



# DustMon

## A Standard Operating Procedure

From the Microtrac Particle Analysis Laboratory (PAL)

SL-AN-44 Revision C

APPLICATION NOTE



## Introduction

This operating procedure is provided to assist the user of the Dustmon in obtaining information on the dust content of powders. The procedure may require operational changes depending upon the dust content and other aspects of the measurement. It is intended as a starting document. The Operator's Manual contains further details and options that may be of value in analyzing the data and should be read prior to making final decisions on a formal SOP that will include data presentation for a given company, material or user. Please contact Microtrac, Inc for specific questions should they arise.



- The entire procedure should be read before starting a measurement unless expertise has been attained in the use of the equipment.
- The purpose of this Standard Operating Procedure is to provide the customer or user the basic concepts required to achieve repeatable data as well as to provide the basic steps required on a daily operating basis.
- This SOP may require changes by the customer since each material sample may require special representative and sample preparation steps that are specific to a given industry and because the Microtrac Dustmon is a general purpose instrument. Its design is intended for use by a wide variety of industries as described in the Microtrac promotional and advertising literature.

## Safety and Sample Handling

- Identify the material to be measured.
- Obtain a material Safety Data Sheet (MSDS)
- Read and understand the requirements for safe handling of the material and fluids to be used.
- Use protective clothing, fume hoods, gloves, safety glasses and other safety equipment as prescribed in the MSDS.
- Obtaining a Representative Sample Prior to Sample Preparation and/or Measurement.
  - Dry powder samples
    - Two methods of mixing.
      - Powders in glass or plastic containers. Mix well by rotating and tumbling at least 10 times in each direction.
      - Powders in bags.
        - ✓ Mix in bag by rotating, tumbling and squeezing.
        - ✓ Transfer to a container and mix accordingly
    - Use sample scoop to obtain sample. Normal size spatulas are too small to make the process of weighing and transfer efficiently done.
- Sample Preparation: No sample preparation is required except as described above for storage.

## General Measurement Considerations

- The standard amount of sample should be 30 grams measured for 30 sec. Longer times may be preferred depending upon the situation. If there is a lot of dust (dust index greater than 20 -

30) and the curve is not decreasing, a longer time should be selected to give a better indication as to how long the dust stays in the air.

- Dust index should be less than 50. Appropriate dust index values are 4 – 12 with the white light. If less than 3, then the laser should be used. If value is greater than 50, then repeatability may not be as good.
- Dust may get trapped in a large mass that drops into the bin and not be released on a repeatable basis. Note a value of 100 is total blockage of the light (i.e. putting your hand in front of the light). Clean the bin and tube after every sample. This avoids “small clouds or poofs of dust” sent up when the new sample hits the old sample thus getting a combination of the two.
- Samples should be stored in a desiccator to prevent humidity effects. Humidity and other agglomeration effects can affect the dust amount.
- Microtrac S3000 and S3500 diffraction particle measurements may show different results than Dustmon.
  - Dustmon is only measuring dust in the air. Microtrac S3500 diffraction instruments measure the entire distribution.
  - There is no attempt to disperse agglomerations in the Dustmon.
  - It has been shown that the dispersion of sample in fluid in diffraction may show more fines in the distribution than indicated by Dustmon. This situation was examined and found that running TurboTrac without any energy showed that the Dustmon was correct since non-dispersed sample was measured. The Dustmon showed more fines with the Arizona Coarse Test Dust than the Arizona Fine Test Dust. TurboTrac (no energy) reported Coarse Test Dust had more fines as well. Thus the TurboTrac (no energy) and Dustman (no energy) concurred for dry measurements. Wet measurements were good for dispersed particle and full PSA.
  - If questions arise about fines as described above, TurboTrac measurement and wet measurements may be required to support Dustmon data.
  - Data (dust amount and particle size) are mostly used to compare to other samples and the relative propensity to produce dust under the conditions of the measurements.

