

Keywords

Aurora 1030W
Parallel Chamber
Single Chamber
TOC
Total Organic Carbon

**Parallel Chamber Configuration for
High Throughput Analysis Using the
Aurora 1030W TOC Analyzer**

Abstract

Increased sampling requirements continue to overburden sample capabilities of many analyzers. The demand for additional analysis capability has been traditionally resolved through addition of duplicate systems including support hardware, i.e., autosamplers and utilities, requiring substantial capital investment. An innovative approach to total organic carbon (TOC) analysis using the Aurora 1030W TOC Analyzer (Figure 1) with a second, parallel reaction chamber configuration, supported by an automated peak detection system, has increased sample throughput by 45% versus a serial method performed on a single reaction chamber analyzer.

Introduction

The ability to process multiple samples quickly continues to be a problem in the laboratory environment. Ever-increasing regulatory demands place higher sample processing requirements on the laboratory. Ensuring Good Laboratory Practices (GLP) through the use of check standards, before, during, and after a sequence is run, adds additional analyzer run time for regulatory compliance. These demands limit the ability of highly trained laboratory staff to operate equipment and interpret data. These issues are aggravated by current industry trends for analyzers to follow a serial process approach resulting in per-replicate analysis times exceeding 15 minutes.



Figure 1. Aurora 1030W TOC Analyzer

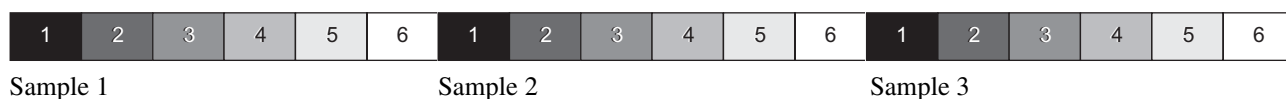
Improving laboratory productivity requires performing more analyses using less direct labor, while maintaining a high standard of data quality. Management has few options to resolve these issues other than adding hardware and personnel to meet demands for increased sample throughput. The cost associated with this approach can be too exorbitant to allow implementation.

Analyzer Issues

A review of current analyzer techniques demonstrates a key limitation to equipment productivity, the serial approach to sample processing. Most analyzers repeat some constant order in a linear fashion with little effort invested in optimization. Compounding this problem is the static approach used during the steps or states of the process. These dynamic states have fixed time requirements to achieve results and are configured for worse case situations. This static approach, while effective, is not usually efficient.

The detection state is a prime example. An analyzer usually has a detection time window set long enough for a large peak to return to baseline. But the analyzer cannot determine when that peak end will occur. As a result, time is lost while the detection state continues to completion. The data required to perform the needed calculations could have been collected in a fraction of the allotted time window.

Figure 2 illustrates the usual process involved in TOC analysis, where the typical system flow is discrete operation performed serially.



| TOC Analysis Steps |
|-------------------------|
| 1. Sample aliquoting |
| 2. Sample preprocessing |
| 3. TIC processing |
| 4. TOC processing |
| 5. Draining |
| 6. Cleaning |

Figure 2. Typical sample processing using a TOC analyzer with a single reaction chamber

Little can be done to expedite the order of steps in the analysis. However, a number of design features can be implemented in a basic system to exploit available efficiencies.

The first of these includes the design of the sample delivery system. A number of instruments use a single device, i.e., a syringe or peristaltic pump, to perform all sample transfer requirements. The use of a single sample delivery device precludes any preprocessing of the following state as the device waits to complete an event prior to acting.

The system outlined in Figure 3 addresses this design weakness. The syringe pump is configured to allow a number of parallel activities. Gas pressure is in place to use for sample transfer, draining, and other utilities. This concept allows the syringe to preprocess the next sample or replicate while the software performs the TOC processing state (step 4 in Figure 2). At the end of the sample sequence, the reactor drains under positive pressure and is ready to accept the next preprocessed sample. By optimizing sample handling, substantial processing time is eliminated.

