Number: SHT-A2PREP-2
Date: October 22, 2013
Pages: 6

SERVICE How To

Model Number:	Originator:	Topic
A2PREP	**Petro van Poppel**	**Instrument Setup Instructions**

Instrument Setup

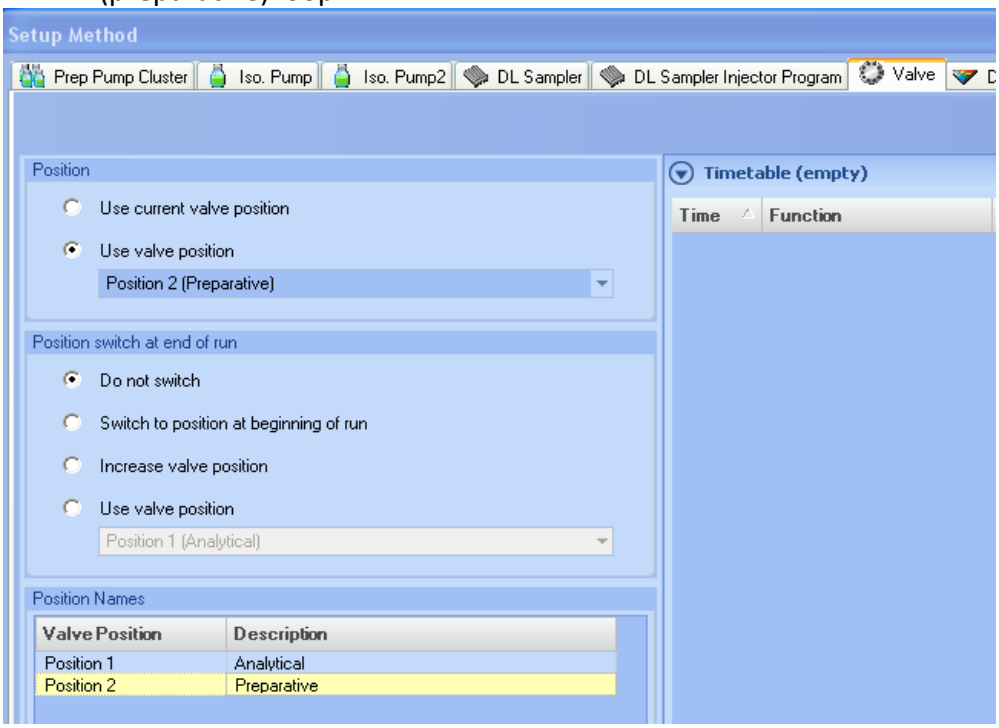
You will need:

- Solvent A: 100% water
- Solvent B: 100% ACN
- Standard Combined 1260 Analytical/Preparative System

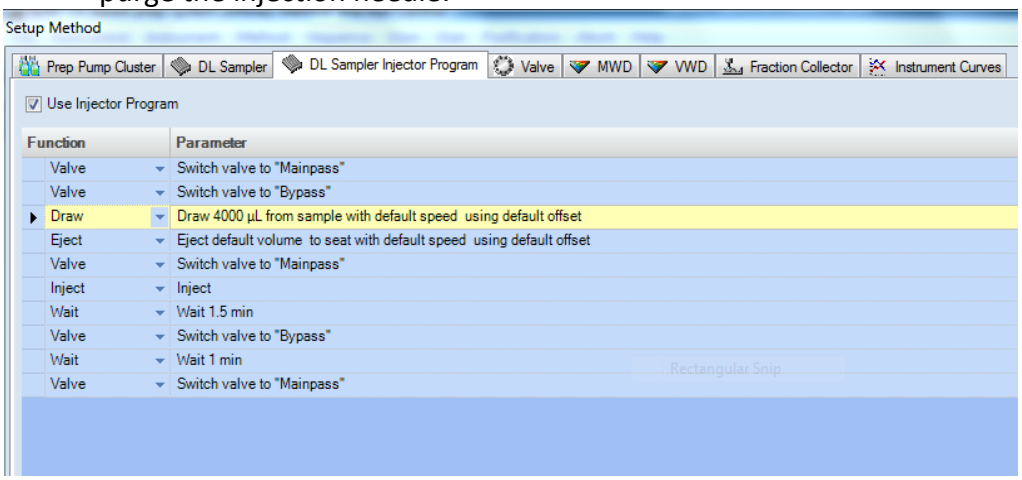
Procedure:

