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**ENVIRONMENTAL APPLICATIONS
OF THE
PARTICLE ANALYSIS SYSTEM**

E. J. Moritz and C. R. Hoffman

Rocky Flats Plant
P. O. Box 464
Golden, Colorado 80402-0464

U.S. DEPARTMENT OF ENERGY
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SUBJECT DESCRIPTORS

Filtration
Particle Counting
Water Monitoring
Water Treatment

EG&G ROCKY FLATS, INC.
ROCKY FLATS PLANT
P. O. BOX 464
GOLDEN, COLORADO 80402-0464

PREPARED UNDER CONTRACT DE-AC04-90DE62349
FOR THE
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ABSTRACT

This study demonstrates the applicability of particle counting technology for analysis of various water treatment systems at the Rocky Flats Plant. The Particle Analysis System described in this study determined the water quality of samples from environmental remediation, stormwater treatment, and drinking water treatment operations. Samples were measured in either discrete or on-line mode. This data showed filtration efficiencies, particle counts, particle size distributions, and real-time treatment system performance.

Particle counting proved more sensitive than the turbidimetric measurement technique commonly used by the water treatment industry. Particle counting is a two-dimensional measurement of counts and sizes, whereas turbidity is a one-dimensional measurement of water clarity. Samples showing identical turbidities could be distinguished easily with the Particle Analysis System.

The Particle Analysis System proved to be an efficient and reliable water quality measurement tool, and it is applicable to a variety of water treatment systems at the Rocky Flats Plant.

INTRODUCTION

The Rocky Flats Plant is a Department of Energy facility in the nuclear weapons complex located approximately 16 miles northwest of Denver.¹ Water discharges are currently regulated by the National Pollutant Discharge Elimination System permit issued under the Clean Water Act and the Agreement in Principle between the Department of Energy and Colorado Department of Health.

These discharges have been an ongoing source of concern to the public.² In a continuing effort to improve water quality monitoring, the operating contractor (EG&G Rocky Flats, Inc.) is assessing advanced technology in particle counting.

This study reports current progress on development of the Particle Analysis System (PAS) and its application to water treatment systems at Rocky Flats. This report meets requirements of the "Proposed Real-Time Monitoring Methodology" required by the "Workplan for the Control of Radionuclide Levels in Water Discharges from the Rocky Flats Plant."³ The Workplan was prepared in response to Section XII of the Statement of Work to the Interagency Agreement between the Department of Energy, Environmental Protection Agency, and the Colorado Department of Health.⁴

Filtration is a chemical-free method of water treatment for removing certain waterborne contaminants from Rocky Flats surface waters. Radiochemical contaminants are of particular concern; removal methods and removal measurement receive a great deal of attention. Because radiochemical contaminants are thought to be associated with colloids and particulates in the micron to sub-micron size range, filtration has been the central focus of a radiochemical removal strategy.

With no real-time analytical methods available for directly monitoring radiochemistry at environmental (sub-picocurie per liter) levels in water, Rocky Flats considered using indicator parameters to provide continuous control of water quality and water treatment processes. Early results of laboratory-scale studies by Los Alamos National Laboratory indicate that filtration through a 0.45- μ filter produces measurable reduction in levels of

plutonium and americium in water.³ One potential parameter is turbidity, commonly measured in drinking water treatment plants to indicate water quality and treatment effectiveness.⁵ Drinking water regulations generally require turbidity measurements less than 0.5 nephelometric turbidity units (NTU) in finished water.⁶ This is the lower range of turbidity measurement sensitivity, and water utilities are increasingly recognizing that this simple surrogate measurement does not give enough information to optimize water treatment.

Particle counting technology, the subject of this study, is well developed for industrial, electronic, and medical applications using readily available commercial products and reasonably understood methods.⁶ Compared to turbidity for monitoring water clarity, particle counting provides more information and greater sensitivity since particles are individually sized and enumerated.⁷ This method produces accurate, precise information below the range of turbidimetric sensitivity.

Particle count and size distribution data have broad applicability for water treatment process control, design, and selection, as well as monitoring of drinking water quality. Other benefits include: measurement of filter efficiencies, chemical process optimization, chemical cost savings, waste sludge minimization, and capability to make real-time measurements.⁸

Like turbidity, particle counting fails to provide a direct measure of radionuclide activity. However, the water treatment process can be monitored for the particle size range thought to be associated with the majority of radiochemical contaminants on an individual sample basis (discrete sample mode) or on-line basis (real-time continuous flow mode). Thus, filtration efficiency can be determined and potentially correlated to the reduction in radiochemical activity.

This study reports the development, implementation, and environmental applications of the PAS for Rocky Flats water treatment systems.

EXPERIMENTAL

Technology

Particle counters are used for counting and sizing particles in process fluids. Samples can be analyzed individually as bottled discrete samples or by on-line monitoring of flow in real time.

The basic components of a particle counter are the sensor (signal generating/detecting device) and the counter (data processing device). For discrete analysis, a batch sampling device is needed. For on-line analysis, a flow controlling device is required.

For analyzing particulates in water, particle size is usually expressed in terms of diameter in micrometers (μ). The implied unit is in equivalent spherical diameter because spherical particles are used in instrument calibration.

Instrumentation

A literature search and several laboratory demonstrations led to selection of equipment for this study. Demonstrations of filter testing, laboratory measurements, and instrumentation were provided by:

Pine Valley Water Treatment Plant,
Colorado Springs, CO

Pall Filter Corporation, East Hills, NY

Interbasic Resources, Grass Lake, MI

3M® Filtration Products, St. Paul, MN

During initial experiments, we used certain equipment already on hand, including a computer, printer, and Hiac Royco 4100 six-channel counter. We evaluated samples from Rocky Flats surface water detention ponds, operable units, and drinking water treatment plant.

