SediGraph particle size analysis

Principles

The SediGraph method of particle size analysis is based on Stokes Law (Equation 1); a particle falling under its own weight in a liquid will reach a terminal (settling) velocity (v), when the gravitational force balances the buoyancy and drag forces on the particle. This behaviour is dependent on the size of the particle (D), the density (ρ) of the particle material, and the density (ρo) and viscosity (η) of the liquid. Put simply, larger particles settle faster than smaller particles.

$$D^2 = \frac{18v\eta}{(\rho - \rho o)g}$$

Equation 1

The SediGraph uses a paralleled X-ray beam to detect changes in suspended sediment concentration during settling, at different vertical distances in the analysis cell, and at specific times during settling. Hence, a concentration measured at a specified point, and at a specific time, will represent the concentration of particles smaller than or equal to the Stokes Law predicted size (for a given velocity (= distance/time)). During the course of analysis, this provides a distribution of concentration for different particle sizes. From this, statistical analysis of the distribution can provide standard grain size statistics (eg mean, skewness, kurtosis).

It should be noted that the underlying particle density and liquid density/viscosity assumptions are fundamental to all particle size and statistic calculations. If these initial parameters are incorrect, all output particle size data are also incorrect.

Sample preparation

Samples to be analysed using the SediGraph should be cleaned (free of organics) and material coarser than 65 µm (i.e. sands and gravels) should be removed. Follow the standard procedure for this [see '**Particle Size Analysis (for soils/sediments)'** in the online methods section of the Laboratory Manual - http://www.geog.ucl.ac.uk/lab/methods.htm#soil]. Where samples contain an important coarse component, the sample should initially be analysed using the sieving method, recording the grain size distribution of the >65 µm component and the retaining the <65 µm for analysis within the SediGraph system. Sieve data can be added to sample information for consideration within the software-based analysis (see page 81 in the SediGraph III 5120 Operator's Manual).

The X-ray source in the SediGraph requires 30 minutes warm-up time. It is therefore advisable to switch the system on (main unit, carousel and then computer; activate the X-ray source) and use the 30 minutes to assign sample file details to the beakers.

MasterTech carousel

For easier access to each beaker, the Perspex sheet can be carefully lifted from the carousel and the stirrer arm lifted by pressing 'Arm – Load' on the front panel (Figure 1).
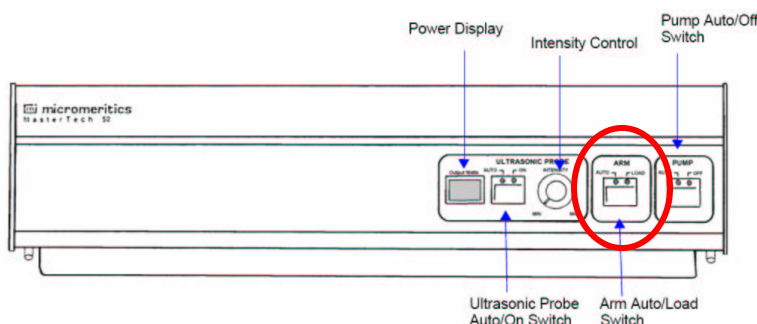

Figure 1 Carousel front panel.

Samples should be put in individual beakers on the carousel [the carousel can be loaded with up to 18 samples for automatic processing]. To each sample should be added:

- 1 drop of Calgon
- deionised water up to the line of the carousel.

It is wise (though not essential) to run a baseline survey as the first 'sample'. To do this, the first beaker should contain just deionised water and a drop of Calgon.

Please ensure that the Perspex is replaced and the arm returned to Auto before proceeding with the analysis.

Performing Sedigraph particle size analysis

All control and management of sediment analysis and processing is done within the SediGraph III V1.04 software. This software is available on the Research Microscope Room (B02) computers. Please use these computers to set up sample file details, and when ready to process a run, these can be copied into the **sedigraph user data directory** on the SediGraph computer.

Setting up your sample files

Each sample file that you need to generate can be based on a default sample or a previously defined sample file, with a suite of parameters covering specifics such as sample material and analysis liquid density, stirrer time, pump speed, number of analyses per sample, and reporting options. This enables the user to create a series of sample files without the need to repeatedly modify options. The software provides a number of default sample types (for example Silica, Kaolin, Cement, Feldspar, Garnet), which may or may not be suitable. It is also possible to set up a new sample type if the density of the material is known.

Set up the first sample file, including specifying particular options. In the **File** menu, click on **Open →**
Sample information. Make sure the directory location is **c:\5120\data\userdata**. First, define the sample filename – use the last 3 letters of your UCL id name followed by 001. This ensures that the file can be linked back to you. Keep a clear record of sample filenames that you create and the samples you are analysing. Click **OK** and agree that you want the file created: a '**.smp**' is created, and this will contain all pre-defined and post-analytical information and data. The following window contains sections (tabs) on **Sample Information**, **Material Properties**, **Analysis Options**, **Report Options** and **Collected Data**. An outline of an example sample file [HBU-001.smp] is provided in Appendix 1, and care should be taken to ensure that all options are appropriately completed.

Sample files for subsequent samples can be generated by importing the information defined in the first sample. Create the file as defined for 001, but increment the number. When the sample information window appears, click on **Replace All**, and then select the original sample file you defined (001) – this will transfer all the specific choices made in the first file into the current file. Individual sample files must be created for all samples to be analysed.