1. Turn On all modules, prime and purge the pumps for 1 minute at 100mL/min.

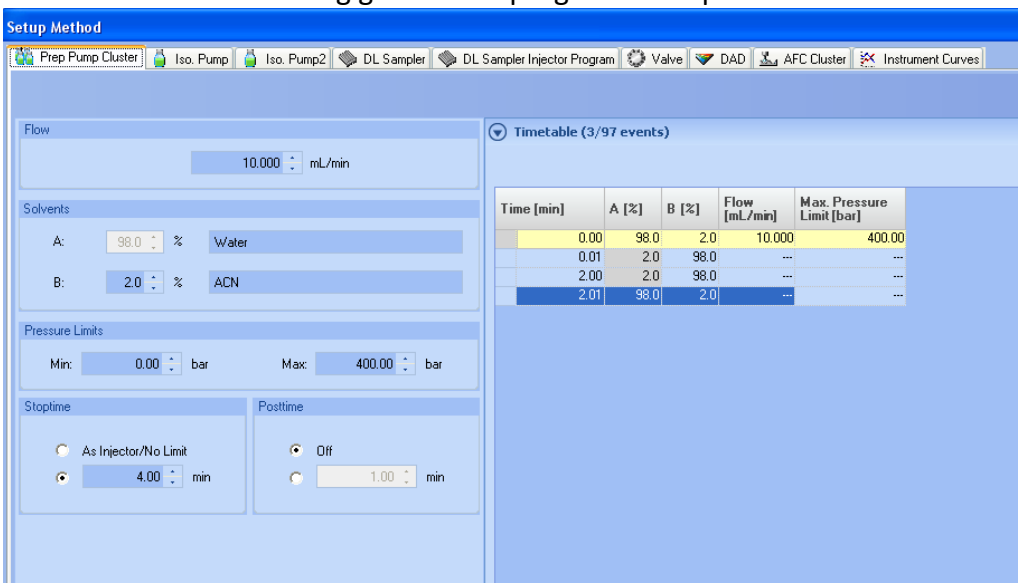
2. Create and run a Purge method for the Dual Loop sampler. Use the following details to create the method:
    a. Use the purge syringe command and purge at least 5 x the dual loop autosampler.
    b. Select the preparative flow path in the method and ensure that the purge method starts with an injection of a blank in the upper (preparative) loop.



c. Write an injector program and inject a blank of 4000 µl of ACN to purge the injection needle.



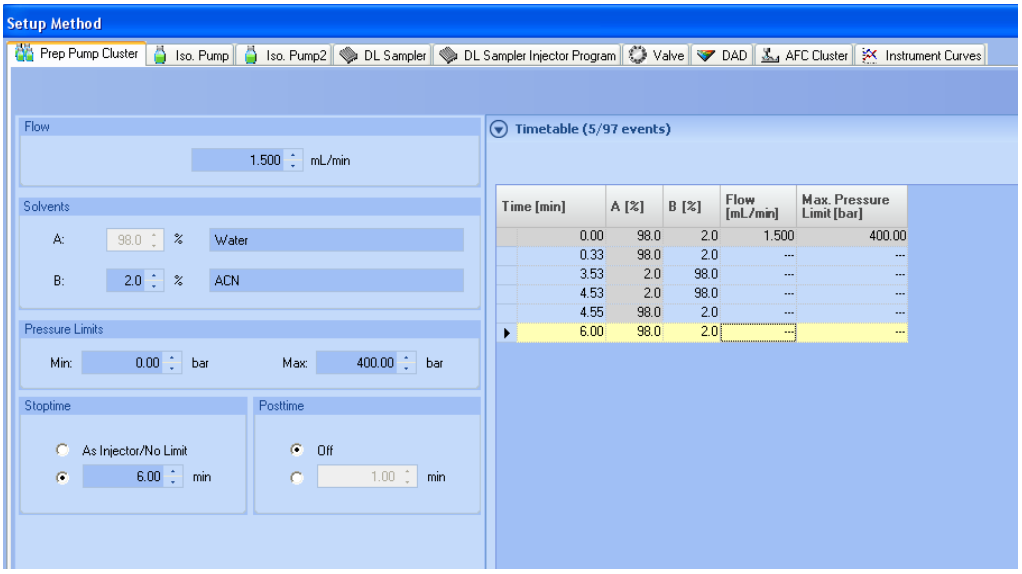
d. Write the following gradient to purge both loops.



