak detection, Confirming Ions detection, Ions overlay, Ions Ratio pass & Calibration curve)**





Ret	Calculated Amt	Theoretical Amt	Sample Amt
Aa	Aa	Aa	Aa
98.171		100.000	98.171
152.592		150.000	152.592
198.882		200.000	198.882
12.450		N/A	24.900

4.7. Report detected compounds concentration according to (AFQP 521) in food test system

5. Shutdown

- 5.1. **Please note that the system should not shutdown unless required for scheduled maintenance**
- 5.2. Go to (TSQ Due Dashboard) and click on (Instrument Control) button
- 5.3. Set values of the (MS transfer line temp.) and (Ion source temp.) to (0 °C) and click (Send) button
- 5.4. Wait until the Ion Source temperature actual value goes below 100 °C (Takes a few hours because of the vacuum)
- 5.5. Click on (Shut Down) button and answer (Yes) to the pop up window
- 5.6. Wait until (Turbo Pump Speed) goes from 100% to 0%

- 5.7. Switch Off the power supply on the TSQ Duo
- 5.8. In case of MS service support open the vent valve and wait about 5-10 minutes to equilibrate the pressure, If no service support is necessary do not open the vent valve
- 5.9. Close the software and switch Off the PC (**If needed**)
- 5.10. Make sure the GC oven is 40 °C or less, and then switch the power **OFF**
- 5.11. Close the valves on all gases supply bottles (**If needed**)
- 5.12. Switch the Autosampler power **OFF**