Microplastics Sample Collection Methodology and Laboratory Processing

Sample Collection Methodology

Sample collection will be conducted at two channel and two near shore locations within Hudson River Park by trawling a Neuston net. Each trawl will have a duration of **15 minutes,** throughout the months of June to October.

1. Trawling sites in Hudson River Park
    a. **Site 1 (C1 & NS1)** – Between Pier 40 and Pier 25
    b. **Site 2 (C2 & NS2)** – Between Pier 76 and Chelsea Piers
2. Trawling Method
    a. **Neuston trawl** from a motorized vessel
        i. Trawl is secured with a rope from the foreword most portside cleat
        ii. Trawl at a consistent speed of **3-5 knots**, ensuring smoothness
3. Trawl time/GPS coordinates and sea/boat conditions are recorded as per *5 Gyres Institute's Citizen Science Protocols for collecting and sorting sea surface samples*.
    a. Field data recorded on 2016 Microplastics Sampling Field Data Sheets.
4. **Trawling Procedure**
    a. Rig Neuston Net and attach collection jar
        i. Bridle attaches at each of the 4 corners of the net's frame
        ii. Thread rope through bridle & **forward most portside cleat**
        iii. Collection jar screws into threaded ring at end of net
    b. Take GPS coordinates & Sea/Boat conditions
        i. Use the **CTD** to take water conditions (see instructions)
        ii. Use Boat's systems/phone for GPS coordinates
        iii. Beaufort Scale to determine sea conditions
    c. Place net in water and begin trawling
        i. Ensure net remains relatively flat and stable in the water; **not under boat**
        ii. Ensure collection jar is submerged and empty of air bubbles
    d. Trawl for 15 minutes
        i. Monitor net to ensure smoothness of trawl
    e. When stopped, remove net from water
    f. Pour excess water from collection jar out through net; rinse with site water
        i. Fill squirt bottle with site water to rinse
        ii. Remove large organics/debris
    g. Transfer sample to a mason jar
        i. Label as follows on lid and side: Site#, Date (ex. C1 7/19/16)
5. **Store samples in commercial isopropyl alcohol solution**

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a. Unless being taken directly to lab for analysis

Laboratory Processing Methodology

This processed is based from: NOAA's **Laboratory Methods for the Analysis of Microplastics in the Marine Environment: Recommendations for quantifying synthetic particles in waters and sediments.** This requires a hooded lab/adequate ventilation.

1. **Wet Sieving**
    - **a.** Pour the sample through a stacked arrangement of 5.6-mm and 0.3-mm stainless steel mesh sieves
    - **b.** Rinse sample with squirt bottle of distilled water to transfer all residual solids to sieves as needed.
        - ***i.*** *Pick out large (leaves/seaweed/macroplastics) or troublesome (unidentified goo/plant fibers) debris*
    - **c.** Rinse sieves thoroughly with distilled water, drain, and sort.
    - **d.** Discard or archive material retained on 5-mm sieve
2. **Transfer Sieved Solids**
    - **a.** Weigh dry 500-mL beaker to nearest 0.1 mg
    - **b.** Transfer solids collected in the 0.3-mm nitrex sieve into tared beaker using spatula and minimal distilled water rinsing with squirt bottle
    - **c.** Ensure all solids are transferred to tared beaker
    - **d.** Place beaker in 90 °C drying oven for 24 hours
3. **Determine Mass of Total Solids**
    - **a.** Determine mass of beaker with dried solids with an analytical balance to nearest 0.1 mg
    - **b.** Subtract mass of tared beaker to provide mass of total solids (all microplastics and natural materials
4. **Wet Peroxide Oxidation (WPO)**
    - **a.** **CAUTION:** This mixture is highly reactive, observe proper safety protocols and perform in a fume hood
    - **b.** Add 20 mL of aqueous 0.05 M Fe(II) and sulfuric acid solution to beaker with 0.3 mm size fraction of solids
    - **c.** Add 20 mL of 30% hydrogen peroxide. **CAUTION:** this solution can boil violently if heated >75 °C
    - **d.** Let mixture stand at room temperature for 5 minutes before proceeding
    - **e.** Add stir bar to beaker and cover with watch glass
    - **f.** Heat to 75 °C on hotplate
    - **g.** As soon as gas bubbles are observed at surface, remove beaker from hotplate and place it in the fume hood until boiling subsides. If there is potential overflow, add distilled water to slow reaction.
    - **h.** Heat to 75 °C for an additional 30 minutes

   i. If natural organic material is visible, add another 20 mL of 30% hydrogen peroxide
   j. Repeat until no natural organic material is visible
   k. Add ~6 g of salt (NaCl) per 20 mL of sample to increase the density of the aqueous solution (~5 M NaCl)
   l. Heat mixture to 75 °C until the salt dissolves