### Computers and Software:

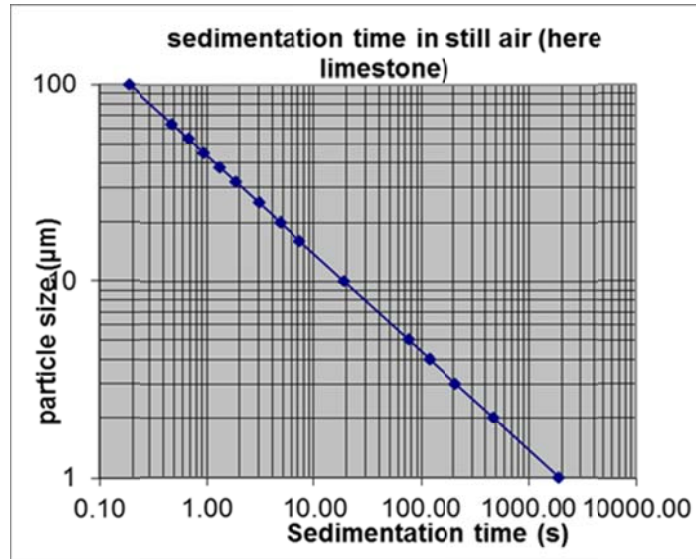
Microtrac/Anatec software is compatible with Windows XP and later computer operating systems. If there is a question, consult Microtrac.

- Assure that the Dustmon bench power switch is ON.
- Turn on all modules associated with the Computer module (monitor and printer).
- Double "click" on the Anatec shortcut icon. Software will open to the data display.
- Click on SYSTEM and click on LOG-IN. Enter the required password. This will allow set ups.
- Set software to visible (white light). Laser is used only when very small amounts of dust are being measured and contained in 30grams.
  - Follow instructions in Dustmon Manual for testing the instrument with Calibrated Neutral Density Filter. The expected value is on the Conformance Sheet sent with each filter assembly. The filter provides a calibration of the amount of the original white light allowed to interact with the dust plume formed. It provides an opportunity to test the instrument to

determine if it complies with the values on the Conformance Sheet. Contact Microtrac service if the value is not within the value and range stated on the Filter Certificate.

- Previous instrument version required that a lens be maintained or kept with a specific instrument. This is no longer required since each neutral density filter is calibrated and labelled with a certificate of conformance. Note that it is not necessary to evaluate the laser using the filter.
- Set-up – Click “System > Preferences” (Settings in some versions of software)
  - “Save Values At”: Enter the following values for Position 1 thru 10: 1, 2, 5, 10, 20, 30, 50, 75, 100, 150,.Position 10 and Measuring time (below) must be the same.
  - “Measurement Time”: 200 sec. Click SET to active the time. Value may return to 0, but the time entered will be used.
  - “Result After 30 Sec”: Remove Tick in box if shown. Enter 200 and then put “tick” into box.
  - “T=0”. Indicates where on the graph the measurement starts. Value should be “0”. Place tick in box if not shown.
  - “Filter Width” – Smoothing. Normal value is 5. Increasing the value will cause many peaks to be shown.
  - “Custom Scaling”: This value is used to force the calculations to match some previous information
  - “Columns to Show in List View”: Be sure all items have tick in boxes. Scroll down to be sure ticks are in boxes named Value 1, 2, etc. Value 1 corresponds to Position 1 in Save Values At.
  - Close Set-Up
  - “Folder”: Establish a folder for customer data
  - “File name”: Enter sample information including File number, sample ID.
  - “Sample ID”: Enter Sample ID and any comments on the sample amount, etc. (Note that sample amount for final data may change depending upon the dust index).
- “Particle Distribution” - Click on tab in middle of screen –
  - The particle size measurement in Dustmon is derived from Stokes Law for sedimentation. The following steps are required. Note that the particle size of the dust is an estimate since most particles are not spheres and Stokes law pertains especially to spheres. The values are extremely useful in a relative sense of comparing the dust concentration and particle size for a given product. If full particle size measurement is needed, it should be performed by another method which will include the dust as well as non-dust particles.