After the preliminary evaluation, certain items were replaced to provide a more powerful, compact, portable PAS (Figure 1) suitable for relocation to remote sampling sites. This system now contains the following items:

Counter:	Hiac Royco 9064. Provides up to 512 sizing channels.
Sensor:	Hiac Royco HRLD-150
Batch Sampler:	Hiac Royco 3000
Software:	Hiac Royco Particle Distribution Analysis Software. Allows sophisticated data manipulation and display options.
Computer:	Modgraph GX-2386C-SX. Compatible with counters; provides ample data storage options in a compact, rugged enclosure.
Power System:	SOLA Mini-UPS. Supplies portable, uninterruptible backup power at remote locations.
Rolling Cart:	Provides portable desktop and houses instruments.

The sensor (Figures 2 and 3) measures particles using the light extinction principle, also referred to as light blockage or light obscuration particle counting.

To date, this particle sizing method has been the most frequently used technology in the field of drinking water treatment. As the sample flows through a sensing zone, a beam of known intensity emitted from a laser diode source shines across the sensing zone to a photodetector. When a particle passes through this zone, some light is scattered and some is absorbed by the particle, so the light intensity reaching the photodetector decreases. The amount of decrease is a function of the particle area (its shadow). The change in light intensity at the photodetector is transmitted

through electronic circuitry as a pulse to the counter, which counts and analyzes pulses.

Particles are counted when the electrical pulse is greater than a specific threshold level. Each particle generates one pulse, and the total number of pulses corresponds to the total number of particles. The threshold for analysis must be above the background electrical noise level of the sensor for the pulse to yield meaningful information. The particle size range depends on the sensor model. Our sensor measures particles ranging from 1.0 to 150 μ . The lower size limit of a sensor is determined by the background noise level. The upper limit is a function of the dynamic range of the instrument and the physical size of the sensing zone with associated tubing.

The background noise level for this sensor is 11.0 mV as determined by the manufacturer according to requirements of the American Society for Testing and Materials (ASTM F658-87).⁹ Particle size standards of 1.0 μ were detected at 18.0 mV, which is significantly higher than the background noise level.

The counter controls various other components of the particle counting system. It receives and stores voltage pulses from the sensor, transforms the input into particle size information, and outputs the data. User-selectable particle size channels are available to sort the data.

Various levels of data manipulation, display, and output capabilities are possible. These range from highly sophisticated to simple output of raw data. Raw particle data can also be transferred from the counter to spreadsheet programs.

Discrete Sampling

Using this counting method, samples are collected individually, placed in containers, and transported to a particle counter. The liquid batch sampler (Figure 4) uses a syringe arrangement to draw the sample from a container and deliver it through tubing to the sensor at a controlled flow rate. The sensor precisely meters the volume of sample

FIGURE 1. Particle Analysis System (PAS)

FIGURE 2. Liquid Sensor, Hiac-Royco HRLD-150

FIGURE 3. Light Extinction Particle Count Sensor

through linear actuators on the syringe itself. Discrete analysis is affected by the sample container, particulate settling and agglomeration, and air bubbles.

The sample container itself can be a source of particle contamination. For calibration of liquid particle counters, ASTM F658-87 requires glass narrow-mouthed sample bottles having caps lined with compatible noncontaminating films or liners cleaned by a specific procedure.⁹ We used 180-ml glass bottles meeting these criteria and certified free from contaminants larger than 1 μ .

As soon as a discrete sample is collected, it is vulnerable to changes in the particle size distribution attributed to settling and agglomeration. Air bubbles can form from dissolved gases, and bubbles are counted as particles by the instrumentation. Hiac Royco suggested three possibilities for redistributing particulates and eliminating bubbles prior to analysis: gentle inversion, ultrasonication, or a combination thereof. We selected gentle inversion because it is simple and requires no special equipment.

Hiac Royco recommended stirring the sample during analysis to keep particles in suspension.

FIGURE 4. Liquid Batch Sampler

The batch sampler (equipped with a stirrer and clean glass-encased magnetic stir bars) was used. Higher stirring speeds causes excessive turbulence and air bubbles, but a slow stirring speed eliminates these effects.

Water samples from the Rocky Flats surface water detention ponds, operable units, and the drinking water treatment plant were measured in discrete mode during initial instrument evaluation experiments. Instruments were operated according to procedures specified in the operating manuals and observed during the demonstrations.

For this study, discrete samples were analyzed six times. The first analysis was considered an instrument rinse; therefore, the average of the next five replicates was reported.

On-Line Sampling

With this particle counting method, the sample flows continuously from the process stream through the sensor. On-line analysis is affected by the length of the counting cycle, the delay interval between cycles, the sample supply line, and the flow control device.

On-line counting cycles are programmed by the analyst and triggered automatically by the counter. Intervals between cycles may be varied from a few seconds to more than an hour. The sample volume analyzed during the cycle is determined by the flow rate through the sensor. Assuming the flow rate is constant during the cycle, a set, reproducible volume of sample is analyzed. Of importance is the length of time during which counting actually takes place. If the period is too short, the number of particles counted will be too low to provide statistically valid data. If the period is too long, the nature of the particulates in the on-line process stream may actually change. Significant amounts of data can be generated with on-line particle counters. A dedicated computer is necessary.

On-line particle counting is not vulnerable to sample contamination from collection bottles and other handling procedures. Contamination is limited to particulates originating from the supply lines between the process stream sampling point and the sensor. Analysis is performed in real time, so changes in particle size distribution resulting from sample transport and storage are avoided. One drawback is: if the sample stream becomes too concentrated for the sensor, dilution is impossible without accessory on-line equipment. Other disadvantages are: the sample is not conserved for further analysis, and the flow rate through the sensor must be constantly and precisely controlled.

On-line particle count installations require flow control devices capable of maintaining sensor flow within an acceptable tolerance of the sensor's calibration flow rate. There is no simple "factor" for correcting sizing and counting errors caused by flow rate deviations from the calibration value. Errors in counting and sizing data escalate with increased deviation from the calibration flow rate. Therefore, data collected during flow rate deviations should be considered invalid.

