PDA3000 Particle & Flocculation Monitor

Operating Manual

Manufactured by:
Rank Brothers Ltd
56 High Street, Bottisham, Cambridge CB25 9DA, England
Tel: +44 (0)1223 811369 Fax: +44 (0)1223 811441
Website: http://www.rankbrothers.co.uk/

Contents

Warranty

We guarantee the manufacture of the instrument and parts against faults for a period of twelve months from the invoice date.

If a fault should occur within this period then we undertake to either:

- Supply free of charge replacement parts for you to fit to the instrument.
- Upon return of the item at your expense, repair or replace (at our discretion) the instrument free of charge and return it to you at our expense.

FCC Notice

This device complies with part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) This device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.

European Union Compliance

The PDA3000 is CE marked and conforms to the following product specifications:

EMC	EN 61326-1:2006 (Immunity) EN 61326-1:2006 (Emissions)

1. Introducing the PDA3000

The PDA3000 is a simple, rugged, but very sensitive monitor for flowing suspensions and emulsions, based on an optical technique developed at University College London. The unit is very compact and ultra-lightweight (width: 200 mm, depth: 40 mm, height: 750 mm, weight: 375 g).

Applications include:

- Selection of optimum coagulant dosages.
- Control of dispersion and emulsification processes.
- Monitoring of floc growth, breakage and re-growth.

Some of the advantages of the PDA3000 over more conventional techniques are:

- Very simple to operate—little attention needed.
- Powered from a computer—no separate power supply needed.
- A very wide range of suspension concentrations can be directly monitored.
- No orifices to clog.
- Minimal problems due to contamination of optical surfaces.
- Flow-through operation, ideal for on-line applications.
- Novel flow cell using inexpensive, disposable plastic tubing—no connection problems.

1.1 Nature of the Technique

The flowing suspension is illuminated by a narrow beam of light perpendicular to the direction of flow. For the PDA3000, a novel flow cell has been developed; transparent flexible tubing fits in a detachable block that houses a light source (miniature light-emitting diode) and a sensitive photodetector. The LED has a wavelength in the near infra-red and the detector response is closely matched to this. The sample flows through transparent plastic tubing located in a slot between the light source and detector. A narrow light beam passes through the flowing sample by means of two aligned pinholes about 1 mm in diameter.

The output from the photodiode is converted to a voltage, which consists of a large average (DC) component, together with a small, fluctuating (AC) component. The DC component is simply a measure of the average transmitted light intensity and is dependent on the turbidity of the suspension. The AC component arises from random variations in the number and size of particles in the sample volume (i.e. the volume illuminated by the light beam, typically of the order of 1 mm^3 with a 3 mm tube). Because the suspension flows through the cell, the actual sample in the light beam is continually being renewed and local variations in particle number and size give

fluctuations in the transmitted light intensity. These fluctuations cease when the flow is stopped.

The root mean square (RMS) value of the fluctuating signal is related to the average number concentration and size of the suspended particles. For fairly uniform suspensions, estimates of particle size and number concentration can be made, but the main use of the PDA3000 is in the monitoring of flocculation and dispersion processes.

The RMS value of the fluctuating signal increases, when aggregation of particles occurs. Measurable changes in the RMS value occur long before any visible signs of aggregation are apparent. Conversely, when aggregates are broken, the RMS value decreases, reaching a minimum when disaggregation (or dispersion) is complete. The DC value (related to the turbidity) is much less sensitive to changes in the state of aggregation, but can still give useful information. In many cases, the Ratio value (RMS/DC) is the chosen parameter, since this is not affected by drift in the opto-electronic components or by 'fouling' of the tube wall by adhering particles. The Ratio is often referred to as the 'Flocculation Index'.

