6.1 Reagents

BioTector TOC analyzer uses the following reagents:

I. **Acid:** 1.8 N Sulfuric Acid (H_2SO_4) Reagent containing 80 mg/l Manganese Sulfate Monohydrate
II. **Base:** 1.2 N Sodium Hydroxide (NaOH) Reagent

Reagents should not contain high levels of organics, nitrates and phosphates. Ideally, the level of organics, nitrates and phosphates should be less than 100 µg/l (ppb) in the deionized water used to prepare TOC analyzer reagents.

Acid and Base reagents are stable up to 1 year. The reagents must be stored in a safe and secure location, where the temperature does not drop below 2°C, in compliance with the local regulations. Table 6 below summarizes the total days each BioTector TOC Analyzer reagent lasts at various system configurations:

Table 6 BioTector TOC Analyzer Reagent Consumption

REAGENTS	Container Size (Liters)	TOTAL DAYS REAGENT LASTS
Acid	19	122
Base	19	122

Above table is derived from several online operation parameters such as 100% online time.

Recommended bunds (reagent spill trays) to contain above quantity reagents are 1x 50 Liters.

6.1.1 Sulfuric Acid Reagent

There are a number of factors which affect the purity of the sulfuric acid solution used by the BioTector. These can take the form of inorganic or organic contamination. Inorganic contamination does not generally occur in concentrated acids, but there could be some dissolved CO_2 present in the deionised water, which is used to make up the acid solution. To remove any CO_2 dissolved in the deionised water used to make up the acid solution, sparge the water with a CO_2 free gas such as Nitrogen.

Organic contamination has two main sources: Organics, which are present in the concentrated sulfuric acid, or dissolved organics in the water used to make up the solution. The manufacturer will specify the level of organics in the concentrated sulfuric acid. For the BioTector, this should be as low as possible. If the deionised water generator is functioning correctly, then there should not be any organics in the deionised water. Ideally, the level of organics should be less than 100 µg/l (ppb).

The procedure for making up sulfuric acid solutions for the BioTector from concentrated acid is as follows:

Mixing Procedure for the Preparation of 1.8N Sulfuric Acid Solution containing 80mg/l Manganese Sulfate Monohydrate:

Concentrated sulfuric acid is dangerous. The preparation of sulfuric acid solutions should only be carried out by persons properly trained in the handling of chemicals.

NEVER POUR WATER INTO ACID, ALWAYS POUR ACID INTO WATER!

Use eye protection and gloves.

A 1.8 N solution of sulfuric acid consists of 88.27 g (48 ml) of 100% sulfuric acid per liter of solution. Typically, concentrated sulfuric acid has purities of 96-98%. The procedures for both 1 liter and 25 liters of acid reagents are described below.

The instructions below assume that the sulfuric acid used has a purity of 98%, which is the standard commercial purity of sulfuric acid.

1. To prepare an acid solution, always use deionised water, free from organic and inorganic carbon. The conductivity of the deionised water used should be less than 0.5 µS/cm.
2. Blow through the deionised water with a CO_2 free gas such as Nitrogen or Oxygen, to purge the water of any dissolved CO_2.
3. Fill 90% of the container with deionised water.
4. a) In order to prepare 1 liter of 1.8 Normal acid solution, add 90 grams (49 ml) of 98% purity sulfuric acid. Mix gently and add enough deionised water to make it exactly 1 liter.
   b) To prepare a 25 liters acid solution, add 2250 grams (1225 ml) of 98% purity sulfuric acid in stages. Adding the sulfuric acid in stages will prevent the solution from heating up. Gently mix and then add enough deionised water to make it exactly 25 liters.
5. Seal the container.
6. Gently shake the container to mix the acid with the water.

7. Add enough Manganese Sulphate Monohydrate catalyst ($MnSO_4.H_2O$), so that the acid reagent will contain 80 mg/l (mg per litre) of $MnSO_4.H_2O$. For instance, for a 25 litres acid solution, add 2 grams of $MnSO_4.H_2O$ and mix well.
8. Allow the container to stand for one hour, and gently shake again to ensure a good mix.

Control of Normality:

In order to check the Normality of the acid solution prepared, take a 12 ml sample from the prepared 1.8 N sulfuric acid solution. Add 2-3 drops phenolphthalein and just enough 1.2 N sodium hydroxide until the color of the mix becomes red. If the normality is correct, 18 ml of 1.2 N sodium hydroxide will neutralize 12 ml of 1.8 N sulfuric acid.

Calculations for Different Purities of Sulfuric Acid:

Table 8 below tabulates the required amounts of sulfuric acid with different purities.

Table 8 Amounts of concentrated sulfuric acid required to prepare 1 and 25 liters of 1.8 N acid solutions at various purities.

	1 Liter		25 Liters	
% Purity of Sulfuric Acid	**(ml)**	**(grams)**	**(ml)**	**(grams)**
100	48.00	88.27	1200	2207
99	48.48	89.16	1212	2229
98	48.98	90.07	1224	2252
97	49.48	91.00	1237	2275
96	50.00	91.95	1250	2299

6.1.2 Sodium Hydroxide Reagent

There are a number of factors which affect the purity of the sodium hydroxide solution used by the BioTector. These can take the form of inorganic or organic contamination. Inorganic contamination has two main sources: Sodium Carbonate (Na_2CO_3), which is present in the concentrated sodium hydroxide, or dissolved CO_2, which can be in the water used to make up the solution. The level of Na_2CO_3 in the concentrated sodium hydroxide will be specified by the manufacturer. For the BioTector, this should be 0.1% or lower. If possible the concentrated sodium hydroxide solution should be obtained from approved manufacturers (e.g. Sigma Aldrich and Fisher Scientific). Carbonates appears to be the main cause of contamination found in sodium hydroxide solutions, and generally the level of Na_2CO_3 is found to be lower in concentrated liquid sodium hydroxide than in sodium hydroxide pellets. Therefore, the use of sodium hydroxide pellets should be avoided when preparing the BioTector sodium hydroxide reagent.