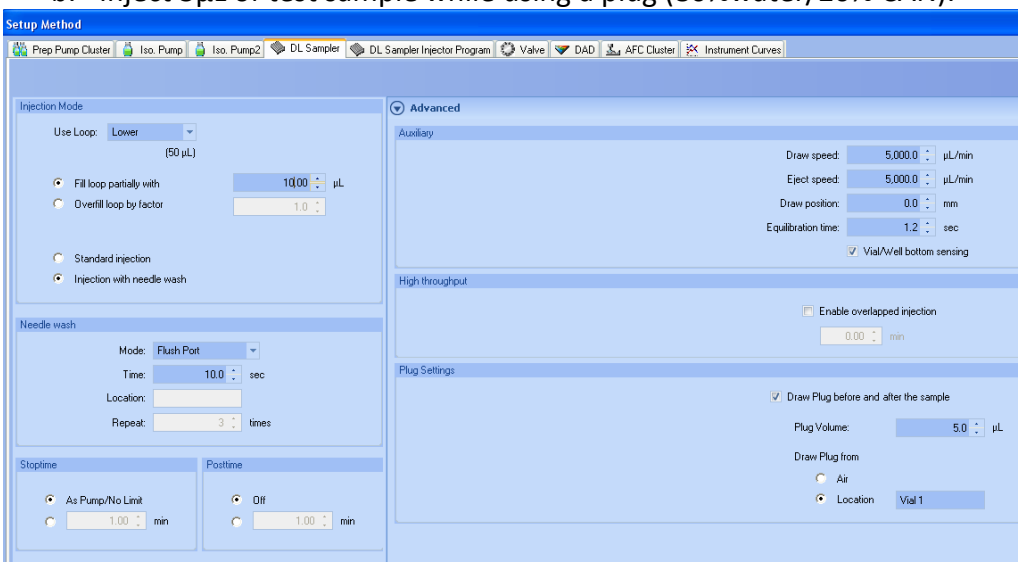
e. Stop method after 4 minutes.

f. Save method as “Purge Injector Method”

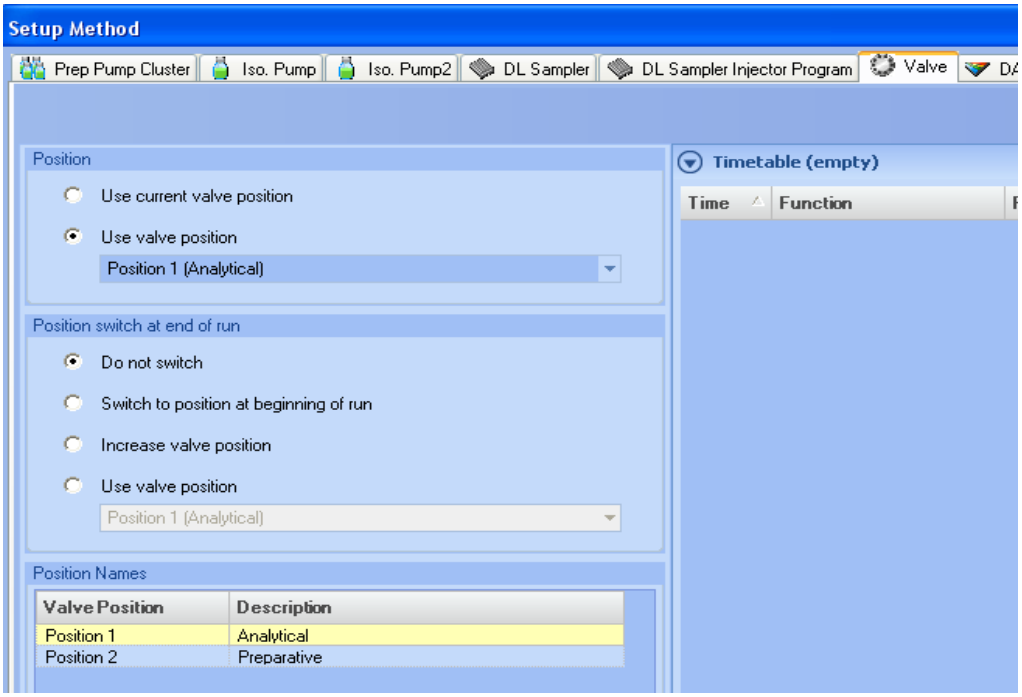
3. Create a Standard Analytical method, with the following details:
    a. Flow of 1.5mL per minute, Gradient from 2% to 98% organics, 6 minute runtime.



