
May 2013

T002: BioTector Series 4 Trouble Shooting

BioTector Series 4 Troubleshooting: if the BioTector does not produce any TIC or TOC CO₂ peaks.

NOTE: The checks detailed in this test program are divided into three sections, TIC, Base Oxidation and TOC. It is important that the following checks are carried out in sequence. That is, even if the problem lies with the TOC phase, *always start with the TIC phase.*

NOTE: Please refer to the manuals supplied with the instrument for additional information.

Before checking the TIC, Base Oxidation or TOC phases, confirm that the settings in the BioTector match those in the configuration printout supplied with the BioTector.

It is also necessary to confirm that the zero adjust in the BioTector has been set correctly – see Maintenance / Zero Adjust. If in doubt, run a Zero Calibration / Check or Base Calibration / Check to confirm the setting.

Section 1: Checking the TIC phase of the reaction.

1. Run a standard with a known concentration of TIC and TOC through the BioTector.
2. Check that the sample pump is pumping correctly. For the SR25 pump, after synchronisation 16 pulses = 6.1ml in ~11 sec. For the WMM60 pump, after synchronization 16 pulses = 7.0ml in ~10 sec.
3. Check that the sample in valve is operating correctly, and that the correct quantity of sample is entering the reactor.
4. Check that the circulation pump is circulating the solution around the BioTector.
5. Check the pH of the solution during the TIC phase of the reaction. The pH should be around 1.

If the pH is incorrect check the following:

- That the acid is connected to the correct port on the BioTector.
- Check the pH of the acid in the container. It should be around 1.
- Check the strength of the acid. (See section CHECKING THE ACID AND BASE.)
- Check that the acid pump is pumping correctly (i.e., 20pulses = 4.2ml in ~13 sec).
- - Check that the sample in valve is operating correctly, and that the correct quantity of acid is entering the reactor.

NOTE: The presence of a TIC peak does not indicate that all the TIC has been purged from the sample. If the pH of the sample is >13, then it is possible that only a proportion of the TIC will have been purged. If in doubt, check the pH in the reactor during the TIC cycle. If it is <1, all the TIC has been purged.

6. Confirm that the flow on the MFC is correct.
7. Check that the CO₂ analyser is operating, with a source of CO₂ gas.
8. Check that the separator and connections linking the cooler to the reactor are not contaminated with base. If the cooler was contaminated with base, then the base will absorb the CO₂ generated by the reaction, and prevent it reaching the CO₂ detector. The separator can be cleaned by removing it from the BioTector and running warm water through it.

Section 2: Checking the base oxidation phase of the reaction.

9. Check that the circulation pump is circulating the solution around the BioTector.

WARNING: For safety, remove the ozone start cable or the plug in ozone relay from the ozone PCB before the start of the base oxidation cycle, when carrying out the next test. This is to ensure that there will be no ozone in the reactor during the test.

10. Check the pH of the solution during the Base oxidation phase of the reaction. The pH should be around 13.

If the pH is incorrect check the following:

- That the base is connected to the correct port on the BioTector.
- Check the pH of the base in the container. It should be around 13.
- Check the strength of the base. (See section *CHECKING THE ACID AND BASE.*)
- Check that the base pump is pumping correctly (i.e., 20pulses = 4.2ml in ~13 sec), and that the correct quantity of base is entering the reactor.

NOTE: When the pH has been checked, and all tube connections tight, the ozone start cable or the plug in ozone relay can be reconnected to the ozone PCB.

11. Confirm that the flow on the MFC is correct.
12. Confirm the ozone generator is working. Measure the input current. This should be around 0.9A. DO NOT adjust the current unless running for at least 10 reactions has warmed up the ozone generator. Any adjustments made when the generator is cold may result in a high input current when the generator warms up. The normal warm running current should not exceed 1.1 amps. If the current is not correct, it can be adjusted using P1 on the OZB PCB. The high voltage transformer also makes a slight hum when running, and this can be heard if the generator is tested with the circulation pump and panel fan switched off. *Do not test the ozone generator with the cover off, as very high voltages are present.*

Resistances of the HV coil are as follows:

- Low voltage, + to -, 2.5 to 3 ohms
- Low voltage (+ or -) to high voltage terminal, 7k to 14k ohms

When the ozone board is not switched on, only the green power LED (LD8) should be lighting. When the ozone board is switched on, 3 green LED's should be on - LD8, LD1 and LD5. Note: the LED's are the same for both the OZB.01.97 & OZB2000.1. If the red LED, LD2 is on, the lockout circuit has activated. If this cannot be reset with the reset switch, contact BioTector Analytical Systems. Always confirm the operation of the ozone board when the wires have been re-connected.

Section 3: Checking the TOC phase of the reaction.

WARNING: For safety, remove the ozone start cable or the plug in ozone relay from the ozone PCB before the start of the TOC cycle, when carrying out the next test. This is to ensure that there will be no ozone in the reactor during the test.

