INTRODUCTION TO LASER NEPHELOMETRY: AN ALTERNATIVE TO CONVENTIONAL PARTICULATE ANALYSIS METHODS

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Be Right™

In memory of

Clifford C. Hach

(1919-1990)

inventor, mentor, leader and, foremost,
dedicated chemist

Table of Contents

SECTION 1 Introduction

Filtration management in the water treatment industry must be optimized to produce water with the quality and consistency necessary for human consumption. In addition, water production must be economical and affordable for the end user. This must be accomplished regardless of the changing condition of the raw water and any related treatment techniques that change with time. Outbreaks of water borne pathogenic diseases have increased the pressure on manufacturers to develop highly sensitive filter effluent measurement tools. The water treatment industry prefers that these tools be very high quality, robust, simple, and economical to use.

The Hach FilterTrak 660™ or FT660 sc Laser Nephelometer (FT660) is an ultra-sensitive nephelometer designed to monitor filter effluent and track any filter event that is detected by a particle counter or a traditional turbidimeter. This instrument combines the basic concepts of traditional nephelometry with advanced technology to produce an ultra-sensitive filter management tool. The instrument is designed specifically for effluent or better quality water and can be applied to the drinking water industry or other industries that require particle detection at exceptionally low levels.

This bluebook discusses current methodologies and instruments used to monitor filter effluent in water treatment plants. The benefits and limitations of each technology are discussed. This leads to a discussion of how the FT660 Laser Nephelometer can serve as either a complementary or replacement technology for monitoring filter performance and subsequent effluent water. Theories on how particles pass through a filter and how the FT660 technology was designed for early detection of filter events, including filter breakthrough will be discussed.

Data collected at local water treatment plants are analyzed with the emphasis on comparing the results obtained by this instrument to those obtained by traditional turbidimeters and particle counters. Again, the overall goal of this paper is to present and demonstrate a new technology that will serve as a simple, yet sensitive tool for optimizing filter management in the water industry.

The FilterTrak 660 meets the requirements of Hach Method 10133, which is EPA approved for the monitoring of Drinking water. Appendix A presents this approved method. Appendix B and C present case studies demonstrating practical applications of the FT660 technology.

1.1 Overview of Filtration

One of the most important goals of water treatment plants (WTP) is to efficiently, consistently, and economically produce high quality water for human consumption. The raw water must go through several stages of production including chemical dosing, mixing, coagulation, settling, and final filtration. These stages eventually remove the particulate material that could potentially affect water quality and safety.

A thorough knowledge of the ongoing performance of the filters in the WTP is an important element of water production. Since particulate matter is often invisible to the human eye, instruments must be used to determine if the particulate matter in the water is at a level that could cause problems. To achieve the most effective monitoring, many WTPs recognize the importance of utilizing all economically feasible tools to track short- and long-term performance. The most common tools include turbidimeters and light obscuration particle counters. The benefits and drawbacks of each of these instruments is presented in *SECTION 5* on page *11*.

SECTION 2 Overview of Turbidity

Turbidity is a qualitative measurement of the light scattering material present in water. All materials scatter light to some degree. Turbidimeters enable the WTP to identify and monitor the levels of materials at different points in the sample stream. Turbidity has been used for monitoring solutions of process control, monitoring final product quality, as a surrogate for health risk assessment, for regulatory control of water quality, and numerous other applications across a wide spectrum of industries.

The water industry has historically used turbidimetric measurement as a marker of consistency and quality of water effluent from water treatment plant filters. The measurements are most commonly made at the filter effluent prior to the sample entering the public distribution system. Comparing the filter effluent turbidity measurement with the turbidity measurement of raw or influent water provides an estimate of the overall efficiency of a water treatment plant.

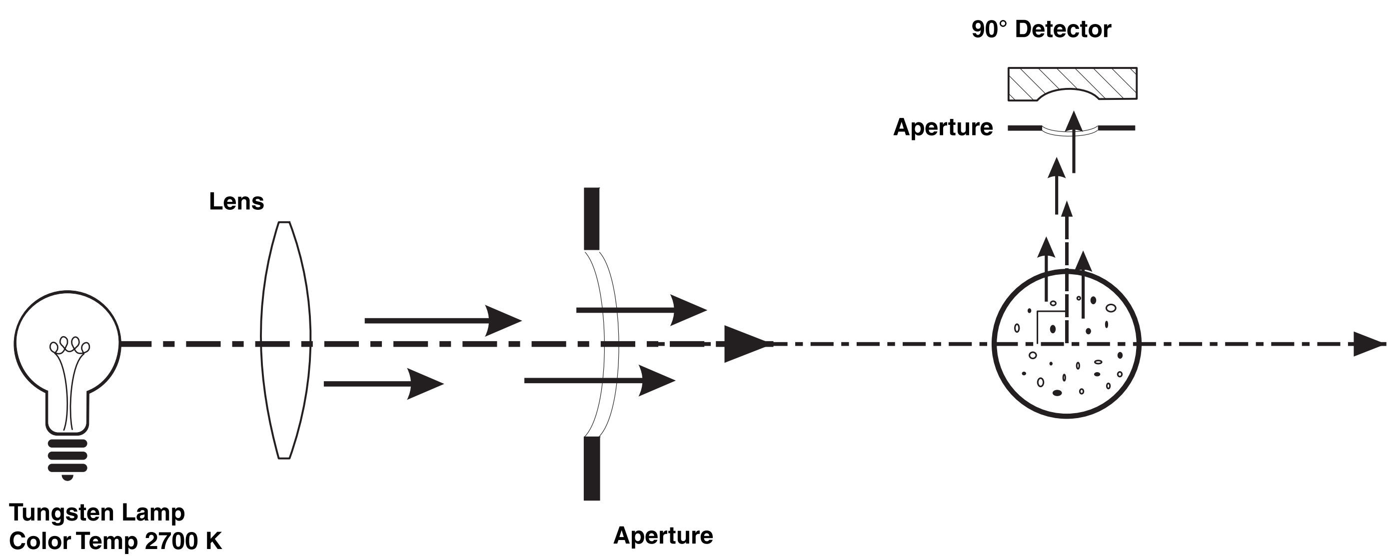
2.1 Turbidity Technology

Turbidity technology has been standardized for use by regulatory agencies as a benchmark for the general quality of water and its relationship to public health. Generally, the fewer particles present in water, the lower the risk that the remaining particulate matter is harmful to public health. With that knowledge, very low turbidity water is desired.

Standardized turbidity procedures and techniques have been developed and utilized for many years to provide a consistent framework of technology. This framework allows WTPs to track their current and historical performance. This standardization has resulted in restrictions within the science of turbidity. Despite regulatory constraints, manufacturers are being challenged to advance the technology.

Figure 1 shows the basic design of a nephelometer (a turbidimeter that only utilizes light scatter at 90 degrees).

Figure 1 Optical System of a Nephelometer



2.2 Turbidity and Regulatory Reporting

Until recently, the regulatory reporting requirements for turbidity have remained somewhat constant. However, the regulations have been recently revised to a more stringent level of 0.3 NTU and further reductions of the reporting levels below 0.2 NTU are being considered. For membrane systems, proposed limits will be set at < 0.15 NTU and a target of < 0.1 NTU.

Recent studies have shown that current turbidity technology is capable of accurately measuring down to 0.3 NTU, when all aspects of turbidity measurement are considered (proper instrument setup, calibration, sampling and analytical technique). As water quality becomes more important to our increasing populations, increased pressure to drop the regulatory upper limit to below 0.3 NTU is being felt. This push is leading many to question if the current technology is indeed capable of effectively measuring turbidity at lower levels. The current standardized technology does have some limitations which will be discussed in *SECTION 4* on page *9*.

SECTION 3 Particle Counting Technology and Application Overview

Particle counting involves counting and sizing of particles in a sample. Counting is the primary, discrete function and sizing is a secondary, less descriptive function. Particle counting has been utilized in many industries including the semiconductor, hydraulic, pharmaceutical, and most recently the drinking water industry.

3.1 Particle Counting Technology

Particle counting uses several types of technology, depending on the application. The three most common technologies are electrical zone sensors, light-scatter sensors, and light-obscuration sensors.

Light-obscuration sensors have been found to be the most applicable to the drinking water industry. These instruments typically measure particles 2 µm and larger and can easily monitor plant effluent water with no instrument modifications. Of the three particle counter technologies previously mentioned, light obscuration sensors are easier to use and more cost-effective than electrical zone and light scatter sensors.

Light obscuration involves the use of a light source of a fixed wavelength and constant intensity. The path of the beam crosses a sample chamber. When a particle passes through the beam, it scatters or absorbs some of the incident light, reducing the amount of light striking the detector (extinction). The bigger the particle, the more the incident light is removed and the greater the reduction in the light striking the detector. The amount of light extinction at the detector is converted to a pulse that correlates to a specific size particle used in the instrument calibration.

This technology is sensitive to counting particles greater than 2 µm in size. Size is a secondary determination of the particle counter and the “sized” particle in the sample is referenced to the standard used in calibration. Sizing is a qualitative function and is influenced by many variables. Several factors, such as the particle’s refractive index, absorption, shape, orientation, and position affect the reported particle size. Size profiles can sometimes be traced to specific types of treatment events and categorizing particles by size allows the WTP to determine if pathogen-size particles (approximately 2–20 µm) are passing through the filter. If no 2–20 µm particles are passing through the sensor, it is assumed that no pathogens of that size are passing through the filter.

Particle counter technology is best applied to the drinking water industry as a tool to monitor filter effluent. Specific applications of particle counters on filter effluent include:

- Characterizing filters
- Identifying particles of a size representing the size of pathogens
- Detecting breakthrough precursors on a filter
- Troubleshooting — Identifying the root cause of an unexpected event or trend in a process

3.2 Particle Counter Limitation

Particle counters do present some limitations when utilized in the drinking water industry. Particle counting was first used in the pharmaceutical and ultra-pure water industries where the sample streams are highly purified and controlled. Since particle counting instrumentation was primarily designed for these sample streams, few problems are seen in this application.

Although filter effluent is the most common treatment stage for particle counter use, they are sometimes used for other stages of water treatment, even on raw water. Care must be taken when using the particle counter on raw water because clogging and biological growth can cause major functional problems at the instrument level. This limits the applications of particle counters in this industry. Attempts to place particle counters upstream of the final filter effluent have proven to be problematic. In fact, several manufacturers discourage the use of particle counters on settling basins or raw water unless the operators are willing to support high maintenance costs.

The drinking water industry is very different from the pharmaceutical and ultra-pure water industry. These differences, outlined below, require additional maintenance and effort by the WTP operator.

- The drinking water industry must produce very large volumes of water. Traditional filtration processes produce a final product capable of clogging the instrument. Fortunately, many plants have their processes highly optimized and instrument clogging is a rare occurrence for effluent samples.
- Cost of ownership, maintenance, and the generation of large volumes of data limit the application of particle counters in the drinking water industry. Particle counters are initially more expensive to purchase than turbidimeters and often require additional accessories to run the instrumentation. Maintenance is another issue; particle counters are difficult instruments to calibrate on-site. If on-site calibration is performed, the cost is high. Calibration verification methods have been developed but often are seen as difficult and not highly accurate. The WTP operator may lack confidence that the results from a particle counter are indeed accurate.

Particle counters are designed to detect a narrow size range of particles. Most used in the drinking water industry are optimized to detect particles in the 2–100 µm range and are not capable of detecting particles below 2 µm. If the precursors of filter events are sub-micron, a particle counter will miss them. In addition, particle counting instruments cannot handle significant spikes in turbidity. A high spike of turbidity can result in coincidence, a condition in which the instrument can no longer detect particles separately.

In summary, when particle counters are properly used on filter effluent water, they can be very effective. Many water plants have adopted this technology and have optimized their treatment processes; claiming production results that could not be accomplished using only traditional turbidimetric techniques. To successfully use particle counters, operators must adhere to proper setup, calibration, and maintenance. Instrument verification is a relatively new concept in particle counting and few methods are available today. The methods available generally require more cost, training, and technique than the verification methods associated with turbidimeters.

SECTION 4 Turbidity Technology and Application Overview

The use of turbidity in the drinking water industry has many known benefits as well as some limitations. Turbidity is a simple qualitative measurement. Basically, the lower the turbidity of the sample, the higher its overall quality. This applies both to the aesthetic and health aspects of the water sample. Through the use of standards and common design criteria, the parameter has been regulated and successfully used as a semi-quantitative analysis parameter. Comparisons between treatment plants are possible along with comparison of current and historical data. Baseline information from one WTP can be referenced and used to determine how a water treatment plant performs compared to other plants over time.

4.1 Calibration

Turbidimeters are typically easy to calibrate. Since all calibration standards are traceable to primary formazin, a basis for consistency and a high level of accuracy throughout the water industry has been established. With the advent of new stable turbidity standards, the operator no longer needs analytical technique training to prepare turbidity standards. The calibration procedures themselves have also become simplified. This, coupled with the premixed standards, has improved the consistency and accuracy of turbidimeter calibration and measurement.

4.2 Calibration Verification

Simple and rapid performance verification is also an advantage when using turbidimeters. The recent development of both stable low-level turbidity standards and dry calibration-verification devices has paved the way for accurate and consistent instrument performance at any turbidity level.

4.3 Theory of Operation

Turbidity is not descriptive with respect to the number or size distribution of particles in a sample. Since it is specifically indicative of the presence of particles in a sample, it is therefore considered a qualitative measurement.

Most turbidimeters use a tungsten filament lamp as the light source. The tungsten filament emits a wide range of wavelengths. Particles with a size less than 2 µm can be detected by turbidimeters if the overall number of particles presents enough light scatter. As a result, turbidimeters are highly sensitive to small particles, even those particles smaller than 2 µm that are not typically “seen” by particle counters.

Overall, turbidimeters require minimal training to operate and understand the technology. The new generations of instruments have simplified user interface menus that make instrument operation easy. Turbidimeter maintenance is also fairly simple. If properly installed, maintenance consists of cleaning the instrument when necessary.

4.4 Turbidimeter Limitations

Turbidimeters have several drawbacks that are listed below:

- Tungsten light sources have a relatively short life compared to solid state laser diode sources and must be replaced at regular intervals. This is normally a simple process that the operator can perform.
- Turbidity measurement is truly qualitative in nature and has not been designed to provide descriptive information with respect to size or number of particles in a sample. As turbidity rises, a correlating increase in the particulate material in a sample occurs. As particulate matter in a sample increases, the possibility that some of the material could pose a health threat also increases.
- As discussed earlier, turbidimeters are widely used in the water industry, which has mandated the instrument design. The regulatory factor has limited the evolution of instrument performance. For example, the current resolution of today's instruments is only specified to the nearest 0.05 NTU by the regulatory agencies. To further push these limitations downward, regulatory restrictions on the design criteria of these instruments must be loosened to encourage the implementation of new technology.
- Turbidimeter instrument response to events, such as filter breakthrough, has historically been thought of as slow and suppressed. This is true in some cases but factors such as flow rate and instrument location can drastically influence response.

Turbidity measurement has been and will continue to be an influential monitoring and regulatory parameter in the water industry. The technology provides a simple and easily applied method of particulate detection that has been in use for decades. Turbidity remains the benchmark parameter regulating filter performance in water treatment plants worldwide. Other technologies, such as particle counting, are emerging. While they have not replaced turbidity, they have benefited the industry when used as a complementary system.

SECTION 5 Complementary Technologies: Turbidity and Particle Counting

When particle counting is used with turbidimeters as an additional analysis tool, positive results are produced. Sometimes, a particle counter will detect an event before a turbidimeter and sometimes the reverse is true. Particle counters used in the drinking water industry detect particles greater than 2 μm. Turbidimeters give information on particles below this size. If the event involves sub-micron particles, the particle counter may miss the event altogether. If the event consists of only a few large particles, the turbidimeter may miss the event. Events are often detected using both technologies, but not usually simultaneously. In many cases, a particle counter will detect an event before a turbidimeter. These examples do show the complimentary nature of turbidity and particle counting, but this often leaves the WTP analyst with additional questions. Which instrument is right? Is there really a problem? How should the treatment process be changed to fix the problem?

Even with complementary technologies, a means of consistently providing information regarding filtration events is sometimes missing. Additionally, using both technologies adds cost and complexity to the water treatment plant. The data is often overwhelming and requires additional resources to analyze. A single tool that could help simplify this complexity would be of great benefit to the end user.

SECTION 6 Developing a New Tool for Monitoring Filter Effluent

Filter effluent is typically monitored by turbidimeters, particle counters, or both instruments. To determine how to detect particulate events more consistently, an understanding of some of the characteristics of natural particle distributions is required.

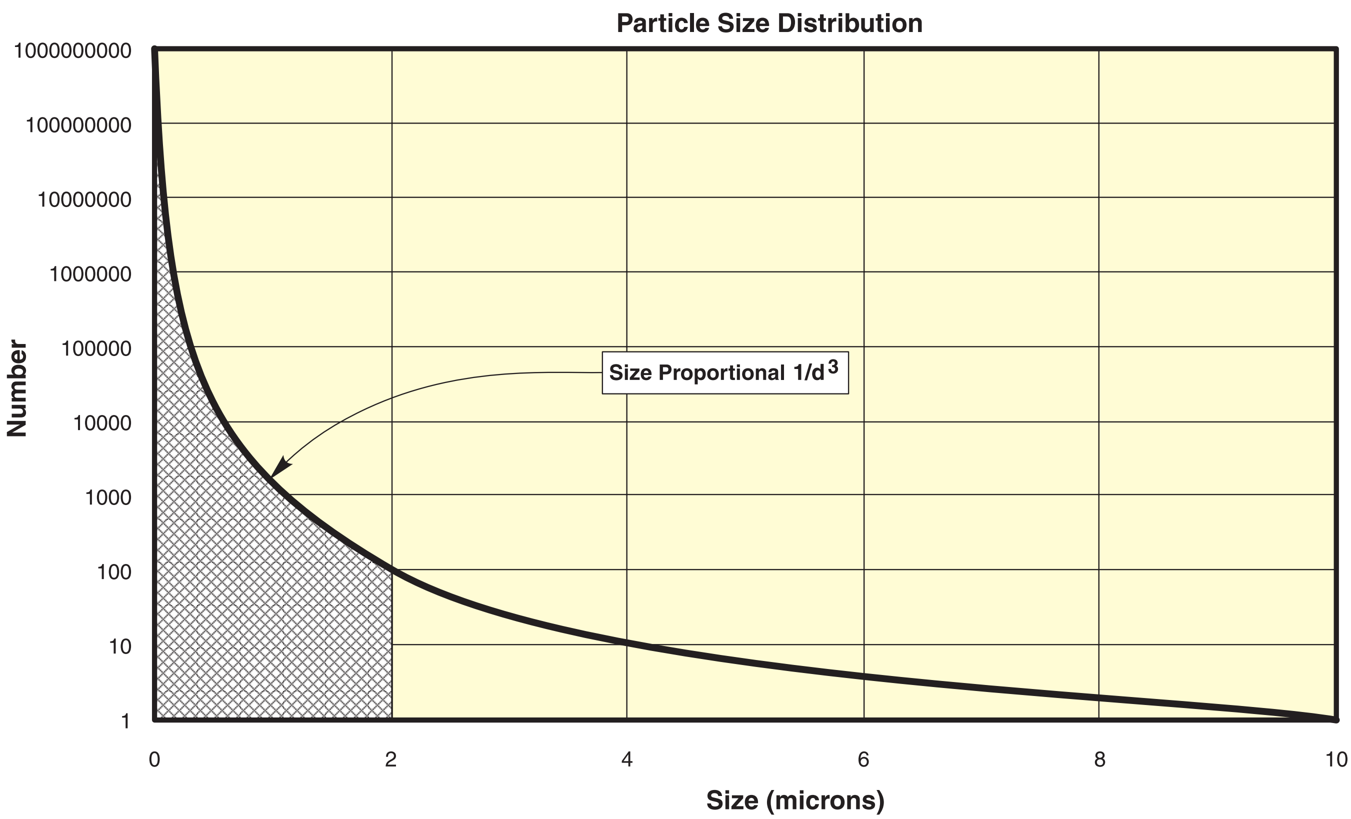
Natural particle distributions typically follow a typical power law. Generally in nature, distributions will follow a $1/d^3$ relationship when correlating size to number of particles in a sample. For example, if a sample contains one 10-µm particle per mL, that same mL of sample would contain one thousand 1-µm particles and one million 0.1 µm particles. The particle counter would only detect those particles greater than two microns in size. *Figure 2* shows a typical power law relationship. Note that all particles below 2 µm in size would be missed by the optical particle counters currently used in the drinking water industry.

When utilizing particle detection as a surrogate for pathogen detection, the most common pathogens such as cryptosporidium and giardia are of a size greater than 2 µm. However, several potentially harmful pathogens such as bacteria and viruses are below 2 µm. It is important to have information regarding the presence of sub-micron particles in the filter effluent.

Again, turbidimeters have specific limitations that have prevented the technology from becoming more sensitive:

- The light source lacks stability and has a very broad wavelength range. The wavelength range of a typical tungsten lamp is between 400 and 1600 nm. Simple light source filters reduce the amount of infrared radiation reaching the sample, but it is still significant in the measurement.