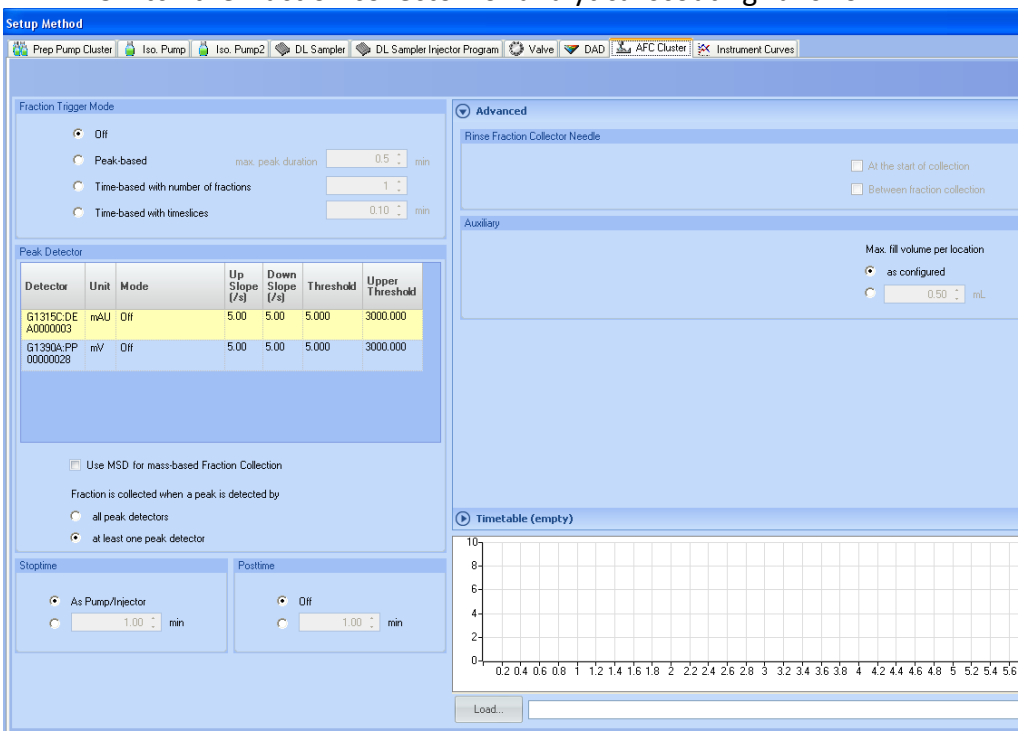
    b. Inject 5µL of test sample while using a plug (80%water/20% CAN).



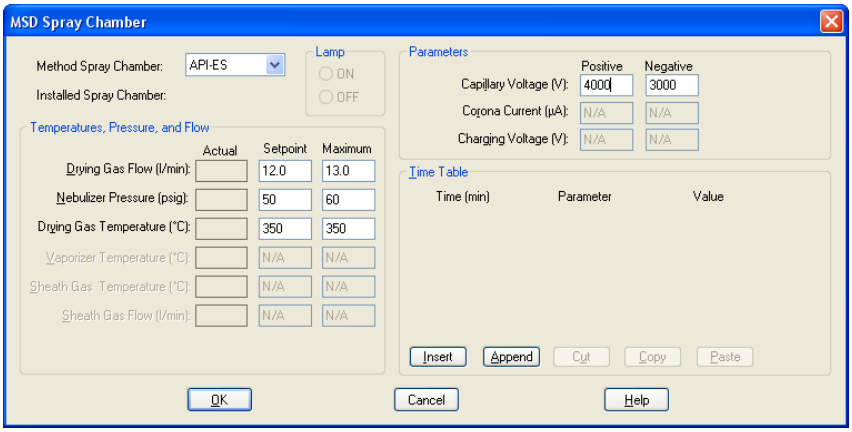
    c. Use at least a 10 seconds needle wash and the injection cleaning (10x) function to reduce carryover.
    d. Set the flow path to analytical.



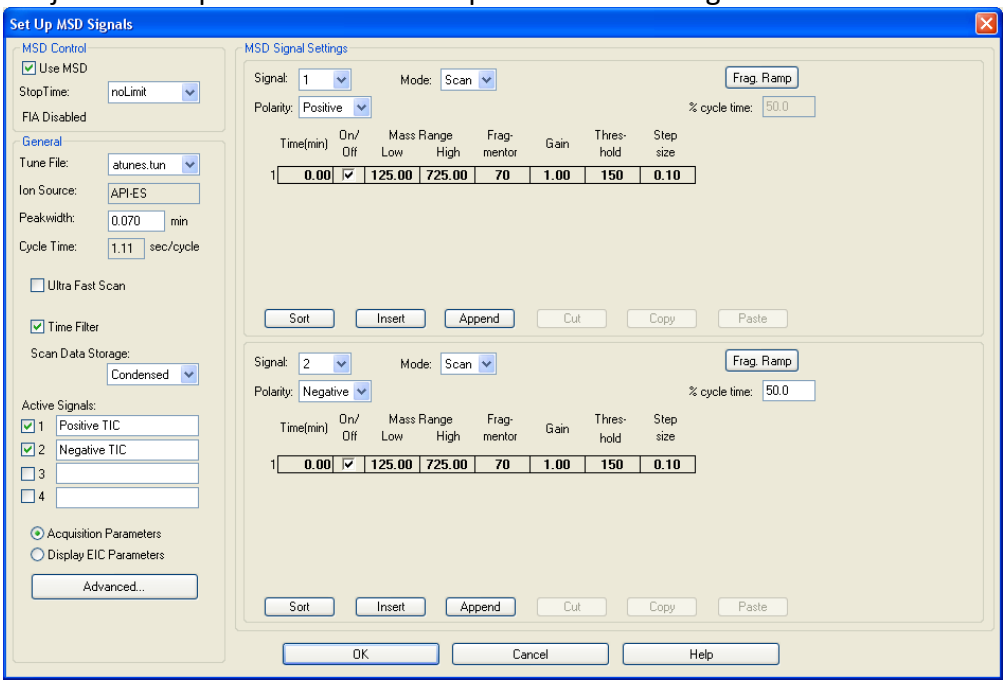
e. Set the UV absorption to 254 nm use a bandwidth of 30 nm.
f. Switch the fraction collector for analytical scouting runs “off”.