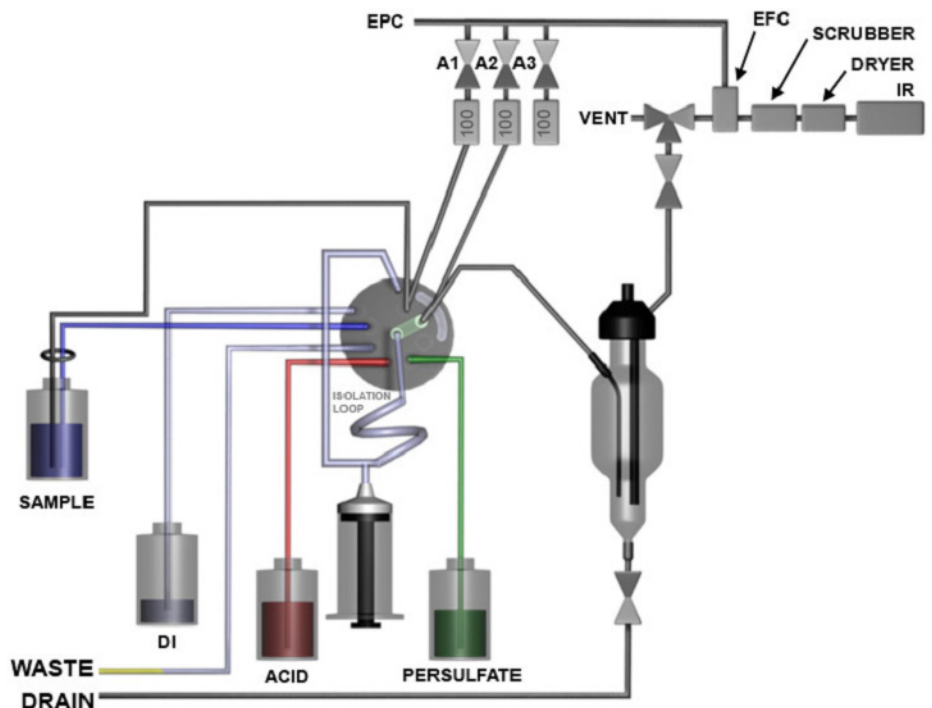


Figure 3. Syringe pump configuration allows parallel activities using a single chamber configuration

Another major design feature provides an advantage through a dynamic processing state. Unlike the static approach described earlier for data evaluation, implementing a dynamic algorithm provides automated peak picking and integration. In the example of the detection state, the time allowed for the state to complete is as great as 10 minutes. The integration algorithm, however, recognizes the sample is complete in less than two minutes, and the software allows the analysis to proceed.

In Figure 4, bars represent the start and end points of the peak, and area is calculated between these point. In this case, the detect time was set to 180 seconds. However, the end points were all detected around 70 seconds. At that time, the software closed the detect window, and the analyzer was allowed to start processing the next replicate.

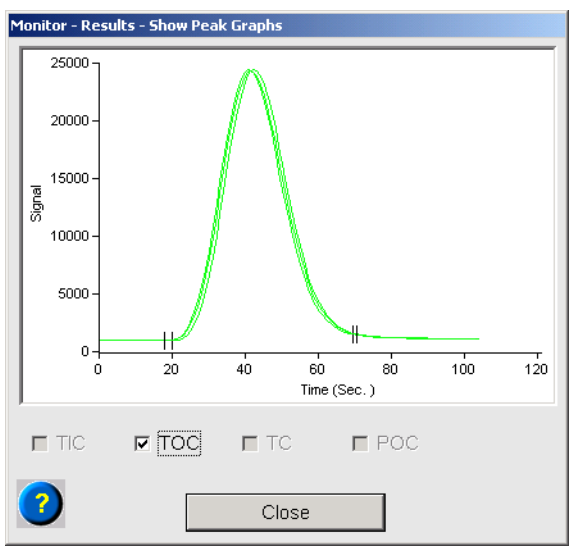


Figure 4. TOC peak detection using the Aurora

Implementing these basic concepts produces substantial improvements in instrument productivity. For more advances in sample throughput, the basic design can be expanded into a parallel configuration (Figure 5).