2. Software

2.1 Requirements

The PDA3000 communicates with your computer via the included Rank Brothers Logger software. This software requires a computer running one of the following operating systems:

- Microsoft Windows 7 SP1 (or later service pack)
- Microsoft Windows 8.1
- Microsoft Windows 10

2.2 Installation

To install Rank Brothers Logger:

1. Insert the included disc into your disc drive.
2. An autoplay window should appear, press the *Run RBLogger* button. (If the autoplay window does not appear then open Windows Explorer, go to Computer and double-click the icon for your disc drive.)
3. If asked, confirm you want to allow the software to make changes to your computer.
4. When the *Rank Brothers Logger Setup* window appears, press *Next*, choose where you want the software to be installed, then press *Install*.
5. The installer may take several minutes to complete, once done press the *Finish* button to complete installation.

3. Operation of the PDA3000

Flow cells available for the PDA3000 can accommodate standard plastic tubing of 1, 3 or 5 mm internal diameter. The choice of tubing depends on the nature of the suspension and the amount available. The 3 mm tubing requires a minimum suspension flow rate of around 10 mL/min and the flow rate should not exceed about 40 mL/min. (For high flow rates, quite high shear rates are developed in tube flow and some breakage of aggregates may occur.) For many practical applications 3 mm tubing would be appropriate, but other widths might be better in certain cases. (See Choice of Flow Rate in the Appendix for more information.)

Insert the tube in the slot and fit the retaining bar by sliding it into the two grooves. (Note: the retaining bar can only be inserted from one side of the cell.) When the retaining bar is correctly fitted, it will slightly compress the plastic tube and hold it firmly in position. The tubing will be flattened against the walls of the slot and this gives a well-defined optical path length.

Connect the PDA3000 to the computer, using the supplied USB cable and run the software by double-clicking the Rank Brothers Logger icon. The screen shown in Figure 1 should appear. The results are displayed in graphical form and can be saved to a file. The saved data are RMS, DC and Ratio (RMS/DC). Any two of these values can be plotted, as Channel 1 or Channel 2. All three of the current sample values are displayed at the bottom of the screen. Since the RMS value can be very small (much less than 1 mV) a gain of 20× is applied to the fluctuating signal before the RMS value is derived by the PDA3000. Also, to enable the Ratio value to be conveniently displayed, a factor of 1000 is applied by the software. Thus, the displayed Ratio values are 20,000 times higher than the ‘true’ values.

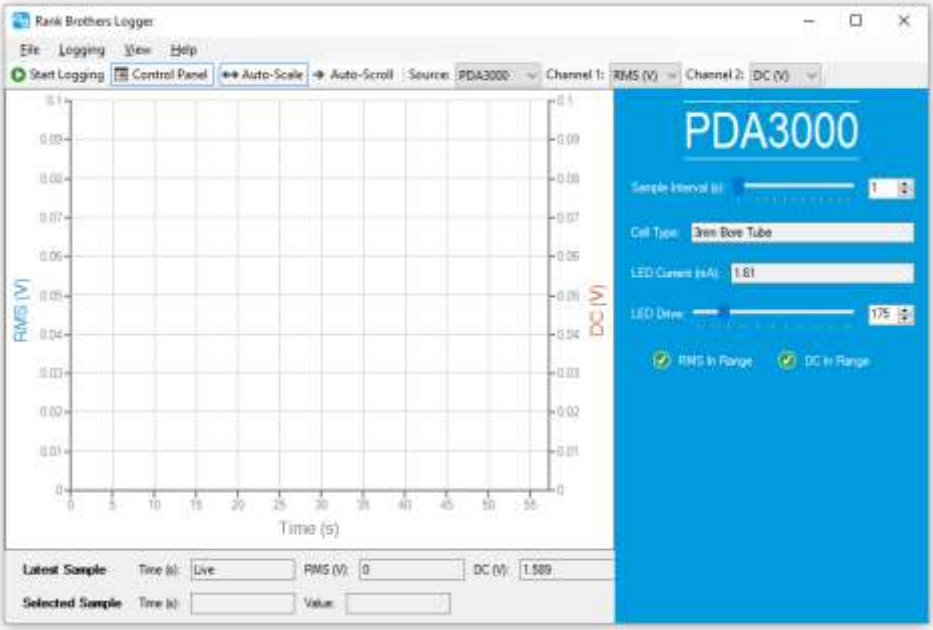

Figure 1 Rank Brothers Logger—Opening Screen

Before monitoring, some settings have to be made in the control panel on the right-hand side of the screen. (If this panel is not visible, press the *Control Panel* button at the top left of the screen.)

