

Unified Fines Method
for minus 44 micron material
and for
Particle Size Distribution

Compiled by COSIA Fines Measurement Working Group

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Items highlighted in yellow are revision from previous version

Preamble

This Unified Fines Method document is provided by Canada's Oil Sands Innovation Alliance (COSIA) as part of an ongoing initiative within the COSIA Tailings Environmental Priority Area (EPA). The creation of this document was initiated by COSIA in 2013, as a result of a request from the Alberta Energy Regulator (AER). The request was partly related to apparent inconsistencies amongst fines measurements reported by Operators and a requirement for a more consistent measurement method specified in Directive-74 issued by the Regulator in 2009. A Fines Measurement Working Group (FMWG) consisting of industry practitioners was created by COSIA, and supported by personnel from the AER, to carry out and direct work to develop the standard method contained in this document. The mandate of this group, with input from AER, was to develop a technically accurate and effective method of measuring mineral solids less than 44 microns in size, with incidental benefit to overall particle size distribution. Within the oil sands industry the minus 44 micron fraction is known as "fines". This document contains the proposed measurement methodology needed to provide technically and statistically defensible oil sands fines measurements, and is aimed to be applicable across all areas of an operating oil sands processing facility.

Existing particle size distribution methods commonly in use by the oil sand industry, as well as established institutional methods that are commonly used in other industries were evaluated with laboratory data as part of this method development. The unique nature of the sample materials requires more sample preparation in terms of cleaning and dispersion than standards already established for soils provide.

Significant challenges remain that impact this method, and include obtaining representative sampling within an operating oil sands processing facility that is outside of the FMWG scope. Specific challenges to the method include obtaining consistent measurement of particles with organic attachment, the impact of sample cleaning and preparation steps on the final result, variation in fine tails constituents affecting particle density assumptions, laboratory occupational health and safety concerns, and scope and cost increases that may be incurred compared to the various laboratory methods presently in use.

Ongoing Work

It is understood that this method **has been refined during 2015 and may further be refined** based on the ongoing method evaluation commissioned by COSIA. This method evaluation has included a Design of Experiment (DOE) study conducted by the NAIT Applied Research Centre for Oil Sands Sustainability (NARCOSS) and an Inter-Laboratory Study (ILS) to be conducted by Alberta Innovates Technology Future (AITF). Both of these studies are managed by the COSIA-FMWG.

These two evaluations allow this standard method for fines measurement to be statistically evaluated. The DOE has allowed optional procedures within the United Fines method to be validated statistically, whilst the Inter-Laboratory Study (ILS) will provide indication of statistical validity with respect to repeatability and reproducibility. The ILS is expected to involve commercial laboratories presently supporting oil sand Operators, as well as the Operator laboratories and laboratories within educational institutions.

The FMWG will evaluate the results of both the DOE and ILS to determine if revisions to the standard method are warranted.

The results of both the NARCOSS and AITF studies are expected to enter the public domain, within a broader COSIA report.

Implementation

It is understood that the COSIA Tailings EPA and or the Alberta Energy Regulator will decide future use of the method. This decision may include a formal implementation plan for this Unified Fines Method to support Directive 74 or other requirements, which may or may not allow for calibration to existing Operator-specific methods.

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1 SCOPE

This method allows for the determination of the minus 44 micron or “fines” fraction as well as the overall particle size distribution (PSD) of solids from Oil Sands operations. These may be solids from oil sand deposits, designated oil sand tailings disposal areas or process stream samples. The primary purpose of this method is to allow for tracking of ‘fine’ solids through a process; other particle size distribution methods may be more appropriate for the characterization of such behavior as settling or flow. Currently, the definitive value for ‘fines’ content is the less than 44 micron value. Each measurement system will assign a single dimensional value to each particle considered; this dimensional value is considered to represent a non-uniform three dimensional particle for the purposes of tracking these solids.

This method allows for the determination of a particle size distribution for particles in the range of below 44 micron through to 2000 micron using a laser diffraction particle analyzer system, sieves or a combination of the two. The reported units of the distribution are percent volume less than a given particle size when using a laser based system or weight percent less than a specified size when using a sieve system. In order to reasonably compare results from the two measurement systems the assumption made is that there is no density difference exhibited by the solids reporting to different size ranges therefore making volume per cent equivalent to weight per cent for the data generated.