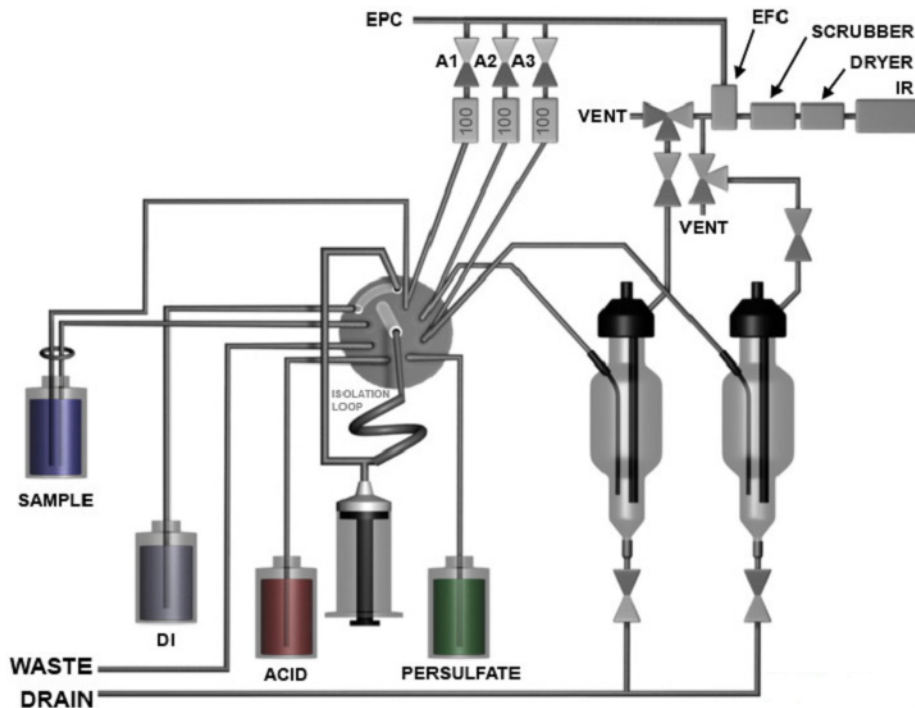
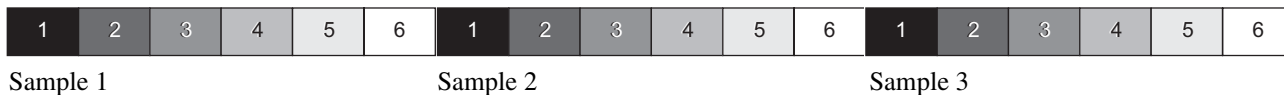


Figure 5. Parallel chamber configuration

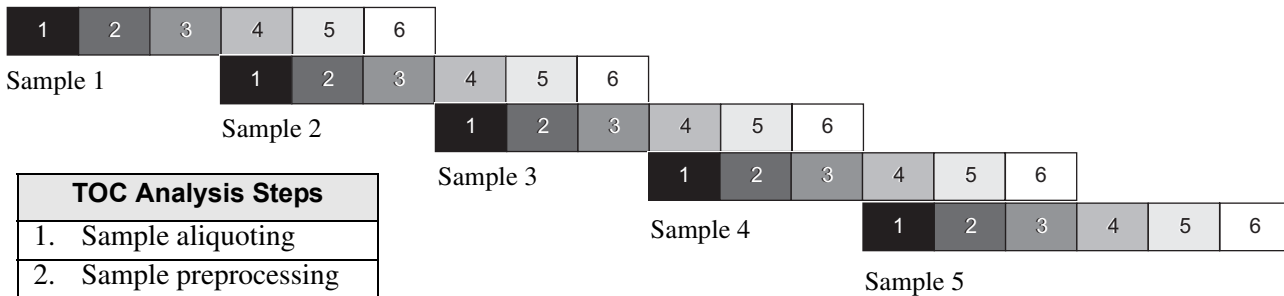
Using this configuration, system operation can be further optimized. In TIC/TOC analysis, the unit can process two samples, which are offset to allow the system to detect carbon dioxide from one reaction chamber while adding sample and reacting in the other chamber. Thus, the potential sample throughput of a parallel chamber configuration can be increased by 45% over a single chamber, depending on the method. The analyzer uses the same valve, select manifold, and drain manifold as the single chamber, providing redundancy that provides the additional benefit of continued operation should one chamber have a failure.

Comparing the basic flow shown in Figure 5 with the sample processing flow of a parallel system clearly illustrates the potential advantage of this system (Figure 6).

Single Chamber Configuration



Parallel Chamber Configuration



| TOC Analysis Steps |
|-------------------------|
| 1. Sample aliquoting |
| 2. Sample preprocessing |
| 3. TIC processing |
| 4. TOC processing |
| 5. Draining |
| 6. Cleaning |

Figure 6. Comparison of sample processing using a single chamber vs. a parallel chamber configuration

Experimental

To test the ability of a parallel chamber configuration to improve sample throughput, the Aurora 1030W TOC Analyzer (Figure 1) was equipped with parallel reaction chambers to allow concurrent sample processing.

Potassium biphthlate (KHP) standards were obtained from OI Analytical (PN 169252). Reagent water is low-level TOC water, also referred to as a 0-ppm C standard. Drinking water purchased from a local grocery store in a one-gallon bottle.

To prepare the 10-ppm C standard, remove 10 mL of the 1,000-ppm C KHP stock solution with a volumetric pipette. Place in a 1-L volumetric flask containing 500–750 mL of reagent water and swirl to mix. Add reagent water to a final volume of 1,000 mL and mix well.

To prepare the 5-ppm C standard, use 5 mL of 1,000-ppm C KHP stock solution and dilute as above.