1. The *Sample Interval* can be set at a value from 1 second upwards. For many applications, frequent sampling is required, but for long-term monitoring samples can be taken at longer intervals.
2. The LED current (which determines the light intensity) should be set by entering a number (typically 250 or less) in the box on the right-hand side of the *LED Drive* section, followed by pressing the *Enter key*. The actual LED current (mA) is displayed in the panel above and this will normally be of the order of a few mA. (Note: the default LED Drive setting is 175 and this typically gives an LED current of 1–2 mA.) It is important not to set the LED current too high or incorrect readings will be given. With clean water (or other liquid) in the tube, the DC value should not be higher than about 3.5 V. When there are very large particles (or flocs) in the flowing sample, it may be necessary to reduce the LED current further. Generally, this may be necessary when the displayed RMS value exceeds around 0.6 V. For very turbid samples, only a small fraction of light is transmitted and it may be necessary to increase the LED current, so that the DC value becomes greater. In practice, the

appropriate LED current needs to be set according to the nature of the sample under test.

The mode of operation of the PDA3000 depends very much on the nature of the test procedure. Essentially, a representative sample of the test suspension must flow continuously through the tube at an appropriate rate. The flow may be once-through or could be recirculated back to the sample vessel. Once-through flow is convenient when there is ample supply of suspension and this can be achieved simply by gravity. Re-circulation requires some form of pump, which should be located downstream of the monitor, to minimise problems of floc breakage. The recirculation method is typically used in a modified 'jar test' procedure, where sample in a stirred vessel is passed through the PDA3000 and then back to the vessel. This method has been widely used to study the action of different coagulants, including floc strength, breakage and re-growth of flocs and others. Optimum dosages of coagulants (and dispersants) can also be easily determined.

With the sample flowing, monitoring is started by pressing the *Start Logging* button.

During a test the chosen parameters are displayed graphically, so that their changing values with time can be easily seen. There is a choice of *Auto-Scale* or *Auto-Scroll* for the display, by pressing the appropriate button above the screen. If *Auto-Scale* is chosen, then the whole record from the start of logging is displayed. With *Auto-Scroll*, only values over the last minute or so are shown (depending on the size of the window). This can be adjusted from the *X-Axis Scale* submenu in the *View* menu.

At the end of a test, press the *Stop Logging* button. Data can then be exported as a text file or as a spreadsheet (including Microsoft Excel) by pressing the relevant item in the *File* menu. When the data are saved, the data logger can be cleared by pressing the *Clear Log* item in the *Logging* menu.

4. Appendix: Theory and Applications

4.1 Theory

The basis of the technique used in the PDA3000 lies in the fundamental non-uniformity of suspensions and emulsions, when examined on a sufficiently small scale [1, 2]. If a small sample is examined microscopically it is apparent that the local particle concentration varies randomly over the field of view. In a flowing sample, local variations in composition are made apparent as fluctuations in the intensity of a narrow beam of light transmitted through the suspension, or, in other words, fluctuations in turbidity (see Figure 2). The method is widely known as the *Turbidity Fluctuation Technique*.

When light transmitted through a suspension is measured (as shown in Figure 2) the turbidity can be defined in terms of the optical path length, L, the particle number concentration, n, and the scattering cross-section of the particles, C. (For simplicity, we are assuming that all of the particles have the same size). If the incident light intensity is I_0, then the transmitted light intensity is given by the Beer-Lambert law:

$$I = I_0 \cdot exp(-\tau L) \qquad (1)$$

Where τ is the turbidity, given by:

$$\tau = nC \qquad (2)$$

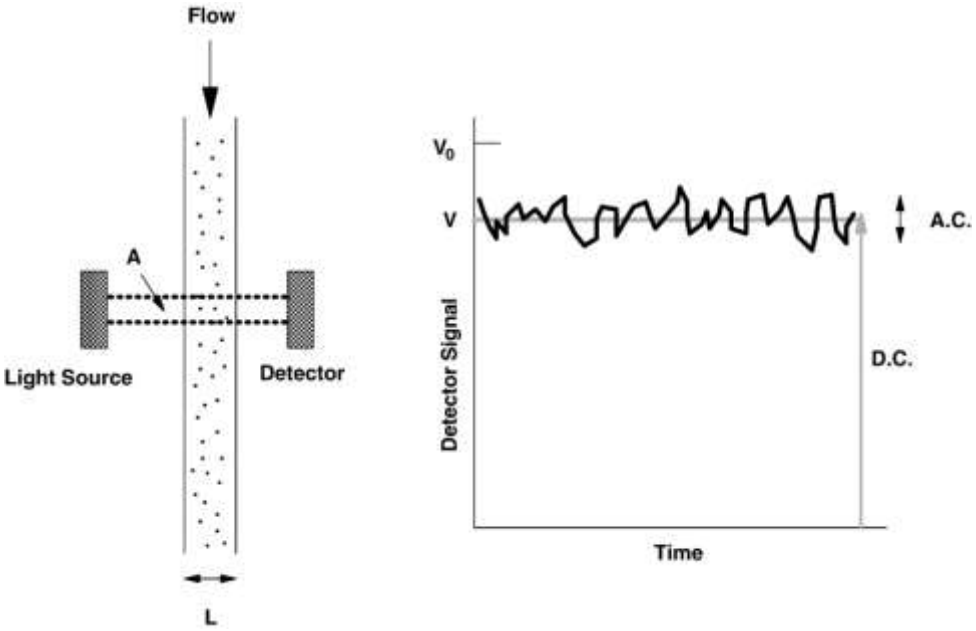

Figure 2 *Turbidity Fluctuation Technique*—Schematic

Since the light intensity is converted to a voltage by the detector, the Beer-Lambert law can be written as:

$$V = V_0 \cdot exp(-\tau L) \qquad (3)$$

Where V_0 is the voltage corresponding to the incident light intensity (i.e. with clean fluid in the light beam) and V is the voltage when there are particles in the light beam. Strictly, V is the average value of a fluctuating voltage and is here called the DC value.

The detector output would typically show a mean (DC) value of a few volts and the fluctuations (AC) might be of the order of only a few mV or less. The normal procedure is to derive the root mean square (RMS) value of the voltage fluctuations and to divide this by the mean (DC) value, to give a Ratio value R. In the PDA3000 the AC component is separated from the DC and then amplified by a factor of 20 before the RMS value is derived.

It can be shown that, for a monodisperse suspension (all particles of equal size,) the Ratio is given by:

$$R = \frac{V_{RMS}}{V} = \sqrt{\frac{nL}{A}}C \qquad (4)$$

Where A is the effective cross-sectional area of the light beam.

During operation of the PDA3000, the optical path length, L, and the beam dimensions remain constant. So, the Ratio value depends on the square root of the particle concentration and on the scattering cross-section of the particles. For particles larger than around 10–20 µm, C is roughly proportional to the cross-sectional area of the particles and so depends greatly on particle size. As particles aggregate, the number concentration decreases and their scattering cross-section increases. In practically all cases, the rise in C outweighs the decrease in n, so that the Ratio value increases as particles aggregate.

Although equation 4 is based on the assumption of equal-size particles, the same general conclusions apply to the more practical case, where there is a distribution of particle size. The Ratio value is still proportional to the square root of the particle concentration, and increases with aggregation. However, larger particles have a greater influence on the Ratio value. Small particles among much larger aggregates will have very little effect on R.

Because both the turbidity and the Ratio value depend on the particle concentration, n, and the scattering cross-section, C, it is possible to determine n quite easily. From equations 2–4 the following expression can be derived:

$$nLA = \left(\frac{ln(V_0/V)}{R} \right)^2 \qquad (5)$$

The term on the left-hand side is the average number of particles in the sample volume, (i.e. in the light beam). If the beam dimensions are not known, then the monitor could be calibrated by passing a monodisperse suspension (e.g. polystyrene latex) of known concentration through the tube and monitoring the DC and RMS values. It is also necessary to measure V_0, when clean water is flowing.