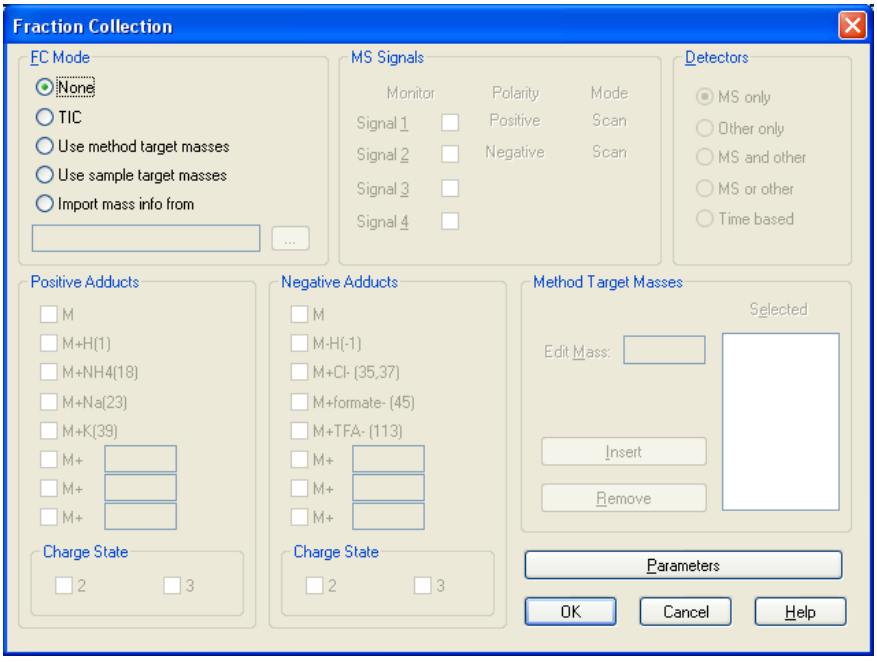
g. Monitor the Solvent Compositions.
h. If an MSD is part of the system, follow steps i to k.
i. Set the correct parameters for the ion source.



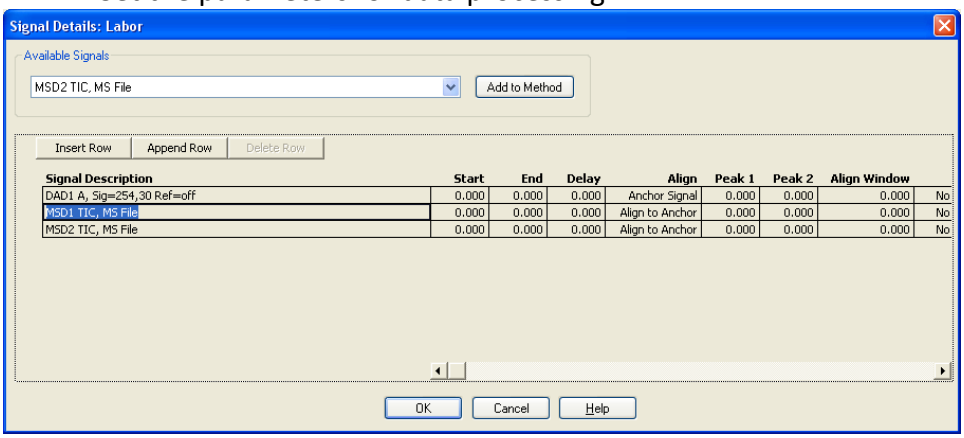
j. Set the parameters of scan speed and scan range.