This method requires submitted samples to be between 40 and 140 grams.

2 SUMMARY

Clean dry solids obtained either by Dean & Stark Soxhlet solvent extraction or repetitive cold solvent washing are analyzed for particle size distribution using a number of clearly defined steps. The clean, dry solids are first disaggregated, then appropriately subsampled, adequately dispersed and finally the particle size distribution is measured.

3 BACKGROUND

The size distribution of oil sand solids is important in characterizing ore bodies and tailings deposits as well as determining processability relationships in extraction plants. There are many different methods for measuring PSD's. All of them rely on various assumptions about the particles being measured (e.g. shape, density, dispersion, settling rates, etc.). As a result, different PSD measurement methods may produce different results based on these assumptions. The results from these different measurement methods may have different levels of applicability to different characteristics or behaviors of the samples being analyzed. It is necessary to understand the underlying science behind the measurement methods to determine which method is most suitable for the generation of information with respect to a given characteristic or behavior. The method described herein is suitable to the mass balance of the less than 44 micron solid fraction around a process. In order to complete the mass balance calculations it is necessary to assume that there is no density difference exhibited by the solids reporting to different size ranges therefore making volume % equivalent to weight % for the data generated.

Prior to PSD analysis, the sample solids must be clean and dry. Sample solids are cleaned to remove bitumen from the system since this bitumen will interfere with the particle size determination. However, it must be acknowledged that the cleaning processes used may result in the loss of some of the finest particles present in the system. The loss of some of the finest solids can be significant for some process stream samples, especially for streams where the majority of the solids in the sample are often very fine (<10 micron). Refer to Appendix A for a discussion of fines losses during cleaning. For oil sands ore body samples, extraction process streams, and tailings pond samples, the loss of very fine solids through the extraction thimble tends to be much less significant. It has been found experimentally that the amount of fine solids which are lost through the thimble during the cleaning process of Dean Stark extraction has a strong correlation to the amount of bitumen present in the thimble but does not exhibit a strong correlation to the amount of fines present in the sample. **The solids passing through the thimble are corrected for in the**

measurement system (see Appendix A). Possible solids lost into the fibers of the thimble are not corrected for.

Sample solids are “fully” dispersed prior to PSD measurement. This is to ensure that the amount of fine particles measured (i.e. %< 44micron) is not a function of how much shear force was applied to the sample prior to measurement.

While laser diffraction, alternatively referred to as Low Angle Laser Light Scattering, instruments provide a rapid and convenient measurement of PSD in the range of 0.02 micron to 2000 micron, the instruments are blind to particles outside this range. This range restriction arises from the angles at which information is collected. Laser Diffraction systems use light scattered from the edges of particles and can ‘see’ particles of all types and colours. Samples with a significant amount of large particles may require an additional screening step using a sieve, with the results corrected to account for the particles retained on the sieve. In this way, very large particles that would otherwise be outside of the analysis range, for example rocks found in reject samples, will be reported.

In addition, depending upon the volume of the recirculation system used with the laser optical bench, it is necessary to sieve out particles above a specific size range to reduce the width of the particle size distribution such that sampling statistics are respected and only representative samples are presented to the laser diffraction system (see appendix B for details).

4 REFERENCE METHODS

Laser Diffraction:

ASTM C1070-01: Standard Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering

With the ASTM method, suitable dispersing agents and sonication procedures are recommended depending on the sample, but an exact dispersing procedure is not specified.

ASTM D4464-00: Standard Test Method for Particle Size Distribution of Catalytic Material by Laser Light Scattering

Within the ASTM method, an unspecified instrument is used to measure the PSD from 30 to 300 microns. For this method, only a vague statement of producing stable dispersions is given, with no recommendations on how to achieve this. The ASTM method recommends caution when using surfactants or other additives, as they strongly affect the results. The interlaboratory results that involved additives were excluded from the ASTM precision study as a result.

UOP 856-07: Particle Size Distribution of Powders by Laser Light Scattering

This standard is for determining the particle size distribution of powders and slurries using laser light scattering for particle sizes in the 1 to 100 micron range, but the instrument is capable of measuring particles from 0.02 to 2800 microns. Samples are analyzed as received or prepared using sonication to disperse agglomerated particles.