Equation 5 shows that the particle number concentration can be determined from the DC and RMS values, together with the beam dimensions, without any knowledge of the light scattering properties of the particles. If the mass concentration of particles is known, then the mass of each particle could be derived and hence the particle size. However, this only applies to suspensions of equal-sized particles. In the more practical case, when there is a range of particles size, the number concentration derived from equation 5 is lower than the actual value. However, for an aggregating suspension,

equation 5 gives a good indication of the relative change in particle number and hence of aggregate mass.

4.2 Choice of Flow Rate

The PDA3000 is operated very simply by passing the suspension under test through a suitable plastic tube located in the gap between light source and detector. As previously mentioned, the choice of flow rate is quite important. In deriving the RMS value, only fluctuations within a given frequency range are used (roughly 1–350 Hz). The frequency range of the AC signal depends on the time of passage of particles though the light beam and hence on the flow rate. If the flow rate is too low, some of the lower frequency contributions are lost, so the measured RMS value is reduced. For higher flow rates, some of the upper frequencies are lost, which again reduces the RMS. Also, at high flow rates, especially with narrow tubes, the shear rate can become quite high and this can cause breakage of aggregates and a reduction in RMS. The following table gives suggested ranges of flow rates for three different tube diameters. At these flow rates the tube flow will be laminar.

Tube Diameter (mm)	Flow Rate (mL/min)
1	0.5–2
3	10-40
5	25–100

If flow rate is in the correct range, then changing the flow rate should not affect the Ratio value.

4.3 Applications

The PDA3000 can be used to gain information on particle dispersions, especially on the state of aggregation of particles. The most common use is with coagulation/flocculation processes. In the laboratory, the PDA3000 can give very useful information to supplement the standard 'jar test' procedure. In the jar test, coagulant is added to a suspension, which is then stirred for a standard period and then allowed to settle. After settling, the turbidity of the supernatant liquid (the Residual Turbidity) is measured. The test is repeated for different dosages of coagulant in order to establish the optimum dosage under given conditions. If the suspension is sampled continuously through the PDA3000, then the process of floc formation can be monitored throughout the stirring period. Also, the breakage and re-growth of flocs can be studied by a brief increase of the stirring speed (and hence the effective shear rate) of the suspension. An example is shown in Figure 3.

In this example a 30 mg/L suspension of silica particles, 1.5 µm diameter, in London tap water was dosed with aluminium sulphate ('alum') at 0.2 mM Al (about 5 mg/L as Al). The coagulant was added at 100 s and the suspension was stirred at 50 rpm. At 450 s the

stirring speed was increased to 400 rpm for 20 s and then reduced to 50 rpm. The growth of flocs is clearly shown by the rapid rise of the Ratio value. This eventually reaches a plateau value where further floc growth is limited by the shear in the stirred sample. When the stirring speed is increased to 400 rpm there is a sudden and rapid decrease of R, because of floc breakage. When the speed is restored to 50 rpm the flocs re-grow, but only to a limited extent. Note that the DC value (and hence the turbidity) changes very little during the entire process. The Ratio value gives a much more sensitive indication of changes in the state of aggregation of particles.

Although the Ratio value is strongly correlated with floc size, it is not possible to derive absolute size information. Nevertheless, results such as those in Figure 3 give very useful information on the relative floc sizes under different conditions.