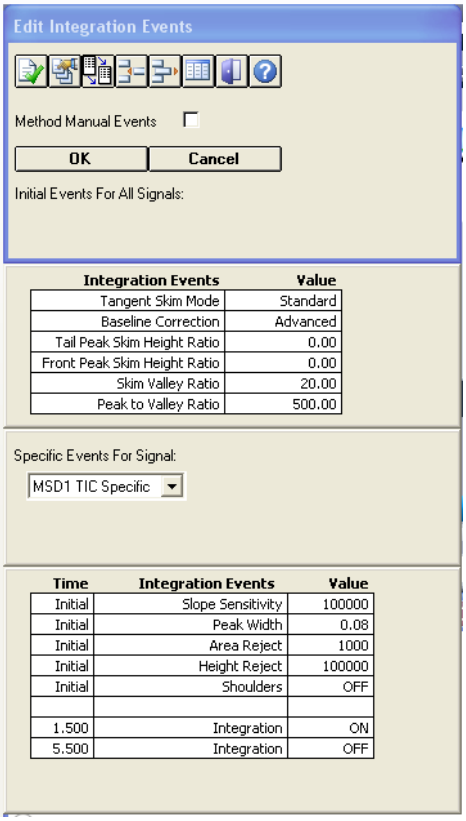
k. Disable Fraction Collection.



l. Set the parameters for data processing.

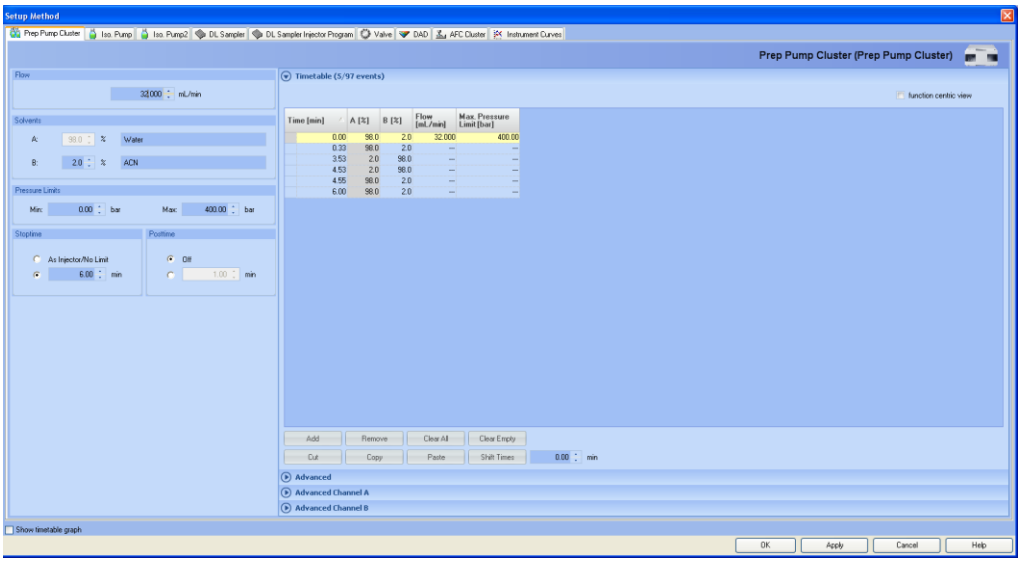


m. Set the correct integration parameters (start/stop value, peak width/height and area reject).