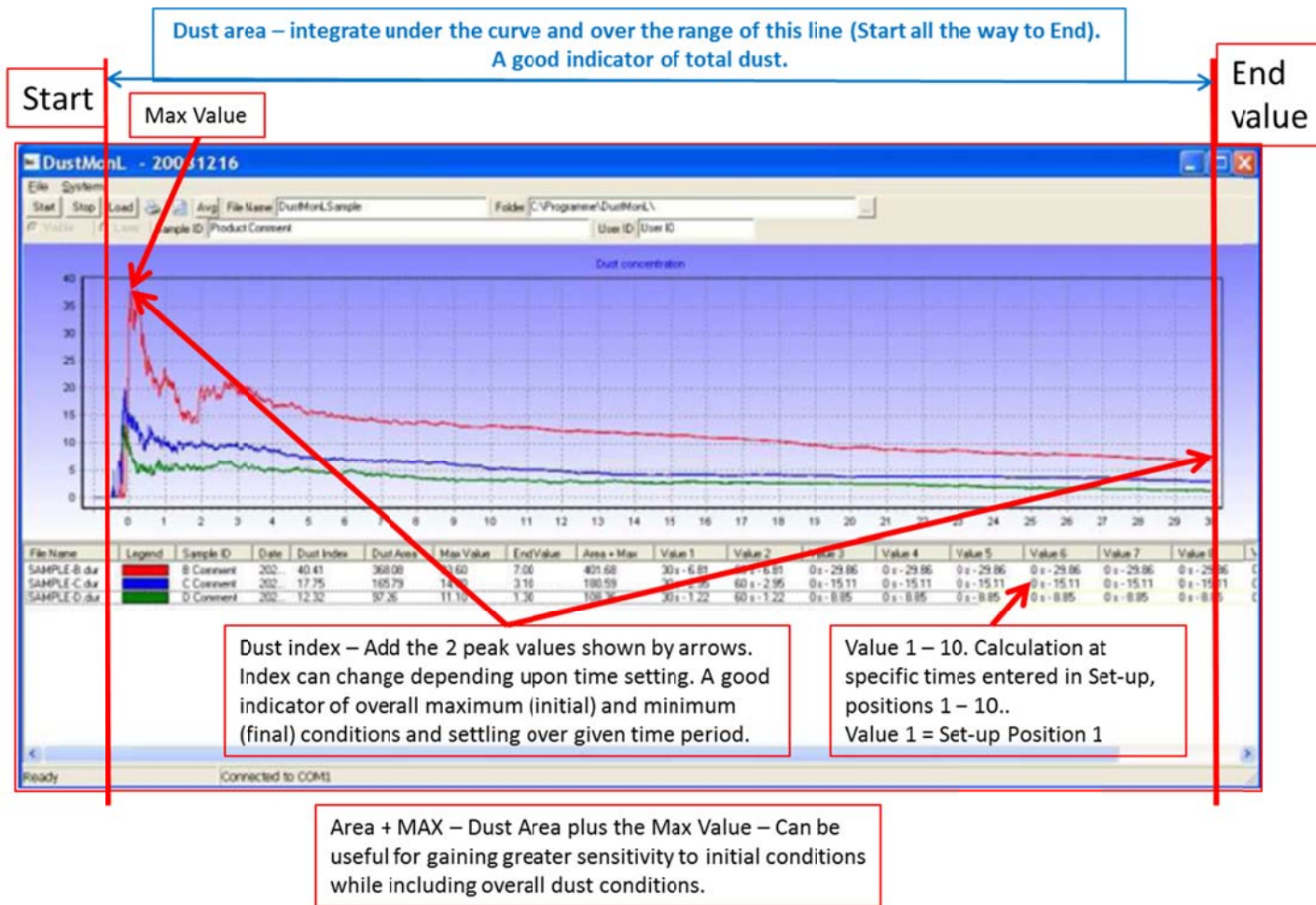


- Sedimentation time is mostly related to the particle density and particle size if all other factors are constant. Since density of particles is generally known, the sedimentation times of various sizes can be calculated.
  - Sedimentation times are calculated for each size chosen based on several factors most notably, the density and sedimentation height.
  - Sedimentation Time = [Constant] [sedimentation height] [viscosity of air] / [particle density][particle size]<sup>2</sup>
- “Sedimentation height”. A constant value based upon the height from the sample bin to the sensor. No user measurements are required.
- Enter “Viscosity” – Use 1.8E-05 - The viscosity of air is used. This value will vary minimally with normal operating temperature and pressures.
- “Particle density” - The only value that should be changed is the particle density. Enter density as Kg/m<sup>3</sup>. Multiply g/mL or g/cc by 1000 to convert to Kg/m<sup>3</sup>.
- “Constant”: 1.835 - An instrument constant.
- Start Time is 2.4. Optimum value to use – do not change. This allows enough time for the dust to form and for particle size calculations as dust settling occurs beyond the peak.
- Extinction coefficient (EC) – The extinction co-efficient is considered constant across the range unless appropriate values are known for each size class chosen. In general, the value proposes little error due to the large difference in refractive index between the dust and air. The value 2 is appropriate for most materials. Use a value = 2. It would require knowing the EC for each size. Thus there can be some inaccuracy in the sizes. Keep in mind that the values are based upon sedimentation in air and NOT a diffraction measurement. Thus, this is another reason the data will not agree with S3500. The Dustmon PSA can be used as monitoring tool for dust production and comparison to other samples.
- Particle Size values–
  - Enter desired particle size values. Particles larger than 100µm rarely produce a dust unless the density is very low or the dust powder is very small. 100, 63, 53, 45, 38, 32, 25, 20, 16, 10, 5 and 4.
  - Note this size is only for dust and most likely will not agree with complete particle size by Microtrac diffraction. The values can be changed. This must be done

before a measurement. There is no method to enter new particle sizes and recalculate. Particle Size – Measures only the dust in the air. So it cannot be expected to match S3500 distribution data which takes into account all particles.