To remove any CO_2 dissolved in the deionised water used to make up the sodium hydroxide solution, sparge the water with a CO_2 free gas, for example nitrogen. Ideally, the level of organics should be less than 100µg/l (ppb).

Mixing Procedure for the preparation of 1.2N Sodium Hydroxide solution, from 50% concentrated Sodium Hydroxide solution:

Concentrated sodium hydroxide is dangerous. The preparation of sodium hydroxide solutions should only be carried out by persons properly trained in the handling of chemicals.

Use eye protection and gloves.

A 1.2N solution of sodium hydroxide consists of 48g of sodium hydroxide per liter of solution. Therefore, when using 50% concentrated sodium hydroxide, 96g of concentrate is required per liter. The instructions below are for 25 liters of solution.

It is important to purge the deionised water (used to make sodium hydroxide base reagent) with a CO_2 free gas prior to the mixing procedure.
As it is not possible to purge the carbonates (CO_3^-) from the base reagent by means of any CO_2 free gas, prepared base reagent should never be purged further with any gas after preparation.
When mixing the sodium hydroxide solution, care should be taken to minimize the amount of atmospheric CO_2 gas being absorbed in the solution. The base container should be fitted with a CO_2 filter to prevent the reagent coming in contact with air. Failure to do so will result in increased background CO_2 readings.

1. Use deionised water, free from organic and inorganic carbon. The conductivity of the deionised water used should be less than 0.5 µS/cm.
2. Fill 90% of the container with deionised water.
3. Blow through the deionised water with a CO_2 free gas such as Nitrogen or Oxygen, to purge the water of any dissolved CO_2.

4. a) In order to prepare 1 liter of 1.2 Normal base solution, add 96 grams of 50% purity sodium hydroxide. Mix gently and add enough deionised water to make it exactly 1 liter.
   b) To prepare a 25 liters base solution, add 2400 grams of 50% purity sodium hydroxide in stages. Adding the sodium hydroxide in stages will prevent the solution from heating up. Gently mix and then add enough deionised water to make it exactly 25 liters.
5. Seal the container.
6. Gently shake the container to mix the base with the water.
7. Allow the container to stand for one hour, and gently shake again to ensure a good mix.

If the base reagent is not mixed fully, then a highly concentrated layer will form in the bottom of the container. As the BioTector takes the base from the bottom of the container, this may create problems during operation, as the base will be too strong relative to the acid. Always mix the base solution for a second time, about 1 hour after it has been manufactured to ensure that it is completely mixed.

Control of Normality

Use a 10 ml sample from the sodium hydroxide container. Add 2-3 drops phenolphthalein and just enough 1.0N hydrochloric acid until it turns clear. If the normality is correct, 12ml of 1.0N hydrochloric acid will neutralize 10ml of 1.2N sodium hydroxide.

6.1.2.1 Sulfuric Acid Strength Testing Procedure

The procedure below describes a simple technique for checking the relative strength of the acid reagent.

Use eye protection and gloves.

In order to check the absolute strength of the acid reagent, a base standard of known strength (1.2N) will be required. The basis for the test is as follows: As the acid being tested is 1.8N, and the base is 1.2N, then 12ml of 1.8N acid mixed with 18ml of 1.2N base should give a pH of 7.

1. In a container, place 12ml of 1.8N acid. Check the pH. It should be 0pH.
2. Slowly add 16ml of 1.2N base. Check the pH. It should be ~1pH.
3. Add 1ml of 1.2N base, (total 17ml of base). Check the pH. It should be ~2pH.
4. Add 1ml of 1.2N base, (total 18ml of base). Check the pH. It should be ~7pH.
5. Add 1ml of 1.2N base, (total 19ml of base). Check the pH. It should be ~12pH.
6. Add 1ml of 1.2N base, (total 20ml of base). Check the pH. It should be ~13pH.

6.1.2.2 Sodium Hydroxide Strength Testing Procedure

The procedure below describes a simple technique for checking the relative strength of the base reagent.

Use eye protection and gloves.

In order to check the absolute strength of the base reagent, an acid standard of known strength (1.8N) will be required. The basis for the test is as follows: As the acid is 1.8N, and the base being tested is 1.2N, then 12ml of 1.8N acid mixed with 18ml of 1.2N base should give a pH of 7.

1. In a container, place 18ml of 1.2N base. Check the pH. It should be 14pH.
2. Slowly add 10ml of 1.8N acid. Check the pH. It should be ~13pH.
3. Add 1ml of 1.8N acid, (total 11ml of acid). Check the pH. It should be ~12pH.
4. Add 1ml of 1.8N acid, (total 12ml of acid). Check the pH. It should be ~7pH.
5. Add 1ml of 1.8N acid, (total 13ml of acid). Check the pH. It should be ~2pH.
6. Add 1ml of 1.8N acid, (total 14ml of acid). Check the pH. It should be ~1pH.