After initial evaluation, the PAS was relocated to Operable Unit 2, which treats Rocky Flats surface water from seeps and runoff to remove radio-nuclides and organic contaminants. The process

stream was monitored during successive days to verify repeatability of the filtration operation, using the PAS in an on-line configuration.

A calibrated rotameter liquid flowmeter and metering valve were installed on the exterior cabinet of the batch sampler downstream from the sensor to allow on-line, real-time flow rate measurement and control. The batch sampler was not required for this configuration, but it was used as a convenient protective housing for the sensor to support the flowmeter/metering valve.

Sample supply and waste lines of 1/4-in o.d. Teflon® tubing were attached to the process line and connected to the sensor. Flow rate was controlled with the metering valve and adjusted to 25 ml/min (rate used during calibration of the sensor). This was verified by placing the end of the sample waste line from the sensor into a graduated cylinder and monitoring the sample volume. Then the metering valve was adjusted until the 25 ml/min flow rate was achieved.

Parameters

The following parameters affect both discrete and on-line analyses.

Instrument Warmup

Adherence to the manufacturer's recommended warmup period is essential. During this time, the optic and electronic components go from powered-up to stable operating conditions. Sufficient warmup minimizes variations in accuracy. Our counter was programmed for self-diagnostic routines to signal malfunctions. A 20-minute warmup was used in this study.

Channel Size and Width

Capability to distinguish among particle sizes depends on the size ranges chosen. In simple terms, the size ranges or channel settings of a particle counter can be viewed as "sorting bins."

The more channel settings the counter has, the greater its potential to define true particle size distribution. For example, a counter with 150 channels set at 1- μ intervals gathers very detailed information. Assuming acceptable sizing capability and resolution, the counter sizes and counts particles in very narrow ranges. A counter with six channels set at 10- μ intervals gives less information since it sorts a broad range of sizes into one channel. In the latter case, all particles from 1 to 10 μ would be counted in the same channel as a 9- μ particle. Since channel widths are selected by the analyst, a single sample analyzed at different settings presents different data sets.

In samples with a random particle distribution, there are more small particles. To provide the best possible definition of particle size distribution from a counter with limited channels, narrow channel widths must be chosen for the lower sizes and wide channels for the higher channels. In this way, the ability to define the size distribution is limited by the sensor resolution capabilities rather than counter channel settings chosen by the analyst.

The Model 4100 counter used in the initial experiments has six channels available, and we utilized settings of 1-2, 2-5, 5-10, 10-20, 20-40, and 40-150 μ . The Model 9064 has 512 channels available, but most of our sampling with this counter utilized only 20 to 50 channels. Channels were set at 1- μ increments for sizes below 10 μ and increased up to 10- μ increments for sizes greater than 20 μ . Using more than 20 to 50 channels did not improve analysis.

Rinsing

The batch sampler was rinsed during warmup and between samples. Focus of the rinsing was on the sensor itself and tubing between the sample and sensor. The objective was to remove air bubbles and dislodge particles from the tubing. Flow rate checks are conveniently conducted during system rinsing. Clean 0.22- μ filtered deionized water was used for rinsing during the warmup. Deionized

water was used for between-sample rinsing. ASTM F658-87 advises analysis of samples from "cleaner" to "dirtier" to minimize rinsing requirements, and this method was used whenever possible.⁹

During warmup, replicates of the rinsing liquid were run through the batch sampler as if a particle count were being performed. The lowest channel was set to the sensor's lowest detection limit. Sufficient replicates were run to reduce the cumulative total particles to the expected level for deionized water. For this study, fewer than 10 particles/ml greater than 1 μ were expected in the deionized water. A trend of decreasing counts became obvious as a dirty system became cleaner.

For on-line applications, a complete rinse of the sample supply line before connecting the sensor minimized chances of clogging the sensor with large particles.

Flow Rate

Precise particle counting requires flow through the sensor at a constant, predetermined, precise rate. Two flow rate concepts must be addressed: the working rate range specified by the sensor manufacturer and the rate used during sensor calibration. The flow rate must be sufficiently slow to allow the electrical signal generated by the particle to develop fully. Also, it must be slow enough to ensure that the electronic impulse created by a particle traversing the light beam is transmitted to and processed by the counter before the next particle passes through.

Once calibrated at a specific flow rate, deviations must not occur. If this specific rate cannot be achieved or an alternate rate is desired, the sensor must be recalibrated. Otherwise, counting and sizing errors occur. The manufacturer of this sensor specifies a 10- to 75-ml/min flow rate and 25-ml/min calibrated flow rate.

In discrete sample particle counting, the batch sampling device generates the flow of the sample

FIGURE 5. Particle Analysis System Using Deionized Water. Concentrated samples were diluted with deionized water; therefore, those particle counts were subtracted as a background.

through the sensor. In on-line particle counting, a flow controlling device downstream from the sensor ensures constant, accurate flow.

Concentration

Correct counting and sizing depend on no more than one particle being in the sensing zone at a time. Otherwise, the measurement reflects the combined electrical response of all particles in the sensing zone. Numerous small particles in the sensing zone could yield the same response as a single large particle. The maximum particle concentration limit for the sensor used is 12,000 particles larger than $1.0 \mu\text{m}$ of sample. Analysis of an overconcentrated sample will yield seriously flawed data.

Overconcentration is related solely to the particle concentration and cannot be compensated for by alteration of flow rate through the sensor. The only way to make particles less concentrated is by diluting the sample.

Dilution

When the particle counts in undiluted samples exceed the concentration limit of the sensor, they

do not reflect the true concentration. The cumulative total particles counted will be an underestimation of the true number, and this leads to inaccurate, imprecise results. Once the concentration reaches the operating range of the sensor, it becomes a linear relationship. Also, replicate samples generate very similar counts at each dilution, showing that the dilutions are reproducible.