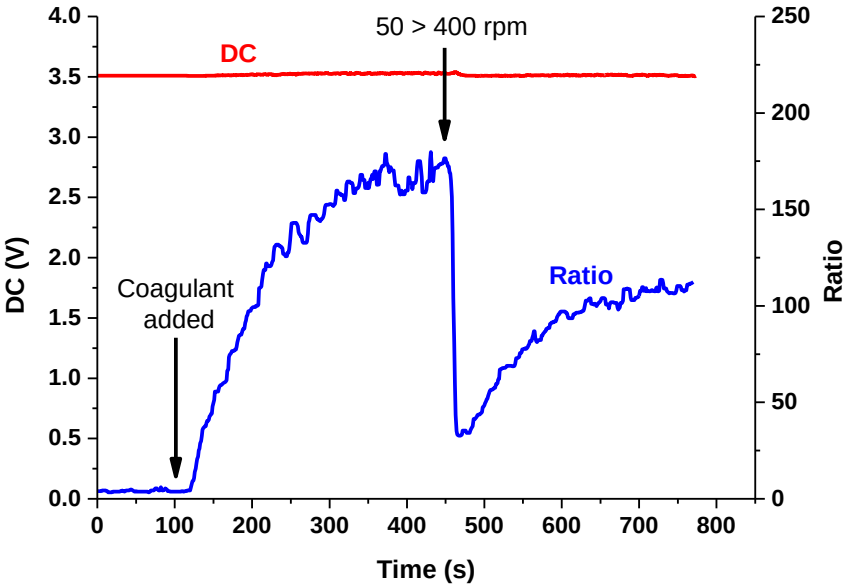

Figure 3 Floc Formation, Breakage and Re-growth—PDA3000 Response

There are many potential applications of the PDA3000, some of which are listed below, with relevant references:

- Papermaking systems [3]
- Asphaltene aggregation [4]

- Flocculation of bacteria [5]
- Creaming and flocculation of oil emulsions [6]
- Water [7] and wastewater [8, 9] treatment
- Floc strength investigations [10]

All of these works used an earlier Rank Brothers instrument—the PDA2000, which is based on the same *Turbidity Fluctuation* principle. The PDA3000 would give equivalent information in a more convenient manner.

5. References

[1] J. Gregory. Turbidity fluctuations in flowing suspensions, *Journal of Colloid and Interface Scienc*e, 105 (1985) 357–371.
[2] J. Gregory. Monitoring particle aggregation processes, *Advances in Colloid and Interface Science*, 147-48 (2009) 109–123.
[3] R. Gaudreault, T.G.M. van de Ven, M.A. Whitehead. Mechanisms of flocculation with poly(ethylene oxide) and novel cofactors, *Colloids And Surfaces A: Physicochemical And Engineering Aspects*, 268 (2005) 131–146.
[4] N.H.G. Rahmani, T. Dabros, J.H. Masliyah. Online optical monitoring of asphaltene aggregation, *Industrial & Engineering Chemistry Research*, 44 (2005) 75–84.
[5] A. Szepesszentgyorgyi, S. Barany, I. Mecs. Flocculation of Bacillus thuringiensis var. israelensis suspension and its efficacy against mosquito larvae, *Acta Biologica Hungarica*, 56 (2005) 151–164.
[6] Y. Cao, E. Dickinson, D.J. Wedlock. Creaming and flocculation in emulsions containing polysaccharide, *Food Hydrocolloids*, 4 (1990) 185–195.
[7] S.G. Kim, K.J. Choi, P.J. Yu, S.H. Kim, Y.D. Lee. Evaluation of mixing conditions using an on-line monitoring technique, *Korean Journal of Chemical Engineering*, 25 (2008) 19–24.
[8] T.P. Nguyen, N.P. Hankins, N. Hilal. A comparative study of the flocculation behaviour and final properties of synthetic and activated sludge in wastewater treatment, *Desalination*, 204 (2007) 277–295.
[9] Z.L. Yang, X.X. Liu, B.Y. Gao, S. Zhao, Y. Wang, Q.Y. Yue, Q. Li. Flocculation kinetics and floc characteristics of dye wastewater by polyferric chloride-poly-epichlorohydrin-dimethylamine composite flocculant, *Separation and Purification Technology*, 118 (2013) 583–590.
[10] P.K.K. Jin, X.C.C. Wang, H.X. Chai. Evaluation of floc strength by morphological analysis and PDA online monitoring, *Water Science and Technology*, 56 (2007) 117–124.