To prepare the 1-ppm C standard, remove 2 mL of the 1,000-ppm C KHP stock solution with a volumetric pipette. Place in a 2-L volumetric flask containing 1,000–1,500 mL of reagent water and swirl to mix. Add reagent water to a final volume of 2,000 mL and mix well.

Results and Discussion

The Aurora was calibrated with reagent water and KHP standards (1, 5, and 10 ppm C) using the single chamber mode. The method was set for NPOC only with internal sparging using default parameters and a sample volume of 8 mL (Figure 7).

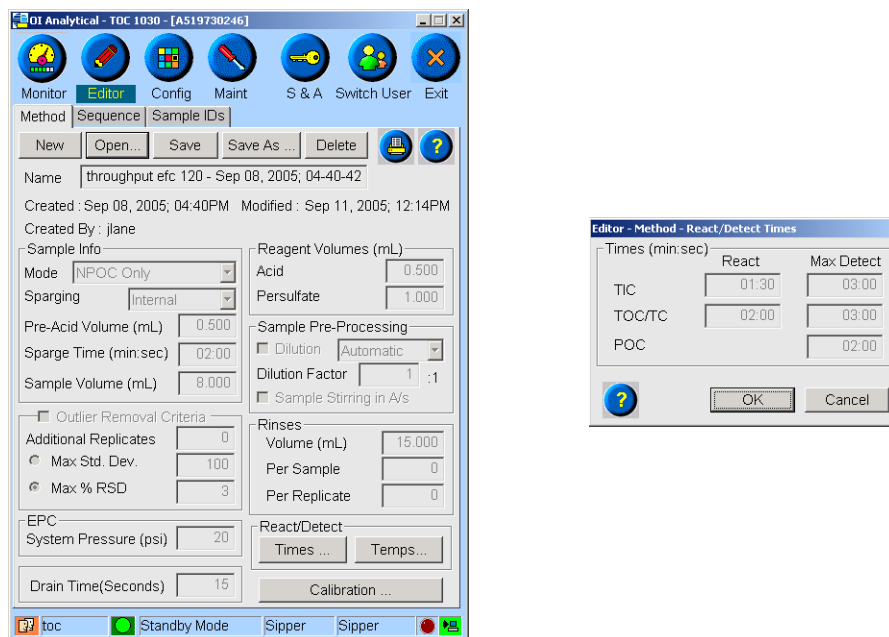


Figure 7. Method parameters used to calibrate the Aurora

Figure 8 shows the results of the calibration.

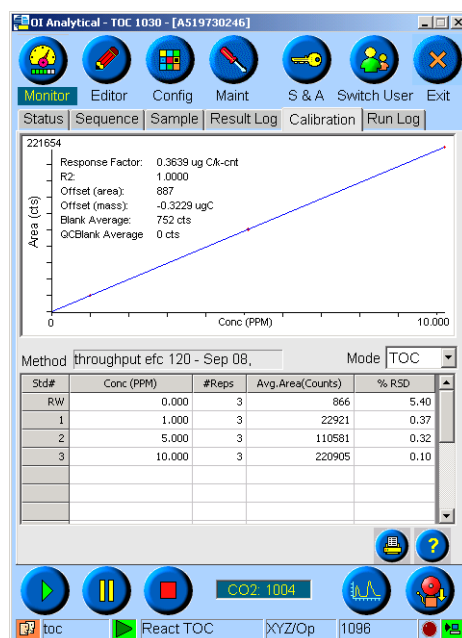


Figure 8. Calibration results

Check standards were then used to test the calibration. Four replicates of a 1-ppm check standard were run on the

Aurora using single and parallel chamber modes. Table 1 shows the results.

Table 1. Check standards

| Configuration | ppm | % RSD | % Accuracy |
|----------------------|------------|--------------|-------------------|
| Single chamber | 1.006 | 0.20 | 0.60 |
| Parallel chamber | 1.014 | 0.43 | 1.40 |

Fifteen samples of drinking water were run with four replicates for each sample. The Aurora was run in single chamber mode and then rerun using parallel chambers. Table 2 shows the results.