Discrete samples analyzed for this study with particle concentrations greater than the sensor limit of 12,000 count/ml were diluted with $0.22\text{-}\mu$ filtered deionized water until the count was well within the operating concentration range. This accepted method for analyzing overconcentrated samples was recommended by Hiac Royco, Interbasic Resources, 3M, Pall, and the Pine Valley Water Treatment Plant. Unfiltered water from the Rocky Flats ponds was typically diluted to one part sample in 99 parts deionized water (100:1). The count generated by the deionized water itself was subtracted from these analyses, and the remaining count was multiplied by 100 to correct for the dilution factor. Figure 5 shows a typical size distribution from the deionized water source.

Most filtered samples required no dilution because the particle concentrations were well within the operating concentration range for the sensor. The

only exceptions were samples analyzed during periods of filter failure; they typically required dilution factors from 5:1 to 100:1.

Calibration

Sensor calibration defines the relationship between the voltage pulse transmitted by the sensor to the counter and the size of the particle that initiated the pulse. For the sensor used, the amplitude of the voltage pulse varies with the area of the particle passing through the sensing zone.

In accordance with ASTM F658-87, size calibration is accomplished by establishing the instrument response to particles of known size.⁹ First, the sensor's working flow rate is determined. Then the calibration medium (suspensions of monodisperse spheres) are prepared in clean deionized water. A certificate of analysis is supplied with each batch of calibration spheres. It specifies the actual particle size (coefficient of variation not to exceed 5%) and certifies use of statistically valid techniques traceable to the National Institute of Standards and Technology (NIST).

At least four sizes of calibration spheres spanning the entire size range of interest are required. One size of calibration sphere at a time at a particle concentration about 25% of the sensor's maximum concentration capacity is run through the sensor.

A particle size distribution results from analysis of a monodisperse suspension and can be depicted as an x/y coordinate log scale plot. The x-coordinate is threshold setting in millivolts, and the y-coordinate corresponds to particle count. The voltage at the median of the particle size distribution is determined. This reading determines the setting for particles the size of the calibration sphere. The procedure is repeated for the other sphere sizes. If contaminated, calibration spheres are unsuitable for use in particle sizing tests. Calibration spheres are stored according to manufacturer's instructions and not allowed to freeze.

A calibration curve for an individual sensor at a specific flow rate is constructed by plotting particle size on the x-coordinate with the y-coordinate corresponding to the millivolt setting for each calibration sphere size. The millivolt response for particle sizes between actual calibration points is determined by interpolation.

The sensor was calibrated by the manufacturer on September 17, 1991 at a flow rate of 25 ml/min. During July and September 1992, two field calibrations were performed at Rocky Flats to verify stability of the sensor. These calibrations were performed according to ASTM F658-87 with suspensions of NIST traceable spheres. Fourteen sizes of calibration spheres ranging from 1 to 112 μ were used.

The curves generated by the factory and field calibrations plotted together are shown in Figure 6. These curves are very similar, indicating the sensor was stable for a year. It also indicates that the data acquired with the sensor for this period can be directly compared.

Resolution

Resolution measures the sensor's ability to distinguish among sizes of particles. Following procedures in ASTM F658-87, the sensor demonstrated a resolution of less than 0.5- μ variance for 9.87- μ spheres. A sensor with poor resolution for samples of 1.0- μ variance for 9.87- μ spheres would generate a wider particle size distribution.

Data Presentation

Particle count and size data can be expressed graphically and mathematically in many ways. The most useful data available from a single particle analysis include the cumulative total particles greater than the smallest size detected and the differential particle count in each size channel. Such data are referred to as particle size distribution. Data collected for a series of particle

FIGURE 6. Stability of Calibration Curve.
NIST traceable standards were used.

count analyses over a period of time (time-trend analyses) are also possible. If count and size data are acquired upstream and downstream from filter media, filtration efficiency can be calculated.

Particle count and size data can be converted into valuable information useful for applications such as treatment plant process optimization and control, treatment plant unit design, and establishment of water quality criteria based on particle counting.

Particle Count

Information from a single sample particle count analysis includes the cumulative total particles greater than the lowest size detected, the differential number of particles in each size channel, and slope of the particle size distribution graph. When particle count data are presented, the channel sizes and volume of sample measured must be specified. In this way, results from different sensors and sample volumes can be compared directly.

Particle Distribution

Particle count within size ranges makes up the particle size distribution in a sample. Graphs, *e.g.*, Figure 5, plot particle size (x-coordinate) against the number of particles (y-coordinate).

Time-Trend Analysis

A particle counter installed on line for real-time monitoring of a process stream or discrete samples from the same source can provide information to track changes in differential and cumulative particle data as well as particle size distribution over time. Time-trend graphs, *e.g.*, Figure 7, plot the elapsed time (x-coordinate) against the number of particles (y-coordinate).

Filtration

When count and size data are acquired upstream and downstream from filter media, percent

FIGURE 7. Memtek® Microfilter On-Line Testing - Operable Unit 2
(Time-Trend Analysis)

filtration efficiency can be calculated by the following formula:

$$\text{Percent Efficiency} = 100 \times [1 - (\text{total downstream count} / \text{total upstream count})]$$

RESULTS AND DISCUSSION

The PAS was evaluated in three separate tests. This testing provided data over a wide range of particle counts and sizes. The majority of particles are smaller sizes and exponentially decrease in quantity to larger sizes.

Application 1

The first PAS application was for a Rocky Flats detention pond water treatment and discharge system. The two-stage filter-bag filtration included two filter tanks in series with six 3M 529 filters in the first tank and six 3M 523 filters in the second tank.

Filter bag efficiency testing of the pond water is presented in Table 1 and Figure 8. Efficiencies are calculated for the first and second filter sets and both sets together. There are six groups of particle size ranges because the particle counter

had only six channels. Turbidity is also indicated. Between 5 μ and 10 μ, the first stage of filters failed to perform satisfactorily. A particulate contaminant of concern in the 5- to 10-μ range would not have been efficiently removed. This could be a characteristic of that type of filter, or it could indicate a leak in the filter seal. Filter performance depends on material, fabric weave, thickness, and seams.

