

## Toluene Water Analysis

September 2013 NAA

For use on water collected after toluene distillation.

### I. Reagents:

- a. 25mL 2-EB (internal standard)
  - i. Add 50mL ddH<sub>2</sub>O to a 100mL volumetric flask and a stir bar
  - ii. Weigh ca. 0.3655g 2-ethylbutyrate (C<sub>6</sub>H<sub>12</sub>O<sub>2</sub>) add quantitatively to flask and mix well
  - iii. Note exact weight of 2EB added to calculate molarity (MW of 2-EB is 116.16)
  - iv. When in solution remove stir bar, rinse and q.s. 100mL with ddH<sub>2</sub>O
  - v. Store in refrigerator

### II. Personal Protective Equipment:

- a. Lab coat
- b. Safety glasses/goggles
- c. Latex gloves

### III. Sample Preparation

- a. If water sample is frozen, thaw and vortex well
- b. Pipet 2mL of vortexed sample into a disposable glass tube
- c. Pipet 0.2mL of 2-EB solution into the same tube and vortex well
- d. Pipet sample into a GC vial and seal, then store in the refrigerator

### IV. GC Analysis

- a. Hydrogen usage (20mL/minute) in closed GC system.

V. Column Specifications and GC Conditions:

a. Column

i. Packing

1. Supelco 11965
  - a. 10% Sp-1200
  - b. 1% H<sub>3</sub>PO<sub>4</sub>
  - c. 80/100 Chromosorb W AW
2. Supelco 12144
  - a. 15% SP-1220
  - b. 1% H<sub>3</sub>PO<sub>4</sub>
  - c. 100/120 Chromosorb W AW

ii. Measurements

1. Length: 8 feet
2. I.D.: 2mm
3. O.D.: ¼ inch

iii. Special Instructions

1. Leave 3 inch space on injector end
2. Leave 2 inch space on detector end

b. GC Conditions

i. Temperature

1. Oven: 145°C
2. Injector: 185°C
3. Detector (FID): 200°C

ii. Flow Rates

1. Nitrogen (N<sub>2</sub>): 20 mL/minute
2. Hydrogen (H<sub>2</sub>): 20 mL/minute
3. Compressed Air: 220 mL/minute

iii. Slope Sens: 0.10

iv. Attenuation: 2<sup>8</sup>

c. Supplies

i. Syringe

1. Hamilton 80358 w/ Hamilton 80458 needle
2. Hamilton 910393

ii. Septum

1. Thermogreen LB-2 Septa 11.0 mm (Supelco 20654)

iii. Inlet Liner

1. PureCol Inlet Liner 2 mm (Supelco 20536)
- iv. Filters
  1. Nitrogen
    - a. Agilent OT3-2
    - b. OMI-2 (Supelco 23906)
  2. Hydrogen
    - a. Agilent OT3-2
    - b. OMI-2 (Supelco 23906)
  3. Air
    - a. Supelco 20618 w/ Supelco 20298 replacement mesh