Figure 2 Typical Power Law Relationship of Particle Size Versus Number in a Natural Water Sample



- Available detection systems lack the high sensitivity to short wavelengths of light. The regulatory design criteria, in essence, states that the lamp and detection systems must be modified so the peak response of the signal is between 400 and 600 nm. This modification is an attempt to increase sensitivity to light scattered by smaller particles.

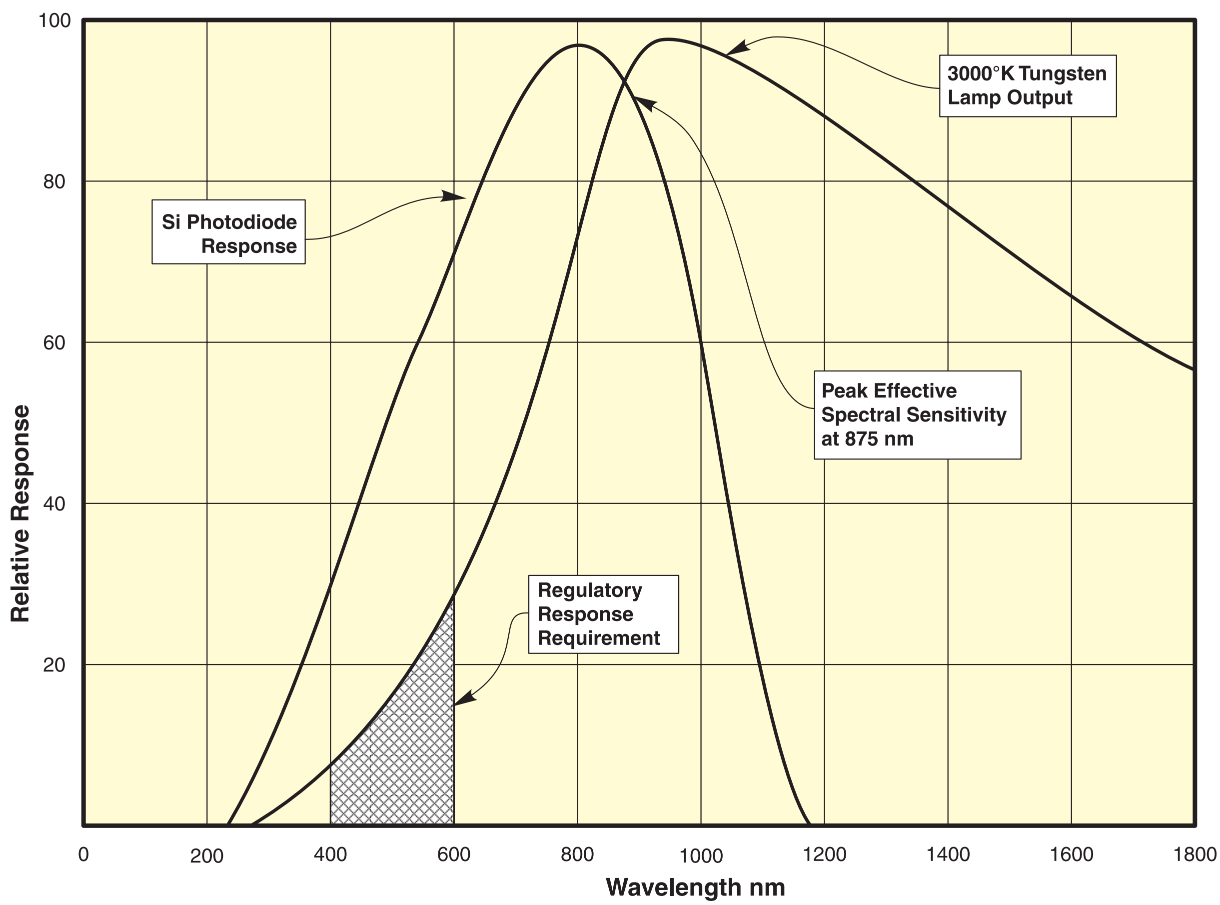
The disadvantage of forcing the detection system to produce a peak within the 400 to 600 nm range, is that the tungsten lamp and the detector are not used optimally. Since the combination of light source and detection system is not optimized, a dramatic impact on sensitivity levels of current turbidity instrumentation occurs.

Figure 3 shows the spectral output and detection of a typical turbidimeter that has been designed to meet regulatory requirements.

The key to improving turbidity sensitivity at low levels is to concentrate on the detection of small particles. Particle counter technology currently allows the detection of particles below one micron but such ultra sensitive particle counters are not utilized in the drinking water industry because of difficulties in application and high cost. Theoretically, there is no limit on particle size detection using a turbidimeter. The concern is that current technology has poor signal-to-noise ratios at low detection levels. Thus, small spikes may be lost in the noise of the instrument.

Figure 3 Effective Spectral Distribution for 3000 K Tungsten Source—Si Photodiode Detector System

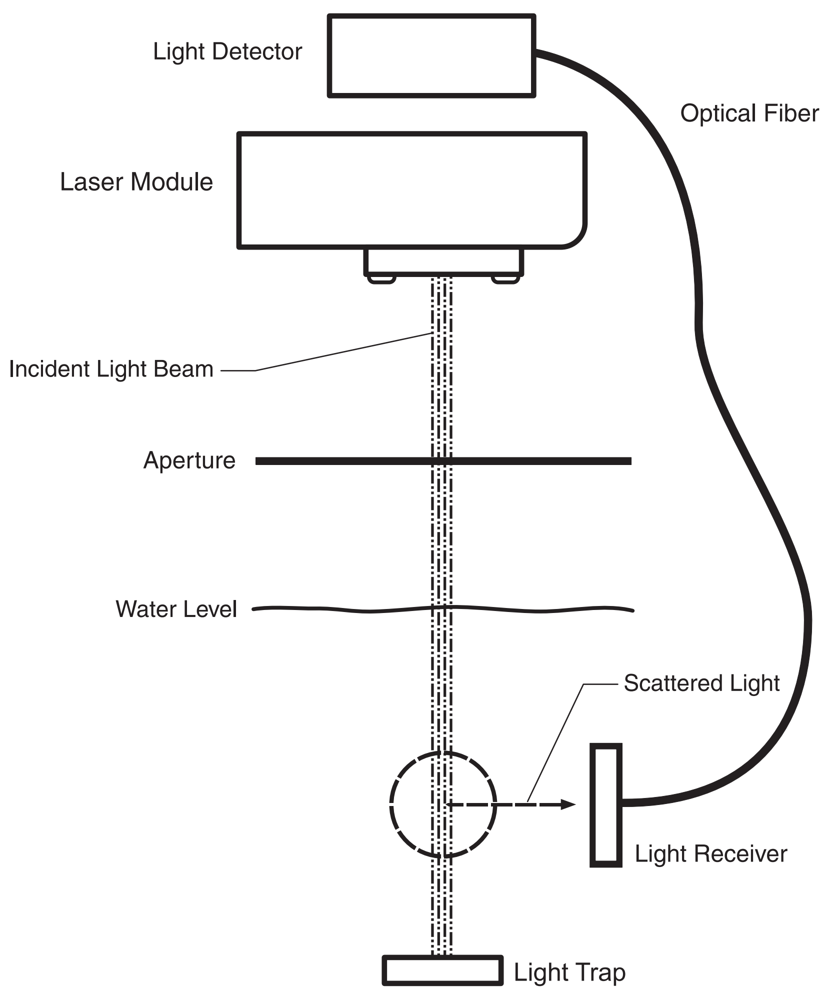
The regulatory requirement specifies a peak response between 400 and 600 nm. To accomplish this, light filters are used to remove most of the energy spectrum emitted from a tungsten lamp source.



Recently, Hach has lead the industry in the development of ultra-sensitive turbidimeters that effectively and consistently detect ultra-low changes in turbidity. The breakthrough instruments, the FilterTrak™ 660 and FT660 sc Laser Nephelometers, have combined a light source with a detection apparatus that utilizes each component at its optimum point of performance. The detection apparatus utilizes fiber-optic technology to carry the scattered light signal to the detector. This produces a very stable signal baseline at the lowest of turbidity levels.

The FilterTrak 660 sc Laser Nephelometer utilizes nephelometric detection of light scatter from particles in a sample. The optical design is shown in *Figure 4*. The geometry of the incident light source and the centerline into the detector receive fiber is fixed at 90 degrees, which is consistent with the detection angle associated with current nephelometry technology. The light source is a laser diode with a wavelength of 660-nm. The response system is optimized to detect this specific wavelength. In addition, the incident light is collimated into a narrow beam, resulting in a relatively high beam power density. This high power density results in higher scattered intensity from smaller particles. In addition, a light trap at the bottom of the nephelometer body absorbs the portion of the narrow beam that passes through the sample without being scattered. These features combined create a measurement system with dramatically lower stray light.
In general, the stray light exhibited by a laser nephelometer is less than 50% that of traditional instruments.

Figure 4 FilterTrak 660 Laser Nephelometer Optical Configuration



The FilterTrak 660 sc Nephelometer detection apparatus is coupled with fiber optic technology to convey the scattered light signal to the detector. The high power source and very sensitive detector combine to produce a strong signal even at the lowest of turbidity levels. The optimization of the optical components results in an increase in sensitivity of more than two orders of magnitude over the sensitivity that is achieved with the best turbidimeters in use today. This allows for a very stable baseline—also referred to as the limit of detection (LOD)—capable of detecting a change in turbidity down to 0.3 mNTU (1.000 NTU = 1000 mNTU). Since sensitivity is defined as the detection of a change in turbidity, the lowest numerical value that an instrument can read is not as significant as the smallest change that it can detect. *Figure 5* displays the spectral characteristics of the FT660 laser and detection apparatus.

6.1 Explanation of the RSD Parameter

The FilterTrak 660 sc offers a complementary parameter to the laser nephelometry measurement. This parameter is known as the Relative Standard Deviation or RSD. This is a dimensionless parameter that provides a quantative assessment of the variability (fluctuation) of the laser turbidity measurement. Studies have shown that that baseline of a turbidity measurement will often increase in fluctuation before the actual laser turbidity measurement will begin to increase in response to a particle event. Second, the RSD parameter has been shown to be more sensitive to a turbidity event in addition to serving as a precursor event to a turbidity spike. Appendix B provides more information on the application and use of the RSD parameter.

The sensitivity of the RSD parameter directly related to the instrument design of the FT660 sc Nephelometer. This is due to the optical creation of a very small analysis volume within the turbidity sensor. (The analysis or view volume is that volume of sample within the turbidimeter body that is in view by the detector window). This volume is small but well defined by the optical design. This volume also contains a high energy density from the incident light beam, which can easily be scattered by a single or low number of particles. When a particle passes through the view volume, there is a rapid increase in the scattered light signal while that particle is in the view volume. When the particle passes out of the view volume, the signal decreases rapidly. The change in signal is infrequent and this instability of signal is quantified by the RSD parameter.

The RSD value is calculated as the standard deviation divided by the mean for a given set of measurements. The result is multiplied by 100 and is expressed as a percent. The equation below provides the calculation used to determine the RSD value:

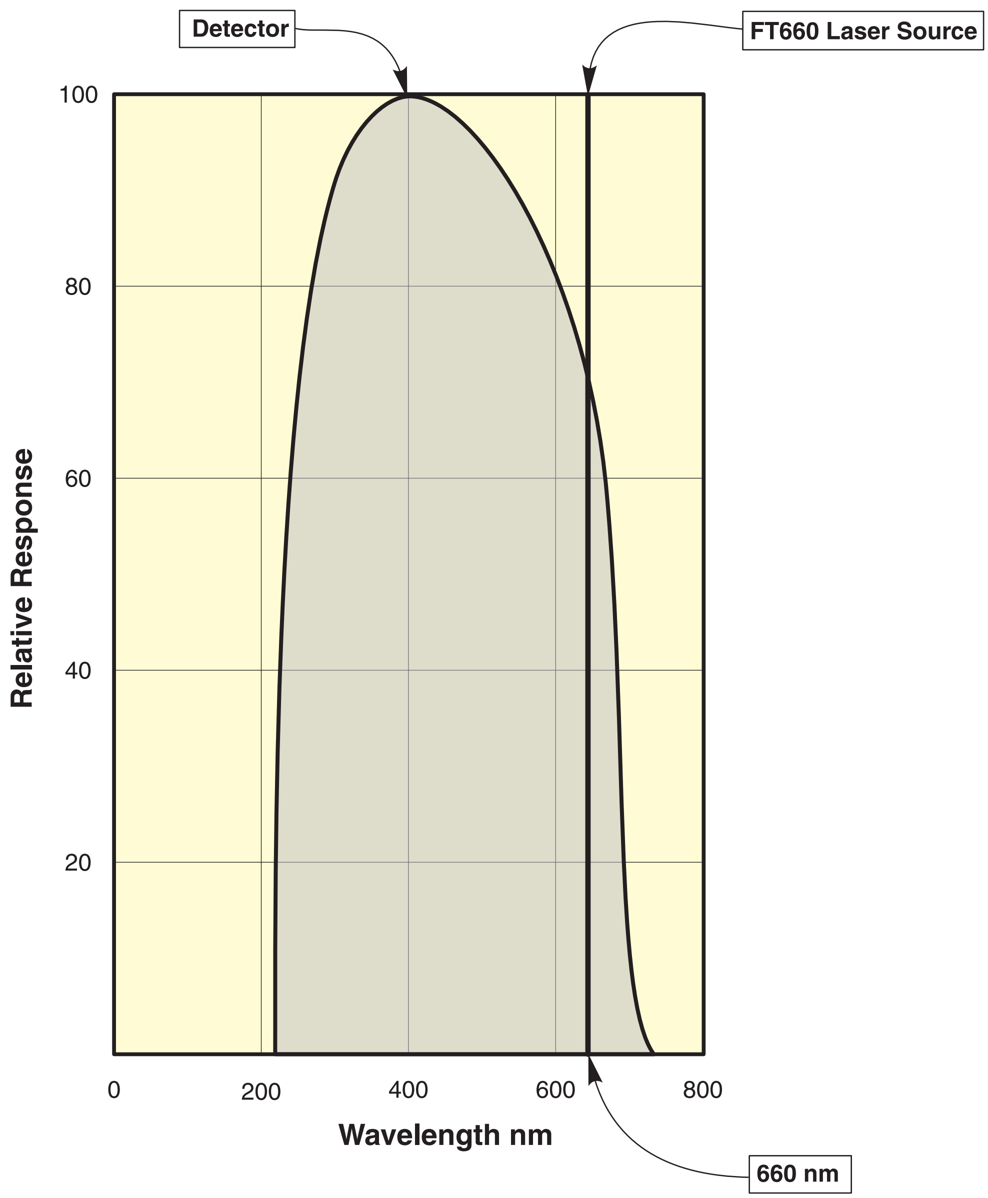
$$\text{RSD} = \left(\frac{\text{Std. Deviation}_n}{\text{mean}_n}\right) \times 100$$

Where n = number of measurements used

The RSD calculation is derived from a block of 10 consecutive turbidity measurements that are logged by the FT660sc. From these 10 measurements, the standard deviation and the average are calculated. The value is then displayed on the secondary measurement line of the sc 100 (see *Figure 5* showing the display information). After the RSD value is calculated, it is held until a new set of data containing 10 new measurements are collected and used to calculate the next RSD value. Thus, the RSD value will be updated at the rate of once every 10 seconds.

Figure 5 Spectral Overlap of the FilterTrak 660 Light Source and Detector

The FilterTrak 660 detector response significantly overlaps the spectra by the light source. In addition these two components are far more sensitive then those utilized in traditional turbidimeters. This results in high signal-to-noise ratios that increases the sensitivity of the FilterTrak 660 Laser Nephelometer.

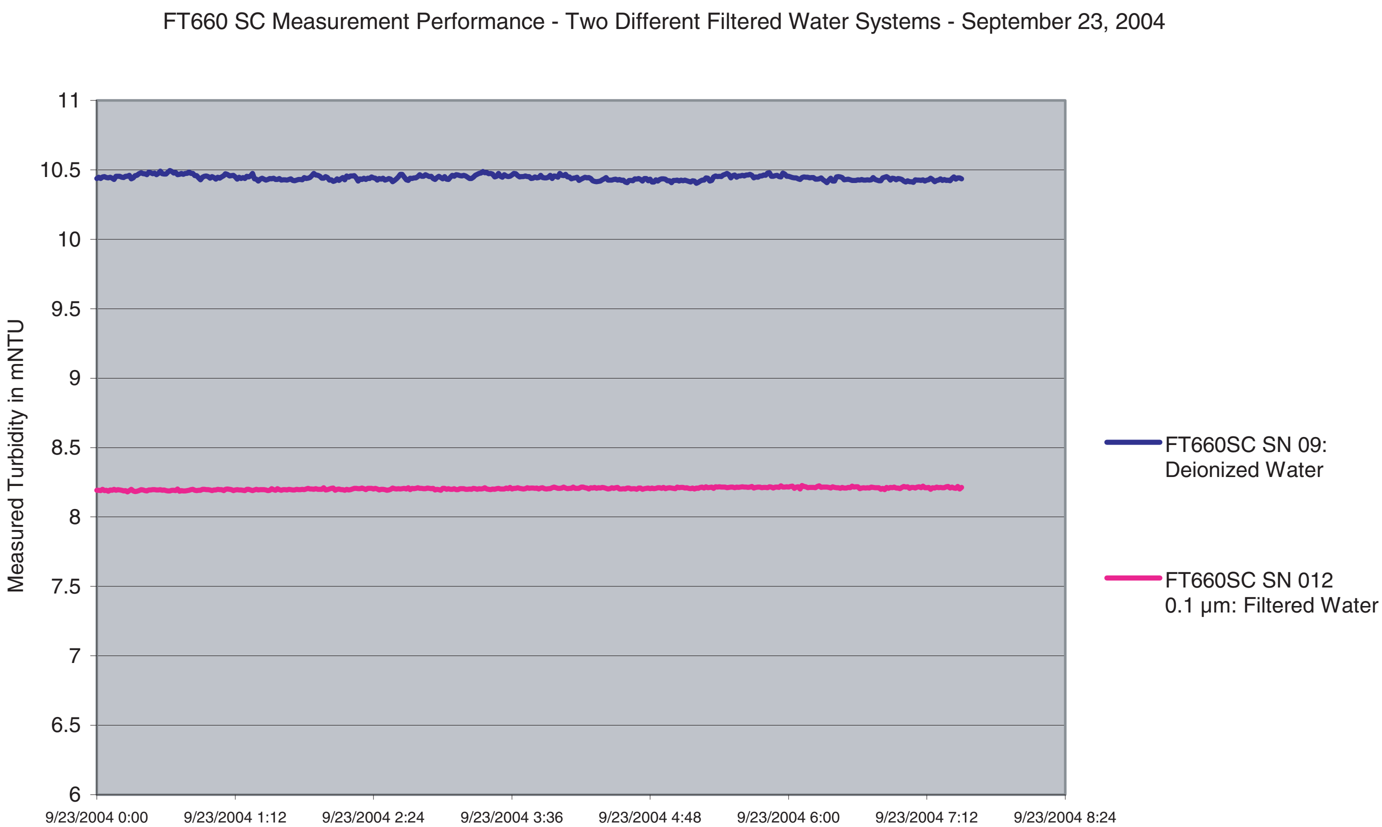


The RSD parameter is treated as a separate and independent monitoring parameter relative to the laser turbidity measurement. The parameter is updated every 10 seconds, where the laser turbidity value will update every second. The parameter is best used as an early warning parameter to an impending turbidity event and as a complementary parameter to the turbidity parameter (a turbidity spike will also be complemented by a spike in the RSD parameter). While the laser turbidity parameter is currently approved for regulatory monitoring, the RSD parameter is not a regulatory approved monitoring parameter.

Figure 6 displays the baseline of two different water sources that are measured with two different FT660 sc laser nephelometers. One sample is tap water that was filtered through a 0.1 µm filter. The second sample was a deionized water sample that has been conditioned by anion and cation exchange columns. Measurements were logged once per minute for a time-span of 7.5 hours. Flow through both instruments was set at 200 ml/minute.

The averaged turbidity for the 0.1 µm filtered water was 8.20 mNTU and the deionized water measured 10.44 mNTU. The measurement variance for the 0.1 µm filtered water was 0.102 percent RSD and the measurement variance for the deionized water sample calculated to be 0.168 percent RSD. The level of measurement resolution and stability provided by this technology allows the differentiation between different waters that are treated to purity using different techniques. *Figure 6* provides such an example.

Figure 6 Baseline Stability of the FilterTrak 660 Laser Nephelometer



6.2 FilterTrak 660 Laser Nephelometer vs. Standard Turbidimeter Technology

One of the greatest advantages of the FilterTrak 660 Nephelometer technology is that it still focuses and derives its results in nephelometric turbidity units (NTU). The instrument has been designed specifically for low turbidity applications with a range of 0–5000 mNTU. The instrument design and the basic concepts of light scatter and detection closely resemble current Hach process turbidimeters. The similarities offer many advantages to the end user.

6.2.1 FilterTrak 660 and 660 sc Calibration Basics

There are two versions of the FT660 Laser Nephelometer. The original version, the FT660 provided the option of performing either a 1-point or a 2-point calibration to cover the range of 0-1000 mNTU. Both curves resulted in equivalent performance. The FT660 sc only requires a single point calibration at 800 mNTU. The use of this single calibration point will accurately adjust the slope of a highly linear relationship between light scatter response and turbidity in the range of 0 to 5000 mNTU.