Alternative filter bags were evaluated for possible improvements. This testing was performed by an independent laboratory (Interbasic Resources) to ASTM industrial standards using PTI® test dust contaminant in a filtered deionized water source.¹⁰ One of these tests is presented in Table 2. Enough data in each range and overall must be acquired to obtain a statistical representation of the sample. The 0.5-μ bag was 95% efficient or better for the manufacturer's rating of 0.5-μ absolute. At terminal pressure, this filter performed slightly below the expected level of 99% efficiency at 0.5 μ. The 10.0-μ bag was greater than 99% efficient for its rating of 10 μ. At terminal pressure, this bag performed slightly above the expected 99% efficiency level at 5 to 7 μ.

The 3M 723 filter cartridges were evaluated for potential replacement of filter bag units at the pond. The raw pond water was pumped directly

TABLE 1. 3M Filter Bags - Rocky Flats Pond Water

TEST 1 - February 1992						
Particle Size (μ)	Sample Point 1 4.51 NTU (diff. count/10 m ℓ)	Sample Point 2 3.72 NTU (diff. count/10 m ℓ)	Sample Point 3 2.02 NTU (diff. count/10 m ℓ)	Sample Points 1-2	Sample Points 2-3	Sample Points 1-3
	Efficiency (%)					
1-2	5,811,670	4,551,180	1,766,860	21.6	61.1	69.5
2	2,370,770	1,406,900	413,240	40.5	70.6	82.5
5	77,710	71,570	25,570	7.0	64.2	67.0
10	10,670	10,370	3,590	2.1	65.3	66.3
20	1,090	840	330	22.9	60.7	69.7
40-150	50	30	10	40.0	80.0	80.0

TEST 2 - February 1992						
Particle Size (μ)	Sample Point 1 5.81 NTU (diff. count/10 m ℓ)	Sample Point 2 5.20 NTU (diff. count/10 m ℓ)	Sample Point 3 2.18 NTU (diff. count/10 m ℓ)	Sample Points 1-2	Sample Points 2-3	Sample Points 1-3
	Efficiency (%)					
1-2	4,960,400	4,564,070	3,024,220	7.9	33.7	39.0
2	2,786,220	1,638,800	417,230	41.1	74.5	85.0
5	84,740	6,4720	16,200	23.6	74.9	80.8
10	10,290	9,490	1,530	7.7	83.8	85.1
20	950	860	130	9.4	84.8	86.3
40-150	40	30	0	25.0	100.0	100.0

Sample Point 1 - Raw water
 Sample Point 2 - After first filtration
 Sample Point 3 - After second filtration

FIGURE 8. 3M Filter Bags - Rocky Flats Pond Water (February 1992)

TABLE 2. Pall Filters Bags. Tests were performed by Interbasic Resources, Inc. according to ASTM standards.

Pall Y050C4 Filter 0.5- μ absolute - 25-psid terminal pressure			
Particle Size (μ)	Upstream from filter (diff. count/10 ml)	Downstream from filter (diff. count/10 ml)	Efficiency (%)
0.5-0.8	20,072	1,180	94.1
0.8-1.0	12,011	439	96.4
1.0-1.5	3,631	182	95.0
1.5-2.0	3,208	148	95.4
2.0-3.0	2,502	86	96.6
3.0-5.0	1,346	54	96.0
Pall Y100C4 Filter 10.0- μ absolute - 25-psid terminal pressure			
1.0-5.0	27,879	431	98.5
5.0-7.0	14,481	153	99.0
7.0-10.0	5,096	27	99.5
10.0-15.0	1,331	4	99.7
15.0-20.0	480	0	100.0
20.0-30.0	123	0	100.0

through the cartridges without prior or subsequent treatment. One discrete water sample was analyzed and used for all the raw water values and efficiency calculations. The treated water samples were taken in time increments during the filtration process.

Table 3 presents the treatment efficiency results. Particle removal efficiencies using 3M 723 cartridges for the raw water exceeded 95% and were markedly better than field-tested filter bags. See Figure 9. Twenty particle size ranges were reported and plotted using a new particle counter chassis and expanded capability. More subtle changes can be distinguished in Figure 9 than in Figure 8, which represents only six channels. Two increases in particle distribution at approximately 4.5 and 18 μ appear in Figure 9. The difference from the influent (raw water) to the filtrate (filtered water) shows the removal of particles throughout the range monitored.

All filtration efficiencies exceeded 95% in the 1.5- to 150- μ size range. The cartridge performance

shows consistent removal over the whole range of particles monitored, unlike filter bags (Figure 8) where a particle range was not filtered very efficiently. This demonstrates how differential particle count data are useful for evaluating size-specific filtration efficiency, whereas total particle count shows only overall performance of the filter. Thus, two filters may remove the same total particle count but may perform differently in certain size ranges. It should be noted that although turbidity remained below 1 NTU in the cartridge-treated effluent, the PAS continued to detect changes in the particle count, indicating increased sensitivity of the PAS as compared to turbidity measurement.