The SediGraph III 5120 Operator's Manual covers the specifics of the operational procedure, and you are advised to consult this for further information.

Initiating an analysis run

To prime the SediGraph analysis, in the **Unit 1** menu, click on **MasterTech Schedule**. This allows you to specify samples (and sample files) for each beaker on the carousel, and initiate the analysis of a series of samples. The following window provides a view of the **Operation** schedule (currently empty). You are also able to view **Instrument Schematic** (illustrates system components and current system state) and **Instrument Log** (a log of system processing and messages) and **Latest Results** (a summary plot of X-ray absorption of most recent data) by changing the view choice at the top of the window.

Within the operation schedule, use **Insert** to specify samples to beakers – the first window to come up will refer to beaker 1, but you can change the beaker number to that required. Within this window, you have the option to define the sample as either *baseline measurement* or *sample analysis*. Remember to define beaker 1 as baseline if your first beaker contains just deionised water and calgon. Click **OK** to save and insert these details into the operation schedule. Continue to insert further sample files (click on **Browse** to navigate to the

individual files), until information/files for samples in each beaker are listed in the schedule. Once this is complete, you can click on **Start** in the MasterTech Schedule window, and the SediGraph will automatically analyse all samples.

Reporting your sample data

Depending on the reporting options chosen in your initial sample file set up, SediGraph will produce a range of datasheets presenting the output particle size information. Click **Reports → Start report** (F8), which will allow you to choose one of the sample files that have been processed. To the right of this output, you can **Save As**, and choose between the SediGraph reporting system (**.rep**), Excel spreadsheet (**.xls**) or ascii text (**.txt**) formats. This will generate a file containing all the information reported – but will not contain other information that you have chosen not to report. Alternatively, you can click File → Export (choose the appropriate sample file and choose Destination – File), to generate a simple ascii file containing the cumulative distribution table (**.rpt**), which can be opened in Textpad or any other text editor.

Completing your analysis

Check that all sediment in the system has been washed out – if sediment is still present, initiate a further rinse to clean through the system. Please turn the X-ray source to stand-by once your analysis has been completed. All beakers used in your analysis should be washed to remove all sediment, and then rinsed in deionised water, dried and replaced in the carousel. Your sample files should be copied (and removed) from the SediGraph computer to your WTS account or a removable drive/disk. Further modification and querying of sample data/reports can be done using the SediGraph software installed on computers in B02. Ensure that the computer, carousel and main unit are turned off when finished.

Appendix 1: Advanced sample information for example sample file hbu001.smp

Advanced sample information	Fields	HBU-001.smp	Explanation and possible options
Sample Information	Sample	**Deben estuayr**	sample character/information
	Operator	**ucfahbu**	use UCL id to specify the system operator
	Submitter	**ucfahbu**	use UCL id to specify the sample submitter
	Types of Data	**Auto**	for all SediGraph based data acquisition, this should be auto
	User Parameters	**Omit**	used to define characteristics for Statistical Process Control (SPC) – see manual for more details
Material Properties	Description	**Minerogenic sediment (quartz-dominated)**	type of sediment
	Sample Material	**Sediment (quartz) 2.6 g/cm^3**	defines the main constituent of the sample, and its density – you can define your own material type if you know the specific material density
	Analysis Liquid	**Water**	defines the liquid within which the analysis is being done – in most cases this is water, but if you think you require another medium, please contact Janet Hope
	Analysis Type	**High speed 60 µm 0.5 µm**	defines the relative priority of speed to resolution, and the starting and ending particle diameters to be considered (if the full range isn't required) – the further into the finer range you choose, the longer the analysis will take (full range is ~ 2 ½ hours per sample) – note that a 'Maximum diameter' possible is calculated for a given material and liquid density
	Analysis Unit	**35°C**	no changes should be made here, but the *Analysis time* is usefully quoted, and will change to when different starting/ending diameters are used
Analysis Options	Mastertech Treatment	**60 High 30**	these are the default settings* for stirrer action – there should be no reason to change these
		tests: 1 6	you can define the number of tests to be done on each sample (if concerned with quality control): the remaining pump and mixing chamber options should remain as default*
	Full Scale Scan	**3 Medium**	pump speed and bubble detection will depend on the type of sediment being analysed … coarse material requires faster pump speeds and coarser bubble detection – finer material requires slower pump speeds and finer bubble detection
	Rinse	**2**	the system should be rinsed through between each sample analysis, so the number of rinses should be at least 1
Report Options	Report options, graphics & title	**Deben estuary - minerogenic sediment**	this simply provides the title information that will be provided at the start of the output data reporting
	Distribution Type	**Mass**	most grain size distributions provide frequency by mass, but here there is also the choice of area and number
		Last	if running multiple tests on each sample, you can decide if you want to report the first, last or the average of these tests
	Reports to Generate	**Combined Report Particle Size Table Cumulative Graph Frequency Graph**	there are a number of reporting options – the user should familiarise themselves with these using the Manual
			each option can also be edited to specify column headings, axis units and particle size statistics
Collected Data			once the analysis is completed, this section will contain the raw distribution data
			you can also input previously analysed sieve data (Merge with external data) if definition of a coarser fraction is required

For default settings* and specific option information, consult the SediGraph III 5120 Operator's Manual.