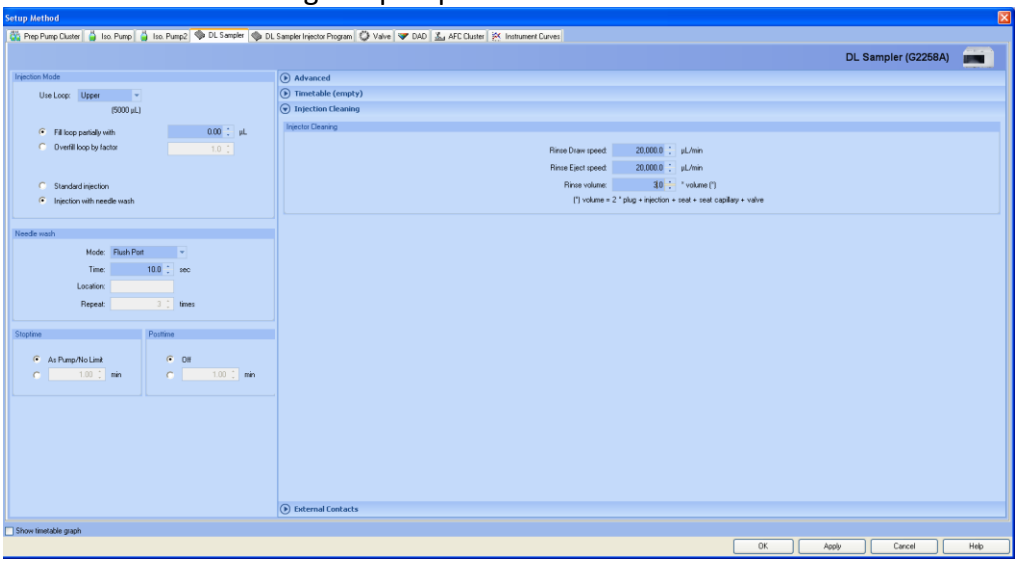


n. Save the method as “Analyt_Basis_Short” and create a sequence of 3 injections.

4. Create a Standard Preparative method, with the following details:
    a. Flow of 32mL per minute, Gradient from 2% to 98% organics, 6 minute runtime.

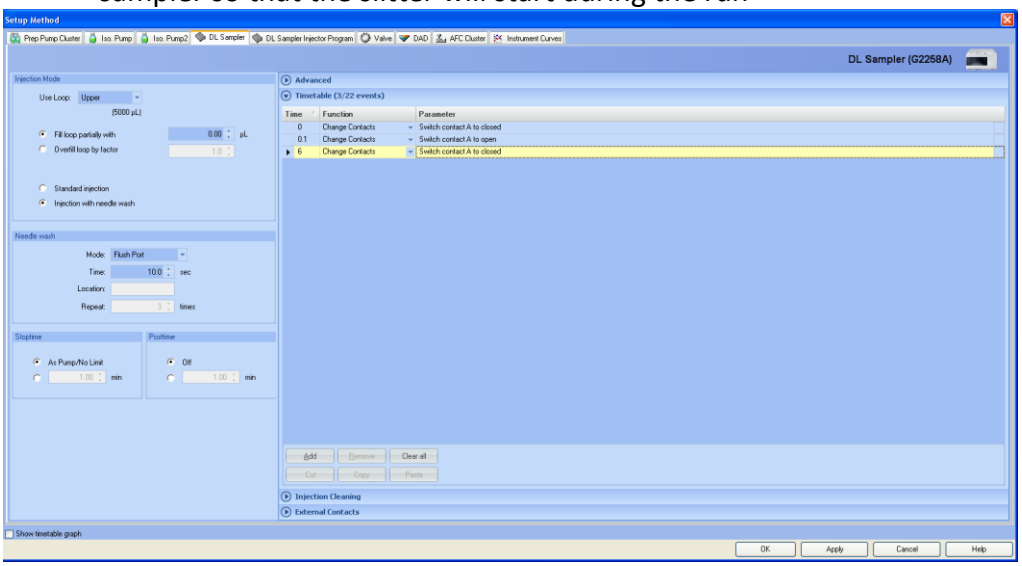


    b. If an MSD is used, set a make-up flow rate of 1.5mL/min, where the make-up solvent contains: 70 % Methanol/ 25% Acetonitrile/4.9 % Water/ 0.1% Formic Acid.
    c. Set the following sampler parameters.

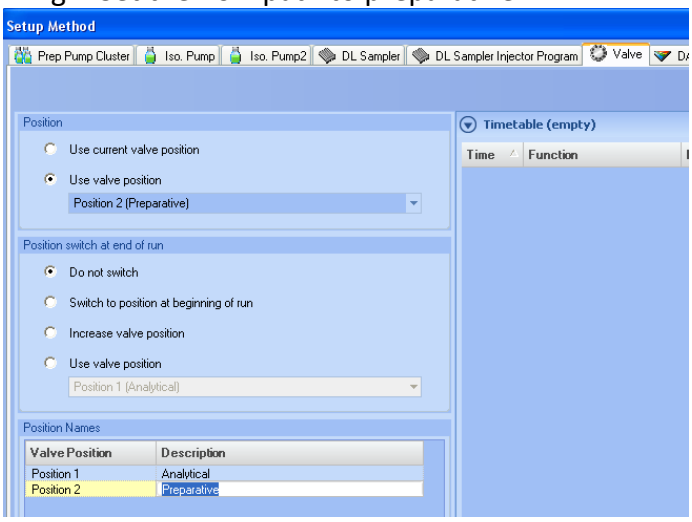


    d. Use at least a 10 seconds needle wash and the injection cleaning (3x) function to reduce carryover.
    e. Set the active flow split to a split ratio of 1:1000