13. Check that the circulation pump is circulating the solution around the BioTector.
14. Check the pH of the solution during the TOC phase of the reaction. The pH should be around 1.

If the pH is incorrect, check the following (Note: It is assumed that all the necessary TIC phase checks have been made.):

- *If the TOC acid valve is not fitted*, check that the sample in valve is operating correctly, and that the correct quantity of acid is entering the reactor.
- *If the TOC acid valve is fitted*, check that this valve is operating correctly, and that the correct quantity of acid is entering the reactor through the ozone port.

NOTE: When the pH has been checked, and all tube connections tight, the ozone start cable or the plug in ozone relay can be reconnected to the ozone PCB.

15. Confirm that the flow on the MFC is correct.

If the BioTector fails to operate when it is put back on line, it is possible that the pH of the sample is too far outside the limits for the BioTector. Generally, the minimum pH of the sample is 2, and the maximum is 12.

Low readings, additional checks which can be carried out.

Checking the circulation pump.

If the heavy-duty pump is installed, and the piston is worn, then some of the sample being analysed in the reactor can leak out through the pump-head during the analysis. Confirm that the pump-head is ok by first disconnecting the electrical cable for the water pump, then measuring any liquid leaking out through the water out port during the reaction. If more than 1ml is collected per reaction, then the pump-head may have to be replaced. When the test is complete, re-connect the water pump cable.

Additionally, the circulation pump flow rate can be tested using the procedure described in information sheet T005.

Checking that the Manganese catalyst is in the acid.

If the manganese catalyst is not included in the acid, then full oxidation will not be achieved, and the TOC result will be reduced to about 66%. It is possible too check if the catalyst has been added to the acid, as the liquid in the reactor at the end of the TOC cycle will have a light pink colour. If this is difficult to see, then collect the sample out from the BioTector in a glass container, where the pink colour should be more obvious.

Checking the tightness of the ARS valve.

If the ARS valve is damaged, then some of the sample which was to be pumped into the reactor can be instead pumped around the bypass loop, resulting in low TIC and TOC readings.

Checking the location of the sample bypass tube.

Ideally, the sample bypass tube should end at the same height as the centre of the ARS valve, and drip freely into a pressure free drain at this height. This will ensure that no sample is drawn through the ARS valve as it rotates. This problem can occur if the sample bypass line is long and at a low elevation when compared to the BioTector, and if the ARS valve is slightly worn.

Confirm that the pinch valves used for taking an on-line sample or calibration / manual sample are switching correctly, opening fully on the open side and sealing on the closed side.

Confirm that there are no bubbles in the sample fluid. This is of particular importance on high ranges where the sample size is small.

Checking the air injection pump / valve.

If this pump does not operate correctly, the sample will not be injected into the reactor. This can result in both lower readings and also cross stream contamination. Cross stream contamination is especially noticeable on high ranges with small samples, as a large proportion of the sample can remain in the injection tube between the ARS valve and reactor if the injection pump does not operate correctly.

Pressure in the reactor due to a blocked ozone destructor can increase this effect.

Contamination in the reactor:

Inspect the reactor. Reactor can have an effect on the pH, for example, acid can be stored inside the possible sludge material, with the result that:

- At the start of the base oxidation phase, the pH is >13 (which is correct).
- As the base oxidation phase progresses, the acid trapped in the sludge slowly reacts with the base.
- The result can be that at the end of the base oxidation phase, the pH could be acidic (<3pH).

In this case, the reactor / glass bead compartment may need to be cleaned using DI Water (or tap water), and if necessary, the glass beads replaced.

Checking the CO2 analyzer.

Assuming that the TIC check on the second page of this guide has not given the desired result, the CO2 analyzer should be checked at zero (with and CO2 free gas), and span (about 75% of full scale) with an appropriate CO2 calibration gas.

Additionally, confirm that the CO2 analyzer is set to its correct range.

Checking the MFC.

Confirm that the MFC is ok, for example with a roto-meter calibrated at 0-100LPH.

Confirm that the ozone level is ok, using the procedure described in T006.

Confirm that the oxygen quality and concentration is ok.

If the oxygen concentration is only 21% (for example in air), your oxidation and recovery will be reduced substantially.

Checking BioTector software settings:

Is the sample being analyzed on the correct calibration range? Typically, the reading should be <75% of full scale of the selected calibration range.

Is the zero calibration ok, and carried out using the optimum calibration range (i.e. the calibration range most frequently used, or representative of the most important readings)?

Is the span calibration ok, and carried out using the optimum calibration range?

Are the COD or BOD factors set correctly (normally set to 1.00 if a COD or BOD factor is not used)?

High readings, additional checks which can be carried out.

Pressure in the CO₂ analyzer.