The FilterTrak 660 and 660 sc Laser Nephelometers are calibrated using formazin-based standards. Hach Company recommends StablCal® Stabilized Formazin Standards, that utilize the formazin polymer to generate light scatter. StablCal standards are custom formulated for use with this instrument. Advanced statistical analysis is performed for each lot produced. To ensure calibration consistency, each standard is defined to the nearest mNTU with a relative standard deviation not to exceed 5 percent.

Proper calibration will result in excellent measurement accuracy, which is better than 5 percent of reading or 5 mNTU on the FT660 and 3 percent of reading or 5 mNTU on the FT660 sc of the same range. To achieve this level of performance, calibration should be performed exactly as described in the respective instrument manuals.

The FilterTrak 660 Laser Nephelometer was designed to be calibrated on-site using the same calibration methodologies as other Hach process turbidimeters. Calibration requires minimal training, but to ensure accuracy, the manufacturer's procedures must be followed. For this instrument, lack of cleanliness is the most probable cause of error. Precautions must always be taken to keep the instrument clean during operation and calibration. Scheduled, closely-monitored cleaning of the instrument body, bubble trap, and detector apparatus is necessary. Store unused instruments in a clean, dust-free environment.

Since calibration is performed using StablCal Stabilized Standards which are directly traced to 4000 NTU primary formazin standards, the instrument reports results in NTU. This provides a direct reference and correlation to turbidity and its respective units in a traditional use.

6.2.2 FilterTrak 660 Instrument Design

The design of the FilterTrak Laser Nephelometer body, bubble rejection apparatus, and optical bench is nearly identical to the widely-used Hach 1720D process turbidimeter. Similarities in features eliminate the need to correlate variances in performance with respect to flow path, volumes, and bubble rejection efficiency.

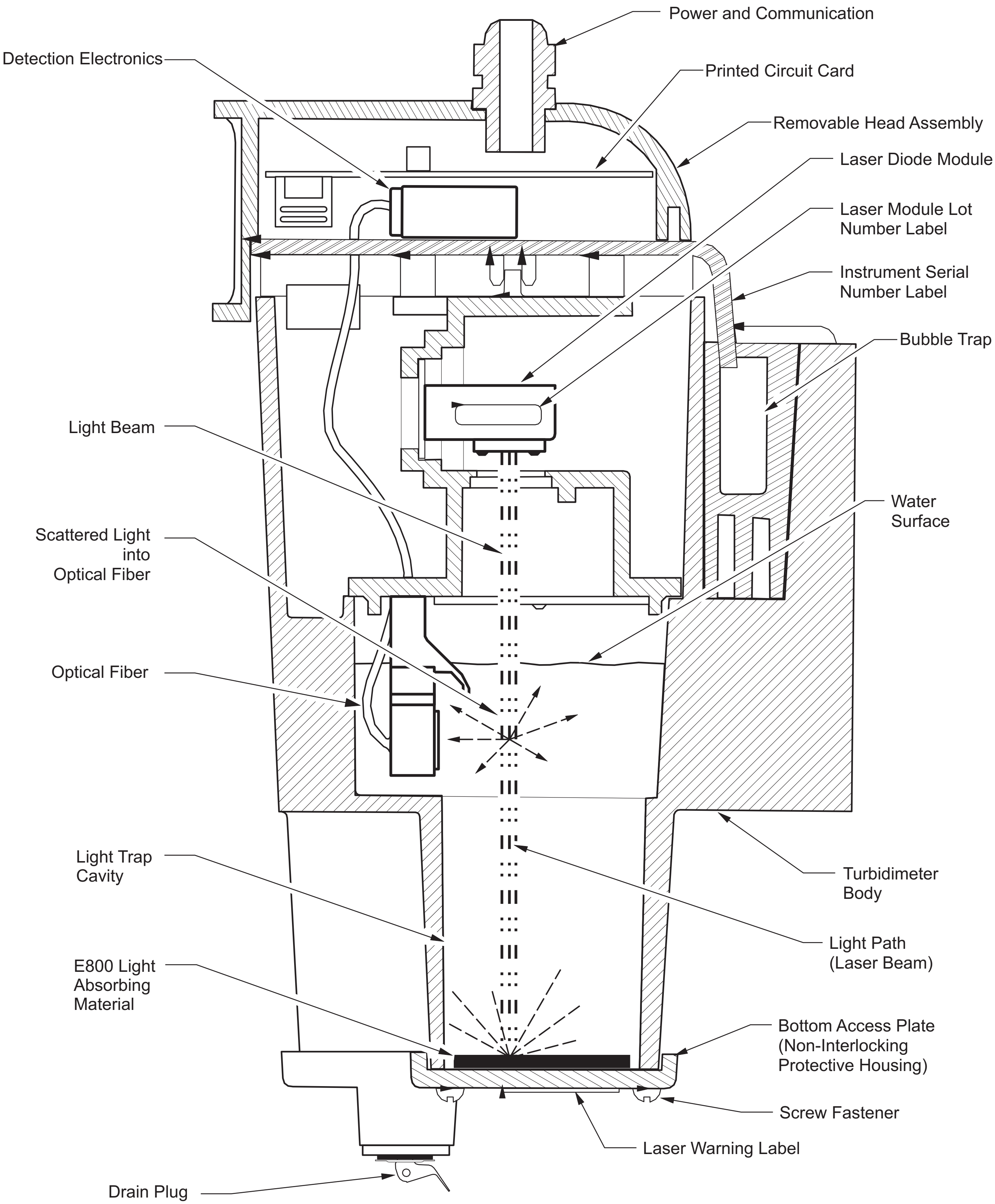
Figure 7 shows a cross-section view of the FilterTrak 660 or 660 sc Laser Nephelometer. The physical design of the instrument body, bubble trap, and sample flow path is very similar to the Hach 1720D Turbidimeter; an instrument that was designed to meet regulatory agency requirements. The body of the FilterTrak 660 contains a light trap that absorbs light at 660 nm. This eliminates nearly all reflectance of incident light and allows for low level measurement—down to 5 mNTU.

6.2.3 FilterTrak 660 Applications

A narrower range of applications exists for the FilterTrak 660 Laser Nephelometer than for traditional nephelometers such as the Hach 1720 Series. This instrument is specifically designed to monitor the quality of water downstream of the filter including the combined filter effluent, clearwell, and points in the distribution system. The instrument is also designed to monitor ultra-clean water such as the effluent from purification and membrane filtration.

This instrument is designed to serve as a tool to help ensure filter performance, predict events, and/or confirm events detected by other particle sensitive instruments. Overall, the FilterTrak 660 Nephelometer will help the WTP operator respond more quickly to events that could result in the release of unwanted material and an increased risk to public health.

Figure 7 Section View of the FilterTrak 660 Laser Nephelometer



7.1 FilterTrak 660™ Laser Nephelometer Performance Data

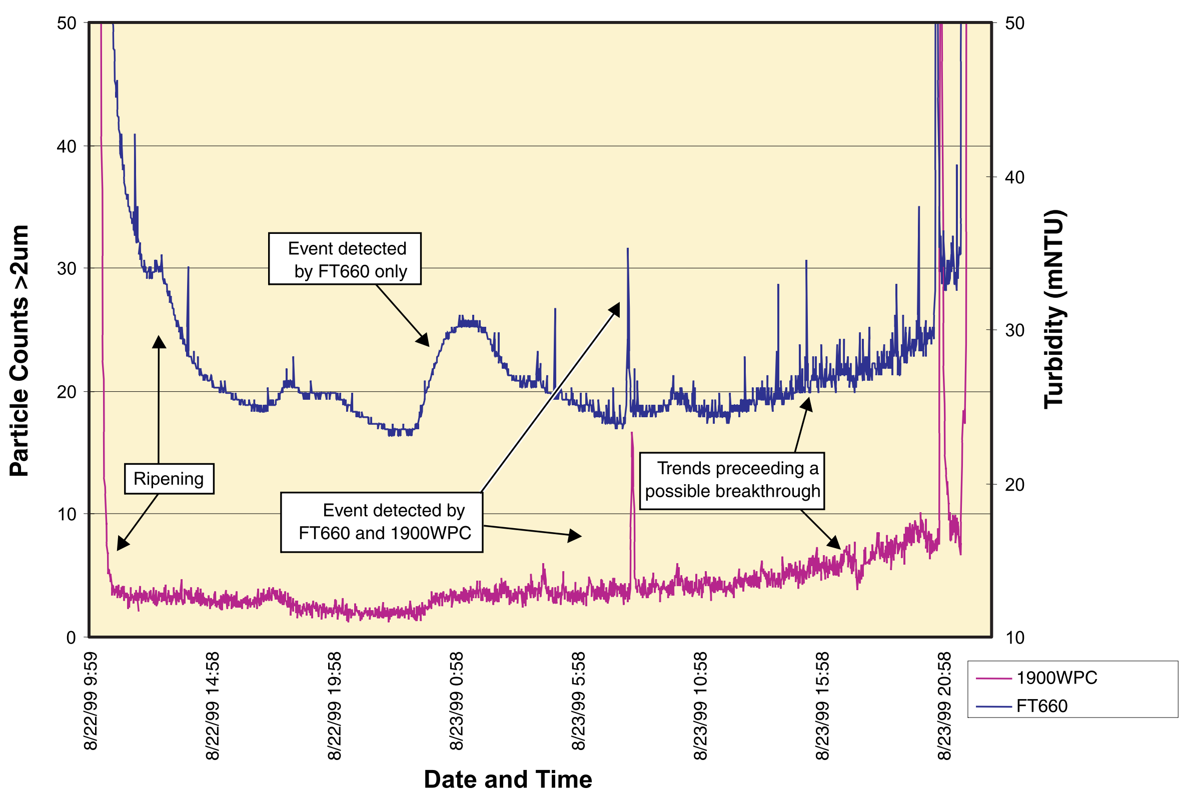
The presented data shows the potential of the FilterTrak 660 Laser Nephelometer for detecting and predicting filter events. In many cases, the FilterTrak 660 Laser Nephelometer closely compares with particle counters. In other instances, the FilterTrak 660 Nephelometer detects events not “seen” using traditional instrumentation.

The FilterTrak 660 Laser Nephelometer has been tested at several water treatment plants to monitor the water directly leaving a filter. A particle counter and a traditional turbidimeter were also placed on the same sample. Flow rates were matched as closely as possible to facilitate direct measurement comparisons.

A typical filter run is shown in *Figure 8*. The data shows particle counts and turbidity from ripening through breakthrough. The FilterTrak 660 and the Hach 1900 WPC particle counter data are presented. The 1900 WPC is a 2-µm sensor that has been very successfully applied in the drinking water treatment industry. Both instruments were monitoring the sample stream in parallel, with similar flow rates (approximately 200 mL/minute).

In *Figure 8*, it is important to note that the detection of the ripening period is different for the two instruments. The ripening period detected by the particle counter is shorter than that detected by the FilterTrak 660 instrument. This occurs because the larger particles (>2 µm) that are detected by the particle counter settle more quickly and the smaller sub-micron particles that are detected by the FilterTrak 660 instrument settle more slowly.

Figure 8 FilterTrak 660 Typical Filter Run



Near the end of the filter run, both instruments indicate an upward trend in both turbidity and particle counts. This is typically the trend observed before a filter breakthrough. At this point the filter run was terminated with a backwash. Both instruments also detected the backwash event.

Within this filter run, some smaller events are detected by the FilterTrak 660 instrument, but are missed by the particle counter (for example the event shown in *Figure 8* on 8/23/99 at approximately 1:21). These are most likely sub-micron events that contain particles sizes below the detection level of the particle counter. Each event detected by the particle counter is also detected by the FilterTrak 660 instrument. Concurrent detection by both particle counter and FilterTrak 660 is solid confirmation that the event is real and warrants further investigation.

Figure 9 shows another typical filter run. This graph also shows data from a 1720D turbidimeter. Again the sample flow rate and distance from the sample point are similar to the other two instruments. The 1720D turbidimeter meets the design criteria for EPA Method 180.1 for regulatory reporting of turbidity. The measurement signal from the 1720D was brought into an external data logging system, causing slightly reduced resolution. The minimum change graphically displayed by this instrument is 3 mNTU (0.003 NTU). This is evidenced by the block-like response changes displayed in the graph. Note that the 1720D still detects the ripening of the filter (left edge of the graph) but then remains relatively stable (plus or minus 3 mNTU resolution) until backwash of the filter begins (denoted by the large spike near the right side of this graph).

The turbidity data shown from the FilterTrak 660 instrument and the 1900 WPC Particle Counter display more filter events than those seen by the 1720D turbidimeter. The ripening period appears to be shorter when only the particle counter data is considered. This indicates that only the more rapidly settling, i.e, larger, particles (>2 µm) are detected by the particle counter.

When several minor events were detected by the FilterTrak 660 instrument, the particle counter reading remained relatively constant. Since all spikes detected by the 1900 WPC Particle Counter were also detected by the FilterTrak 660 instrument, the complementary nature of these two technologies is evident.

An event detected by both the FilterTrak 660 and 1900 WPC occurs on 8/22/99 at approximately 4:52 in *Figure 9*. This portion of the graph is expanded and displayed in *Figure 10*.

As shown in the graph, the FilterTrak 660 detects the event approximately 10 minutes before the particle counter. This indicates that the sub-micron particles of this event are indeed a precursor to the larger particles eventually detected by the particle counter. Remember, the sample point and flow rate for all the instruments shown in *Figure 10* are nearly the same.

Observe the data from the 1720D. This instrument, which utilized the technology mandated by the regulatory methods, does not have the sensitivity to detect the event seen by the other two instruments with any confidence. Accepted practice within the water treatment industry is for the operators to ignore very small changes in turbidity. The observed change of 0.003 NTU would likely be assumed to be noise and ignored by most operators. In most situations when using current technology, readings below 1 NTU are rounded to the nearest 0.05 NTU.

Figure 9 FilterTrak 660 Typical Filter Run with 1720D Turbidimeter and 1900 WPC Particle Counter Data

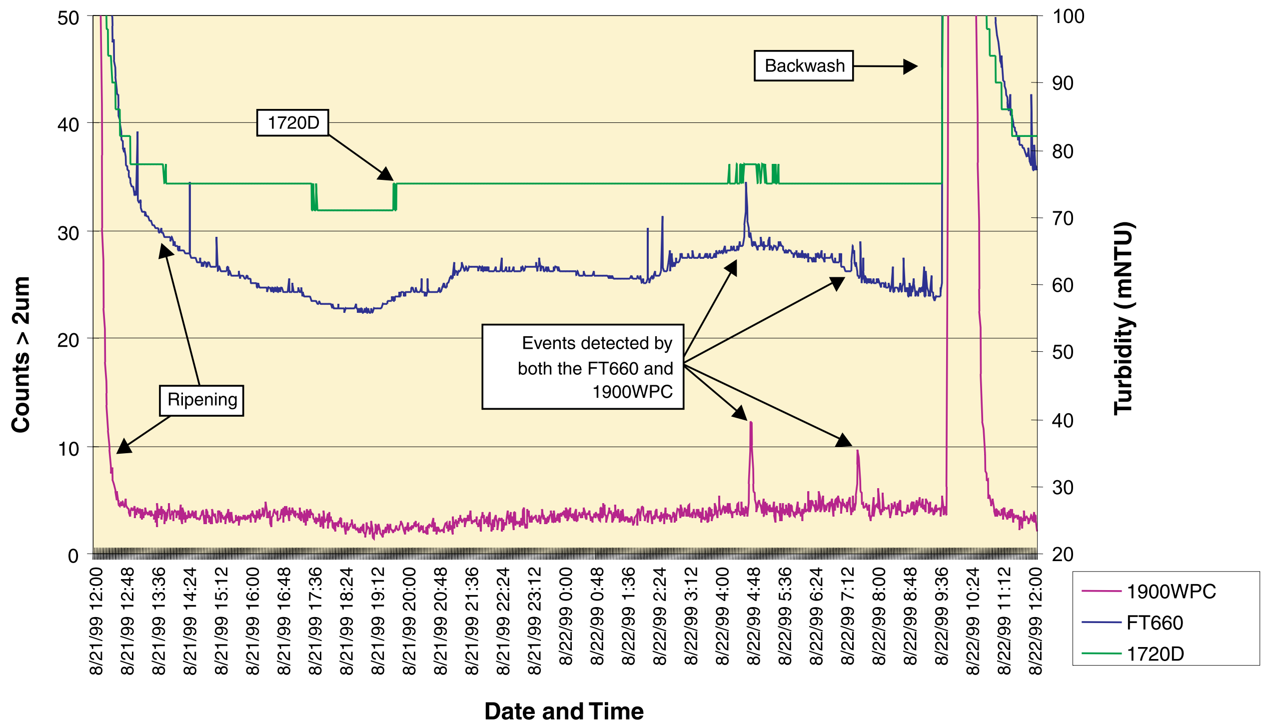
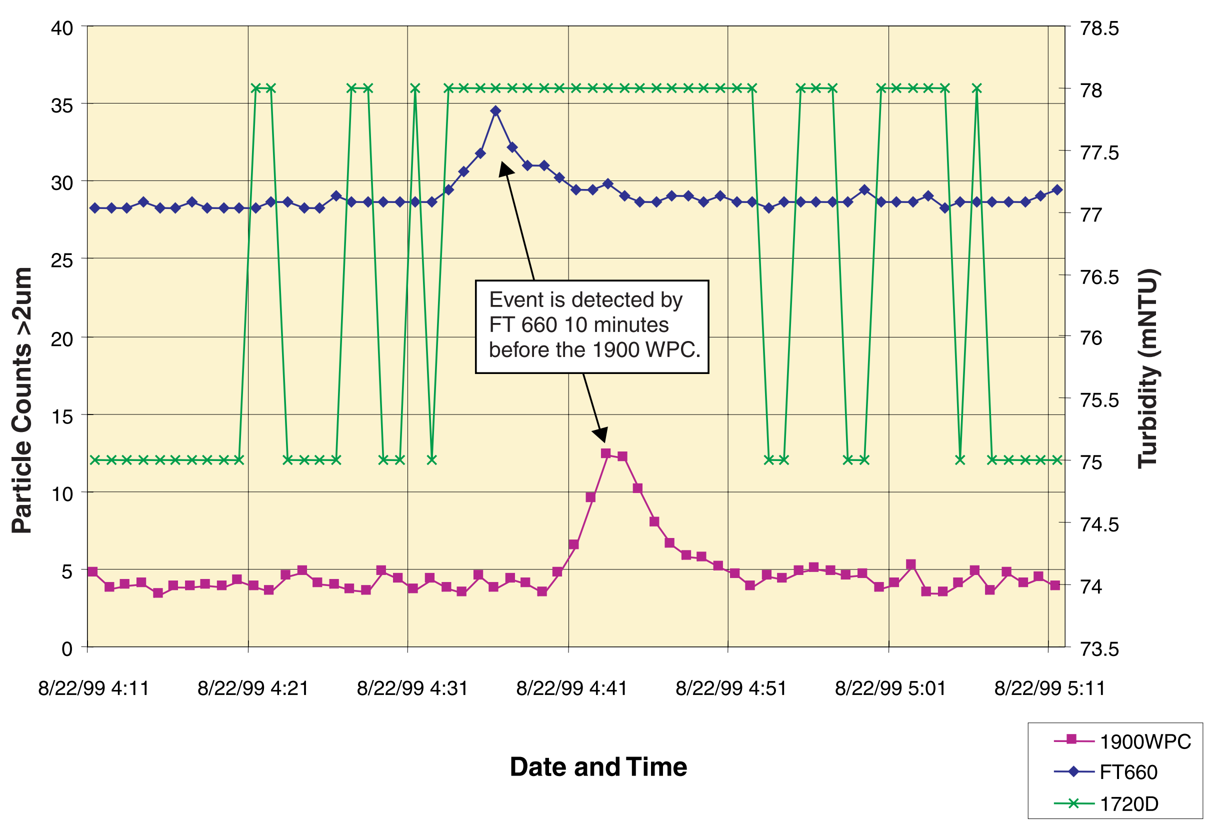


Figure 10 FilterTrak 660 Typical Filter Run with 1720D Turbidimeter and 1900 WPC Particle Counter Data—Expanded View



SECTION 8 Summary

The FilterTrak 660™ Laser Nephelometer has demonstrated the ability to track particle counter events and often detects events before the particle counter sees them. This correlates to accepted power laws that are used to model particle distributions of natural waters—as the size of particles decrease, the number of particles increases substantially. The ability of the FilterTrak 660 to detect of small particles is a precursor to the detection of larger (>2 μm) particles seen by the particle counter.

The FilterTrak 660 complements the particle counter technology that is currently being applied to the drinking water industry. Since the FilterTrak 660 instrument detects the same spikes or events detected by the particle counter, the operator has confidence that such an event is indeed occurring. If additional detail regarding an event is needed, the particle counter can provide useful information with respect to count and size distributions.

The FilterTrak 660 also complements standard, familiar technology. The instrument is a pure nephelometer and is calibrated using the same standard that is used with today's turbidimeters. The FilterTrak 660 reports results as mNTU. Those results are traced to primary formazin standards. Like other turbidimeters, the instrument is easy to calibrate, verify, and maintain. It does not have application problems (such as clogging, cleaning difficulties, etc.) that are sometimes associated with particle counters. In addition, the data are easy to interpret and store. The additional sensitivity of the FilterTrak 660 Laser Nephelometer provides the WTP operator with yet another window into the analysis of their treatment methods.

In conclusion, the FilterTrak 660 Laser Nephelometer is designed as a filter management tool. The instrument is intended to provide the operator with another method to detect events and to a limited degree, to determine the nature of an event. Coupled with current turbidity technology and particle counter technology, the FilterTrak 660 can help the operator optimize the efficiency of a water treatment plant. It can also provide another level of confidence for confirming that the water leaving the water treatment plant is indeed safe for the consumer.

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APPENDIX A Hach Method 10133 — Determination of Turbidty by Laser Nephelometry*

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of turbidity in finished drinking, filtered effluent, distribution, ultra-pure, micro-process and any colorless water with turbidity less than 5.0 NTU.

1.1.1 Note: colorless water is any water exhibiting a Platinum-Cobalt color value that is less than 50 color units when measured at 455 nm using a 1-inch sample cell.

1.2 The applicable range is 0 to 5000 milli-nephelometric turbidity units (mNTU).**
Note: 1 NTU = 1000 mNTU.

1.3 This method is to be used for compliance monitoring under the Safe Drinking Water Act (SDWA).

2.0 SUMMARY OF METHOD

2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in mNTU's, are made using a laser nephelometer designed according to specifications given in section 6.1. Primary standard suspensions are used to calibrate the instrument. These are referred to as instrument calibration standards. Calibration verification standards are used to check instrument performance and verify the instrument is operating correctly. Primary standards and Calibration Verification Standards are listed below:

2.1.1 Primary standards:

2.1.1.1 Formazin polymer is used as a primary turbidity suspension for water because it is more reproducible than other types of standards previously used for turbidity analysis.

2.1.1.2 StablCal® Certified formazin turbidity standards are formazin polymer suspensions that have long term stability. These standards are assayed to the nearest mNTU and are designed for calibration of the instrumentation used in this method. These standards are available from Hach Company.

2.1.2 Calibration Verification Standards:

2.1.2.1 A calibration verification standard suspension is used as a daily calibration check. This standard can be a dry apparatus or a stabilized formazin standard.

2.1.2.2 The dry calibration verification module (CVM) is designed for daily monitoring of the calibration of the instrument without the use of wet standards. The CVM is calibrated against primary standards. The CVM must be designed specifically for the instrumentation used in this method. Such devices should be re-certified on a regular basis.

2.1.2.3 StablCal® stabilized formazin standards can be used for calibration verification.

3.0 DEFINITIONS

3.1 CALIBRATION BLANK (CB) — A volume of filtered reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards or surrogates analytes.

3.2 CALIBRATION VERIFICATION STANDARDS (CALVER) — Commercially prepared, stabilized liquid, gel turbidity standards, or dry opto-mechanical devices calibrated against properly prepared and diluted primary formazin, or stabilized formazin. These CALVER standards are for calibration verification of the instrument.

* Revision 2.0, January 7, 2000

** The Hach FilterTrak 660 sc Laser Nephelometer has an extended range to 5000 mNTU. Extending the range of the instrument does retain linearity and can be used for regulatory use.

3.3 INSTRUMENT CALIBRATION STANDARD (ICAL) — A standard that can be used for the calibration of instrumentation that conforms to this method. These include: a suspension prepared from the primary formazin stock standard suspension, commercially available stock formazin standards, and StablCal® stabilized formazin turbidity standards. The ICAL suspensions are used to calibrate the instrument response with respect to analyte concentration.

3.4 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) — A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.5 LABORATORY REAGENT BLANK (LRB) — An aliquot of reagent water or other blank matrices that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.6 LINEAR CALIBRATION RANGE (LCR) — The concentration range in which the instrument response is linear.

3.7 MATERIAL SAFETY DATA SHEET (MSDS) — Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data including storage, spill, and handling precautions.

3.8 QUALITY CONTROL SAMPLE (QCS) — A solution of the method analyte of known concentrations that is used to fortify an aliquot of LRB matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.

3.9 STOCK STANDARD SUSPENSION (SSS) — A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension or pre-diluted stabilized Formazin suspensions can be used to prepare calibration suspensions and other needed suspensions.

4.0 INTERFERENCES

4.1 The presence of floating debris and coarse sediments that settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

4.2 The presence of true color, that is the color of water that is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.

4.3 Light-absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 Refer to all Material Safety Data Sheets (MSDSs) prior to preparing or using standards and before calibrating or performing instrument maintenance.

6.0 EQUIPMENT AND SUPPLIES

6.1 The turbidimeter shall consist of a nephelometer, with a light source for illuminating the sample, and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be designed so that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.

6.2 Differences in physical design of turbidimeters will cause differences in measured values for turbidity, even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:

6.2.1 Laser Nephelometer Optics

6.2.1.1 Light source: Laser Diode operated at a wavelength of 660 ± 30 nm.

6.2.1.2 There shall be no divergence from parallelism at the incident radiation and any convergence shall not exceed 1.5°.

6.2.1.3 Distance traversed by incident light and scattered light within the sample tube if needed: Total not to exceed 10 cm.

6.2.1.4 Detector/Light Receiver: Centered at 90° to the incident light path and not to exceed ± 2.5° from 90°. The receiver, if used, must be coupled to a photomultiplier tube (PMT) using a fiber-optic cable.

6.2.1.4.1 The PMT must have a spectral output that encompasses the complete spectral output of the light source.

6.2.1.5 Fiber-optic cables may be used to carry light from the light source to the sample or to carry scattered light from the sample to the PMT detector or both.

6.2.1.6 Equipment: Examples of Hach Company's turbidimeters which meet or exceed these specifications are as follows: the FilterTrak 660™ Laser Nephelometer.

6.3 The sensitivity of the instrument should permit detection of a turbidity difference of 1 mNTU or less in waters having turbidities less than 5000 mNTU units. The instrument should measure from 0 to 5000 mNTU turbidity units.

6.4 Balance — Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.5 Glassware — Class A volumetric flasks and pipets as required.

6.5.1 All glassware must be scrupulously cleaned and rinsed with reagent water (see 7.1 below) immediately prior to use.

7.0 <u>REAGENTS AND STANDARDS</u>

7.1 Reagent water, turbidity-free: Pass de-ionized distilled water through a 0.2 mm or smaller pore size membrane filter. Reverse osmosis filtered water is suitable for use in this method. Such prepared water must have a turbidity of between 0.02 and 0.03 NTU (20-30 mNTU). This value should be considered when preparing calibration standards.

7.1.1 During cleaning, use this water for the rinsing of any surface of the instrument that comes into contact with the sample. This includes bubble removal devices, sample chambers, sample cells, sample lines, etc.

7.1.2 Use this water for final rinses of any glassware used in the preparation of calibration standards or in the measurement of samples.

7.2 Stock standard suspension (Formazin):

7.2.1 Dissolve 1.00 g hydrazine sulfate, $(NH_2)_2 \bullet H_2SO_4$, (CASRN 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask.

7.2.2 Dissolve 10.00 g hexamethylenetetramine (CASRN 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask. In a 100 mL volumetric flask, mix 5.0 mL of each solution (7.2.1 + 7.2.2). Allow to stand 24 hours at 25 ± 3°C, then dilute to the mark with reagent water. The turbidity of this solution is defined as 400 NTU.

7.3 Instrument Calibration Standards (ICAL)

7.3.1 Prepared Primary Standards

7.3.1.1 Mix and dilute 2.00 mL of stock standard suspension (7.2) to 1000 mL with reagent water. The turbidity of this suspension is defined as 800 mNTU plus the turbidity of the reagent water (20-30 mNTU). For other values, mix and dilute portions of this suspension as required.

7.3.1.2 A new stock standard suspension (7.2) should be prepared each month. Primary calibration standards (7.3) should be prepared immediately before use.

7.3.2 Commercially Available Standards

7.3.2.1 Formazin in commercially prepared primary concentrated stock standard suspension (SSS) may be diluted and used as required. Dilute turbidity standards should be prepared immediately before use.

7.3.2.2 Pre-diluted stabilized Formazin suspensions (StablCal® Certified Standards) primary standards are available for use in all instruments and require no preparation or dilution prior to use.

7.3.2.2.1 StablCal® primary standards are assayed and reported to the nearest 1 mNTU at the factory.

7.3.2.2.2 StablCal® primary standards are the recommended by the manufacturer for standardization of the instrumentation used in this method.

7.4 Calibration Verification Standards (CALVER)

7.4.1 May be acceptable as a daily calibration check, but must be monitored on a routine basis for deterioration and replaced as required. The CALVER standards must have traceability to formazin or StablCal® primary standards.

7.4.2 A dry calibration verification module (CVM) is an opto-mechanical device that is designed to simulate a specific turbidity value is suitable for calibration. The simulated turbidity value is defined using a formazin or stabilized formazin primary standard.

7.4.2.1 The CVM must be designed specifically for the instrumentation type used with this method.

7.4.2.2 The CVM should be factory re-certified using formazin or StablCal primary standards on an annual basis.

7.4.2.3 The CVM can be verified on-site using the laser nephelometer and primary standards.

7.4.3 ICAL Standards can be used for calibration verification of the instrument.

8.0 SAMPLE COLLECTION AND INSTRUMENT SETUP

8.1 This method is only applicable to samples of 5000 mNTU (5.0 NTU) or less. Such samples can be susceptible to fine environmental changes, therefore, all samples should be analyzed immediately and cannot be preserved for later analysis. This method is designed for on-line instrumentation.

8.2 Instrument setup: Set the instrument up according to the instrument instruction manual. Additional guidance for set-up is described below.

8.2.1 Instrument location: The instrument should be located in clean surroundings and as close to the sample point as possible. The location should be such that the instrument is easily accessible for cleaning, maintenance and calibration. The location should be free from significant vibration and the temperature should always be between 0-40 degrees C. A temperature of 15-30 degrees C is optimal.

8.2.2 Instrument mounting: mount instrumentation to a pole, wall or panel. The instrument must be mounted level. Do not mount to any surface that vibrates.

8.2.3 Sample lines: Use clean sample lines made of polyethylene, polypropylene or Teflon®. Minimize the distance of the sample lines between the source and the instrument.

8.2.3.1 Install sample line taps into larger process pipes to minimize interference from air bubbles or pipeline bottom sediment. A tap projecting into the center of the pipe is ideal.

8.2.4 Sample flow: The sample flow should be set within the instrument specifications. Slower flow rates can reduce excessive noise should bubble saturation be of concern. A valve should be used to control the inlet flow and will also minimize the effects of sample surges.

8.2.5 Instrument parameter setup: For optimum performance, set the signal averaging to the longest time possible, set bubble rejection to "On."

8.2.6 Initial instrument cleaning: After the installation process is complete, clean all surfaces that will come into contact with the sample according to the instrument manual. Follow cleaning with a rinse of all surfaces using turbidity-free reagent water. See the instrument manual for specific cleaning information.

8.2.7 Calibration: Immediately perform instrument calibration after cleaning of the instrument has been completed. Refer to the instrument manual for specific calibration instructions.

8.2.8 Verification: After calibration and at regular intervals, verify the performance of the instrument using a CALVER standard. The frequency of verification should be no longer than one month and more frequent if deemed necessary.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and other solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated.

9.2 Initial Demonstration of performance

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS).

9.2.2 Linear Calibration Range (LCR) — The LCR must be determined initially and verified every three months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient ICAL or CALVAR standards to insure that the resulting curve is linear. One standard should in the 50-200 mNTU range and the other standard should be between in the 700-900 mNTU range. If any verification data exceeds the initial values by ± 25 mNTU, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.2.1 If the observed response displays positive bias, the result is most likely due to contamination of either the standard and/or the instrument. At this point, the instrument should be thoroughly cleaned as per the instrument instruction manual followed by re-measurement of a fresh standard.

9.2.2.2 If the observed response displays negative bias, it may be due to an impending component failure. In this case, re-calibration and re-verification of the instrument should be conducted to insure performance is intact.

9.2.3 Quality Control Sample (QCS) — When using this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. Run the QCS to determine if the method performance meets the stated acceptance criteria of the QCS. If the determined concentrations are not within the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Nephelometer calibration: The manufacturer's operating instructions should be followed using instrument calibration standards (ICAL). Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales.

10.2 Nephelometer verification: After calibration verify the instrument using either ICAL or CALVER standards.

11.0 PROCEDURE

11.1 Turbidities less than 1000 mNTU: All instrument parameters should be set to insure constant sample flow is fed to the instrument. This will insure bubble interferences are minimized. Refer to the manufacturer's instrument manual for the set up of measurement parameters and flow rates.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Report results as follows:

mNTU	Record to Nearest:
0 - 5000	10

Note: To convert to NTU, divide the mNTU reading by 1000.

13.0 METHOD PERFORMANCE

13.1 In a single laboratory, using filtered water samples at levels of 108, 27.7, and 21.3 mNTU, the standard deviations were 25.0, 1.1, and 5.6 mNTU respectively.

13.2 The inter-laboratory precision and accuracy data in Table 1 were developed. Values are in mNTU. The intra-laboratory precision and accuracy data in Table 2 were developed using various Hach instruments.

Table 1

Theoretical Spike (mNTU)	Averaged Reading	Standard Deviation	Percent Recovery
62.1	65.3	16.40	105.2
104.0	112.0	10.38	107.7
106.0	104.2	14.01	98.3
414.0	458.7	33.50	110.8
619.2	770.3	27.57	124.4
634.0	756.5	26.24	119.3

Table 2

Theoretical Spike (mNTU)	Averaged Reading	Standard Deviation	Percent Recovery
62	70.0	16.3	112.7
104	118.2	8.7	113.6
106	111.8	12.2	105.5
414	456.8	32.8	110.3
619	776.8	23.5	125.5
634	768.5	28.2	121.2

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

Whenever feasible, laboratory personnel should use pollution-prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect air, water and land by minimizing and controlling all releases from hoods and bench operations; complying with the letter and spirit of any waste discharge permit and regulations; and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

For further information on waste management consult "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

16.0 REFERENCES

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APPENDIX B Using Baseline Monitoring Techniques to Assess Filter Run Performance and Predict Filter Breakthrough*

Abstract

Determining if a filter run is approaching a breakthrough condition is a daily challenge for water treatment plant (WTP) operators. Current techniques look for upward trends in either turbidity or particle counts of the filter effluent. However, this does not consistently predict actual filter breakthrough.

This study's objective is to determine if data from different particle detection technologies can be better utilized to characterize filter performance. Simple statistical techniques will be used to interrogate the stability of the baseline filter effluent water values. This study utilizes both traditional and new particle detection technologies to monitor filter effluent for the entire filter run. Each type of technology (particle counters, regulatory turbidimeters, and laser nephelometers) will be evaluated separately on each filter run to determine which generates the best correlation between baseline stability and filter performance. The ability of each of these particle detection technologies to predict filter breakthrough will be evaluated. Ultimately, this study will determine if such correlations can provide a definitive means of interpreting filter performance and can then be used to predict filter breakthrough.

Data from complete filter runs at a pilot-scale plant will be used. The data from the filter runs, which were allowed to proceed through breakthrough, will help determine if this information can actually predict breakthrough. In addition, effluent data from several full-scale water treatment plant runs were analyzed and an example was presented. This information will be used to confirm the pilot-plant data and increase the credibility of the pilot study model.

Results have shown that when laser nephelometers, and (to a lesser degree) particle counters, are used to monitor filter effluent, the measurement baseline stability decreases (the noise level increases) as the filter run progresses. The decrease in measurement stability, when observed during filter effluent monitoring is often attributed to electronic noise in the instrument. This study provides evidence that the "noise" is not attributed to the laser nephelometer, but instead, is due to subtle changes in the sample.

Challenges in Predicting Filter Breakthrough

A major challenge and a primary goal for water treatment plants is the maximization of filtration output while simultaneously providing the consumer the highest quality water possible. Studies have shown that when filter effluent turbidities and particle counts are kept low and constant, the risk of microbial contamination at the filter is low and the overall water quality is high.

All water treatment plants want to avoid a filter breakthrough event. Because of the concern surrounding filter breakthrough, stringent regulations continue to be placed on the effluent water to minimize the risk of pathogen breakthrough. The Enhanced Surface Water Treatment Rule[1] is now implementing regulations specifying that turbidity must be monitored on every filter. This specific rule does two things:

* By Michael J. Sadar, Application Scientist, Hach Company and Kathleen Bill, Water Operations Specialist, City of Aurora.

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- First, it offers continuous profiling of every filter run and provides insurance that the filtration processes are performing well.
- Second it provides a more rapid and direct troubleshooting solution when a filtration problem occurs.

Since each filter is now being monitored, it would be beneficial to use the data already being collected to predict filtration problems before they actually occur.

Even in the midst of these new filtration-monitoring requirements for turbidity, most well run drinking water plants (DWP) do not rely heavily on rising turbidity levels to predict when the filter run should be terminated. Instead of using rising turbidity to predict filter breakthrough, they attempt to be more proactive and terminate the run based on events other than monitoring the filter effluent. Three accepted methods for determining timing for termination of a filter run are:

1. Loss of head pressure

2. A timed run – a set filter run duration based on past filter performance

3. An increase in turbidity levels – Regulated!

Although the run is terminated when any of the above occurs or dictates, for any of the filter run termination methods cited above, the breakthrough condition could have already occurred. To avoid this, most plants apply a conservative filter run time and terminate the run even if the filter continues to perform well. This has proven to be the safest and most proactive approach, and the practice is still backed by monitoring the filter's performance.

Using particle detection instrumentation to predict a filter breakthrough prior to the event actually occurring would be a proactive and beneficial approach to filter management. If successfully applied, this information would provide the water treatment plant additional throughput by delaying backwash where appropriate and more time to react to an unforeseen filter problem leading to a breakthrough of particles. Instrumentation such as particle counters and turbidimeters can be used to detect filtration problems, but are often not used consistently enough. Insufficient data has been gathered by most water treatment plants and so does not aid them to predict every event.

Issues Associated with Regulatory Compliant Particle Monitoring Technologies

One problem associated with the current instrumentation and the associated monitoring method is that the particle event is likely to be occurring before the water treatment plant can react. In many situations, if breakthrough does occur, the effluent has been contaminated and the risk of pathogenic contamination has increased. History has proven that it is not very beneficial to react to a turbidity spike after it is detected.

A second problem is related to instrument sensitivity limits. Turbidimeters designed to comply with EPA 180.1[2] specifications may lack the sensitivity necessary to see low-level filtration problems. A prime example is the 1991 Cryptosporidium outbreak in Milwaukee, Wisconsin. According to reported turbidity measurements, the combined effluent turbidity levels never exceeded the 1991 regulatory limit of 0.5 NTU[1]. Part of the problem may be that the instrumentation in use at that time was not sensitive enough to provide conclusive data on very low-level turbidity events.

Current turbidimeter designs are often unable to detect ultra-low level turbidity changes because of instrument technology limitations. Previous turbidity regulations mandated a sensitivity level to 0.050 NTU and most instruments are able to detect differences well below this level (at least down to 0.010 NTU). However, at levels below 0.010 NTU, changes are often attributed to instrument noise and are usually discarded.

If, when designing instrumentation, the baseline sensitivity was increased and instrument noise was held to extremely low levels, minor turbidity changes and their source would be easier to trace. If the detection sensitivities were increased and if the instrument could consistently and dependably sense the smallest changes in turbidity, then the measurement confidence would also increase. Using this instrumentation, low-level turbidity changes could predict filter spikes. Unfortunately, current regulatory turbidimeters have not successfully provided consistent performance at ultra-low turbidity levels.

In summary, basing the initiation of a backwash on a spike in turbidity or particle counts has been difficult. Historical data is often inconsistent across multiple runs and this inconsistency makes it difficult to determine the necessary parameters to initiate a backwash. To compound the problem, the data obtained from particle counters and traditional turbidimeters does not always agree with respect to event detection. Also, when an event does occur, the determination must be made as to whether it is a minor or major event. If the event is minor and is of short duration, initiating a backwash may be unnecessary. The goal to applying particulate detection instrumentation to filter effluent is to consistently predict major particle spikes or breakthrough events using defined criteria.

Applying New Technologies to Low Level Measurements:

A new generation of instrument technologies may meet the requirements of ultra low level monitoring, and in doing so, restore user confidence when applying the data to real-life effluent monitoring. One breakthrough technology, laser nephelometry, combines a stable, defined, light source with a highly sensitive nephelometric detector.[3] This combination yields a nephelometer with an exceptionally stable baseline due to very low instrument noise. The optical design of the laser nephelometer provides high sensitivity to light scatter from particles that are less than 1 µm in diameter and minor turbidity changes are easily detected against the stable baseline. Hach Company offers the FilterTrak® 660 (FT660) laser nephelometer.

A second instrument, the particle counter, has been applied to filter effluent with good results. The most common on-line particle counters are those with particle size sensitivity down to 2 µm in diameter. Studies have shown particle counts often begin to trend upward prior to a filter breakthrough. A regulatory turbidimeter often will not see the same upward trend until later in the filter run. However, the particle counters have not been shown to consistently predict breakthrough and so the confidence level in them predicting filter breakthrough by detecting ultra low level events has not been high. Those who have spent large amounts of time collecting and interpreting data have achieved the most highly successful implementation of particle counting for breakthrough prediction.

Figure 1 displays a typical filter run monitored by a particle counter, a regulatory turbidimeter, and a FT660 laser nephelometer. Overall, the run is very quiet and the turbidity baselines are very stable. At first glance, the data shows that the filter was performing well within its regulatory limits. However, a closer look at the data from each instrument may provide additional information that could be benefit the operator.

One feature of Figure 1 is the amplitude of the turbidity baseline. As the run progresses, the amplitude increases very slightly. This amplitude is often is referred to as baseline noise and is often attributed to instrument (electronic) noise. In this graph however, the amplitude is not consistent throughout the duration of a filter run—which would be the case if it were instrument noise. Instead, this change in amplitude continues to increase as the run progresses. This information points to a slow but deliberate change in the filtration mechanism or in the process prior to filtration.

The data showing the change in amplitude is clearly apparent for the laser nephelometer and to a lesser degree is observed on the regulatory turbidimeter. In a limited fashion, the change in amplitude is also shown for the particle counter. The amplitude change is not as significant on the latter two instruments since part of the fluctuation is lost in the baseline noise. It is interesting to note that the laser nephelometer easily displays the increasing amplitude of the baseline. Figure 1 displays the effluent baseline fluctuations for laser turbidity and particle counting during an exceptionally quiet run. In most cases, the change in amplitude is far more dramatic.

Figure 1 WTP Filter # 12 Effluent Particulate Monitoring, May 5, 2000

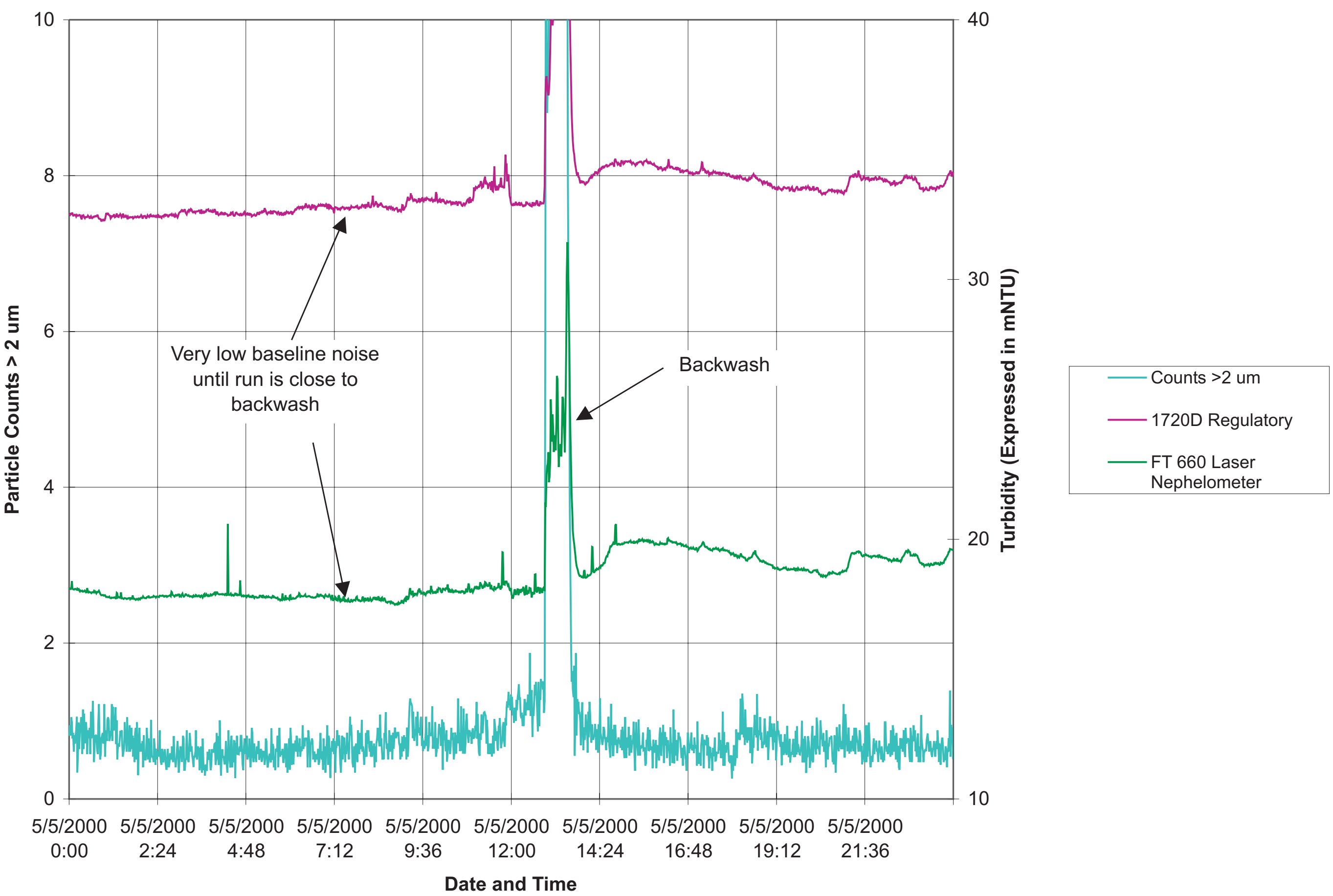
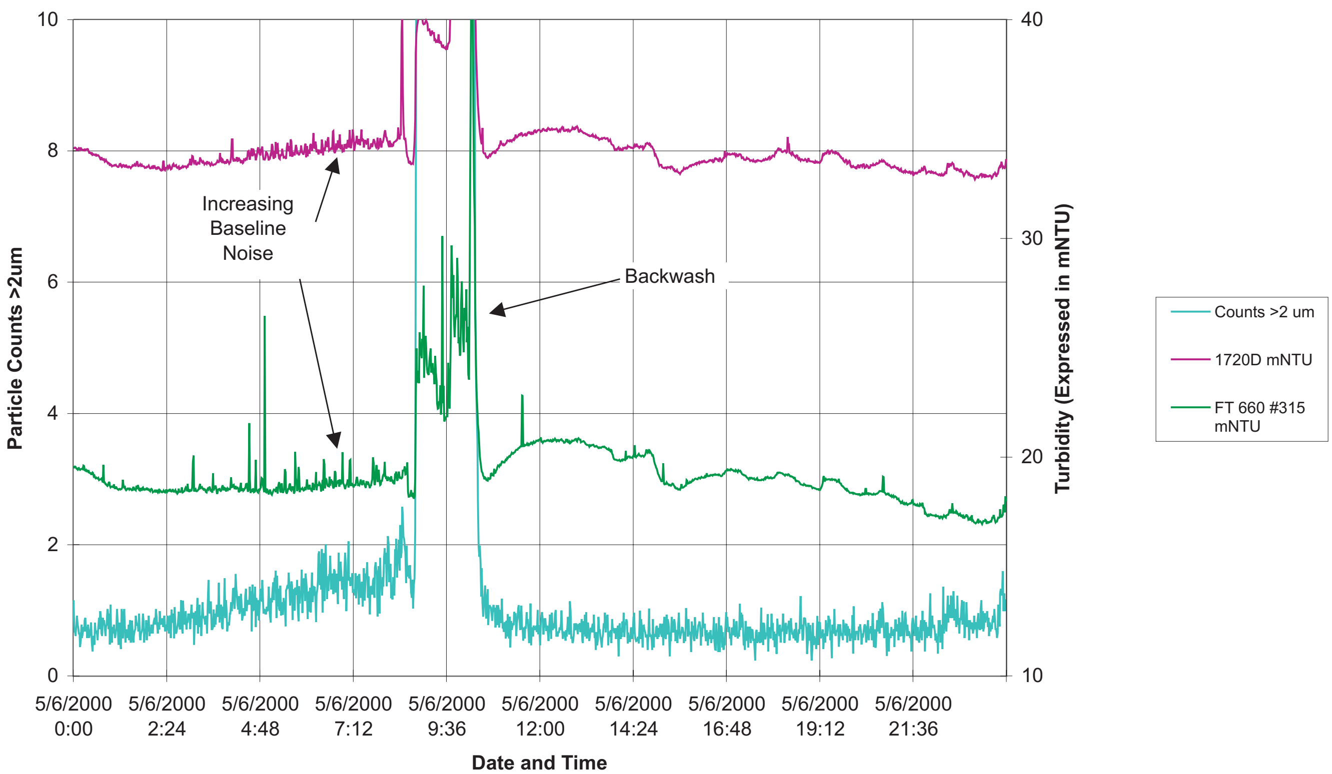


Figure 2 shows a more typical filter run (the next 24-hour monitoring period of the same filter shown in Figure 1). The filter effluent was monitored using the same three particle detection instruments. During this run, the turbidity amplitude increases dramatically as the run progresses. The particle count noise appears to increase slightly and shows a slight increasing trend in counts from a baseline of 2 to 3 cts/mL. The same minor upward trends are observed with the FT660 and regulatory instruments; however, it is the baseline noise that is dramatic.

One question that is to be considered is the cause of this progressively unstable turbidity baseline that correlates to filter run length. A possible explanation for this phenomenon could be linked to the optical design of the FT660 laser nephelometer and to the nature of particle scatter.

Figure 2 WTP Filter # 12 Effluent Particulate Monitoring, May 6, 2000



The Theory Relating to the Increased Amplitude of the Turbidity Baseline

The FilterTrak 660 optical design incorporates an incident light beam with a very defined, coherent, laser-light source. This beam has relatively no divergence when it passes through ultra-low turbidity water. The incident light that reaches the bottom of the sample chamber is absorbed into a special material, which virtually eliminates all internal incident light reflections. Because the residual stray light from this optical system is minimal, an exceptionally stable, low-noise baseline is produced. The incident beam has a small diameter, but contains a very high level of energy. A higher incident light beam power density results—much higher than in a traditional turbidimeter. The result is a substantially higher signal to noise ratio for the FT660. With a higher signal to noise ratio, the instrument sensitivity to ultra-low turbidity fluctuations (those less than 0.001 NTU) can be reliably detected. When this superior incident light system is coupled with a highly sensitive PMT detector, the sensitivity to light scatter from particles is further enhanced.[3]

Referring back to Figure 2, we observe an increase in the amplitude of the turbidity signal (baseline noise) as the filter run progresses. A plausible explanation for this noise is due to the detection of a relatively small number of larger sized particles that may be detaching from the filter media as the run progresses. The theory behind the mechanism of detachment is below.

A part of the process for the pilot plant and the full-scale water treatment plant was to incorporate a filter aid polymer as part of the filtration. The polymer essentially provides a "sticky" coating on the filter media that is created by the polymer charge. Polymers can be negative, neutral, or positively charged. In this study, the polymer charge was neutral. As the filtration progresses, the particles remaining in the settled water will, in theory, adhere to the "sticky" surfaces of the filter media and enhance the filtration process. At the beginning of a typical filter run this process is very efficient and nearly all of the particles adhere to the filter material. As the run progresses, more and more particles bind to the material until most of the binding sites are occupied. With fewer binding sites available, two scenarios can occur.

1. Since all the binding sites are taken, additional particles cannot bind to the media and so they begin to work through the media and eventually, into the effluent.
2. When the media is agitated as hydraulic forces are applied in the filter, some particles that are currently attached to the media and break free.

The result in both situations is that some of the particles will begin to work their way through the filter and with enough time, make their way to the effluent stream. The chance for particles either breaking off the media, or working their way through the filter becomes greater as the filter run progresses. Eventually, particles will begin to trickle through the filter in very low numbers, and it is these few particles that are being detected and seen as the increase in the amplitude of the baseline turbidity.

The reason the FT660 is so sensitive to low numbers of particles is due to the beam geometry and high light scatter efficiency of this instrument (described previously). As a particle moves into the incident beam, it may be of a size that is large enough to result in some detectable light scatter. Because particles are consistently moving in and out of the beam, the resultant baseline noise, such as that seen in Figure 2 increases. As more and more particles enter the effluent sample, the fluctuations will increase, but the overall baseline of the turbidimeter and the particle counter will increase. At this point, the filter is beginning to lose its effectiveness and an upward trend in turbidity and/or particle counts may be observed, indicating that filter breakthrough is approaching.

Study Goals

The application of particle sensitive instrumentation may be used to assess filter performance as the filter run progresses. This study will determine if either particle counters or laser nephelometers can be used to correlate the baseline turbidity or particle counting fluctuations to filter performance. Several process algorithms, that measure the standard deviation based on a fixed number of consecutive running measurements, will be used to assess and quantify the filter run performance. This algorithm will be referred to as the RSD algorithm (Relative Standard Deviation) and will be applied as a process calculation on both the laser nephelometer and the particle counting data from the filter effluent stream. In addition, the algorithm will be applied to the data to determine if it could signal the approach of a catastrophic filtration event, such as a breakthrough.

The final goal will be to apply those algorithms to both the full-scale filter data and the pilot scale filter runs. These pilot scale filter runs are runs where a filter breakthrough condition was actually observed. Again, we will determine if the algorithms can be applied to either particle counter or laser nephelometer data with a high level of success.

Materials and Methods

This study was separated into two phases. Phase 1 involved the use of a pilot-scale plant modeling a full-scale direct filtration plant. During this phase, three of the pilot filter runs concluded with a breakthrough of the filter. Phase 2 involved the collection and analysis of data from over 50 consecutive filter runs conducted at a full-scale water treatment plant in the summer of 2000. A standard filter run was selected from this collection of data and was used to determine if the same algorithms used in the pilot study could be used to quantify the performance of the full-scale filter run.

Phase 1

The pilot study was conducted at a direct-filtration water treatment plant for the city of Aurora, Colorado. The pilot plant is designed to simulate the direct filtration processes of this WTP. The water flowing through the pilot plant simulated 40 million gallons per day (MGD). The raw water first flowed into the pilot plant flocculation basin. As this water entered the basin, it was injected with three chemicals. The first is a polyelectrolyte cationic polymer (PEC) with a resultant concentration of 1.6 mg/L. The second chemical is alum at 7 mg/L, and the third is chlorine, with a concentration of 4 mg/L. After a detention time of approximately 1 hour, the flocculated water flows to two filters, labeled #3 and #4.

Filter #3 is a coarse dual-media filter. This filter is comprised of a 12-inch sand layer with a media diameter of 0.55 to 0.65 mm. Covering the sand layer is a 60-inch anthracite layer. The diameter of the anthracite particles is between 1.2 and 1.4 mm.

Filter #4 is a fine dual-media filter. Like filter #3, it consists of a 12-inch sand layer covered by a 60-inch anthracite layer. The diameter of the filter #4 media is slightly smaller—the sand diameter ranges from 0.50 to 0.60 mm and the anthracite particle diameter range is between 1.0 and 1.2 mm.

A total of three breakthroughs, referred to as A, C, and D, were successfully forced on these filters. Each filter was monitored through breakthrough using a laser nephelometer (the FT660), and a particle counter (2200PCX). Data was recorded at 1-minute intervals throughout the entire filter run. The logged data was pulled into a small SCADA system that was designed specifically for this pilot plant. From the SCADA system, data could be downloaded as a CSV file into an Excel®* spreadsheet where further analysis was conducted. Table 1 summarizes the mechanism used to cause each of the breakthroughs.

Table 1 Summary of the Direct Filtration Pilot Plant Breakthroughs

Filter Run/ Breakthrough	Filter – Date	Failure Mechanism
A	#3 – 6/9/01	Alum feed was stopped
C	#3 – 6/10/01	All chemical feeds were stopped
D	#4 – 6/10/01	All chemical feeds were stopped

* ® Copyright Microsoft Corporation.

For each of the runs resulting in breakthrough, the logged data was transferred to an Excel spreadsheet to verify that the instruments monitoring the effluent water did, in fact, see the breakthrough. Next, four different process algorithms (each based on relative standard deviation (RSD)) were performed on this data to determine if any of these algorithms could assist in predicting the breakthrough and further assess the effluent quality during the run. The RSD is simply the standard deviation based on a set number of measurements divided by the mean (average) of the same measurements. The resulting value was expressed as a percent. A total of four RSD algorithms were evaluated on the data. These were based the number of measurements taken to generate the RSD—3, 6, 12, and 20 measurements.

The algorithms were applied to the data in a way designed to simulate their use as a potential real-time application. These algorithms were used in a process format and recalculated the RSD value each time a turbidity or particle counter measurement was performed. This technique was created to attempt to demonstrate that statistical methods can be applied real-time to data as it is collected. Real time statistical results can be used to help judge a filter run's performance as the run progresses.

After the RSD calculations are performed on each of the filter runs, we will determine which algorithm (based on 3, 6, 12, or 20 measurements) is most suited for the assessing the noise of the filter run if it were applied real-time. The algorithm that shows the greatest sensitivity (largest RSD values) would be most suited for this assessment. Each algorithm will be tested to see if the breakthrough event could have been predicted. In addition, we will determine if the assessed data from either the particle counter or the laser nephelometer offers an advantage in evaluating filter performance and predicting breakthrough.

Phase 2

The full-scale water treatment plant is a 30 MGD plant that uses dual-media filtration. The treatment of the raw water involved flocculation, followed by lamella plate sedimentation. A non-ionic filter aid is applied to the filter after each backwash.

The filter effluent from each run was analyzed in triplicate by concurrently using three FilterTrak 660 (FT660) laser nephelometers. In addition, the effluent was monitored for total particle counts with a size greater than 2 µm. All four instruments were installed on the effluent stream in parallel, with flow settings that were within 10% of each other on the FT660 instruments, and a flow of 100 mL/minute on the particle counter. Prior to data collection, the laser nephelometers were calibrated using primary calibration standards.

During this study, over 50 filter runs were evaluated to determine if the baseline noise increases as the filter run progresses. From this data, the authors selected a total of seven consecutive filter runs to determine if the same algorithms used in Phase 1 of this study could be applied to this real-world data. The data was analyzed to determine if the algorithms are appropriate for use on this data and if so, which algorithms are appropriate for assessing the "noise" level in the effluent stream. In addition, the evaluated data was examined to see if any of these algorithms could be applied to the data to predict a filter breakthrough.

Results and Discussion – Phase 1

The plant operator initially observed that breakthroughs A, C and D were only detected by the particle counter. After further investigation, it was determined that this was due to improper scaling of the SCADA inputs from the laser nephelometer. The FT660 measurements were converted to the proper mNTU scale and the resolution of the nephelometer was correctly presented. After the corrections, the data showed that the breakthroughs were easily detected by the laser nephelometer.

The filter run in which breakthrough A occurred is displayed in Figure 3. This run is characterized by a defined ripening period at approximately 1400, followed by a steady-state filter run that lasts approximately 60 minutes. At 1500, the alum feed pump is halted to force a breakthrough condition. The particle counter and laser nephelometer both begin to observe the breakthrough at approximately 1534 and start to trend upward simultaneously. At 1554, the regulatory turbidity limit of 0.3 NTU (300 mNTU) is exceeded. The time when the regulatory limit is exceeded will used as the reference point for the breakthrough event.

The relative standard deviation (RSD) was calculated for both the turbidity values and the particle count values throughout the course of the filter run shown in Figure 3. The RSD values were calculated based on a set number of running measurements, which were used to calculate the average and standard deviation values. (These values were then used to generate the RSD value.) Four measurement averages were taken beginning with 3 (denoted RSD-3), 7 (denoted RSD-7), 12 (denoted RSD-12), and 20 (denoted RSD-20).

Figure 3 Filter Breakthrough Data –Run A – Aurora Pilot Plant, June 9, 2001

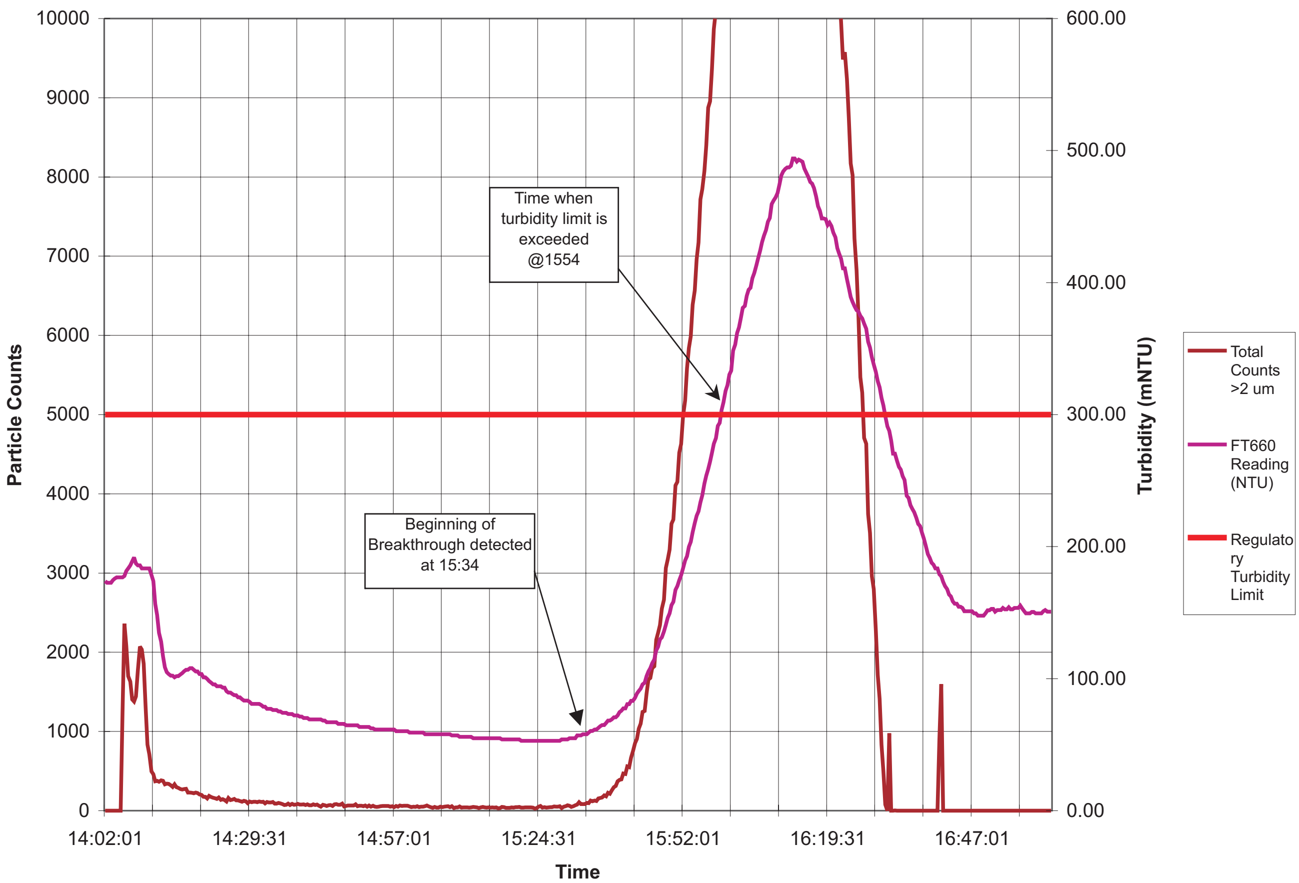
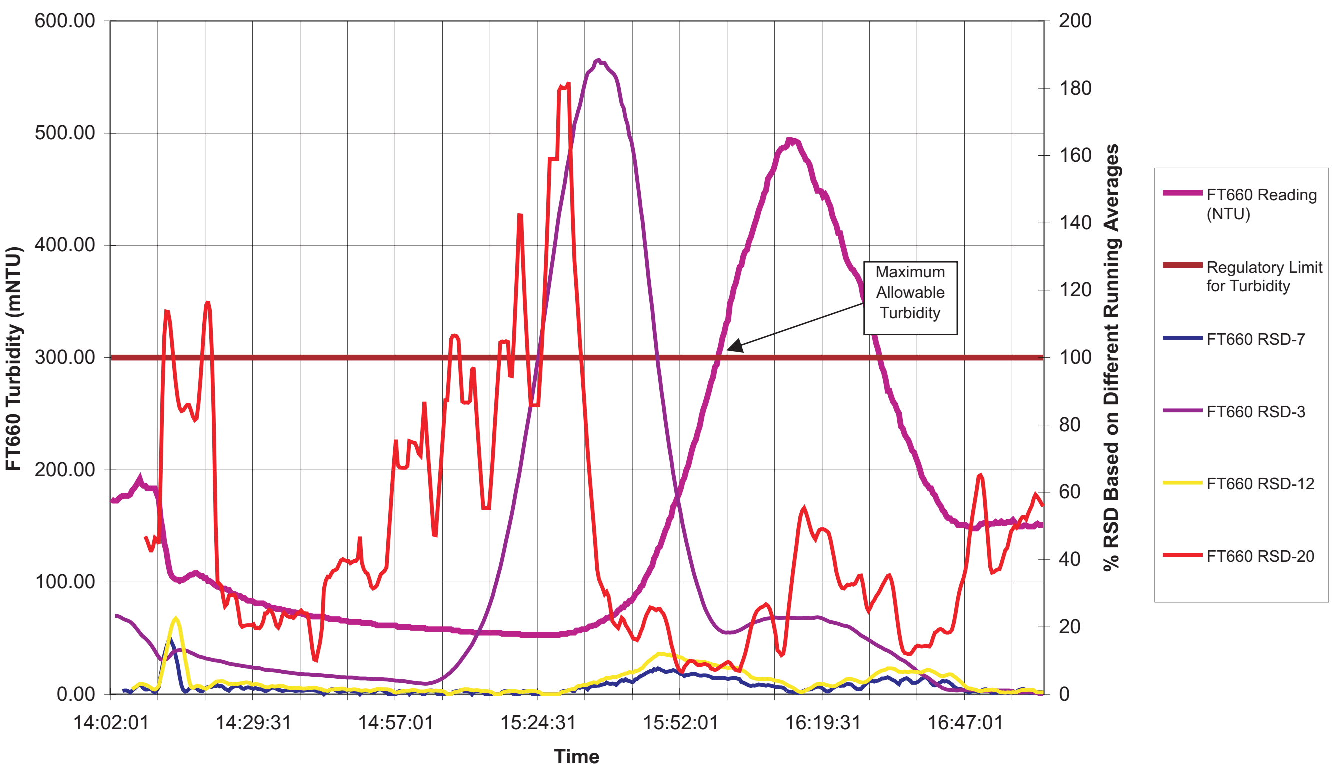


Figure 4 displays the RSD algorithms, which are overlaid with the turbidity measurements during the filter run. Figure 5 displays the plotted particle count data.

In Figure 4, the turbidity of the filter run is displayed on the left y-axis and the RSD algorithms are plotted on the right y-axis. Two dramatic RSD peaks, RSD-3 and RSD-20, are displayed prior to the breakthrough time of 1554. The RSD 20 peak resulted from turbidity changes during the ripening period and if using just this data would be interpreted as a false positive spike. The confusion is due to the use of a large number of samples to generate the RSD values. On the other hand, the RSD-3 peak appears to be more indicative of the actual breakthrough event. This RSD-3 peak does overlap the turbidity spike as well. Further, the beginning of the RSD-3 peak was approximately five minutes after the alum pump “failed” (at 1500). The magnitude of the RSD-3 peak indicates a significant change in the baseline noise, which may reflect the changes to the process stream from the pump failure.

The other RSD algorithms shown in Figure 4 (RSD-7 and RSD-12) also display far less dramatic peaks as a result of the breakthrough. The two algorithms begin to show a peak approximately 5 minutes before the start of the turbidity peak upward trend. In this case, the larger number of samples used to generate RSD values results in a dampening effect on the turbidity noise of the baseline. Because of this, these two algorithms were not effective in predicting the breakthrough. Overall, the shortest RSD algorithm was the only one that was beneficial in this application.

Figure 4 Turbidity Breakthrough Data — Using Relative Standard Deviation to Predict

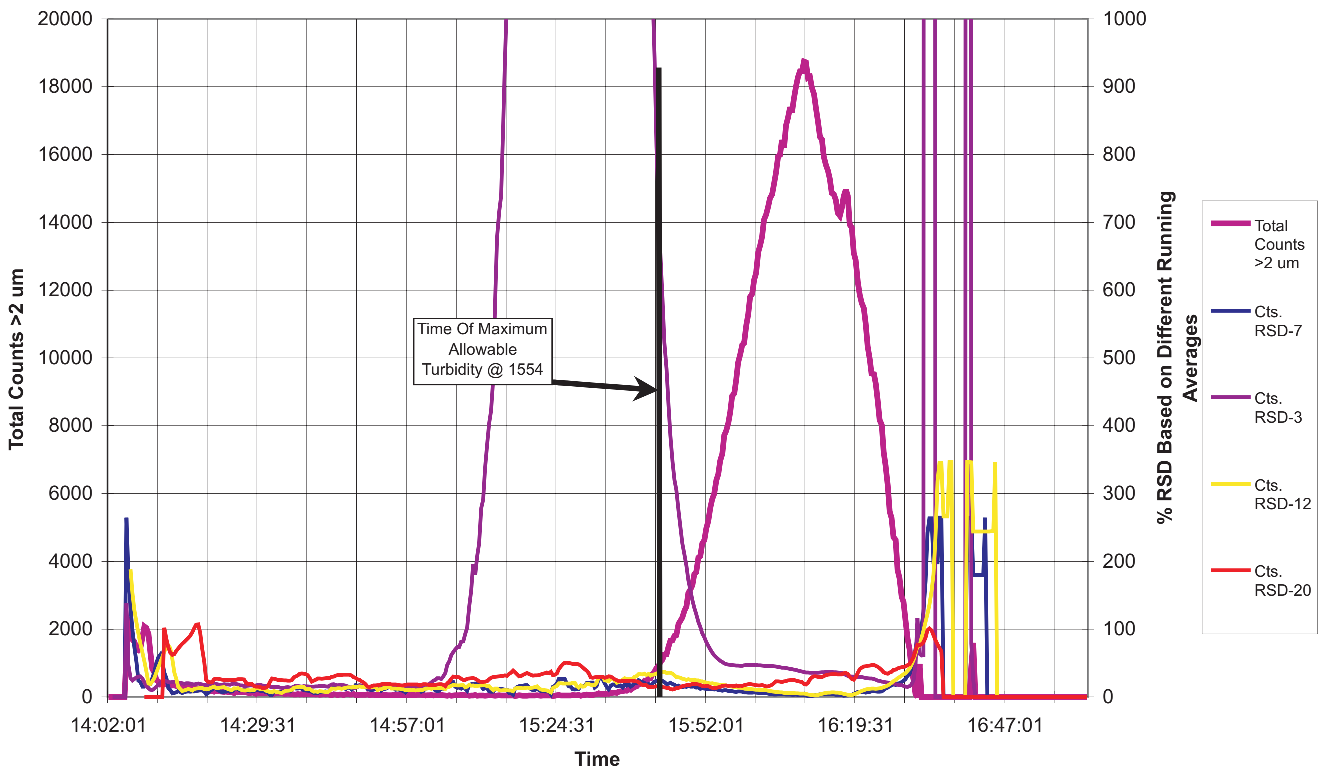


The particle count and RSD overlay is displayed by Figure 5. Here the total particle counts are on the left y-axis and the RSD values are plotted on the right y-axis. In this plot, only one algorithm, RSD-3, showed a significant RSD peak prior to the actual breakthrough at 1558. The RSD-3 peak is significant, the maximum value 7802 vs. a baseline value of 18. This peak began to trend upward within 2 minutes after the alum pump "failure; " an excellent indicator of an upset in the process before the actual breakthrough spike was seen.

The particle counter RSD algorithms from RSD-7, RSD-12, and RSD-20, are also displayed in Figure 5. Slight increases that coincide with the actual particle count spike are observed, but could easily be misinterpreted as baseline noise.

Similar results were observed with breakthroughs C and D. However, it was much more difficult to interpret their RSD spikes because of shorter steady-state conditions during these two filter runs. The shorter steady-states led to much higher RSD baselines and made determining distinct RSD peaks prior to breakthrough much more difficult for the RSD-7, RSD-12, and RSD-20 algorithms. The only algorithm that showed any consistency for both the particle counter and the turbidimeter was the RSD-3 algorithm. Using this algorithm resulted in significant RSD peaks before the turbidity and particle count values started trending up for the breakthroughs.

Figure 5 Particle Count Breakthrough Data — Using Relative Standard Deviation to Predict Breakthrough — Aurora Pilot Plant Run A, June 9, 2001



In summary of Phase 1, the pilot scale data allowed us to apply the RSD algorithms to data in a simulated real-time application. However, due to the short duration of the steady-state condition of two filter runs, only one reasonable application resulted. This filter run, designated A did show that the process algorithms could be applied and could assist in predicting the breakthrough without the chance of a false positive peak. The best algorithm to apply to both the laser nephelometer and the particle counter readings was the RSD-3.

The three filter breakthroughs were easily detected at the exact same time by both instruments that were monitoring the effluent stream. It is critical to appropriately scale the laser nephelometer analog output signal to allow full use of this instrument's detection sensitivity. Initially, the improper scaling of the laser nephelometer measurements led the operator to believe that the instrument was blind to the breakthrough.

Results and Discussion – Phase 2

In phase two we examined seven consecutive filter runs from the full scale WTP. During each of these runs, the effluent turbidity and particle counts were recorded at 1-minute intervals. The turbidity measurements were performed with the laser nephelometer (FT660). This instrument exhibits higher sensitivity and greater baseline stability than the traditional process instruments and allows the turbidity to be measured in mNTU units.

The same approach to the data used in Phase 1 was applied in Phase 2. The four RSD algorithms were applied to the full-scale plant data. After these calculations were complete, a plot was generated showing the RSD values overlaid on the turbidity measurements. Figure 6 provides an example of the overlay turbidity graph.

In Figure 6, the filter run for May 6, 2000 was again analyzed for its turbidity and the assessment of its respective baseline noise (See Figure 2). The left y-axis represents the turbidity and the right y-axis represents the RSD calculations of the four different algorithms, over the course of the filter run. For this run, the backwash event began at 1018. As the run approached backwash, the baseline turbidity became progressively noisier. Immediately after the backwash, the noise of the baseline recovers and remains relatively stable for several hours. The RSD values closely model this baseline noise. Regardless of the algorithm, the frequency of the RSD calculation increased along with the measurement prior to the backwash and consistently exceeded 2 percent. Just before backwash, the RSD algorithms spike dramatically, indicating a major change in the performance of the filter. After the backwash, the RSD algorithms became stable again and were consistently below 2 percent.

In summary, the RSD algorithms displayed a semi-quantitative magnification of the baseline noise exhibited by the actual turbidity measurement. The algorithms that were based on fewer numbers of measurements provided the greatest magnification of the baseline noise. This enhancement was consistent throughout the entire study.

Like the turbidity measurements, the particle counter measurements were also analyzed for the seven runs. The same filter run is displayed for the particle counter measurements in Figure 7 with the particle counts displayed on the left y-axis and the RSD algorithm calculations displayed on the right y-axis.

Figure 6 Filter Run Turbidity vs. Relative Standard Deviation for WTP Effluent

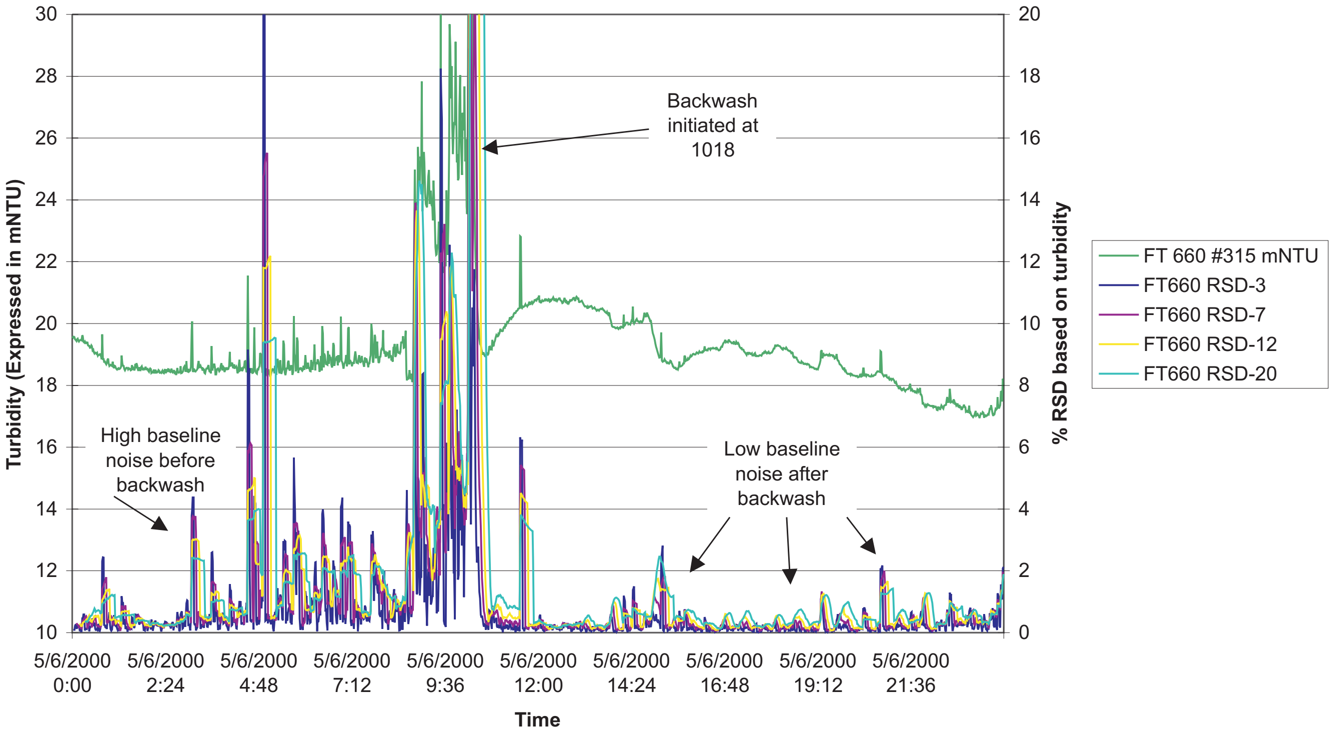
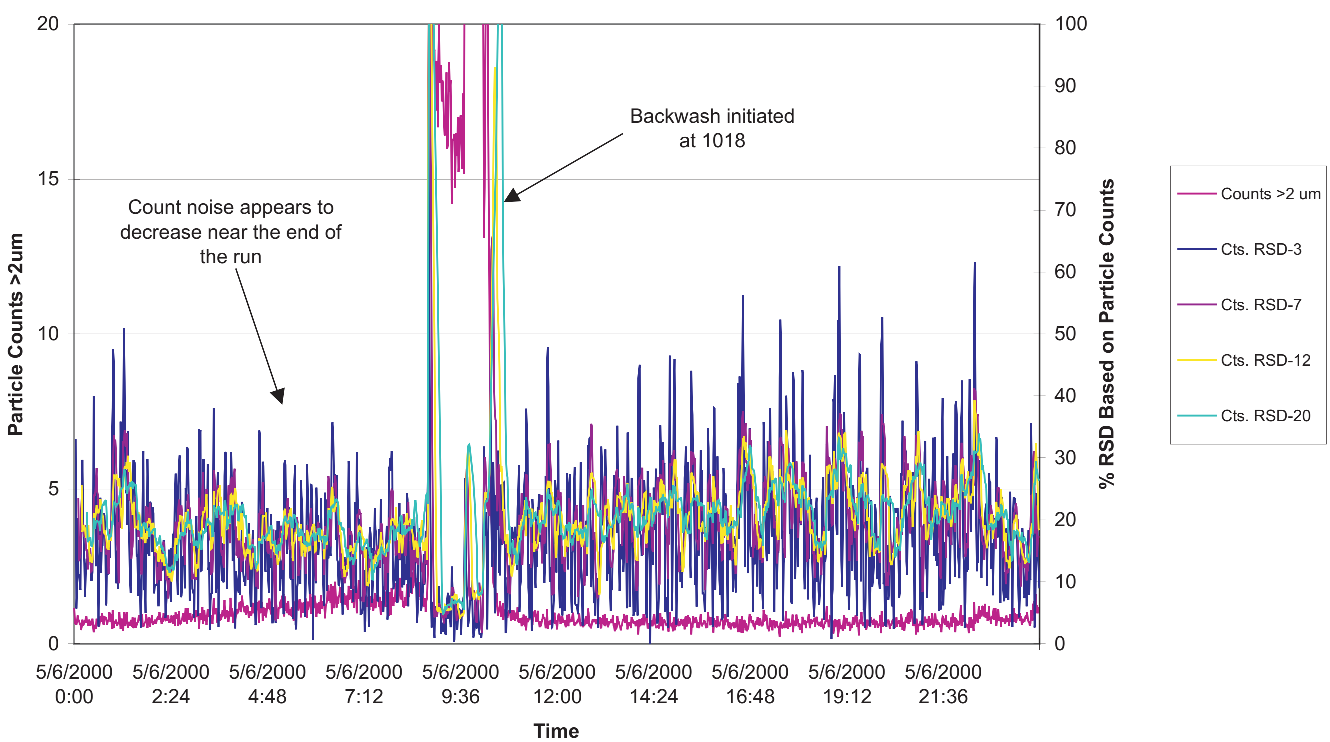


Figure 7 Filter Run Particle Counts vs. Relative Standard Deviation for WTP Effluent



Unlike the turbidity measurements, the particle counter baseline (in Figure 7) appears to remain very consistent for most of the filter run (until a few minutes prior to backwash). It is very difficult to observe an increase in the baseline noise of the particle counter as this run progresses. An increase in the baseline noise is also difficult to see in the RSD algorithms of the particle count data. The four algorithms exhibited significantly more noise than the algorithms generated from the FT660 data, and showed no growth in the amplitude of the noise as the run progressed. Basically, no correlation was found in any of the seven data runs analyzed. The particle counter baseline noise and the associated RSD algorithms did not show any correlation to the quality of the filter run.

Conclusions

During the first part of this study, the RSD algorithms that were applied to the effluent laser turbidity and to the effluent particle counts were demonstrated to be successful in helping to predict the breakthrough of a filter run. However this success was limited to the shorter measurement-based algorithms, namely the RSD-3 algorithms. These were the only algorithms that consistently produced significant spikes immediately before seeing the actual increasing trends in turbidity and particle count levels that preceded the breakthroughs. It is possible that these RSD-3 algorithms could predict oncoming particle events minutes before the actual changes are observed. The large measurement-based algorithms often resulted in false positive spikes due to the lack of a significant steady-state condition of the filter run prior to breakthrough. It was recommended that more breakthroughs be conducted on the pilot-scale plant, where the filter run times would be more representative of that of the full-scale filter run times.

The full-scale data consistently provided information regarding the baseline turbidity that could prove to be beneficial in the assessment of filter performance during the course of a filter run. In all the cases examined, the turbidity noise increased as the filter run progressed toward backwash. Then, after backwash, the noise immediately fell to a steady-state condition and remained there until the next run neared its backwash. This noise was easy to detect using the higher sensitivity nephelometer, but was much more difficult to detect consistently using the regulatory instruments.

The particle counter information did not exhibit the same cyclic trends between baseline noise and filter run time in the full-scale filter runs. As a result, the RSD algorithms did not exhibit any trend or correlation to this as the baseline changed during the course of the filter run. A possible explanation as to why the laser nephelometer appears to correlate and the particle counter appears not to, may be linked back to the theory discussed in the introduction of this paper and also, the particles that create the nephelometric baseline changes may be smaller than the 2-µm threshold limit of the particle counter.

In conclusion, the use of high sensitive laser nephelometry may provide another means of predicting filtration problems through the course of a filter run. The addition of a short measurement-based RSD algorithm will also help to magnify the significance of the baseline noise, which appears to be reflective of the filtration process. Thus, the amplitude of the noise can be used to help detect problems at their very infancy.

Acknowledgements

The authors would like to express their gratitude to the individuals who helped conduct this study. A significant amount of effort was put forth by the employees of the Wemlinger Water Treatment Plant in the maintenance, data collection, and other tasks associated with the pilot plant studies. Those efforts are greatly appreciated. Special thanks go to Leah Harrington, Daria Patterson, and Debby Diehl for their efforts in helping to complete this study.

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APPENDIX C Application Note — A Strategy for Optimizing Water Treatment Plant Preformance Using Light Scatter Technologies*

Executive Summary

Two new particle detecting technologies have been developed to help optimize filter performance at water treatment plants (WTP). One nephelometer was designed to give simple, accurate, and rapid response to turbidity changes during a backwash cycle and while monitoring the filter effluent. The other nephelometer was designed specifically to monitor filter effluent. During this study, the instrumentation was primarily used to monitor particle events during the WTP process. The ultimate goal is to optimize plant performance by identifying and reducing particle events that occur either before or after final filtration.

Optimization of the filter run was defined as the production of a stable effluent stream (characterized by low and consistent turbidity and low and consistent particle counts). Particle shedding from the filter into the sample was minimal for the duration of the run.

Two new instruments were used in this study, a laser nephelometer and a probe turbidimeter. The laser nephelometer, which is designed to detect very small changes in turbidity, was combined with a particle counter and regulatory turbidimeter on the filter effluent. The probe turbidimeter exhibits quick response and contains an 860-nm infrared light source, making it immune to color interference. The probe was positioned in the influent immediately above the filter. Collectively, this instrument distribution allows for more in-depth profiling of each particle event as it moves through the filter.

The study involved the participation of a local water treatment plant that is a member of the Partnership for Safe Drinking Water. For the past year, this plant has been heavily involved in the development and testing of this laser nephelometer. The data collected from that testing was used as a baseline for comparison to data generated in the study.

Site Profile

The water treatment plant where this study was conducted is located near Fort Collins, Colorado. A member of the Partnership for Safe Drinking Water, the plant has the capacity to produce 30 MGD using 12 filters. For the purpose of this study, a single filter was evaluated. The plant's current goal is to not exceed 0.1 NTU in the effluent, even during a backwash event. This plant's processes are under excellent control but the management and operators are interested in continuous improvement by further optimizing their filter runs.

During this study, the plant undertook an expansion project to increase its production capability to 50 MGD. Also, the raw water source, a reservoir, experienced significant change due to severe drawdown (draining of the reservoir). The geographical area supplied by the plant experienced significant drought conditions that required the plant to run at or near capacity for the duration of the study.

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* By Michael J. Sadar, Application Scientist, Hach Company.
Presented at the 2000 Water Quality Technical Conference, Salt Lake City, UT, 11/2000.

Introduction

Particle events are often viewed as surrogates for the quality of water produced in a WTP. Fewer events indicate a higher filter performance and therefore, better water quality. In this study, particle events detected by either a particle counter, laser nephelometer, or standard regulatory turbidimeter in the effluent stream were examined. Monitoring for particle spikes was performed at two points prior to filtration. A standard turbidimeter (1720C) was used to monitor the water as it exited the sedimentation basin and an OptiQuant™ Suspended Solids and Turbidity analyzer was used to monitor the water just before passing through the filter.

The instruments were strategically positioned in the treatment stream to help determine if spikes that were detected leaving the sedimentation basin traveled through the filter. If they did travel through the filter, the goal was to determine if the spikes changed before and after filtration. The magnitude and duration of each spike was also analyzed at different phases of the treatment process.

Laser nephelometers, regulatory turbidimeters, and a particle counter were used to monitor the filter effluent in an effort to determine if the instruments are complementary (which instruments identify the same particle event) or if they detect different events. This comparison of instruments provides WTP management insight into which instrument technology will help them optimize their filtration management. In addition, overall plant performance during plant expansion and geographical drought can be evaluated.

During this study, process monitoring was conducted for a total of 66 continuous filter runs on a single filter. The goal was to focus on the collection, preparation, and analysis of the data without impacting the day-to-day plant operation. All monitoring was passive and the data was analyzed after collection.

Four primary goals were set for this study:

1. To evaluate the role of different technologies in filter optimization and continuous improvement in this DWP.
2. To provide more insight into the WTP processes and the impact, if any, on particle events as they move through a water treatment process.
3. To determine which technologies are better suited for the detection of particle spikes before and after the filter.
4. To investigate the relationship between influent spikes and final effluent turbidity and particle counts.

Materials and Methods

Instrumentation

Three types of instruments were used for effluent monitoring event detection: a particle counter, a low-level regulatory-approved turbidimeter, and three laser nephelometers. All instruments monitoring the effluent were run in parallel with regular sampling.

- The 1900 WPC Particle Counter used in the study has size sensitivity down to 2 microns. For consistent and reliable application of the instrument, it was positioned on the effluent side of the filter.
- The regulatory turbidimeter was a Hach 1720D. The 1720D is commonly used in WTPs for regulatory filter effluent monitoring. This instrument meets all instrument design criteria specified by the USEPA method 180.1.
- FilterTrak™ 660 Laser Nephelometers were also used. These instruments are approximately 150 times more sensitive than traditional turbidimeters and will confirm particle events that might otherwise be interpreted as noise on a traditional low-level turbidimeter. The FilterTrak 660 measures turbidity in mNTU units (where 1 mNTU = 0.001 NTU).

Above the filter, two types of instruments were used:

- A Hach 1720C turbidimeter, owned by the WTP, monitored the sample as it left the settling basin.
- A new turbidimeter, the OptiQuant Suspended Solids and Turbidity analyzer, was installed on the settled water immediately above the filter. This probe design instrument utilizes ISO method 7027 design criteria for turbidity monitoring. Characterized by its quick response, the probe turbidimeter is often used for profiling events, including the turbidity of backwashes.

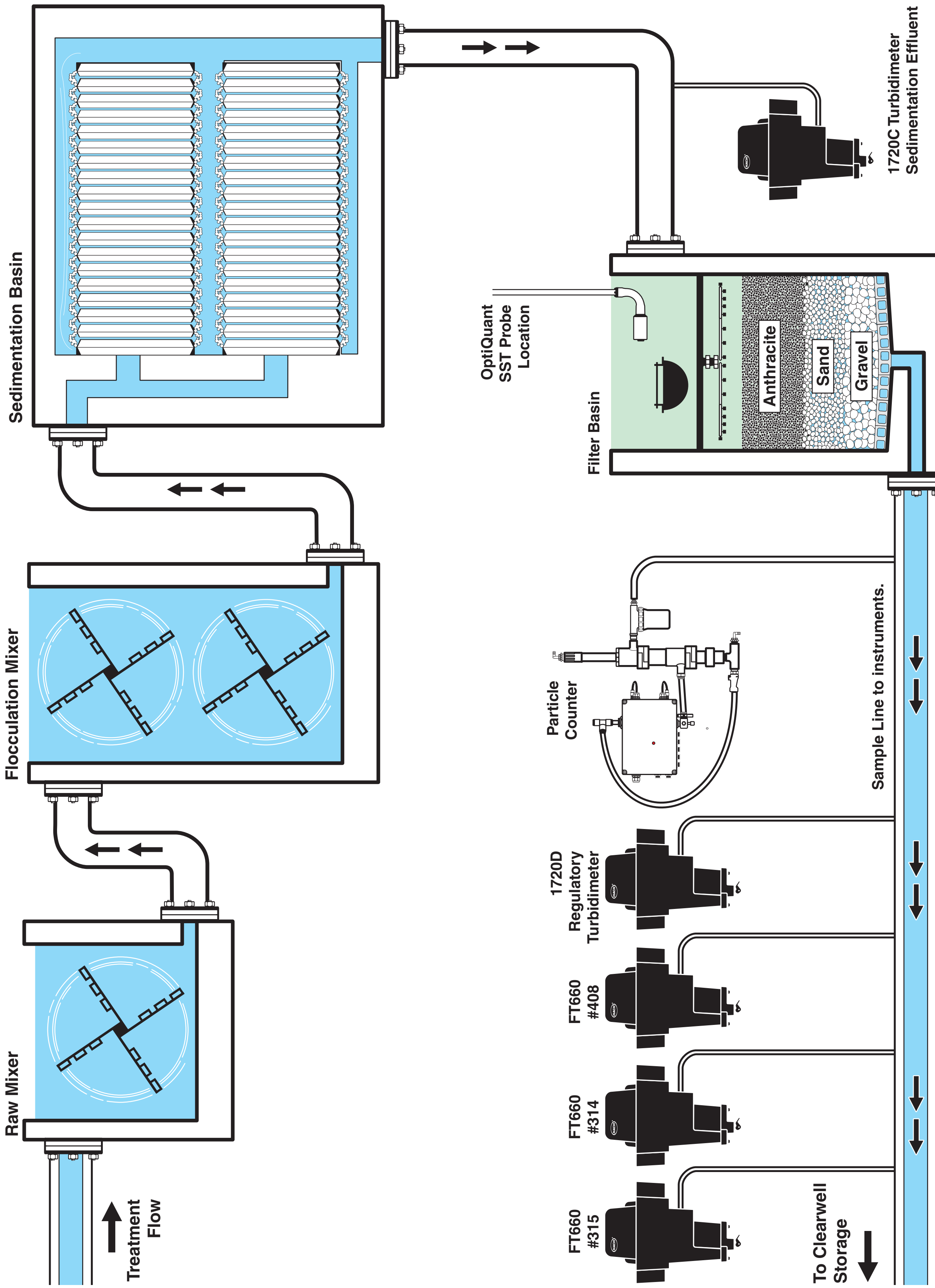
Table 1 summarizes the instrumentation used in the study. Figure 1 shows the strategic location of the instruments in this study.

All instruments were polled simultaneously at 1-minute intervals and data were logged to a computer using digital data networking protocol to minimize errors in measurement and transcription. Microsoft® Excel® was used to analyze and graph the data.

Table 1 Instrumentation Used in the Study

Location	Instrument	Primary Application
Filter Effluent	1900WPC Particle Counter	Counts and profiles particles that are >2µm in size
Filter Effluent	1720D Turbidimeter	Regulatory low-level turbidity
Filter Effluent	FilterTrak 660 SN 408	Low-level spike detection
	FilterTrak 660 SN 314	
	FilterTrak 660 SN 315	
Settled Water	1720C Turbidimeter	Turbidity in the 0.5–5 NTU Range
Applied to Filter	OptiQuant SST Analyzer	Fast Response in 0.3–1000 NTU Range

Figure 1 Instrument Location in the Water Treatment Plant



Redundant testing using three FilterTrak 660 Nephelometers was performed to increase confidence in the new technology, to confirm the detection of minor events, and to isolate interferences such as bubbles or contamination.

Once installed, all instrumentation was calibrated according to the manufacturer's instructions. After calibration, the instruments were allowed to run continuously from May 6 to July 15, 2000 until the 66 filter runs were completed. At approximately four-day intervals, the data was downloaded and analyzed for particle events and other significant criteria.

Particle Events:

Events are characterized as either major or minor. For this study, a major event is categorized as a turbidity spike that is greater than 5 mNTU and that lasts longer than 5 minutes. For particle counting, a major event is any sustained count spike that is greater than 2 counts per mL. Using this criteria ensures that bubbles are not identified as events. A turbidity minor event is any spike that is between 1 and 5 mNTU, or any change between 1–2 counts per mL above the baseline on a particle counter. These criteria only apply to the filter effluent. The events are summarized in Table 2.

Table 2 Particle Event Characterization in Filter Effluent

Instrument	Major Event	Minor Event
FilterTrak 660 or 1720D	>5 mNTU above baseline	1–5 mNTU above baseline
1900WPC Particle Counter	>2 cts/mL above baseline	1–2 cts/mL above baseline

Note: *Natural particle distribution follows a $1/d^3$ relationship with respect to number and size. Each order of magnitude decrease in particle size shows approximately 10^3 more smaller particles.*

Depending on which instrument detects the event, the event profile can be determined. If the event is seen only by the turbidimeter and not by the particle counter, the particles are assumed to be sub-micron. If the event is seen only by the particle counter, then the particles of that event are greater than 2 μm in size and exist in very low numbers. Events that are observed on both instruments indicate that the spike contains a natural size distribution after passing through the filter. An example of this distinction is shown in the data section.

Event detection using laser nephelometer technology required simultaneous detection of the particle spike by all three FilterTrak 660s in the study. When all three instruments detected the spike, the presence of the spike was confirmed.

Data

Once the data was collected and plotted, several pieces of information (metrics) were entered into a master matrix. These include the following:

- Run number (sequenced in chronological order)
- Run time: Time from the end of the ripening period to the start of backwash of the filter
- Date and time of the filter run
- Day of the week for the respective filter run
- Number of major and minor turbidity events

- Number of major and minor particle counter events
- Number of spikes in the filter influent
- Significant changes in baseline noise
- Baseline trends
- Peak turbidity value during backwash measured on the influent of the filter
- Measured turbidity value at backwash on the filter effluent stream

Using this master matrix we were able to quickly identify filter runs that contained events and compare them to filter runs that contained no particle events. From the perspective of this study, an ideal run is one that is free of particle events, trends, or significant baseline noise during the period of time starting at the end of the ripening period and ending at the start of the backwash cycle. In short, the run is stable. Events due to the backwash cycle or within the ripening period were not considered part of the filter run and are not included in the data. Figure 2 shows a "good" filter run, typical of the runs observed over the duration of this study. During this study, 66 percent of the runs were deemed good and had no particle events or trends over these runs.

Filter run termination is primarily determined by time at this water treatment plant. Termination occurs on a regular timed schedule if no breakthrough or loss of head occurs during the prior 20 to 26 hour time frame.

Of the 66 filter runs, a total of 22 showed at least one particle event. These 22 runs were tabulated into a scaled-down matrix that is summarized in Table 3 . The runs are ranked according to the number of total events detected starting with the highest number of events. Within this table, five of the runs had a precursor event that was detected in settled water prior to filtration. An asterisk identifies those runs.

Figure 2 Water Treatment Plant Filter #12 Effluent Particulate Monitoring (05/22/00)

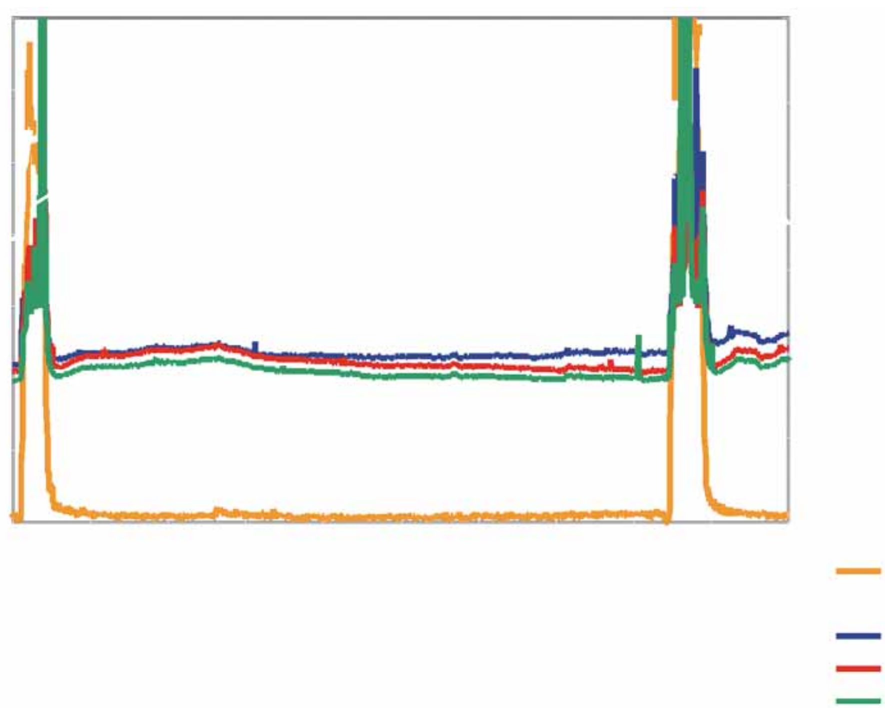


Table 3 Filter Runs showing at Least One Particle Event

EFFLUENT										
			Major Filter Events			Minor Filter Events				
Run	Run Length (hr.)	Total Number of Events	Particle Counter	FT660 #315	1720D	Particle Counter	FT660 #315	1720D	Event Date(s)	Backwash Termination Turbidity (NTU)
21*	24.98	3	3	1	1		2	2	6/1/00	1.74219
54*	24.30	3	—	1	1	2	2	2	7/5 & 7/6	1.10880
58	19.29	3	1	—	—	2	3	3	7/9/00	000.000
2*	26.8	2	2	2	2	—	—	—	5/8/00	0.99504
5	27.05	2	2	—	—	—	2	2	5/17/00	1.48070
22*	31.55	2	2	—	—	—	2	2	6/2/00	0.81269
49	23.95	2	—	—	—	2	2	2	7/1/00	1.05519
52	20.72	2	1	—	—	1	2	2	7/4/00	1.13972
1	20.33	1	—	—	—	1	1	1	5/7/00	1.11836
4	21.23	1	—	—	—	1	1	1	5/16/00	1.17717
12	17.28	1	—	—	—	1	1	1	5/23/00	1.54982
14	22.04	1	1	—	—	—	1	1	5/25/00	1.05917
15*	26.33	1	1	—	—	—	1	1	5/26/00	1.07300
25	22.87	1	—	—	—	1	1	1	6/5/00	1.17753
32	23.19	1	—	—	—	1	1	1	6/13/00	0.94590
34	26.04	1	1	—	—	—	1	1	6/15/00	0.93263
36	27.63	1	—	—	—	1	1	1	6/17/00	0.78922
38	24.79	1	1	—	—	—	1	1	6/19/00	0.69477
44	24.94	1	1	—	—	—	1	1	6/25/00	0.85205
46	35.52	1	—	—	—	1	1	1	6/28/00	0.87059
59	16.53	1	—	—	—	1	1	1	7/10/00	1.05735
63	20.53	1	1	1	1	—	—	—	7/13/00	1.10095

* Runs with an event in the filter influent and in the filter effluent that were detected by the nephelometric turbidimeters 100% of the time and by the particle counter 60% of the time.

Figure 3 and 4 show filter runs that have either particle events or excessive noise (when compared to the criteria of a good filter run). Figure 3 shows that the events (at 6:30, 8:40, and 9:45) are detected by each of the instruments on the effluent stream. However, the last event before backwash (at 9:45) is detected earlier on the turbidimeters than on the particle counter. This indicates that the sub-micron particles (detected by the turbidimeters) are precursors to larger particles (detected later by the particle counter).

Figure 4 does not distinguish separate events, but the baseline noise is substantial throughout the run and the particle count trends do not follow the turbidity trends. When comparing all the runs in the study, this run stands out due to the high level of noise and lack of complementary data on the instruments. Reviewing the log books may lead the operator to the cause of the noise.

Figure 3 Water Treatment Plant Filter #12 Effluent Particulate Monitoring (05/31/00-06/1/00)

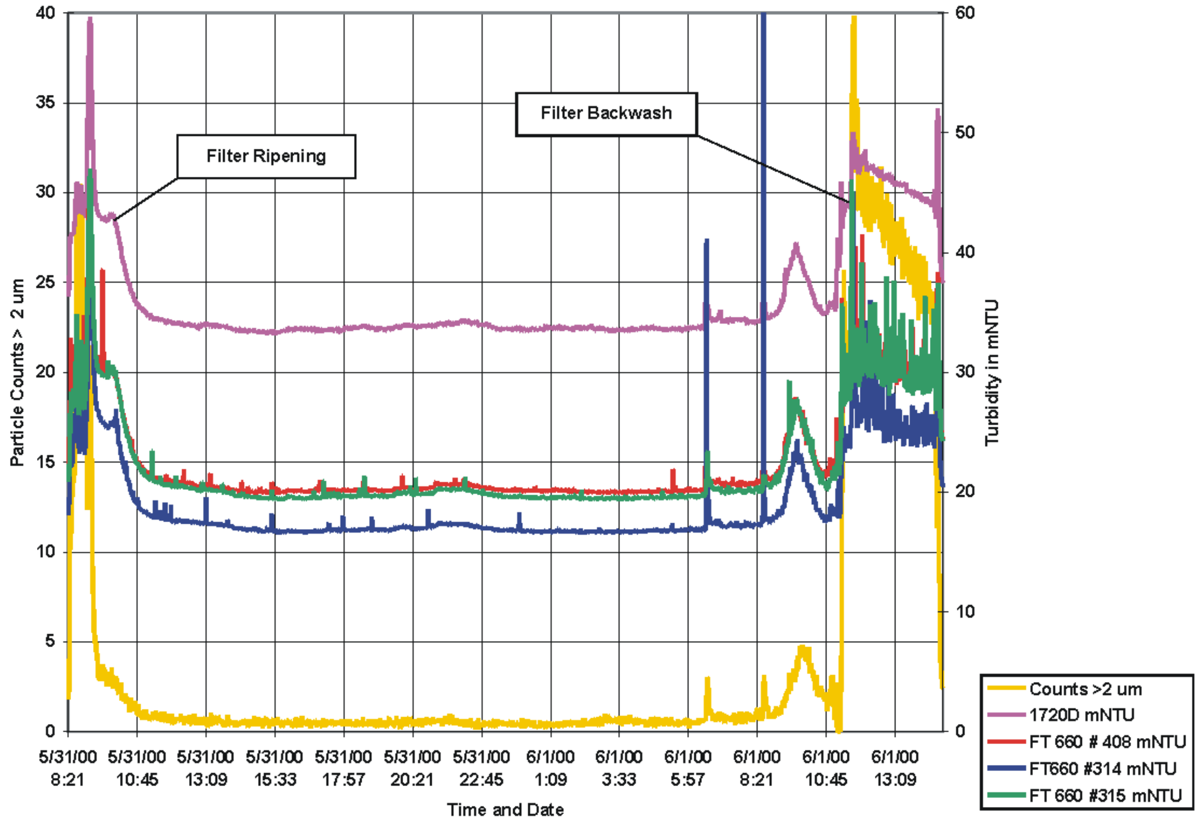
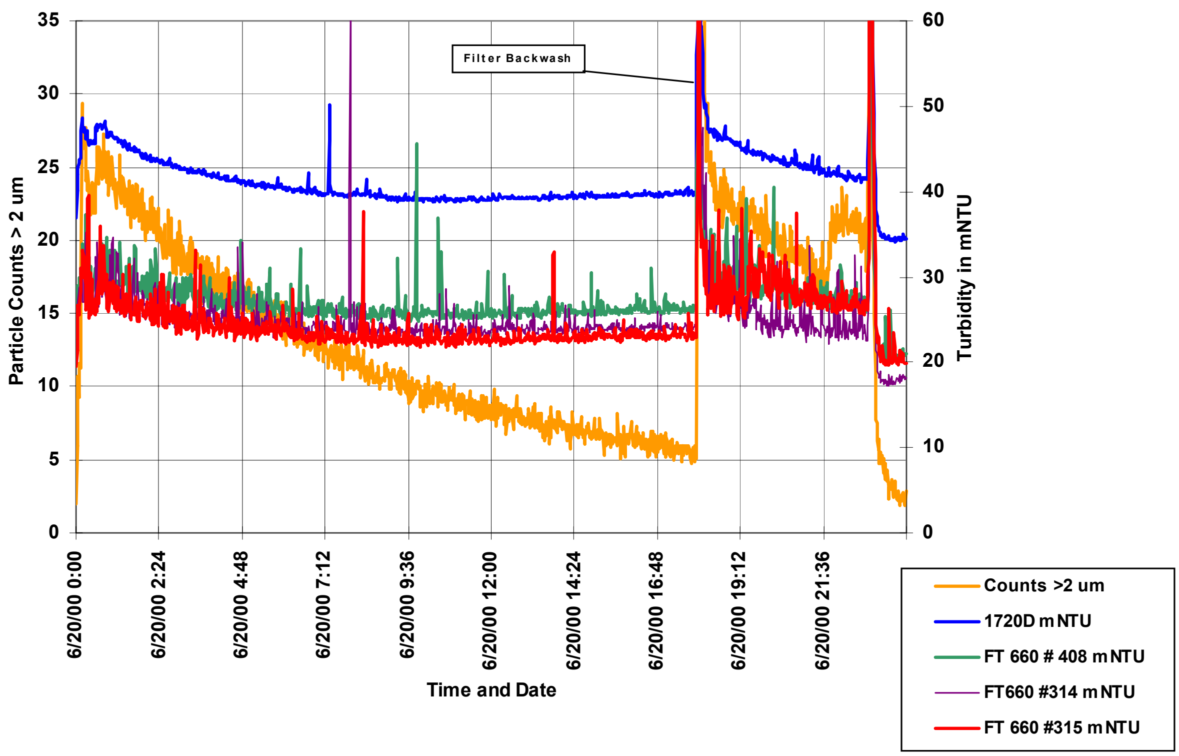


Figure 4 Water Treatment Plant Filter #12 Effluent Particulate Monitoring (06/20/00)

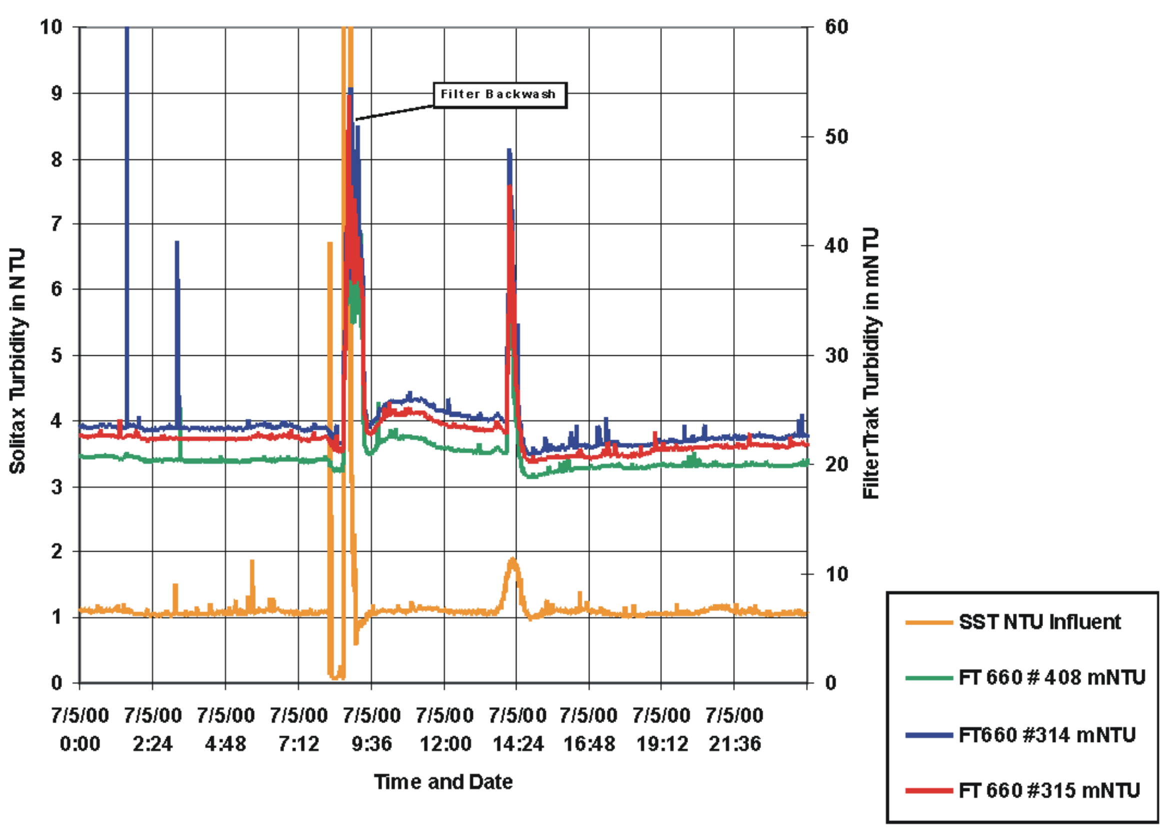


Of the 22 runs that contained particle events, five of the runs contained influent spikes that could be detected in the effluent as well. In all five cases where the spike is detected above the filter, a similar spike in the effluent is seen within the next couple of minutes. Figure 5 shows that the particle event in the influent appears to be a precursor to the particle event that is immediately observed in the effluent with the turbidity technologies. During this run, the spike (at 13:50) in the settled water immediately above the filter increased from 1.2 to 1.9 NTU, a 0.7 NTU increase. The effluent event increased approximately 0.02 NTU, indicating that the filter did remove the majority of this spike. In all five cases, particle spikes that were observed above the filter were easily detected by the laser nephelometer.

Since both the OptiQuant SST and the FilterTrak 660 Nephelometer are calibrated using formazin, the light source differences between the two instruments are minimized. Positioning the instruments on both the influent and the effluent sides of the filter allows log removal calculations to be performed based on the turbidity differential across the filter. Color interferences in the influent are eliminated by the 860-nm wavelength of the OptiQuant SST Probe Turbidimeter. Color is not an interference in the low turbidity levels of the effluent stream.

Several spikes that were recorded at the settled water basin by the 1720C turbidimeter were not detected by the instrumentation downstream from this sample point. Hydraulic surges are the suspected cause and these events are short-lived. The particle spikes that were investigated in this study were those that are tracked through the filter into the effluent.

Figure 5 (07/05/00) **Water Treatment Plant Effluent Particulate Monitoring and Settled Water Applied to Filter #12**

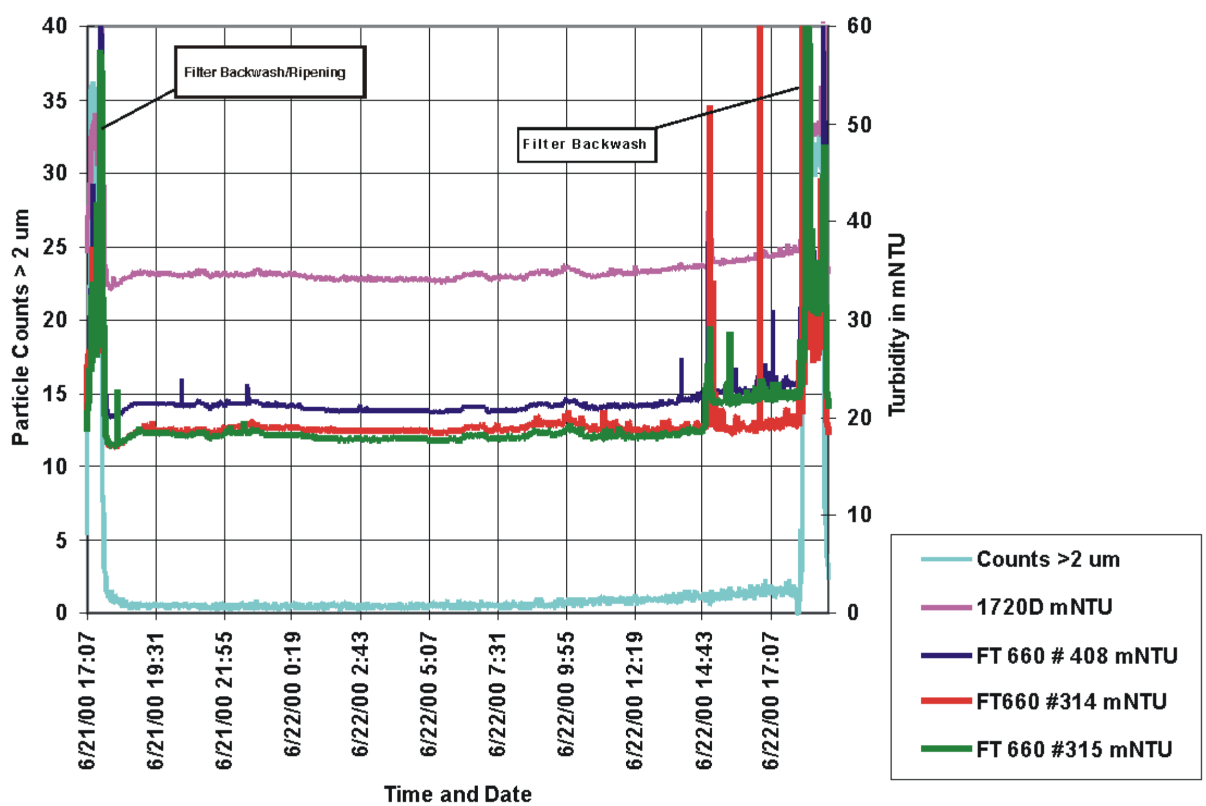


In the majority of the 66 filter runs in this study, an additional occurrence was seen. At the beginning of a typical good run, the FilterTrak 660 baseline showed very low noise. The low noise is maintained until the run is between 65 and 75 percent complete. The noise appeared to increase dramatically as the run progresses toward termination. Figure 6 shows a typical run in which the three FilterTrak 660 Nephelometers all display the same magnitude of baseline noise throughout the run. For 10 randomly selected “good” filter runs (defined as runs without spikes), we looked at the relative standard deviation for the first 75 percent of the run compared to the last 25 percent of the run up to backwash. The baseline relative standard deviation for the last 25 percent of a filter run increased 2.35 times the baseline relative standard deviation over the first 75 percent of the same run.

It is speculated that large particle detachment from the filter media may be the cause of the increase in background noise as the filter run progresses. If this is true, then monitoring background noise may be another means of predicting breakthrough.

When looking at Figure 6 it is interesting to note that there is one particle event that is detected by the turbidimeters, but is missed by the particle counter (at 15:00). This indicates that the event is primarily sub-micron and is below the detection threshold detection limit of the particle counter.

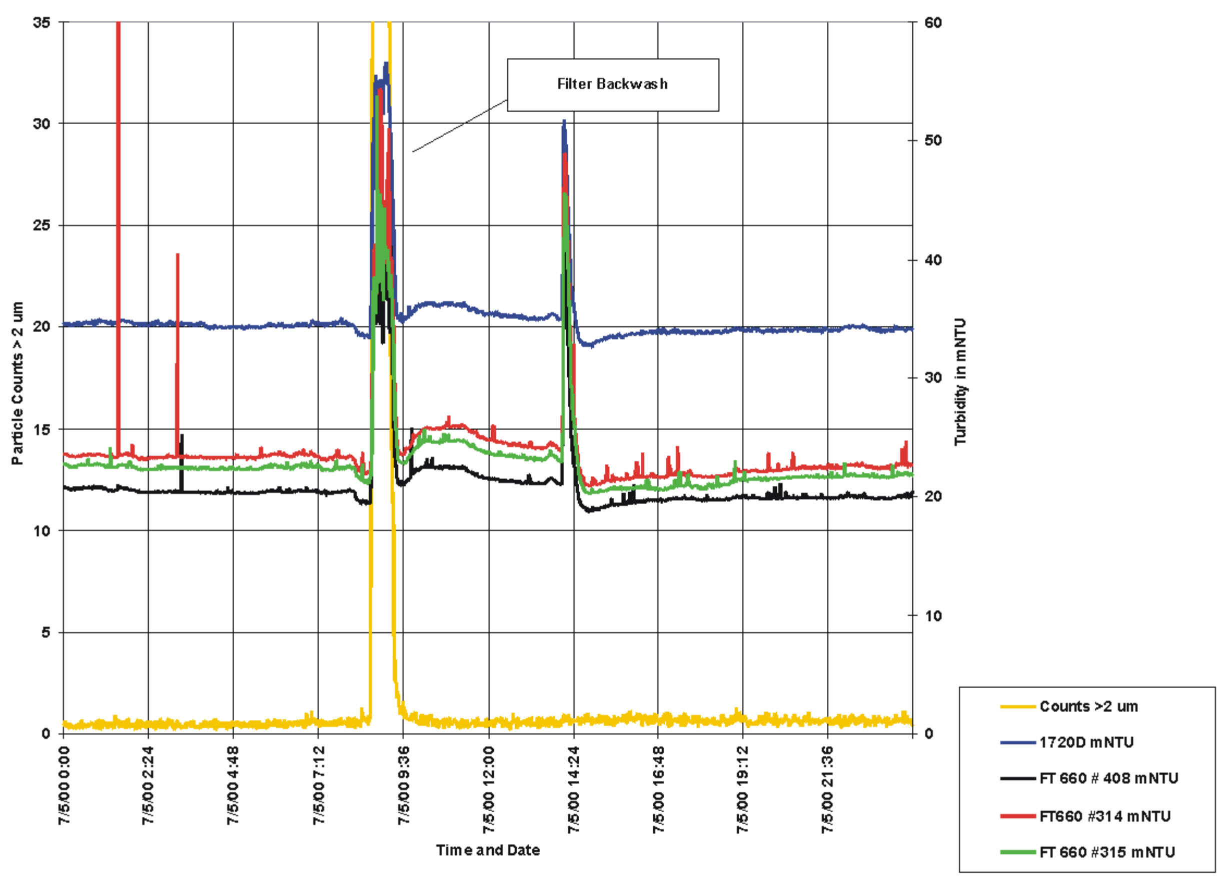
Figure 6 Water Treatment Plant Filter #12 Effluent Particulate Monitoring (06/21/00–6/22/00)



In the large majority of the filter runs logged, the two technologies—turbidity and particle counting—complemented each other when detecting events in the filter effluent. However, in a couple of cases, events that were detected by the turbidimeters were totally missed by the particle counter. Figure 7 is the same filter run displayed in Figure 5 but includes the data from the particle counter. This figure shows that the precursor event detected above the filter was seen by the effluent turbidimeters, but was not detected by the effluent particle counter. Because the particle counter missed the event, we can surmise that this event is sub-micron in nature.

Correlations between the turbidity value at the termination of backwash and filter events were also examined. The turbidity values at backwash termination were in a very narrow range between 0.69 and 1.74 NTU. It can be speculated that the tight range of values at backwash termination predicts the overall consistency of the filter runs over time. No correlation was found between these values and particle events. The correlation between the peak turbidity at backwash and subsequent filter events of the proceeding run was also investigated. Again, there was no correlation between these two parameters.

Figure 7 Water Treatment Plant Filter #12 Effluent Particulate Monitoring (07/05/00)



Conclusions

Of the 66 continuous filter runs that comprise this study, 33 percent contained particle events as they were defined at the beginning of this study. Of the 22 runs that contained events, 23 percent appear to have a precursor event detected by the OptiQuant SST probe turbidimeter that was monitoring the pre-filter sample.

The FilterTrak 660 detected the majority of events that the other effluent instruments detected and also detected some events that they missed. In addition, the FilterTrak 660 baseline standard deviation increases as the run progresses. This may be a precursor to breakthrough and warrants further investigation.

Events that are detected in the pre-filtered water were also consistently seen by the FilterTrak 660 and particle counter. All five settled water precursor events were also seen in the effluent. The turbidity of these influent spikes, which range between 0.5 and 2 NTU, were reduced significantly as they passed through the filter. The resulting events in the effluent were very small with turbidity changes ranging between 0.005 and 0.030 NTU (5-30 mNTU) and the finished water was maintained far below the requirements of the Partnership for Safe Drinking Water.

The impact on construction and the seasonal drought in the area did not appear to correlate to the frequency of events. Runs with events did occur in an apparently random order during the 66 runs.

As was discussed earlier, having both a particle counter and a laser nephelometer provides information as to the composition of a particle event. Event detection that is complemented by both instruments indicates a natural distribution of particles roughly following the $1/d^3$ relationship. In these cases, the nephelometer will detect the particles slightly before the particle counter because small particles move more rapidly through a filter. If only the laser nephelometer detects the event, the composition of the particles is most likely sub-micron in nature. If the particle counter alone detects the event, this indicates a non-natural distribution of large (2 µm) particles and may indicate a change in the conditions within the filter or a contamination issue. In all cases, the use of two instruments provides further insight into the particle sizes of respective events.

This WTP filter effluent did not exceed the Partnership turbidity limits throughout the entire study (including backwash runs). However, the instrumentation did show both good, clean filter runs along with runs with definitive particle events. Though it may be challenging, the WTP management can investigate their logs to see if the runs that contained events relate to any changes in the treatment upstream of the filter. This is continuous improvement at its best.

Note: *RSD Parameter is part of the FT660 sc and can be used for enhanced sensitivity to impending particle spikes. The study discussed in Appendix B was the foundation for implementing the parameter. The RSD parameter can serve as a critical tool for optimization of a particle run and in predicting particle spikes.*

The WTP showcased in this study is, in reality, a best-case scenario. Its processes were optimized for the duration of this study and are under very tight control at all times. However, a WTP that does not have consistent filter runs, or one that often has particle spikes could use this instrumentation to detect, analyze and eventually reduce or eliminate such events. The intent and anticipated use of the study instrumentation goes beyond regulatory requirements and will help plants achieve production of water characterized by high quality and consistency.

We plan to continue monitoring this filter for the benefit of the plant management. Due to structural problems on the dams for the raw water source, the source will be drained significantly throughout the summer and fall of 2000. We will continue to see if changes to the raw water source have an impact on particle event occurrence at this sample site.

NOTES

The late **Clifford C. Hach**, founder of Hach Company, was a graduate of Iowa State University. Widely respected in the water analysis industry as an inventor, progressive innovator and research scientist, Mr. Hach held numerous patents and many of his papers appeared in technical industry publications.

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