The advantage of the PAS for pond water treatment operations is its capability to determine filtration efficiencies for particle removal in the 1- to 150- μ micron range, which is potentially associated with radiochemical activity. Investigation¹¹ shows that approximately 60 to 75% of plutonium is associated with the size fraction greater than 0.45 μ . The loading, unloading, and

TABLE 3. 3M 723 Filter Cartridge - Rocky Flats Pond Water

Sample 1			
Particle Size (μ)	Raw Water (diff. count/10 mℓ)	Treated Water (diff. count/10 mℓ)	Efficiency (%)
1.0-1.5	5,660,804	296,764	94.8
2.5	596,663	26,433	95.6
3.5	319,177	13,662	95.7
4.5	607,063	28,408	95.3
5.5	181,687	6,907	96.2
6.5	63,398	1,648	97.4
7.5	70,498	1,468	97.9
8.5	49,697	827	98.3
9.5	34,897	517	98.5
10.5	28,098	408	98.5
11.5	23,199	339	98.5
12.5	15,397	262	98.3
13.5	11,900	300	97.5
14.5	9,999	194	98.1
17.5	29,999	674	97.8
22.5	8,599	149	98.3
27.5	3,000	60	98.0
35	1,600	30	98.1
45	1,400	0	100.0
50+	600	5	99.8

Sample	Particle Size (μ)	Raw Water		Treated Water		Efficiency (%)
		(total count/10 mℓ)	(NTU)	(total count/10 mℓ)	(NTU)	
1	1-150	7,717,674	17	379,054	< 1	95.1
2	1-150	7,717,674	17	159,034	< 1	98.0
3	1-150	7,717,674	17	243,264	< 1	96.8
4	1-150	7,717,674	17	176,404	< 1	97.7
5	1-150	7,717,674	17	181,789	< 1	97.6

FIGURE 9. 3M 723 Filter Cartridges - Rocky Flats Pond Water

filter change-out rates can be determined by monitoring the filtration efficiencies.

Application 2

Operable Unit 2

Operable Unit 2 is the Interim Measure/Interim Remedial Action Water Treatment System used to remove contaminants such as metals (including radionuclides) and organic chemicals from Rocky Flats surface water streams and seeps. Two phases of the Operable Unit 2 process were tested. Phase 1 used filter bag filtration followed by granulated activated carbon. Phase 2 eliminated the filter bags but included a chemical addition step followed by a Memtek® membrane microfilter with the subsequent granulated activated carbon.

Phase 1 - Two tests of Operable Unit 2 filter bag efficiency results are presented in Table 4. The first test uses Strainrite® AP-5 filter bags. The second test uses 3M 523 filter bags. These bags were also used in the second-stage testing of pond water (Table 1). Three filter bags were set up in parallel and each filter's performance was measured individually. All water samples were analyzed using the discrete sample method. The Test 1 efficiencies dropped off significantly in the

20- to 40- μ range in each of the three parallel filters. However, the first filter performed better than the others in the lower micron ranges. Great variability is evident in less than 20- to 40- μ particle count data, which may be caused by inherent bag characteristics or leaks in the seal. Test 1 filter bags are needle-punch, sewn-seam fabricated from a felt polyester material. Test 2 filter bags show good performance exceeding 90% efficiency down to the 5- to 10- μ range. They performed better than Test 1 bags and showed a marked improvement for all ranges monitored. Figure 10 shows raw and treated water results obtained from the third Test 2 filter bag. Again, only six channels were measured because of the limited capacity of the Model 4100 counter.

Phase 2 (Discrete Sampling) - In Phase two, the filter bags were eliminated. Addition of chemicals was followed by a membrane microfilter. Table 5 presents results of Operable Unit 2 membrane efficiencies performed in a discrete mode. Most efficiencies exceeded 99.0% except in the 1.0- to 1.42- μ range. The Model 9064 counter was used to generate these data with 20 channels concentrating on the measurable particle ranges. Figure 11 depicts the membrane batch test. With the increase in channels, most variations in particles are distinguishable. Figure 11 illustrates the two orders of magnitude reduction

TABLE 4. Strainrite AP-5 and 3M 523 Filter Bags - Operable Unit 2.
Three filter bags were positioned in parallel for each test.

	Raw Water	Filter 1		Filter 2		Filter 3	
Particle (μ)	(diff. count /10 ml)	(diff. count /10 ml)	Efficiency (%)	(diff. count /10 ml)	Efficiency (%)	(diff. count /10 ml)	Efficiency (%)
Test 1 - Strainrite AP-5 Bags							
1-2	2,820,000	2,650,000	6.0	2,530,000	10.3	2,370,000	16.0
2-5	1,530,000	774,921	49.4	1,390,000	9.2	1,510,000	1.3
5-10	316,000	64,815	79.5	230,000	27.2	310,634	1.7
10-20	30,235	19,117	36.8	22,820	24.5	26,438	12.6
20-40	4,107	977	76.2	2,255	45.1	1,940	52.8
40-150	112	4	96.4	11	90.2	9	92.0
Test 2 - 3M 523 Bags							
1-2	2,920,000	269,089	90.8	267,561	90.8	845,811	71.0
2-5	1,230,000	37,541	97.0	41,727	96.6	190,711	84.5
5-10	69,005	1,842	97.3	1,706	97.5	7,584	89.0
10-20	28,113	181	99.4	54	99.8	1,717	93.9
20-40	2,576	11	99.6	3	99.9	188	92.7
40-150	96	0	100.0	0	100.0	15	84.4

FIGURE 10. 3M 523 Filter Bags - Operable Unit 2

on a log scale in particles with a 99.0% treatment efficiency.

Phase 2, On-line Sampling - The first use of the PAS in an on-line mode is presented in Table 6.

It contains on-line results of membrane efficiency testing performed with the particle sensor connected to the effluent pipe followed by a flow control device. Normal filter operation is compared with filter operation immediately after

TABLE 5. Memtek Membrane Discrete Sampling - Operable Unit 2

Particle Size (μ)	Raw Water (diff. count/10 ml)	Filtered Water (diff. count/10 ml)	Efficiency (%)
1.0-1.42	435,835	4,711	98.9
2.25	62,900	618	99.0
3.08	13,700	110	99.2
3.91	24,100	193	99.2
4.74	16,395	102	99.4
5.57	6,335	39	99.4
6.40	2,680	13	99.6
7.23	1,470	6	99.6
8.06	985	3	99.7
8.89	805	3	99.6
9.72	800	4	99.5
10.55	675	3	99.5
11.38	400	3	99.3
12.21	290	0	100.0
13.04	205	0	100.0
13.87	175	1	99.4
14.07	125	0	100.0
15.53	95	0	100.0
16.36	70	1	99.6
17.19-150.0	225	0	100.0
	(total count/10 ml)	(total count/10 ml)	Overall Efficiency (%)
1-150	568,265	5,810	99.0

