TQD User Policies

As agreed upon at User meeting on 11.17.11 (Updated 11.04.13)

To communicate with other users:

- **DL-SOP-Triple Quad**
- To get added to this user list, email help@rx.umaryland.edu

Sign up

- Any free time can be reserved by signing up on the calendar
- Changes and/or cancellations to your reserved instrument time should be communicated to other users by using the distribution list
- A one (1) hour grace period is given to users according to their reserved start time. If a user has not begun/arrived by that time, other users are free to use the instrument on a first-come-first serve basis.

Sign In

- Log your name, PI, time used and number of samples on log book
- Fill out sample form (will include brief description of what you are doing, type/matrix of samples, and an assessment of instrument performance)
- Have your contact information available (email and phone number where you can be reached) if you leave samples running while you are not there

Bring:

- Your own solvents. You need to use **LC-MS grade solvents**.
- Your own column.
- Other assorted supplies you may need (gloves, pipets, vials, etc.)

Every-Use Cleaning:

- When you are done you need to flush out the system.
- If using buffers, must flush out whole system and the reservoir line with pure H2O
- Leave in 50:50 ACN:H20 or some comparable condition. Please note what solvent composition you have left in the instrument.
- Remove your sample vials and column when completed
- If you arrive at the instrument and someone else's samples/solvents/column are present, you can remove them and place them on the shelf above the instrument.

Operator's Guide

- A hardcopy of the Operator's Guide is located on the shelf above the instrument in a blue binder.
- The TQD Operator's Guide and other user guides are also available online at: http://www.waters.com/waters/support.htm?lid=1853123&cid=511442&type=USRM

Maintenance Log

- Write down any cleaning, maintenance, issues in the log book so that we have an accurate record of the instrument. Also record any steps you have taken to remedy the problem.
- Types of things to record:
    - communication errors (log exact error message and time/date)
    - loss of sensitivity
    - leaks or other plumbing issues
    - other errors of note

Periodic Cleaning

- At the monthly sign-up meeting, users will also sign up for weekly cleaning on a rotating basis.
- **Weekly:** Clean sample cone and gas cone as per instructions in Operators Guide section 5-20 "Cleaning the sample cone and gas cone"
- **Occasionally, as needed:** Depending on usage and sample composition the following cleaning may also need to be performed according to Operators Guide sections 5-30 and 5-47 "Cleaning the ion block, isolation valve, and extraction cone" and "Cleaning the source hexapole assembly".
- **Occasionally, as needed:** The flowcell for the UV detector also needs to be cleaned periodically following the protocol starting on page 45 in the PDA user's manual. http://www.waters.com/waters/support.htm?lid=10173117&cid=511442&type=USRM

Waste

- All users should share in monitoring the waste
- Please note what solvents you use, so that the approximate composition of the barrel can be determined
- There should be an EHS barrel for the waste (and an empty backup barrel) at all times.
- If you see this waste becoming full, it is your responsibility to fill out the EHS form online and request a pick-up of the waste and a replacement barrel
- http://www.ehs.umaryland.edu/Waste/waste_removal_request_forms.cfm

Essential Information Before you Start:

- Sample plate format: you MUST choose the **"ANSI 48 vial holder -2 ml".** The injector can be damaged if you use the wrong format.
- To make a new method:
    - You need to create three things all **WITH THE SAME NAME**
        - A tune file
        - a method set
        - a method
    - Then you can use intellistart to auto-tune your compound parameters
- Solvent reservoirs
    - To prime the sample reservoirs, use the controls in the Acquity console
    - Make sure you fill the wash reservoir(s), too.

Solvents and Caveats for LC/MS

Solvents are typically chosen based on a compound of interest's solubility and compatibility with various ionization techniques used in LC/MS. Volatility and the solvent's ability to donate a proton are important in ESI and other atmospheric ionization techniques.

Protic primary solvents like MeOH and mixtures with H_2O, such as 1:1 MeOH/H2O or 1:1 ACN/H_2O, are used. Water's relatively low vapor pressure can be detrimental to sensitivity when employed at 100%. Better sensitivity results when surface tension is decreased through addition of a volatile organic solvent. Surfactants with higher proton affinity, though they increase ion liberation from nebulized droplets, can also reduce sensitivity.

Aprotic co-solvents like 10% DMSO in water and isopropyl alcohol improve solubility for some compounds. Formic acid is often added at low levels (0.1%) to facilitate ionization by ensuring the analyte is more basic than the solvent. Even in small amounts, however, some acids, like TFA, though necessary for otherwise insoluble compounds, can limit sensitivity.

In the ESI ionization mode, **buffers and salts (Na+, K+, and phosphate) cause a reduction in the vapor pressure and consequently a reduced signal**. The increased surface tension of the droplets, and resultant reduction of volatility, can be remedied by using relatively more volatile buffers like ammonium acetate, formed by a weak acid-base pair.

Solvent considerations

- Useful Solvents
    - **Water**
    - **Acetonitrile (ACN)**
    - **Methanol (MeOH)**
    - Ethanol
    - Propanol
    - Isopropanol
- Acceptable additives
    - Acetic acid
    - Formic acid
    - Ammonium hydroxide
    - Ammonium formate (salt concentration = 10 mM or less)
    - Ammonium acetate (salt concentration = 10 mM or less)
- Nonvolatile salts (phosphate, borate, citrate, etc.)
    - Can deposit in source and plug capillaries thus requiring more cleaning and maintenance operations
    - Modern source designs can handle nonvolatiles better than older designs
- Surface-active agents (surfactants/detergents) suppress electrospray ionization
- Inorganic acids are corrosive
- Trifluoroacetic acid (TFA)
    - To some extent suppresses positive-ion electrospray at levels exceeding 0.01%.

    - Greatly suppressed negative-ion electrospray.
- Triethylamine (TEA)
    - High PA (232 Kcal/mole) yields an intense [M+H]+ ion at m/z 102
    - Suppresses positive ion electrospray of less basic compounds.
- Tetrahydrofuran (THF)
    - 100% THF is highly flammable, so APCI and most interface techniques use nitrogen as the nebulizer gas. (Using air creates an explosion hazard).
    - Reacts with PEEK® tubing.

Weak and Strong Wash Solvents:

Weak Wash: ACN/H_2O 10:90 (v/v)

Strong Wash: ACN/H_2O 90:10 (v/v)

Sample Form: Place in log book after each session

Name:
PI:
Date:
Start & End time:
Number of samples :
Total net usage time (including setup and post-run cleaning) in hours:

Sample and Analysis Information
Type of samples you are analyzing (e.g., extracted cells, plasma, tissue, etc.):

What type of sample prep or reference to procedure?

Injection solvent:
Injection volume:
Column used:
Flow rate:
Back pressure generated:

Chromatographic Solvents used: please record what you have used.
A:
B:
C:
D:
Wash reservoirs:

Instrument Performance:
Please comment on the instrument performance that you have encountered.