5. **Density Separation**
   a. Transfer WPO solution to density separator (glass funnel with tubing and clamp)
   b. Rinse WPO beaker with distilled water to transfer remaining solids to separator
   c. Cover loosely with aluminum foil
   d. Allows solids to settle overnight
   e. Visually inspect settled solids for any microplastics; if present, drain settled solids from separator and remove microplastics using forceps; archive or discard.
   f. Drain settled solids from separator and discard
   g. Collect floating solids in a clean 0.3-mm sieve
   h. Rinse separator several times with distilled water to transfer all solids to 0.3-mm sieve
   i. Allow sieve to air dry while loosely covered with aluminum foil for 24 hours
6. **Microscope Examination**
   a. Transfer to glass vial for transport to HRPT
   b. Under dissecting scope at 40x, use forceps to collect identifiable microplastics from 0.3-mm sieve and transfer them to a tared vial
      i. Use **Hot needle test** to determine identity of unknown items
         1. Heat pin/dissection poker with lighter and press into material
         2. If it melts/warps, it is plastic.
      ii. Use counters to tally plastic pieces by type (see below)
7. **Gravimetric Analysis**
   a. Weigh mass of vial and microplastics to nearest 0.1 mg
   b. Subtract mass of tared vial to provide mass of microplastics collected on sieve (mass of all microplastics)
8. **Concentration of Microplastics**
   a. **Concentration in km^2 = (distance trawled x width of net)/# plastics**

Types of plastics (NY/NJ Baykeeper Plastics Report February 2016)

Fragment – unidentified hard piece of plastic
Foam – also known as polystyrene or “Styrofoam” used to make single use coffee cups, eatery to-go boxes, and packaging peanuts

Nurdle – Hard, rigid, fixed shape
Line – fishing line and clothing fibers
Pellet – plastic spheres such as microbeads from personal care products or preproduction balls of

plastic known as nurdles
Film – flimsy, thin plastic likely from plastic bags and shipping plastic wrap

Materials and Equipment

Collection

- Neuston net (330 µm mesh)
- Motorized vessel with GPS
- GPS/GIS device
- Castaway CTD
- PFDs (1/person)
- 4 Mason Jars
- 4 Collection Jars
- Basket/bag (carrying samples)
- 2 Buckets
- 2016 Microplastics Sampling Field Data Sheets
- 2016 Microplastics Methodologies
- Pens
- Sharpies
- Label Tape
- Squirt Bottle
- Extra Gallon of Fresh Water (tap is fine)

Analysis

- Stainless steel sieves, 8 in (diameter) and 2 in (depth)
    - 5.6 mm mesh
    - 1 mm mesh
    - 0.3 mm mesh
- Squirt bottle containing distilled water
- 500-mL glass beaker
- Analytical balance (precise to 0.1 mg)
- Metal spatula
- Drying oven (90°C)
- Iron (Fe(II)) solution (0.05 M)
    - Prepared by adding 7.5 g of $FeSO_4 \circ 7H_2O$ (=278.02g/mol) to 500 mL of water and 3 mL of concentrated sulfuric acid
- 30% Hydrogen peroxide
- Stir bar
- Hot plate

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- Watchglass
- NaCl (commercial)
- Metal forceps
- Density separators, constructed from
    - A glass funnel
    - segment of latex tubing on the bottom of the stem
    - pinch clamp to control liquid flow from funnel
- Retort stand
- O-ring
- Spring clamp (2-inch)
- Aluminum foil
- Custom PVC and .3mm nitrex mesh sieves
- 4-mL glass vials (1/sample)
- Dissecting microscope (at least 4X mag)

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NOAA Laboratory Methods for the Analysis of Microplastics Flow Chart p. 14