FIGURE 11. Memtek Membrane Discrete Sampling - Operable Unit 2

TABLE 6. Memtek Membrane On-Line Sampling - Operable Unit 2

Elapsed Time (min)	Particle Size (μ)	Raw Water (total count/10 ml)	Treated Water (total count/10 ml)	Treated Water (NTU)	Efficiency (%)
Before Backflushing					
3.5	1-150	568,265	23,305	3.0	95.9
7.0	1-150	568,265	6,295	2.2	98.9
10.5	1-150	568,265	2,568	1.1	99.5
14.0	1-150	568,265	1,460	0.5	99.7
17.5	1-150	568,265	1,433	0.3	99.7
21.0	1-150	568,265	1,128	0.1	99.8
24.5	1-150	568,265	868	0.0	99.8
28.0	1-150	568,265	756	0.0	99.9
31.5	1-150	568,265	662	0.0	99.9
35.0	1-150	568,265	482	0.0	99.9
38.5	1-150	568,265	618	0.0	99.8
42.0	1-150	568,265	552	0.0	99.9
45.5	1-150	568,265	552	0.0	99.9
49.0	1-150	568,265	514	0.0	99.9
52.5	1-150	568,265	447	0.0	99.9
56.0	1-150	568,265	538	0.0	99.9
After Backflushing					
3.5	1-150	601,772	115,106	*	80.9
7.0	1-150	601,772	63,010	*	89.5
10.5	1-150	601,772	44,184	*	92.7
14.0	1-150	601,772	35,055	*	94.2
17.5	1-150	601,772	27,929	*	95.4
21.0	1-150	601,772	23,790	*	96.1
24.5	1-150	601,772	20,178	*	96.7
28.0	1-150	601,772	17,740	*	97.1
31.5	1-150	601,772	15,501	*	97.4
35.0	1-150	601,772	13,108	*	97.8
38.5	1-150	601,772	11,343	*	98.1
42.0	1-150	601,772	9,583	*	98.4
45.5	1-150	601,772	7,967	*	98.7
49.0	1-150	601,772	6,755	*	98.9
52.5	1-150	601,772	5,778	*	99.0
56.0	1-150	601,772	5,086	*	99.2

*Turbidimeter malfunction

backflushing. These data are further depicted in Figure 7. Only one raw water sample was taken at the beginning. It was used to represent raw water total particle count. The standard filter run required approximately 21 minutes to attain water clarity of 1000 total particle counts. However, the backflushed filter took approximately 80 minutes to attain the same count (as measured before cleaning). Thus, backflushing tripled the time necessary to reach the maximum filtration level. Total particle count of the treated water fluctuated and showed filtration changes after exceeding the lower detection limit of the turbidimeter. The turbidimeter failed to function correctly after backflushing, and thus no data are presented. With this specific system, less than 1000 total particle counts resulted in a reading of approximately 0.0 NTU.

Operable Unit 1

Operable Unit 1 removes metals (including radionuclides) and organics from alluvial groundwater intercepted by a French drain system. The treatment process includes organic destruction by hydrogen peroxide and ultraviolet light, as well as ion exchange to remove metals and radionuclides.

Figure 12 depicts treated effluent particle counting of a tank in Operable Unit 1. Preliminary discrete samples were analyzed from the treated effluent discharge pipe. Particle distribution is similar to Operable Unit 2 data and of comparable clarity.

APPLICATION 3

Rocky Flats Drinking Water Treatment Plant

The drinking water treatment plant influent and filter effluent were monitored to determine filtration efficiencies. Samples were collected on three dates and analyzed with the PAS in discrete sample mode (Table 7, Figure 13). Except for two ranges, the treatment efficiencies exceeded 90%. These data are similar to those from Operable Units 1 and 2. The additional channels produced a more detailed picture of the particle distributions in the water (Figure 13).

In the first two tests, the finished water's final turbidity (0.20 NTU) was the same, while the total particle count was different. Test 3 shows a total particle count reduction, but not as significant as the turbidity values indicate. This demonstrates increased sensitivity obtained with the PAS compared to turbidity.

FIGURE 12. Treated Effluent - Operable Unit 1

TABLE 7. PAS Testing - Rocky Flats Drinking Water Treatment Plant

Particle Size (μ)	Raw Water (diff. count/10 m ℓ)	Treated Water (diff. count/10 m ℓ)	Efficiency (%)
Test 2 (4/13/92)			
1.0-1.34	803.200	15.347	98.1
1.99	141.500	2.527	98.2
2.65	13.500	207	98.5
3.30	15.900	310	98.1
3.96	20.900	421	98.0
4.61	15.600	430	97.2
5.27	8.500	275	96.8
5.92	4.100	151	96.3
6.58	2.100	84	96.0
7.23	1.600	64	96.0
7.89	1.200	46	96.2
8.54	700	40	94.3
9.20	800	30	96.3
9.85	800	32	96.0
10.51	400	48	88.0
11.16	500	28	94.4
11.82	500	34	93.2
12.47	600	14	97.7
13.12	500	22	95.6
13.78	300	12	96.0
14.43	200	14	93.0
15.09	100	10	90.0
15.74	100	11	89.0
16.40	100	6	94.0
17.05	100	6	94.0
17.71	100	10	90.0
18.36	200	2	99.0
19.02	100	2	98.0
19.67	100	1	99.0
20-150	400	16	96.0

	Particle Size (μ)	Raw Water		Treated Water		Efficiency (%)
		(total count/10 m ℓ)	(NTU)	(total count/10 m ℓ)	(NTU)	
Test 1 (4/9/92)	1-150	1,151,100	4.3	24,035	0.2	97.9
Test 2 (4/13/92)	1-150	1,034,700	3.6	20,200	0.2	98.1
Test 3 (4/29/92)	1-150	674,200	3.3	14,865	0.07	97.8
Mean Value	1-150	963,333	3.7	19,700	0.16	97.9

FIGURE 13. PAS Testing - Rocky Flats Drinking Water Treatment Plant (4/13/92)

Pine Valley Water Treatment Plant

The Pine Valley Water Treatment Plant is a municipal operation at Colorado Springs. Filtration efficiencies from this discrete particle counter system are presented in Table 8 and Figure 14. The data were provided by the Pine Valley plant and represent a "snapshot" of its product water operation.