- Dust Index – Value is calculated by adding the max value to the end value (end of measurement cycle). The diagram shows the end value for 30 seconds. See the information below (Decisions to Make) for more information on the optimum measuring time.
  - Complete blockage of the light sensor will report a value of 200. Example: 100 (max value) + 100 (end value). Total = 200.
  - If no dust forms, then the Dust index will be zero. Max value will be "0" and End value will be "0".
  
- Making a Pre -Measurement
  - Assure sample bin is empty and clean (check to be sure no sample and dust free)
  - Assure lab valve assembly (sample beaker, valve, delivery pipe) is clean using long handle brush.
  - Weigh 30 grams of material into a plastic weighing boat that has a pouring tip. This will provide the best means to transfer sample to the lab valve beaker.
  - Sample preparation is not performed for these measurements.
  - Transfer sample into the lab valve beaker.
  - Press START. A delay occurs for background measurement. Then a "click" is heard as the valve opens and sample drops down the delivery pipe into the sample chamber. Dust rises and falls again. Light obscuration measurements are taken and data displayed as the information accumulates.
  
- Decisions to make based upon pre-measurement
  - Initially there is a sharp peak. Then the peak decays as shown during the measurement.
  - The graph will become more horizontal as measurement ensues and dust settles.
  - When measurement is completed, note Dust Index. If greater than 50, reduce the amount. If value is higher repeatability can suffer since dust may get trapped in the mass falling into the bin. Best values are 4 – 12 but may be as high as 20-30.
  - If dust index is less than 3 change settings from Visible to Laser so that Laser has a tick in the box (DustMon LS model only). Re-measure sample.
  - If dust index stays high (20 – 30 or more) and does not seem to be decreasing, then longer measurement time is needed.
  - If dust index and the graph drops somewhat quickly, then 30 seconds may be adequate.
  - If dust index decreases more gradually, then observe data for large differences (example below).
  - The object is to use the shortest measuring time while showing the largest differences in data from various samples.
  - Long measurement times are useful as an indicator of how long the dust can remain in the air when there are no air currents or other factors that can remove the dust.
  - Below is shown a sample data display when using a PC for instrument control.





Above data graph:

- This is an example of comparison data for three different samples. Data show that 30 second measuring time is adequate for measurements for this sample material. Measuring time/period may be extended or reduced depending upon the time required for dust settling. No Dust Index values exceed 50. The greatest difference among the samples is at 2 to 5 seconds. The slope of the red curve after 7 to 10 seconds is steeper than the other two. The dust for blue and green becomes very horizontal and is not changing very much after about 20 seconds. The dust in the red sample is slowly catching up to the dust level of the blue and green. It is slow since the concentration of the dust is higher than in the other two. Perhaps at longer measuring times, the curves will superimpose. Also, dust in red color may have more dust but a larger particle size causing the slope of dust disappearance to be more rapid although starting at a higher level.
- It is possible to observe a higher Max Value before time = 0. This is because, *occasionally*, there is small period of time when the sample is still falling from the lab valve to the sample collection container. This can cause higher values than those during the measurement period. It is important to recognize that the MAX VALUE is determined once measurement timing has started (time > 0).
- Final Measurement
  - Make changes to measurement parameters as needed.
  - NOTE: How to recall data and overlay data as required below:
    - Click on Load. A list of files will be shown.
    - Click on desired measurements – included files will become highlighted.

- Measure three samples and obtain the average. (Recall three measurements using LOAD and click AVG. The graph will show average of three measurements.) The table of values will also show the average of the values.
  - If several samples are submitted, prepare a comparative graph using the averaged values and table by clicking LOAD and selecting proper measurement files.
- Modify graph to use as much of the “white space” as possible.
- Use comparative graph to determine at what measuring times need to be used or to describe to the customer comparative indications.
  - Change in slope of graphs.
  - Rapid decline in any area of the graph compared to other locations
  - Faster setting of dust.
  - Select from the table appropriate values that delineate one sample from the other.
- Cleaning
  - Use a long handle brush that fits neatly into the pipe and contacts the sides.
  - Remove lab valve assembly (delivery pipe) from the instrument
  - Push and twist the brush through the pipe until the brush extends beyond the bottom of the pipe.
  - Pull the brush through the pipe in the direction of which cleaning was performed.
  - Clean sample bin thoroughly.