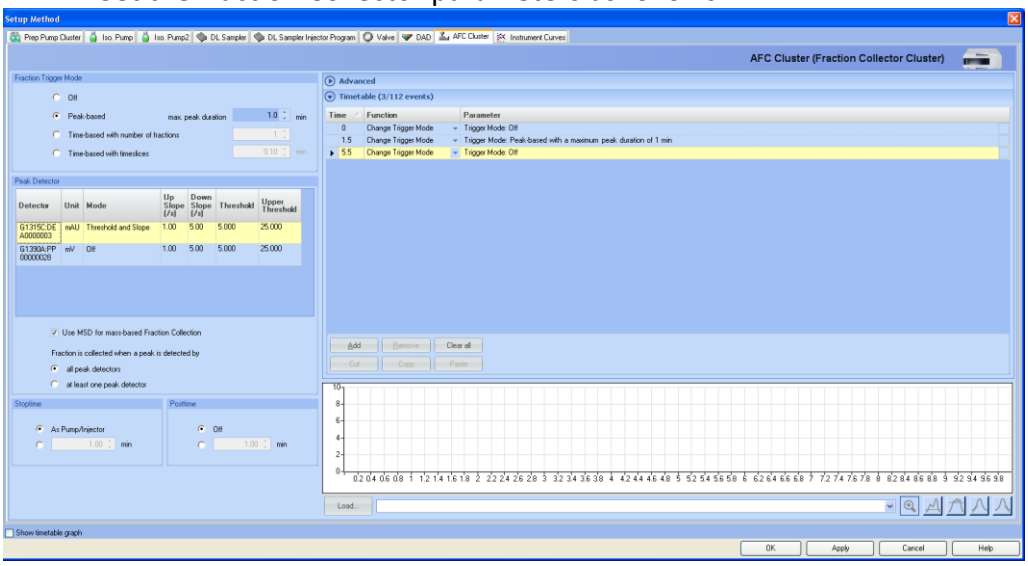
f. Start and stop the active flow split by an external contact event on the sampler so that the slitter will start during the run



g. Set the flow path to preparative.



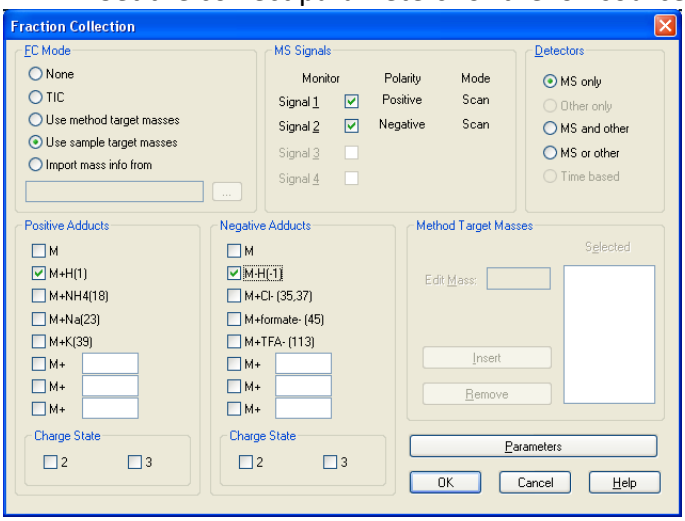
h. Set the fraction Collector parameters as follows.



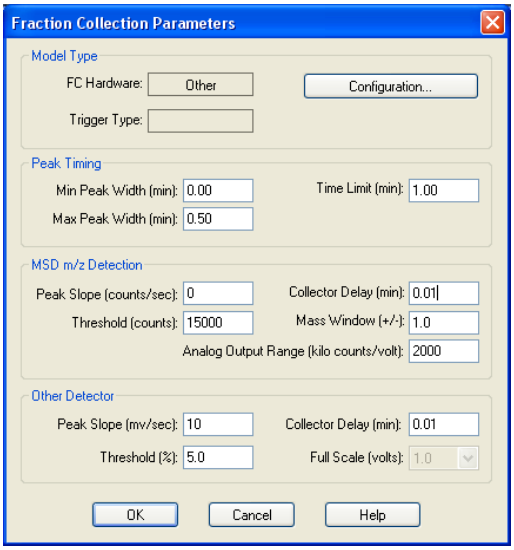
i. Set the UV absorption to 254 nm use a bandwidth of 30 nm.

j. If an MSD is part of the system, follow steps i to k.

k. Set the correct parameters for the ion source.



l. Set the threshold and slope parameters for mass based fraction collection.



m. Leave the delay value as is, it would have been adjusted from the purification software (Delay Calibration).

n. Save the method as “Prep_Basis_Short”.