Table 2. Drinking water samples

| Sample | Single Chamber | | | Parallel Chamber | | |
|----------------|-----------------------|--------------|------------------|-------------------------|--------------|------------------|
| | ppm | % RSD | ppb error | ppm | % RSD | ppb error |
| 1 | 0.040 | 1.83 | 0.73 | 0.047 | 2.18 | 1.02 |
| 2 | 0.040 | 6.67 | 2.67 | 0.046 | 3.49 | 1.61 |
| 3 | 0.044 | 1.58 | 0.70 | 0.048 | 1.13 | 0.54 |
| 4 | 0.041 | 4.55 | 1.87 | 0.047 | 3.35 | 1.57 |
| 5 | 0.042 | 2.02 | 0.85 | 0.048 | 5.19 | 2.49 |
| 6 | 0.041 | 2.53 | 1.04 | 0.046 | 1.80 | 0.83 |
| 7 | 0.039 | 3.22 | 1.26 | 0.049 | 6.00 | 2.94 |
| 8 | 0.041 | 1.74 | 0.71 | 0.047 | 2.24 | 1.05 |
| 9 | 0.042 | 3.48 | 1.46 | 0.045 | 4.11 | 1.85 |
| 10 | 0.041 | 3.14 | 1.29 | 0.048 | 0.53 | 0.25 |
| 11 | 0.043 | 3.36 | 1.44 | 0.049 | 2.76 | 1.35 |
| 12 | 0.042 | 2.59 | 1.09 | 0.046 | 0.87 | 0.40 |
| 13 | 0.042 | 2.86 | 1.20 | 0.047 | 1.79 | 0.84 |
| 14 | 0.042 | 1.34 | 0.56 | 0.049 | 4.58 | 2.24 |
| 15 | 0.042 | 2.93 | 1.23 | 0.045 | 1.92 | 0.86 |
| Average | 0.041 | 2.92 | 1.21 | 0.047 | 2.80 | 1.32 |

The parallel chamber provides consistent results similar to the single chamber configuration with minimal loss of accuracy. Even though the % RSD exceeded 2% at these low TOC concentrations, the average ppb error was less than 2 ppb for both the single and parallel chamber configurations.

The sampling times for this data are presented in Table 3.

Table 3. Sample processing times

| Single Chamber | | Parallel Chamber | |
|---|-----------------------------------|---|-----------------------------------|
| Assay Start Time | Sample Time Duration (hr:min:sec) | Assay Start Time | Sample Time Duration (hr:min:sec) |
| 5:05:24 AM | — | 5:01:10 PM | — |
| 5:37:31 AM | 0:32:07 | 5:27:49 PM | 0:26:39 |
| 6:09:35 AM | 0:32:04 | 5:50:25 PM | 0:22:36 |
| 6:41:29 AM | 0:31:54 | 6:13:01 PM | 0:22:36 |
| 7:13:34 AM | 0:32:05 | 6:35:39 PM | 0:22:38 |
| 7:45:38 AM | 0:32:04 | 6:58:16 PM | 0:22:37 |
| 8:17:31 AM | 0:31:53 | 7:20:52 PM | 0:22:36 |
| 8:49:30 AM | 0:31:59 | 7:43:28 PM | 0:22:36 |
| 9:21:37 AM | 0:32:07 | 8:06:02 PM | 0:22:34 |
| 9:53:43 AM | 0:32:06 | 8:28:40 PM | 0:22:38 |
| 10:25:44 AM | 0:32:01 | 8:51:18 PM | 0:22:38 |
| 10:57:49 AM | 0:32:05 | 9:13:55 PM | 0:22:37 |
| 11:29:51 AM | 0:32:02 | 9:36:30 PM | 0:22:35 |
| 12:01:50 PM | 0:31:59 | 9:59:07 PM | 0:22:37 |
| 12:33:54 PM | 0:32:04 | 10:21:43 PM | 0:22:36 |
| 1:05:58 PM | 0:32:04 | 10:44:21 PM | 0:22:38 |
| Sample Average | 0:32:02 | Sample Average | 0:22:53 |
| Replicate Average (Sample Average ÷ 4) | 0:08:01 | Replicate Average (Sample Average ÷ 4) | 0:05:43 |
| Total Time | 8:00:34 | Total Time | 5:43:11 |

The results in Table 3 indicate an additional two hours and 17 minutes or 39% is required in the single chamber mode. Sample throughput times were reduced by nearly ten minutes and replicate time was improved by more than two minutes and 15 seconds. Overall time saved was over two hours and 15 minutes for fifteen samples. For a complete run of 88 samples, the time saved would exceed 12 hours. Sample throughput may actually be improved further if using the TC mode or NPOC with external sparge mode. The time saved is attributable to the analyzer's ability to preprocess the next available sample.

Conclusion

Implementing the innovative parallel chamber processing approach provides a number of benefits and options:

- Reduces manpower requirements, allowing reallocation of resources to other tasks, essentially adding six man-hours per day.
- Maximizes returns on capital investment in instrumentation.
- Provides backup capability of running a single chamber should a component failure occur.

