

DETERMINING CORRELATION OF NEPHELOMETRIC  
TURBIDITY MEASUREMENT TO SUSPENDED SOLIDS  
IN INDUSTRIAL SAMPLES

Michael J. Sadar, Research Chemist  
Terry L. Engelhardt, Marketing Manager  
HACH COMPANY  
P.O. Box 389  
Loveland, Colorado  
1-800-227-4224

Turbidity measurement often can be used as a substitute for gravimetric solids measurement in the monitoring and controlling of industrial processes.

A procedure using a laboratory turbidimeter has been developed to determine whether an acceptable correlation can be established between turbidity and solids on a given sample. This paper will explore many instrumental and sample variables which must be considered and detail the steps necessary to establish correlation. Data developed during a two-year study on a variety of sample types will be presented.

Turbidity measurement determines the amount of light scattered by particles in a sample. Instrument variables include the number and placement of detectors. Traditionally, turbidity is measured with an instrument with a single detector positioned at 90° from the incident light path. Measurement with the single detector will become less sensitive to solutions that are highly colored, contain light absorbing particles (i.e. activated carbon) or are very turbid. Each of these conditions, while increasing the turbidity, will decrease the amount of scattered light which can reach the detector. They are negative interferences. The measured turbidity will be low. This condition often is referred to as "going blind."

Turbidimeters with multiple detectors equip the instruments to provide accurate measurements even in the presence of color, light absorbing particles or high turbidity. The multiple detector instruments are commonly referred to as "ratioing" instruments. Because these interferences are likely to be present in many industrial samples, a ratioing instrument should be used in most studies of suspended solids versus turbidity. Once the sample has been studied, a determination can be made whether a ratioing or non-ratioing instrument will be necessary for control measurements.

Sample variables include temperature, pH and the chemical and physical nature of the particles in suspension. The amount of light scattered depends on the number of particles. The size, shape, composition and refractive index of particles are other factors which contribute to the amount of scattering. Factors such as temperature and pH may determine the type of instrument appropriate for use, and the sample conditioning steps which may be necessary prior to measurement.

An increase in solids generally can indicate one or more changes have occurred in a process. Chemical or physical changes in the process may result in filter breakthrough or an increase in turbidity of clarifier effluent leading to process contamination. Thus, the monitoring of solids yields critical information about the efficiency and quality of an industrial process.

Solids analyses, usually completed by gravimetric methods, are time consuming and technique sensitive. It may be difficult to obtain a representative sample. The procedure of gravimetric analysis is very time consuming - - typically taking two to four hours to complete. Indeed, the gravimetric solids test will tell the operator there is a process problem but, by the time the operator knows this, the problem cannot be corrected easily. This leads to costly down time and repairs to fix the problem.

Techniques in sampling and measurement are important in the gravimetric procedure. The sample procedure must be consistent from one time to the next in order to obtain useful and reliable results. The sample must be large enough and sufficiently well mixed to make sure it is representative.

A rapid response to changing operational conditions can be achieved by using a turbidimeter as a surrogate for solids measurements. But a study should be conducted to determine how well changes in turbidity relate to changes in solids content. It will be impossible in nearly all samples to make a quantitative conversion from turbidity measurement in Nephelometric Turbidity Units (NTU) to mg/L of solids. However, it should be possible to establish

sufficient correlation in most samples to be confident that trends in turbidity measurement are representative of trends in solids content.

The turbidimeter ideally should have wide measurement range so the raw sample can be monitored without dilution. Dilutions may be used. However, samples requiring dilution will take more time to measure. Accordingly, the response time to changes will increase.

The sample to be used in the correlation study must fit several criteria. The sample must be miscible to the point that dilutions of the sample can be made. Viscous samples may be difficult to monitor because entrained gas bubbles cannot be easily removed. Gas bubbles scatter light and cause a false high turbidity reading.

The sample cannot possess any excessively buoyant particles. Samples cannot be filtered. (This would not be a representative sample.) Each sample is site-specific. Thus, correlation established on a sample from one site may not be applicable to a sample from another site either within the same plant or between plants.

Sample temperature is critical to a successful correlation. The less sensitive the sample is to temperature, the more consistent the correlation will be over time. A temperature profile for the sample should be used to determine whether temperature will lead to erroneous results. If the nature of a sample is temperature-sensitive this correlation may become more difficult to accomplish or it may even be impossible to determine. Ideally, sample temperature during turbidity measurement should be the same as the temperature of the process stream. Temperature changes may affect the solubility or settling characteristics of particles in suspension.

Sample constituents should be well defined. One must know what is in the sample, the chemical reactions in the sample that can change its characteristics, and the conditions of the sample that indicate a process problem. One should know what threshold value is required for process control. The more one knows about the sample of interest, the easier it will be to determine correlation between turbidity and solids measurements.

Obtaining a truly representative sample may be the most important consideration. The sample must be consistent throughout. And, the grab sample must come from the actual process stream. Efforts to establish a correlation on a contrived sample will not be successful. It may be impossible to achieve a correlation on samples not meeting one or more of the above criteria.

The procedure for determining the correlation between turbidity and total suspended solids (nonfilterable residue) can be sorted into four major steps. They include sample dilution, determination of turbidity of each dilution, determination of the total suspended solids of each dilution, and the mathematical calculation which will verify the accuracy and linearity of the turbidity to suspended solids correlation.

The first step is to obtain a representative sample and then determine the amount of dilution required to bring the sample onto scale of the instrument. If the sample is to be diluted, the sample must be well mixed. For the most consistent results, a magnetic stirrer should be used and all dilutions must be made from a solution that is mixed continually. The mix rate should maintain homogeneity but should not introduce air bubbles into the sample. Never shake the sample to mix. Shaking introduces air bubbles that will be mistaken for turbidity.

Once the original sample has been diluted, read the turbidity of the sample. Again, be sure the sample is well mixed. Mixing by inverting the sample cell is sufficient in order to obtain the homogeneous sample. Immediately place the sample in the turbidimeter and record the turbidity of the sample over a short time period. A recording of the turbidity readings every five seconds during the time interval between 15 and 45 seconds is recommended. Average the values and use this average as the turbidity of the sample.

When the first sample reading is recorded, one must be sure the turbidimeter is responding to the sample correctly. If a sample is highly colored, the turbidimeter may become blind due to high light absorption and will not show any response to turbidity changes with dilutions (very common with 90° nephelometers). The sample must be diluted to the point that it is on scale of the turbidimeter and is responding to dilutions.

If the original sample had to be diluted initially to be within the range of the instrument, record this dilution. Dilute all solutions with a low turbidity solvent, usually water. If water is the appropriate diluent, deionized water should be used. This is the master dilution from which all further dilutions will be made in order to complete the correlation study. All dilutions must be prepared from the same dilution water or other solvent.

Once the sample has been diluted onto the working range of the turbidimeter, make at least four more dilutions from this solution (for a total of at least five). Defining the master dilution as 100 percent, the other dilutions should cover the expected range of measurement, i.e. 20, 40, 60 and 80 percent. The volume of each dilution must be suitable for

replicate tests by the turbidimetric and gravimetric methods.

The temperature of the sample must be consistent throughout the preparation and measurement of all dilutions. This is where a temperature profile would indicate if additional sample considerations must take place.

Remember, the original solution from which dilutions are made must be mixed continuously in order to make consistent and homogeneous dilutions. After each dilution has been prepared and mixed, the turbidity and total suspended solids may be determined.

After the turbidity of each sample has been measured and recorded, a gravimetric determination of solids must be made. The remaining solution from each dilution flask is to be used in the solids determination. A volume of at least 50 mL of each dilution should be used in the solids determination. Again, it is important that the sample be well mixed while the 50 mL aliquot is taken. Also, each dilution must be treated in the exact same manner (mixing time, speed) during the solids determination. The lack of a reproducible technique will result in a less accurate measurement and make correlation difficult. Replicate measurements are recommended when adequate sample volumes are available.

If a solution is very low in solids, a larger aliquot of sample may be taken to increase the accuracy and repeatability of the gravimetric solids test. The adjusted volume of each dilution must be accounted for if this is done. Never change the aliquot size within the same set of dilutions.

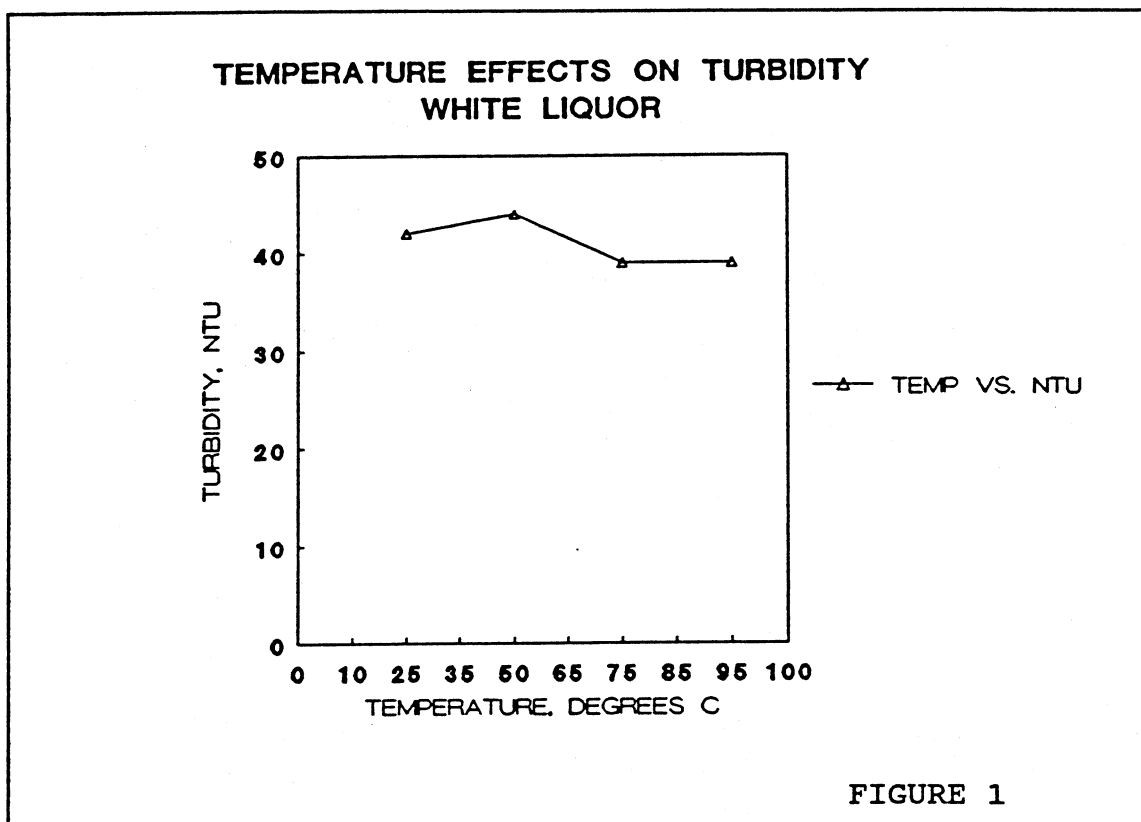
For each dilution, plot the turbidity on the y-axis and the total suspended solids on the x-axis. If this is a usable correlation, several aspects from this graph will be immediately apparent. First, as the total suspended solids values increase (x-axis) the corresponding turbidity must increase (have a positive slope). Next, the graph should show a consistent relationship between turbidity and total suspended solids. The plot need not be perfectly linear, but should display a smooth relationship between points.

The slope of the plot is important. The greater the slope, the more sensitive the turbidity measurement is with respect to total suspended solids, and a turbidimeter could be used reliably for monitoring the solids of the liquor. The lower the slope, the less sensitive is the relationship of turbidity to total suspended solids and the more difficult it becomes to use a turbidimeter to monitor the solids of a solution. A curve exhibiting a flat or nearly flat slope may indicate the need for additional dilution.

On some samples, there may be two or more distinct changes in slope along the curve. Areas of the curve exhibiting a greater slope generally indicate good correlation. Areas where the curve is not as steep are generally those where more dilution may be required. Further dilution of the sample may be necessary to establish correlation. A least squares statistical evaluation can be used to determine the best fit relationship. In general, a correlation coefficient of at least 0.9 will result in a workable relationship.

Example: A sample of white liquor was used to determine if a relationship between total suspended solids and turbidity could be established. The white liquor sample contained a small percentage of dregs.

The first step in the study was to complete a temperature profile of the sample. The sample was first diluted to a 20 percent solution. Then turbidity was measured on a ratio turbidimeter as the temperature was varied. The graph below, Figure 1, illustrates temperature had a negligible effect on the turbidity of this sample. Thus, it was determined variation in sample temperature would not cause difficulty during the balance of the study.



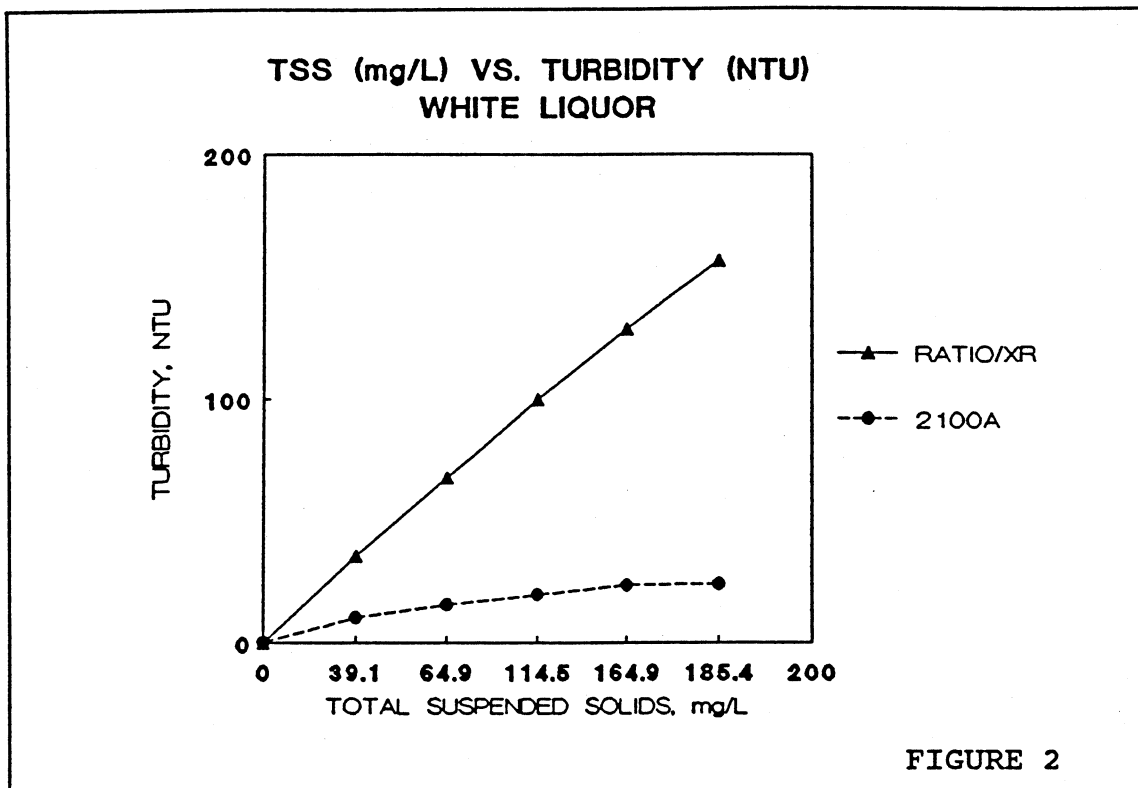
Next several dilutions of the concentrated sample were prepared with deionized water. The concentrated sample was read on the scale of the laboratory turbidimeter (Hach Ratio/XR, Model 43900, range 0-2000 NTU). Therefore, no initial dilution was required. Working dilutions were made directly from the original sample. Each aliquot of the original sample was diluted to 250 mL total volume to provide sufficient sample for turbidimetric and gravimetric measurement.

After dilution, turbidity of a aliquot of each dilution was determined on ratioing (Ratio/XR) and non-ratioing (Hach Model 2100A) turbidimeters. Thus, whether a ratioing-type instrument would be required also was determined.

A gravimetric determination of total suspended solids (in mg/L) was completed for each dilution. The procedure for the gravimetric method was according to Standard Methods for the Examination of Water and Wastewater, 17th edition, with drying at 103° C. The data from the turbidity results and the total suspended solids residue are summarized in Table 1 and Figure 2.

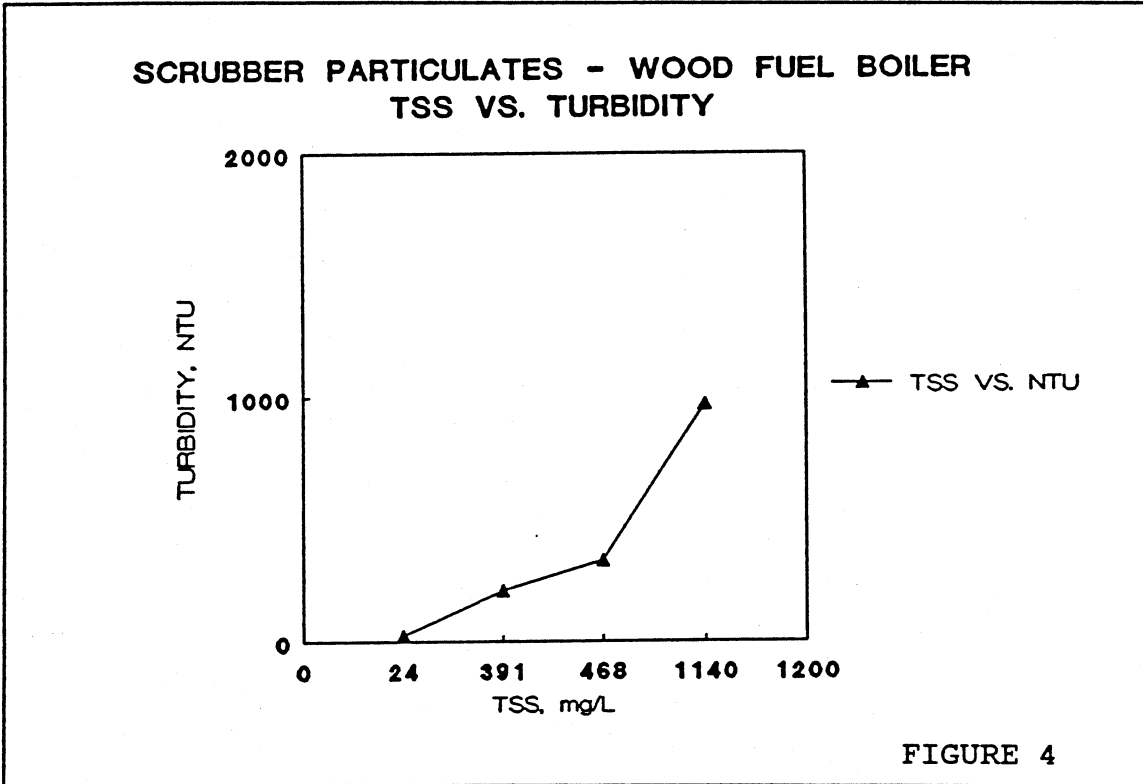
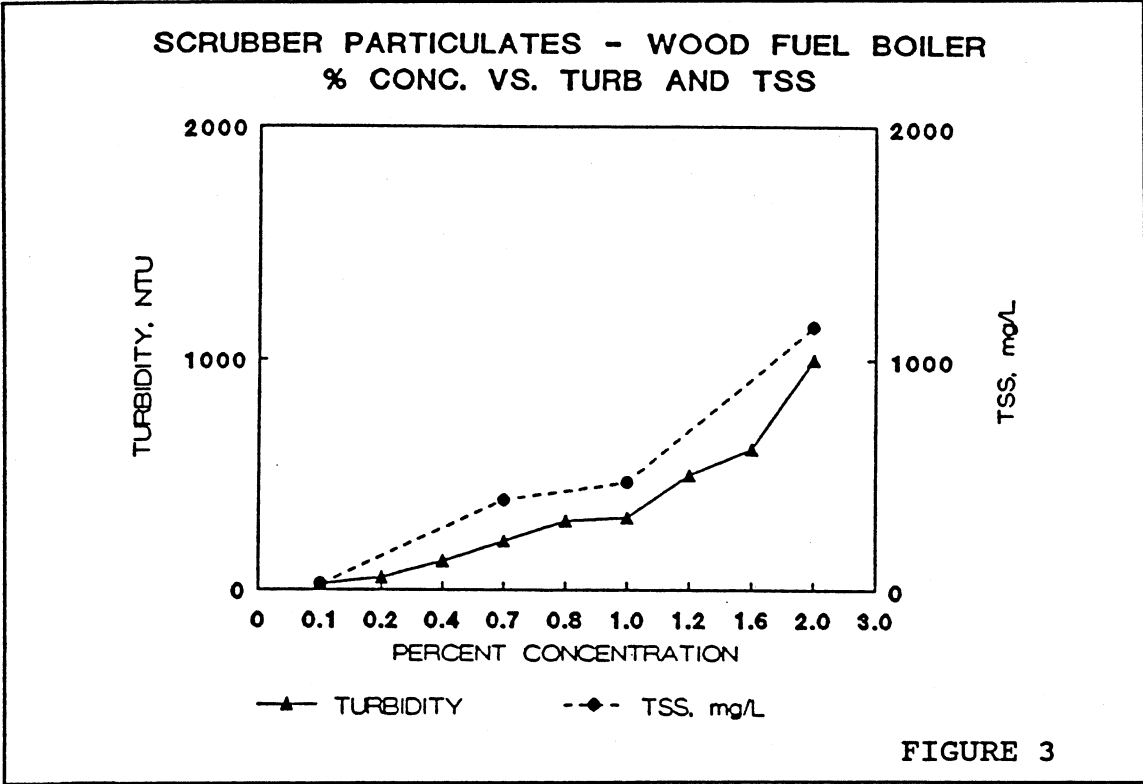
TURBIDITY VS. TSS FOR WHITE LIQUOR					
Dilution Number	Sample Conc.	Turbidity Ratio XR	Turbidity 2100A	TSS mg/L	
1	100%	156.8 NTU	24.3 NTU	185.4	
2	80	128.7	23.8	164.8	
3	60	100.0	20.0	114.5	
4	40	67.7	15.8	64.9	
5	20	35.7	10.6	39.1	
6	0	0.2	0.4	0	

TABLE 1

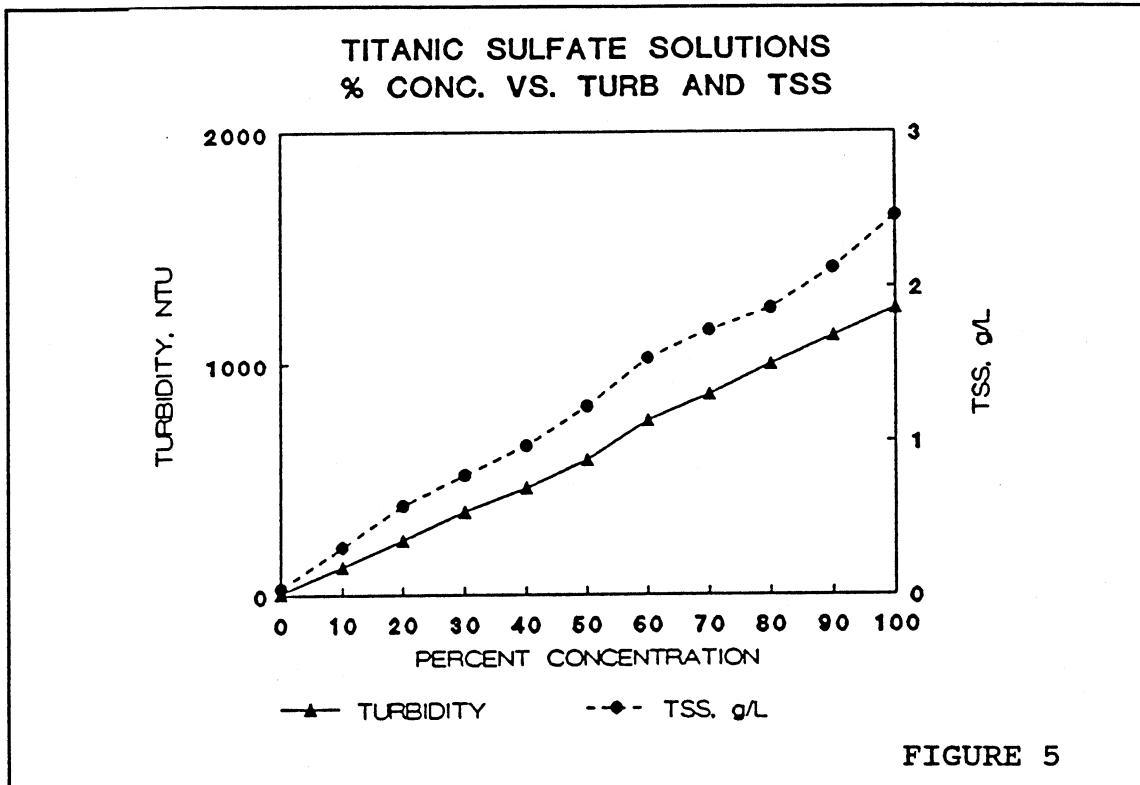


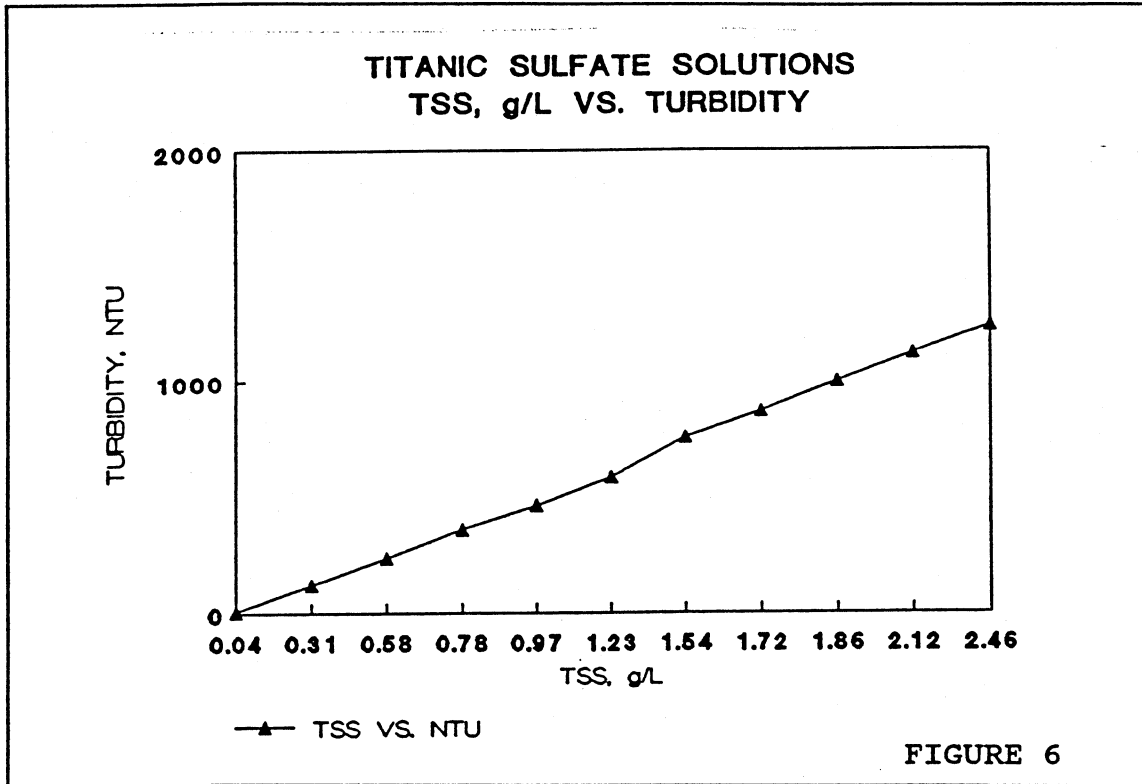
As illustrated in Figure 2, the turbidity readings with the ratioing turbidimeter (Ratio/XR) show a linear relationship to total suspended solids (TSS) with a relatively steep slope over the entire range of dilutions. Turbidity readings from the non-ratioing turbidimeter (2100A) show a linear relationship. However, the relatively flat slope indicates poor sensitivity and will make correlation more difficult. The ratio system is able to better compensate for sample variables. For this sample, use of a ratioing-type instrument will equip the user to monitor the liquor with good correlation to total suspended solids.

Example: A wood processing plant wished to monitor the concentration of fly ash in a stack scrubber from a burner. Excess concentration of fly ash in the scrubber water caused a decrease in scrubber efficiency and unacceptable particulates in the exhaust gas. At the same time, the plant wanted to limit the amount of water added to the scrubber. For this experiment, the original sample had to be diluted 1:50 (a 2% solution by volume) to bring the sample within range of the instrument. Figure 3 illustrates the relationship of turbidity and TSS to the percent dilution. Figure 4 illustrates the relationship of the turbidity to TSS.



Example: A manufacture wished to use turbidity measurement to detect and control the amount of titanic sulfate in a process stream. For this sample, no initial sample dilution was necessary and the sample was not temperature sensitive. Figure 5 illustrates the relationship of percent dilution to turbidity and TSS. Figure 6 illustrates the relationship between TSS and Turbidity.





Example: A manufacturer of titanium sulfate wished to monitor a filtration process used after precipitation of titanium dioxide from solution. Two samples were tested. The first sample was filtrate (First Moore Wash Filtrate). An initial dilution of 50 percent was required. Figure 7 illustrates the relationship between percent concentration, TSS and NTU for the wash filtrate. The second sample, First Moore Pickup Filtrate, the filter cake, was more concentrated requiring initial dilution to a one percent solution. Figure 8 illustrates the relationship between percent concentration, TSS and NTU for the pickup filtrate. Figure 9 illustrates the relationship for both samples between TSS and NTU.

TITANIUM SULFATE CONC. BLACK LIQUOR  
% CONC. VS. TURB & TSS, 1ST MOORE WASH

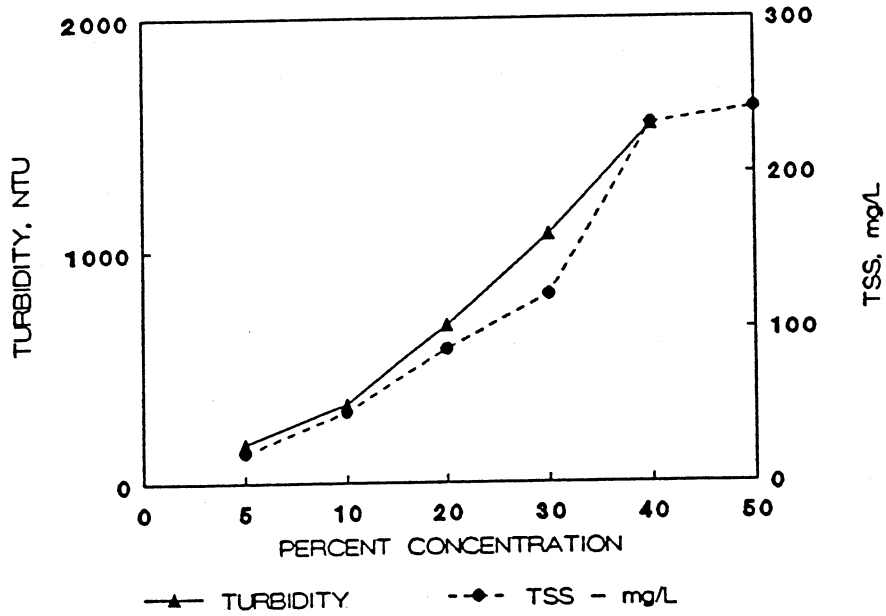


FIGURE 7

TITANIUM SULFATE CONC. BLACK LIQUOR  
% CONC. VS. TURB & TSS, 1ST MOORE FILT.

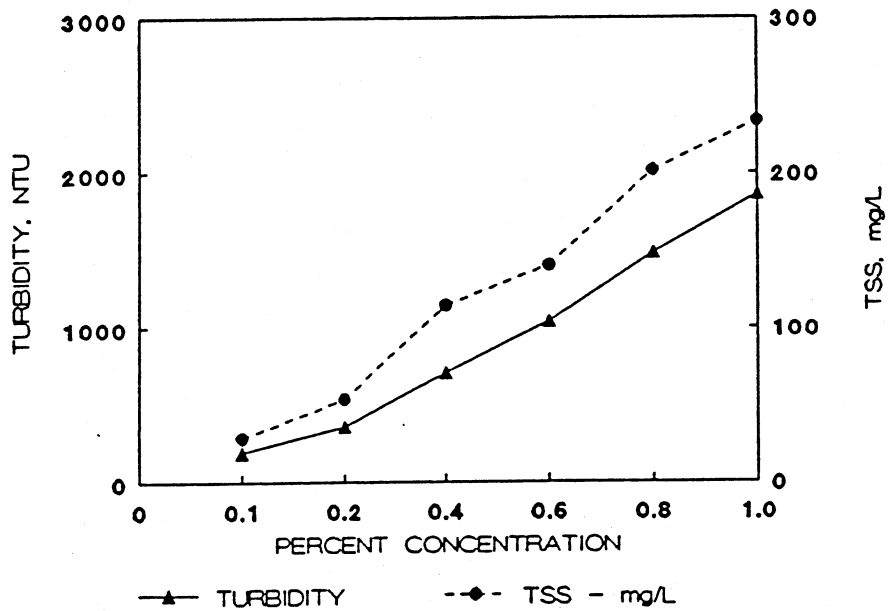
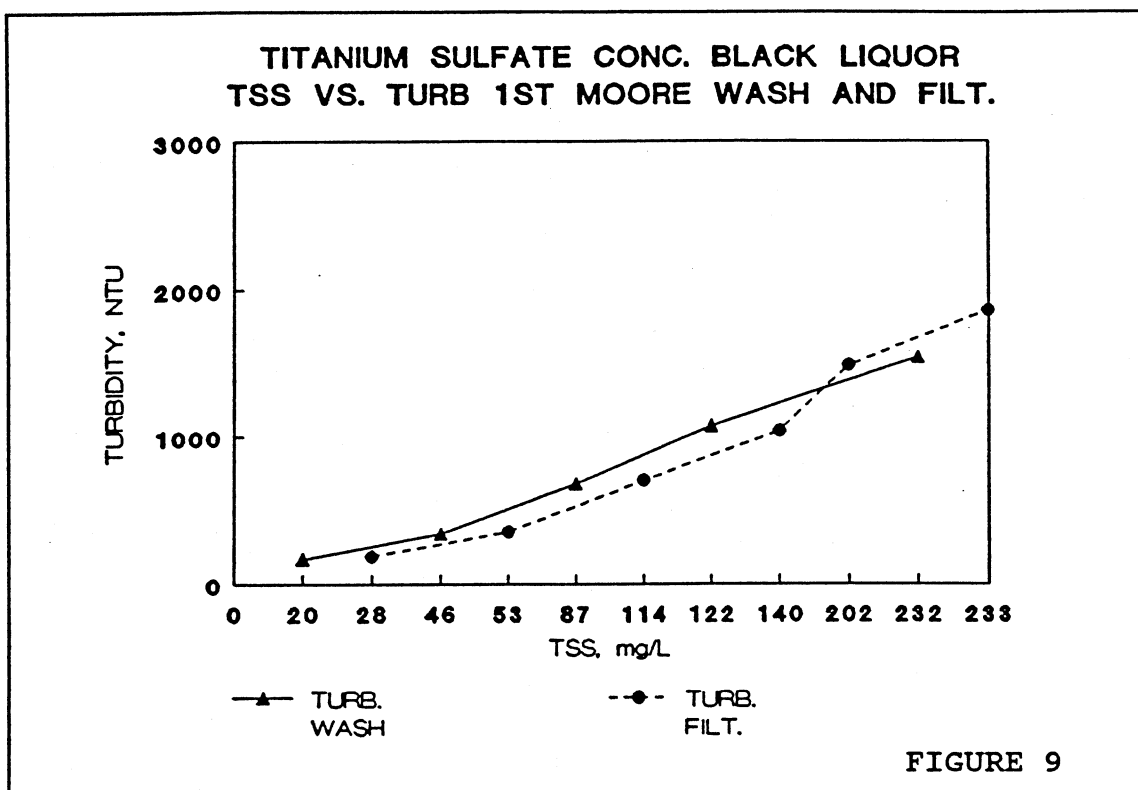


FIGURE 8



### Conclusion

A turbidimeter can be used as a surrogate measure of suspended solids if sample and instrumental variables can be properly controlled. The keys to obtaining an accurate and reliable relationship between turbidity and total suspended solids are:

1. The sample must be a true representation of the sample stream from which it came.
2. All dilutions must be treated the same throughout the study.
3. Consistent technique must be used.
4. A well mixed sample and dilutions of the original sample must be used in all sample manipulations (pipetting, measuring turbidity, transferring to sample cells).
5. Environmental conditions must be consistent throughout the test to reduce variability in the instruments and in the sample. When possible, the temperature of dilutions should be the same as the sample will be in the process environment.

6. The correlation study must be done in a timely manner. The longer it takes to perform the correlation study, the more chance exists for the sample, instrument, or environmental changes to occur.

Establishing a correlation can be time-consuming. However, the result can save not only time but a significant amount of product and process down-time. The response to a process upset is much faster and the upset is corrected more rapidly, resulting in even greater savings in time, labor and product, if a correlation is established.