6.1 Reagents

BioTector TOC analyzer uses the following reagents:

I. **Acid:** 6.0 N Sulfuric Acid (H_2SO_4) Reagent containing 350 mg/l Manganese Sulfate Monohydrate
II. **Base:** 4.0 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	239
Base	19	239

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 deionized water, which is used to make up the acid solution. To remove any CO_2 dissolved in the deionized 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 deionized water generator is functioning correctly, then there should not be any organics in the deionized 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 6.0 N Sulfuric Acid Solution containing 350 mg/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.

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. When sulphuric acid at different purities are used, please refer to table 7 below.

1. To prepare an acid solution, always use deionized water, free from organic and inorganic carbon. The conductivity of the deionized water used should be less than 0.5 µS/cm.
2. Blow through the deionized water with a CO_2 free gas such as Nitrogen or Oxygen, to purge the water of any dissolved CO_2.
3. Fill 75% of the container with deionized water.
4. a) In order to prepare 1 liter of 6.0 Normal acid solution, add 300.2 grams of 98% purity sulfuric acid. Mix gently and add enough deionized water to make it exactly 1 liter.
   b) To prepare a 25 liters acid solution, add 7505 grams 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 deionized 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 350 mg/l (mg per liter) of $MnSO_4.H_2O$. For instance, for a 25 liters acid solution, add 8.75 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 6.0 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, 60 ml of 1.2 N sodium hydroxide will neutralize 12 ml of 6.0 N sulfuric acid.

Calculations for Different Purities of Sulfuric Acid:

Table 7 below tabulates the required amounts of sulfuric acid at different purities.

Table 7 Amounts of concentrated sulfuric acid required to prepare 25 liters of 6.0 N acid solutions at various purities.

% Purity of Sulfuric Acid	Sulphuric Acid quantity per 25 Liter container (grams)
100	7356
99	7430
98	7505
97	7583
96	7662

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 0.04% (or lower if possible). 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.

To remove any CO_2 dissolved in the deionized 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 100 μg/l (ppb).

Mixing Procedure for the preparation of 4.0N 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.

The instructions below are for 25 liters of solution.

It is important to purge the deionized 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 deionized water, free from organic and inorganic carbon. The conductivity of the deionized water used should be less than 0.5 µS/cm.
2. Fill 60% of the container with deionized water.
3. Blow through the deionized 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 4.0 Normal base solution, add 320 grams of 50% purity sodium hydroxide. Mix gently and add enough deionized water to make it exactly 1 liter.
   b) To prepare a 25 liters base solution, add 8000 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 deionized 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

Take a 10 ml of sample from the 4.0 N sodium hydroxide container. Add 2-3 drops phenolphthalein and just enough 1.0 N hydrochloric acid until it turns clear. If the normality is correct, 40 ml of 1.0 N hydrochloric acid will neutralize 10 ml of 4.0 N sodium hydroxide.

Mixing Procedure for the preparation of 4.0 N Sodium Hydroxide solution, from 100% Sodium Hydroxide pellets:

1. Use deionized water, free from organic and inorganic carbon. The conductivity of the deionized water used should be less than 0.5 µS/cm.
2. Fill 80% of the container with deionized water.
3. Blow through the deionized 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 4.0 Normal base solution, add 160 grams of 100% purity sodium hydroxide pellets. Mix gently and add more deionized water to make it exactly 1 liter.
   b) To prepare a 25 liters base solution, add 4000 grams of 100% purity sodium hydroxide pellets in stages. Adding the sodium hydroxide pellets in stages will prevent the solution from heating up. Gently mix to dissolve the sodium hydroxide pellets and then add more deionized 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.