PROTOCOL MSMC-001 version 1 GENERAL PROTOCOL FOR CLEANING GLASSWARE Contributed by staff of MSU Mass Spectrometry and Metabolomics Core Last modified January 16, 2018

Purpose: cleaning of glassware (*e.g.* HPLC solvent bottles, graduated cylinders, round bottom flasks) used for trace analysis of organic chemicals by GC/MS or LC/MS. The protocol is not intended for cleaning of plasticware.

Note: some glassware may show visible evidence of contamination that cannot be removed using the procedure described below. This may consist of insoluble inorganic residues (e.g. spots) or organic contaminants that do not extract well (evident from unusual "beading" of water on the interior of the container. In such cases, it is recommended that a small amount of concentrated sulfuric acid be added to the container, followed by turning the container so that the sulfuric acid makes contact with all interior surfaces. The sulfuric acid can then be drained from the container and disposed of properly (separate from organic wastes). Mixing of concentrated sulfuric acid with water generates heat, and should be performed slowly by adding acid to water (e.g. draining into a much larger volume of water).

Safety precaution: wear appropriate personal protective equipment (lab coat, safety goggles, nitrile gloves)

- 1. Scrub glassware with detergent in hot tap water using a brush appropriate for the container size. Alconox detergent is recommended, and can be prepared in solution form ($\sim 0.1-1\%$) in tap water in advance. Clean interior and exterior of vessel.
- 2. Rinse six times by filling container with warm/hot tap water and emptying vessel into sink drain.
- 3. Rinse six times by filling container with Milli-Q water (> 18 M Ω) and emptying vessel into sink drain.

Steps 4-6 should be conducted in a chemical fume hood.

- 4. Rinse vessel three times with methanol (~ 1/10 of container volume); dispose of waste properly
- 5. Rinse vessel three times with acetone ($\sim 1/10$ of container volume); dispose of waste properly
- 6. Rinse vessel three times with hexane ($\sim 1/10$ of container volume); dispose of waste properly
- 7. Allow glassware to dry on a drying rack or drying oven; openings to glassware may be loosely covered using hexane-rinsed aluminum foil (aluminum foil has a waxy coating that should be removed before use).