If there is a partial blockage in for example the ozone destructor, then the pressure in the CO₂ analyser will be increased. An increase in pressure in the CO₂ analyser will give a corresponding increase in the TOC reading. To confirm that there is no blockage in the BioTector, a pressure meter can be used to measure the pressure in the BioTector with a flow of 80 LPH on the MFC. This pressure should be below 15 mbar at 80 LPH.

Typical pressures would be 11mbar at 80 LPH, or 1.5mbar at 20 LPH.

Start up range.

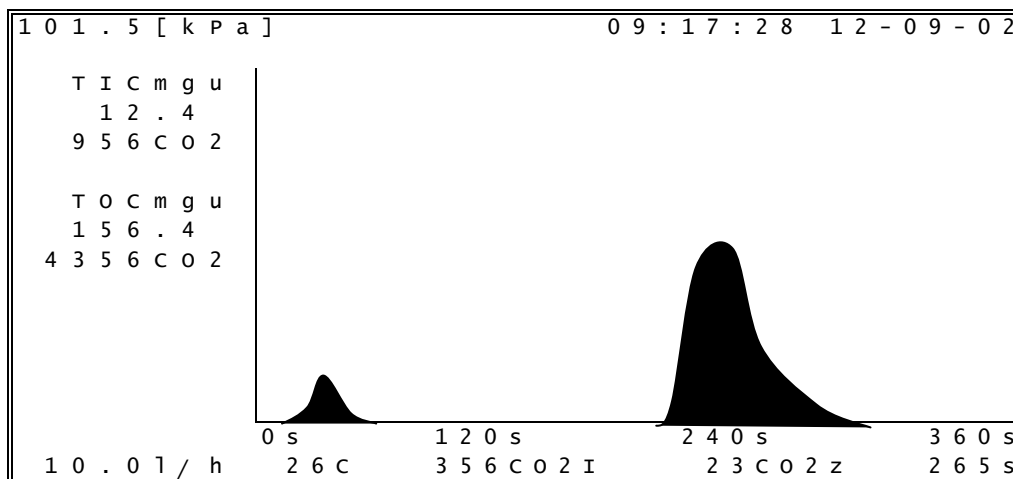
If the standard or on-line sample significantly exceeds the start up range, then the sample will not be completely oxidized. Therefore, although the range will change up (unless the range is locked), the TOC will most likely be higher than the result expected from the standard even on the next reaction due to carryover from the un-oxidized sample left in the reactor from the previous reaction.

Zero incorrectly set.

If the zero has not been set, and additionally if there is a high level of CO₂ in the base, then the TOC could give high results. Solution, if necessary replace the base if the level of CO₂ in the base is high and run the zero calibration.

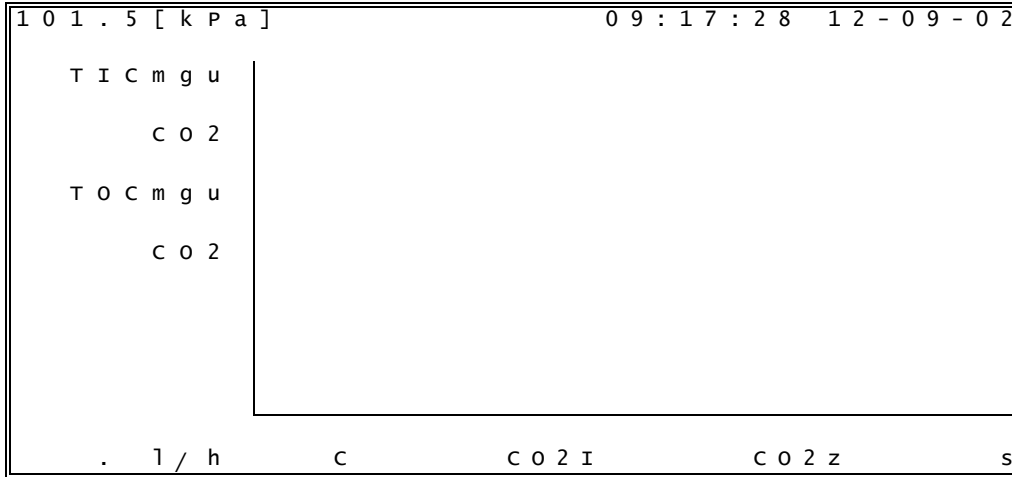
REACTION GRAPH SCREEN

The following shows a typical BioTector reaction graph screen.



After having carried out all the tests detailed on the previous pages, should the BioTector fail to perform correctly, please draw a sketch of the reaction graph that it displays on the blank reaction graph screens provided in the following pages. Fill in as much information as possible in the blank spaces, give the location /serial number of the BioTector and fax back to BioTector Analytical Systems Limited.

Reaction Graph screen, typical:



BioTector S/N _____

Located at _____

Date _____

Comments: