Spotlight

Environmental Control & Water Monitoring

In many industries turbidity represents a fundamental and versatile parameter for quality control. In addition to the classic determination of undissolved particles in drinking water, ie. it is also employed for the control and verification of process sequences.

Unlike other measurement parameters, not only standardised methods for measurement are generally accepted in the analysis of turbidity, but also empirically established procedures as well as industry and company-specific processes.

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TURBIDITY MEASUREMENT - NOT ONLY NEPHELOMETRIC AT 90°!

Turbidity can be described only with limitations as a physical and technical measurement category. The unusual nature of the turbidity measurement in comparison to other parameters can be recognised already in its historical development: Initially a procedure was introduced by Jackson and Whipple around 1900 determining turbidity by means of candlelight under a glass tube. Dilution series of diatomaceous earth (diatomite) with its high level of diatoms were used as calibration standards. The turbidity value was determined visually from the top view. Admittedly a somewhat subjective procedure with the measuring unit of JTU, where 1 Jackson Turbidity Unit corresponds to 1 ppm SiO_2. However, this was providing the basis for the development and continuous improvement of turbidity measurement with meaningful results.

For reaching a more objective interpretation of results and minimising sources of errors the modern optical and electronic technique have improved today's options notably. With the introduction of microprocessors in instrument construction, the measuring ranges have been expanded: Turbidity is linear just in sections, not over the entire measuring range and especially not up to the 10,000 NTU offered today. Therefore, in contrast to the usual chemical understanding, a sample dilution does not necessarily result in a mathematically calculable turbidity value. Particle size and increasing concentration influence the final turbidity value essentially, as the effects of backscattering, multiple scattering and cancellation of each other by shielding effects will be recorded accordingly as signal on the photocell, which anyway can be evaluated by means of microprocessors.

SCATTERED LIGHT EFFECTS AS A FUNCTION OF PARTICLE SIZE

For water, a value of approx. 0.02 NTU is representing the natural base line because of scattering effects. They are caused by the molecular properties of the water itself (at 560 nm) and found their way into the definitions of standard methods for the analysis of drinking water. Thus, the common value for the low-end calibration standard of many laboratory instrumentation has been put to 0.02 NTU. Accordingly, measurement values smaller than 0.01 NTU should be recognised as a more nominal than actually realisable resolution – however, differences in the online and laboratory methods must certainly be taken into account. Particles with a smaller size in "diameter" than the wavelength used for the measurement (less than 560 nm using a tungsten lamp or 860 nm using IR) scatter in all directions symmetrically though somewhat dimmer. This is changing as soon as an interaction of scattering subjects is given, and/or a regular structure is performed.

In case of larger particles besides the absolute size and the shape and structure are influencing the scatter effects in the light beam. Here it is a matter of fact that the forward scattering prevails. Larger particles include agglomerates, Kieselguhr and cell layers and are identified as entities in respect of the detector. *Figure 1* shows the significant role of light scattering angle and the influence of potential measuring angle position in the case of bigger particles. Whereas in the case of smaller particles a more or less uniform scattering takes place in all directions, larger particles perform an unsymmetrical distribution of scattered light including back and forward scattering. For smaller angles of light scattering, e.g. between 10 and 45° the scattering effect is considerably higher. Many industries take advantage of this varying scattered light response for different processes, i.e. the brewing trade: Filters can be monitored in the process by determination of the required amount of kieselguhr and control of filter breakage or filtration itself. Choosing the nephelometric measurement at 90°, the presence of colloids can be checked for the important beer quality attributes of brightness and stability, not being disturbed by other turbidity effects of large particles such as yeast or kieselguhr. This kind of measurement is mostly performed online or inline. WTW is offering two sensors for online measurement: VisoTurb for nephelometric online measurement mainly in water applications and the sensor ViSolid for measurement of total suspended solids by recording back scattering effects.

WHAT ARE MEASUREMENTS WITH THE RATIOMETRIC METHOD?

Ratiometric measurement in general means simultaneous detection of signals at different angles by setting the results into correlation to each other before output. The degrees chosen as well as the number of angles and the applied algorithms are not subject to any national regulation or standard method and therefore are application or manufacturer specific! Measurements in ratio mode must always be specified with an additional ID for Ratio, i.e. NTU (Ratio), and state a manufacturer, trade or company specific "in-house" result, not necessarily being comparable to others.

Results in the ratio mode for small turbidity values up to 40 NTU approach the nephelometric value and deviate in the upper range in a progressive way. Reasonably, all standard methods implement the nephelometric procedure of 90° scattered light measurement for the lower application ranges such as found in the standard regulations of turbidity measurement for drinking water.

In earlier times when improving turbidimeter, the better linearity and the compensation of coloration were seen as advantages of ratio measurement. Nowadays, however, neither of these factors have a big impact: The linearity - given only in sections anyway – is realised by means of modern electronics and microprocessors for all instrument models and measuring procedures. To exclude coloration effects of watery solutions, the choice of an infrared light source is already sufficient for most of the applications.

In total, the ratiometric procedure does not provide any advantages for measurements up to approx. 1000 NTU because particle density and insignificant multiple scattering allow reliable results absolutely with the nephelometric measuring method.

MEASURING RANGES ABOVE 4000 NTU: NO REFERABLE PROCEDURE

For all measuring ranges above 4000 NTU neither a certain measuring method nor a specific hardware configuration is favoured: For this application range there are simply reference standards available; manufacturing instructions are specified only for formazin ending at 4000 NTU according to both, DIN ISO and US EPA standard methods. The extrapolation or any mathematical calculation of values based on 4000 NTU formazin proves to be complex or difficult due to the missing linearity in this measuring range caused by the backscattering and shielding effects between particles. The measuring range between 4000 NTU and 10,000 NTU cannot be referenced with anything: This makes the comparison between different instruments and their results impossible, taking the manufacturer-specific algorithms for ratio mode in this measurement range of turbidity into consideration, as previously mentioned.

Nevertheless, specific turbidity levels and scattered light effects can certainly be recorded in the range > 4000 NTU allowing useful statements about production processes and quality control. Therefore many producing companies have turbidimetric measurement systems following a company routine up to 10,000 NTU which provide reproducible results for specific applications. In this case, the focus is set on company-internal interests rather than on comparison of actual measurement results between instrumentations.

In particular, it can be very challenging to investigate time and effort experimentally in a new company standard for quality assurance by means of turbidity measurement. Application ranges reach from food industry (quality) throughout cell cultures (growth) up to petrochemistry (additives). With the

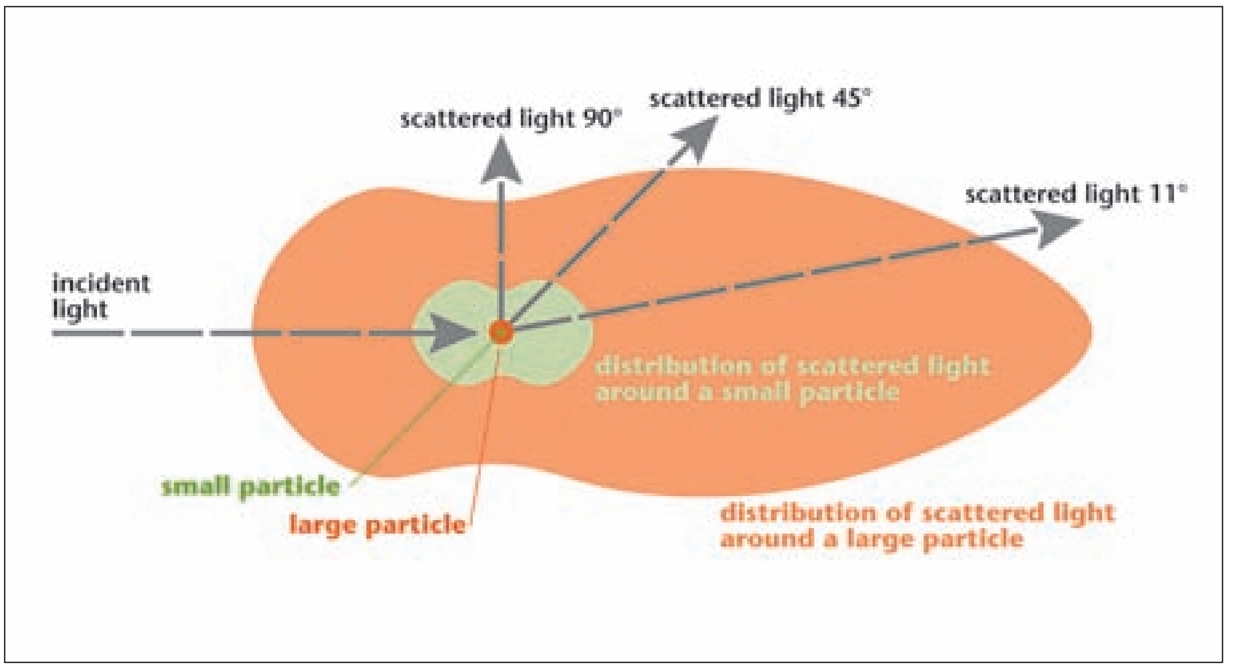


Figure 1. Schematic diagram of the distribution of scattered light (polar diagram) depending on particle size. Large particles exhibit strong forward scattering whereby additional minima and maxima occur according to the angle that have been neglected here.

Turb 430 series, WTW is offering a versatile turbidity measuring instrument for laboratory and field use at the same time providing DIN ISO and US EPA compliance. Besides nephelometric measurement, the lab model Turb 555 offers also ratio mode as well as transmission measurement.

WHAT CAN THE TURBIDITY MEASUREMENT ACCOMPLISH?

Taking a look at the diversity of applied optical systems as well as techniques, the difficulty of comparing different measuring instruments and even samples becomes evident.

The results from measurements with different measuring angles can be compared just as little as the results of various instruments with different light sources or different measurement modes such as ratio versus nephelometric measurements.

Results and their verification above 4000 NTU cannot be traced back to any reference standard or standardised method.

This does not mean, however, that this kind of measurement would be meaningless or procedural errors of instrument manufacturers could be stated.

On the whole, turbidity measurement affords great services for process and quality control within company sample environment by providing comparable and reproducible results for these specific tasks. Also sectoral procedures have been established in individual industries i.e. such as the above mentioned application angle in brewing trade. Moreover, the full potential applications and application fields have not yet been tapped!

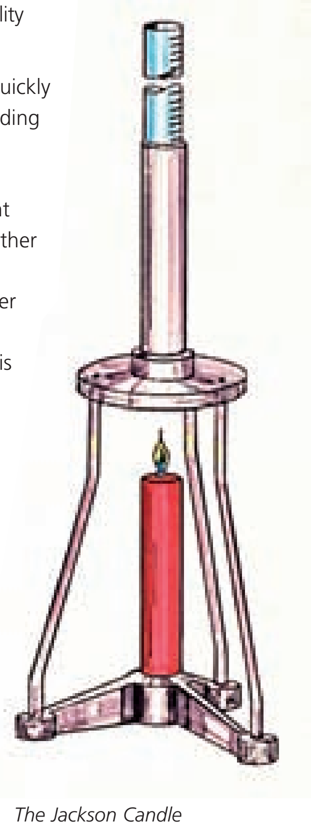
Turb 555

HISTORY: THE JACKSON CANDLE TURBIDIMETER

Based on a stock solution of diatomaceous earth with 1000 ppm silicic acid suspension a dilution series has been made. The diatomaceous earth consists of sedimentary layers from million of years old the sea containing fossil diatoms with enclosed silicon dioxide in their shells. This silica finally is present in form of silicic acid in diatomaceous earth.

For "calibration" a glass tube with flat bottom and a candle below has been calibrated by means of the dilution series: The standards were poured in slowly, looking from the top through the glass tube. With increasing turbidity the visible candle flame started to appear gradually as uniform shimmer, which resulted in the respective calibration mark or – for an unknown sample - in a turbidity value by comparing it with the respective calibration mark. There is no direct comparability with present-day units.

This transmission method quickly revealed limitations in recording low turbidity values (below 25 JTU). For this purpose, nephelometric measurement became accepted in the further development: This applies especially for analysing water with low turbidity values. Today, diatomaceous earth is known as "kieselguhr" and is used in pre-processed form as auxiliary material for filtration in filtration equipments.



The Jackson Candle Turbidimeter

COMMON MEASURING METHODS

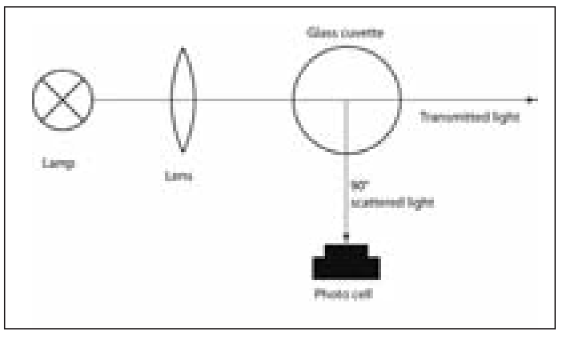
- Scattered light measurement: nephelometric measurement with an angle of 90°
- Measurement with forward / backward scattering: measurements at different angles (e.g. 11°)
- Light transmission method: with 0° as attenuation = absorption of the light
- Two angle instruments: application and manufacturer specific instruments offering ratiometric measurement by putting the result of nephelometric measurement into correlation with result of other angles before displaying final result with "ratio" mark.

The figures show diagrams of various measurement methods with different detector arrangements measuring the incoming signal:

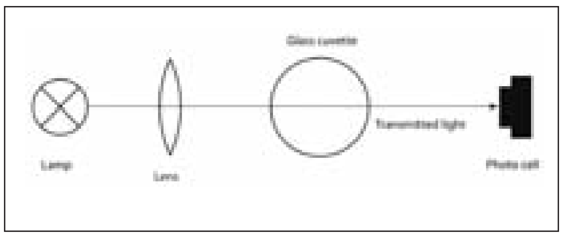
a) nephelometric method at 90°

b) transmitted light at 0° (transmission/absorption)

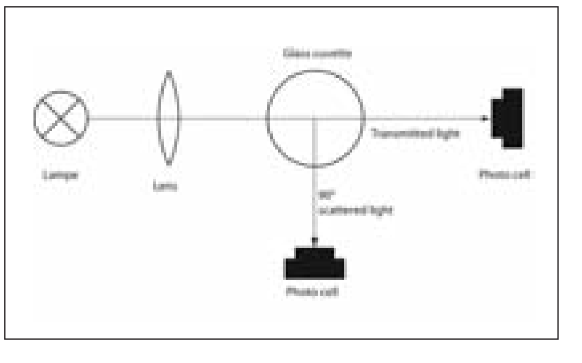
c) ratiometric method with setup of 90°/0°



Turbidimeter (a)



Turbidimeter (b)



Turbidimeter (c)