Hg Analyzer
DMA – 80 (tri-cell)
Standard Operating Procedure
(Revised 01-18-2011)

I. Start Up

1. Sign into the logbook.
2. Turn on computer FIRST
3. Turn instrument on (button is on front of instrument)
4. Open Oxygen tank regulator (high purity O2 not necessary)
    A) Set regulator line pressure to 65 psi
    B) Instrument flow rate = 6-8 liters per min.
    (Adjust flow with knob on back of instrument)

5. The instrument needs to warm up for 30 minutes.

6. Leave clock on 24 hour (military time) or it messes up software

7. Open the method file you want to use.
   Method tab => open file => open method

8. Open the calibration file that you want to use.
   Calibration tab => open file => open calibration

9. Enter samples: they can be run one at a time, or as a group.
   Measure tab => sample tab => click multi sample => click add + 1 sample => enter sample name (enter sample position if different that auto) => enter sample weight.

10. Always run 6 blanks first thing to "clean" instrument (first blank without sample boat to determine if system itself is "clean".

11. Use weight of 1.0 for the blanks
    - If blanks stay high, might need to change the catalyst
12. Run calibration
    - 2% HCL matrix is suggested
    - Transfer standards into brown glass bottles
    - Hg stds are unstable in plastic or clear bottles
    - Store stds in a fridge

13. Milestone recommends to use "square fit " for curve calculation

14. Cell 0 = standards that are less than or equal to 1 ng Hg
    Cell 1 = equal to 1 ng Hg to 20 ng Hg
    Cell 2 = greater than 20 ng Hg (up to~ 1000ng)

    - Blank should be less than 0.009
    - Detection limit is 0.1 ng to 1000 ng Hg
    - Specific voltage should be 3.5 V to 4.0 v

Signal analysis= [X] Maximum
[ ] Integral
[X] Ext shutter
[ ] Signal beyond zero
[X] Peak detection

15. Save data, methods, calibration => my documents => 10020815 => data

II. Shut Down

1. Turn instrument off FIRST (button is on front of instrument)
2. Turn computer off

III. Maintenance

1. Consumables = amalgamate and catalyst
2. After replacing consumables, always check flow rate
    a. should be 6-8 Liters per hour
    b. adjust this with knob on back of instrument
3. Re-calibrate

IV. Creating a new method (must be administrator, not user)

1. Open an empty method file (first save open method)
2. Methods are saved with .m80

<u>Drying and Decomposition</u>

3. Samples with high water content – first drying step = 200*C
4. Samples with liquids with boiling point under 100 *C require an accordingly lower temperature
5. After drying, sample decomposition at high temperature (burned in the oxygen flow)
6. A decomp temp of 650*C ensures a complete decomposition and release of Hg. Only at a sufficiently high temp the entire Hg is set free and can be measured.
7. Example: Pos #1 30 sec. @ 200 *C
    #2 3.0 min @ 200 *C
    #3 1.0 min @ 650 *C
    #4 2.0 min @ 650 *C

<u>Purge Time</u>

8. This is the time elapsing between the end of drying/decomp and the start of the Hg measurement.
9. Standard purge time is 60 seconds

<u>Amalgam Heater Time</u>

10. This is the time required by the amalgam heater to completely release the Hg collected in the absorption cell.
11. Standard setting is 12 seconds
12. Concentrate-in this measuring mode only the last of a series of samples is heated

<u>Recording Time</u>

13. In this time is noted and evaluated the measuring signal of the spectrometer.

14. Standard setting is 30 seconds
15. Concentrate-in this measuring mode only the signal of the last of a series of samples is recorded.
16.