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**TITLE: Identification and Analysis of CI-Agent in Sea and Fresh Waters**

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**1.0 SCOPE AND APPLICATION:**

- 1.0 This method is used to both identify and determine the concentration of CI-Agent a (block co-polymer) in sea and fresh waters, using a gravimetric procedure. It should be noted that this method is not product specific as it analyzes for a broad class of plastic polymers which have a specific solvent solubility coefficient.
- 1.1 The method is applicable to sea and fresh waters containing plastic polymers and block co-polymers. A liter water sample is filtered to remove the polymers, air dried and washed with chloroform to remove oils and other soluble matter. The polymers are then dissolved in hot tetrachloethylene, which is filtered. Addition of cold methanol causes a precipitate which is isolated and weighed as polymers and block co-polymers.
- 1.2 This method can be used to identify block co-polymers if it is found in a neat form. The neat material will easily dissolved in tetrachloethylene and will form a white flocculant precipitate when added to methanol. The neat polymer will have a specific gravity less than 0.90 and be found floating on the surface of fresh and salt waters.

**2.0 APPARATUS:**

- 2.0 Filtering flasks, Vacuum, 2liter, 500 ml.
- 2.1 Funnels, Buchner, California modified, size 7.0 cm. ID obtained from Labconco, 8811 Prospect Road , Kansas City Missouri. Porcelian size #1, 56mm.ID.
- 2.2 Filter paper, Glass fiber-7.0 cm and 5.5cm.: Reeve Angel #934-AH or Whatman Type GFA Predried at 105C., Store in desiccator. Whatman #4-5.5cm., or equivalent.
- 2.3 Wash Bottles, Guth Universal, 500ml. hand operated with Kool-Grip neck or equivalent.
- 2.4 FilterAid, Diatomaceous earth of A.O.C.S. official.
- 2.5 Drying oven, 105 C.
- 2.6 Hot plate.
- 2.7 Analytical balance capable of measuring to 0.0001 grams.
- 2.8 Cold water bath, 10 C. Max

**3.0 REAGENTS :**

- 3.0 Chloroform, Reagent grade
- 3.1 Tetrachloroethylene, Reagent grade
- 3.2 Methanol, Reagent grade

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#### 4.0 PROCEDURE.

- 4.0.1 It is recommended that aqueous samples be collected in 1L amber glass bottles. Duplicate volumes should be provided for reanalysis or QC analysis.
- 4.0.2 Attach the California Buchner funnel to a 2liter filtering flask. Place 2 pieces of 7.0cm. glass fiber paper in the funnel. Pre-wet filter with distilled water and add 2grams of the filter aid, again wash the filter and filter aid with distilled water. Apply gentle suction and pour the pre-measured water sample through the filter funnel, taking care to keep it from going dry.
- 4.0.3 Wash the bottle with 3 times using 30 mls. of methanol. Air dry for 2 min and wash the filter with 3 times with 30 mls of chloroform, to remove any fats, oil and grease. discard the filtrate.
- 4.0.4 Bring 75 ml. of tetrachloroethylene to a boil on a hot plate in a 400 ml beaker. Remove with gloves from the hot plate and transfer the filter paper from step 4.3 to the beaker and stir for (2) minutes, with a glass rod.
- 4.0.5 Attach the porcelain Buchner funnel, to a clean 500 ml. filtering flask. Place a disk of 5.5 cm. Whatman #4 filter paper, two discs of 5.5cm. glass fiber paper in the funnel in that order.
- 4.0.6 Replace the 400 ml beaker from step 4.4 onto the hot plate and bring to a boil.
- 4.0.7 Pre-wet filter paper with tetrachloroethylene prior to filtration and apply gentle suction to the flask and then rapidly pour the hot tetrachloroethylene solution thru the filter. Using a wash bottle, rinse the beaker, and paper four times with 10-15 ml. portions of near boiling tetrachloroethylene. Disconnect the funnel from the filtering flask.
- 4.0.8 To the contents of the filtering flask add 175 ml. of precooled methanol 10C. Swirl to disperse thoroughly and let stand for 10 minutes in the cold water bath to completely precipitate the co-polymers.
- 4.0.9 Weigh accurately 2 pieces of 7.0 cm. moisture free glass paper and place the in the California Buchner funnel. Place the funnel on a 2liter filtering flask and with gentle suction filter the solution from step 4.8. Using a wash bottle, wash the flask, and paper four times with 20 ml portions of methanol.
- 4.0.10 Pull air through the fiber glass paper for 2 minutes. Carefully remove the paper from the funnel and dry in an oven at 10C. to constant weight. Cool in a desiccator and reweigh the paper and contents.

#### 5 CALCULATIONS:

- 5.1 PPM OF CO-POLYMER ANALYZED= Wt. of precipitate in mg/ml of water filtered x 1000=ppm. With a probable accuracy of 2.0 ppm.

#### 6 PRIMARY MATERIALS USED

- 6.0.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. *NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.*

<b>Material (1)</b>	<b>Hazards</b>	<b>Exposure Limit (2)</b>	<b>Signs and symptoms of exposure</b>
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Chloroform	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
TETRACHLOROETHYLENE	Carcinogen Irritant	22 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
<b>1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.</b>			