Table 8 shows the total particle count and turbidity at three stages: the raw water, after flocculation, and final effluent. All measurements are less than 0.5 NTU with the effluent water at 0.02 NTU. This is the low end of measurable turbidity. The filtration efficiency shown is calculated using total particle count for the complete system.

From the raw water to the intermediate step (after flocculation), the reduction in total particle count was 76.9% with turbidity reduction of only 20.5%. From the intermediate step to the final effluent, the total particle count was reduced 96.4% while turbidity was reduced 94.3%. Thus, while the filtration system worked quite well based on total particle count, turbidity values showed

little reduction due to flocculation. This leads to the assumption that the process should be altered by adjusting the flocculent dosage.

Where the measurement needs the most sensitivity (during addition of chemicals), turbidity does not provide enough information. If prompted by turbidity data to add more chemicals, using total particle count to monitor filtration efficiency would produce immediate cost savings and reduce sludge.

The particle counting system at Pine Valley is relatively new, and preliminary data show an expected payback in less than two years with chemical savings, general plant optimization, and meeting or exceeding regulatory requirements. Furthermore, these accomplishments help establish a better public image.

Raw water at the Rocky Flats drinking water treatment plant has higher turbidity and particle count values than at Pine Valley. Rocky Flats data were collected in April while Pine Valley data were collected in January. Seasonal changes and water sources may alter influent raw water quality,

TABLE 8. Pine Valley Water Treatment Plant (1/9/92)

Particle Size (μ)	Raw Water (diff. count/10 mℓ)	After Flocculation (diff. count/10 mℓ)	Multimedia Filtered (diff./count/10 mℓ)	Efficiency (%)
2	28,770	6,890	130	99.5
3	10,920	2,770	110	99.0
4	4,020	720	50	98.8
5	1,440	270	30	97.9
6	690	110	20	97.1
7	420	70	10	97.6
8	270	40	10	96.3
9	180	20	10	94.4
10	150	20	10	93.0
11	120	10	0	92.0
12	90	10	10	89.0
13	60	10	0	100.0
14	50	10	0	100.0
15	40	0	0	100.0
16	30	0	0	100.0
17	20	0	0	100.0
18	20	0	0	100.0
19	10	0	0	100.0
20+	20	0	0	100.0

Particle Size (μ)	Raw Water		After Flocculation		Multimedia Filtered		Efficiency (%)
	(total count/10 mℓ)	(NTU)	(total count/10 mℓ)	(NTU)	(total count/10 mℓ)	(NTU)	
1-150	47,320	0.44	10,950	0.35	390	0.02	99.7

FIGURE 14. Pine Valley Water Treatment Plant (1/19/92)

which in turns affects the water treatment filtration process. Seasonal changes were also noted in the Rocky Flats pond water testing.

CONCLUSIONS

Particle counting is a cost effective, viable technique for water monitoring and treatment. In environmental and drinking water treatment applications, the PAS proved to be an innovative, advanced technology with tremendous potential.

Particle count and size distribution analyses provide a detailed picture of treated water quality and clarity. Changes in filtered particle count and particle sizes were detected even when simultaneous turbidity analysis revealed little or no fluctuation. The varying quality of filtered water with identical turbidities could be distinguished. The filter loading phase and steady-state filter monitoring indicated changes not detected by other methods. Slight changes in filter performance are discernable through corresponding changes in particle count and size distribution. Particle counting is effective for comparing performance of filter media. It is a sensitive measurement applicable in discrete and real-time on-line modes.

The relationship between radionuclide activity and particle counting is being investigated. The PAS demonstrates that water of equal clarity is not always of equal quality.

The PAS used in this study provides the best available technologies packaged from components that are commercially available. Further, its portability allows field testing suitable for many applications. The PAS stood alone as a powerful monitoring device for evaluating water quality. Reliable data were obtained over a one-year period with unquestionable stability in the calibration curve.

Application 1 testing indicated that influent water particle size and concentration characteristics have a direct effect on filtration efficiency. Influent particle size and concentration are affected by seasonal changes, which also affect filtration

efficiencies. The pond water results are variable even when using the same type of filters. Cartridge filter performance was better and more consistent than bag filtration over the particle size range monitored. Sample dilution, handling, and background techniques were evaluated and utilized. Filtration efficiencies and particle distributions were determined for use in evaluating the pond water treatment systems as part of this application.

In Application 2, particle counting indicated water quality changes after the range of 0.0 NTU was achieved. In Operable Unit 2 on-line monitoring, less than 1000 total particle count resulted in turbidity of 0.0 NTU, but filter efficiency improved to approximately 500 total particle count with no change in turbidity.

Filter removal efficiency improved with time because the previously retained particles serve as additional collectors for other suspended particles. This was shown by comparing filtration before and after backflushing. Particle counting can be used to evaluate the filter cleaning procedure (back-flushing) capability by measuring immediate post-filter performance. On-line sample flow rate is critical and must be closely controlled. The flow rate must be the same as the sensor calibration flow rate. Filtration efficiencies, particle distributions, improved monitoring sensitivity, and filter loading from normal operations and backflushing were determined from this application.

In Application 3, the difference between turbidity and particle counting was evident. The PAS indicated changes undetected by turbidimeters, which had also been reported by others.⁷ The PAS monitors water two-dimensionally by counting and sizing particles.

Cost savings for chemicals and optimization of treatment operations based on total particle count has been demonstrated and has also been demonstrated by others.⁸ Particle counting supports increasingly stringent drinking water and environmental treatment and discharge requirements.

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