Sample Preparation for Laser Diffraction:

ISO 13 320 (2009): Particle Size Analysis: Laser Diffraction Methods

This Standard covers the Laser Diffraction Measurement of particle size distribution through the analysis of two-phase systems (powders, emulsions and suspensions in liquids) where particles are analyzed for their light-scattering properties. This Standard includes the areas of theoretical background of Laser Diffraction, recommendations for instrument specifications as well as accuracy and precision of a test method. This Standard does not specify the requirements of particle size measurement of any particular materials and. The particle size range to be measured under this Standard varies from 0.1 micron to 3000 microns. This standard specifies that “for non-spherical particles, a size distribution is reported, where the predicted scattering pattern for the volumetric sum

of spherical particles matches the measured scattering pattern as it assumes a spherical particle shape in its optical model. The resulting particle size distribution is stated as different from that obtained by methods based on other physical principles (e.g. sedimentation, sieving).

ISO 14 487 (2000): Sample Preparation – Dispersing Procedures for Powder in Liquids

This Standard is administered by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*. This Standard is related to the sample preparation methods for particles analyzed to be by Laser Diffraction and covers the dispersion of powder in the liquid. This method applicable to particles size ranging from 0,05 micron to 100 micron.

ISO 14 488 (2007): Particulate Materials – Sampling and Sample Splitting for the Determination of Particulate Properties

This Standard is managed by Technical Committee ISO/TC 24 and describes the sub-sampling procedures for a test sample from a powder, paste, suspension or dust relevant to the measurement of particle size distribution.

Sample Preparation for Sieve Analysis:

ASTM D421-85(2007): Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants (referenced by ASTM D422)

ASTM D75 - Practice for Sampling Aggregates (referenced by ASTM C136, ASTM C117, and ASTM D1140)

ASTM C702 – Practice for Reducing Samples of Aggregate to Testing Size (referenced by ASTM C136, ASTM C117, ASTM D6913, and ASTM D1140)

ASTM Standards for Sieve Analysis:

ASTM D 422-63 (2007): Standard Test Method for Particle-Size Analysis of Soils

This Standard is regulated by ASTM Committee D18 on Soil and Rock and widely used by Geotechnical Laboratories. The test method under this Standard includes both Sieve and Hydrometer. The smallest wet sieving mesh size is #200 (74 micron), measurement of particle size smaller than 74 micron is done using Hydrometer, and the amount of material finer than 44 micron is interpolated from PSD plot.

ASTM D6913-04(2009): Particle Size Distribution (Gradation) of Soils Using Sieve Analysis

This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock. Only test method D6913 includes qualifications on the types of materials for which this test method is applicable: it specifically excludes “soils containing extraneous matter such as organic solvents, oil, asphalt, wood fragments, or similar items. Such extraneous matter can affect the washing and sieving process” (ASTM D6913-04, 2009, p.1).

ASTM D1140-00(2006): Amount of Material in Soils Finer than No. 200 (75-µm) Sieve

This standard is regulated by ASTM Committee D18 on Soil and Rock and manages two methods for measurement of fines finer than 75 micron: A-Test and B-Test. This standard states that “the method to be used shall be specified by the requesting authority” (ASTM D1140-00, 2006, p.1). A-Test Method requires no dispersion prior to wet sieving. *B-Test Method requires* dispersion by soaking in water mixed with deflocculant prior to wet sieving.

ASTM C117-13: Standard Test Method for Materials Finer than 75- μ m (No. 200) Sieve in Mineral Aggregates by Washing (previous version was ASTM C117-95)

This standard is administered by ASTM Committee C09 on Concrete and Concrete Aggregates and widely referenced in the SOP of commercial Oil Sands Laboratories.

This test method provides the guidelines for measurement of fines smaller than 75 micron (No. 200) sieve by wet sieving. This method includes two procedures: Procedure A uses plain water for washing and Procedure B uses water mixed with a wetting agent.

ASTM C136-06: Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates (previous version was ASTM C136-96a)

This test method is regulated by ASTM Committee C09 on Concrete and Concrete Aggregates and covers the determination of the particle size distribution by dry sieving. This standard however is intended for aggregate testing and assumes a relatively coarse sample with low fines content. This standard clearly specifies that if there is a requirement of determination of material smaller than 75 micron, then a combination of both ASTM C136-06 and C117 should be used.

STP 447B (1985): Manual on Test Sieving Methods, “Wire-Cloth Sieves for Testing Purposes”

This is a general guideline on sieving methods and is intended for use as a supplement for published ASTM standards relating to the sieve analysis.

ASTM C429-01(2006): Sieve Analysis of Raw Materials for Glass Manufacture

This Standard is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is related to sieve analysis of raw materials for glass manufacture: sand, soda-ash, limestone, alkali-alumina silicates, etc.

5 REPORT FORMAT AND REPORTING LIMITS

Reporting results from Laser Diffraction:

- Results are reported in the form of cumulative volume % undersize for a standard series of sizes of interest to the data user. Two sizes which MUST be reported are the 44 micron and 2000 micron cumulative volume % undersize. Percent less than a given particle size values are reported to the nearest 0.1%.
- For the purposes of this standard it is assumed that the density of the particles in any size fraction is identical to the particle density in each other size fraction thus allowing the reported values to be considered equivalent to cumulative mass % undersize.
- The data software allows for users to select many different statistical values which can describe the distribution and which the user may find useful. Any such values selected should be reported to the nearest 0.1 micron.

Reporting results from Sieve:

- Results are reported in the form of cumulative mass % undersize for a standard series of sizes of interest to the data user. Two sizes which MUST be reported are the 44 micron and 2000 micron cumulative mass % undersize. Percent less than a given particle size values are reported to the nearest 0.1%.

6 ACCURACY AND PRECISION

6.1 ACCURACY

Particles are three-dimensional objects for which three parameters are required to completely describe their size. The clay minerals associated with the oil sands; kaolinite, illite, glauconite and chlorite in the ore body and, in addition, with montmorillonite or smectite in the waste, are all phyllosilicates. The term phyllosilicate (literally “leafy silicate”) indicates that for all of these clays one dimension of the basic particle is much smaller than the other two dimensions. Thus the particles being sized in the experiments described herein are highly irregular in shape and have been reported as having aspect ratios on the order of 10:1. The physics upon which these sizing measurement systems are based assumes that the particles are highly regular in shape thus allowing them to be adequately described by a single dimensional value. This incongruity shows that an accuracy statement is not possible to define.

6.2 PRECISION

This section will be finalized upon receiving the data will from the ILS

AS ILLUSTRATION OF TYPICAL CONTENT:

THIS DATA AND TABLES WILL BE REPLACED WITH APPROPRIATE DATA FROM THE ILS

Table 1. Example of data to be found here after ILS

	Mean (µm)	Median (µm)	% < 88 µm	
Glass beads n = 35	96.0 ± 0.7	106.2 ± 0.6	23.1 ± 0.8	
	Mean (µm)	Median (µm)	% < 5.5 µm	% < 44 µm
Fuller's Earth n = 51	9.7 ± 0.9	9.7 ± 1.5	39.5 ± 2.0	77.1 ± 2.8

NOTE: The error shown is 2 standard deviations (95% confidence). These precision results were collected over several months on a single instrument (LS 13 320).

7 SAMPLE CONSIDERATIONS

Destructive: Yes.

Amount required: The amount of sample required depends upon the selection made for the measurement step and upon the breadth of the size range of the particles in the specific sample. The total error of a method is made up of several cumulative errors, but in general, the total error equals fundamental error + sub-sampling or segregation error + analytical error. A minimum representative sample mass for measurement is calculated in order that all particles in that received subsample have equal opportunity of being selected in the sub-sampling procedure. In general, the larger the subsample's mass the lower the fundamental error.

While this is true for any measurement on a subsample the concern here is most marked for measurement systems which can accommodate only very small subsample masses. The design of current laser diffraction systems is such that all have a recirculating sample chamber of a fixed size recirculating the particles to be assayed through an optical bench system. The size of this recirculator impacts the mass of the subsample which may be analyzed and thus how well the result obtained represents the sample. Please consult Table 3 for guidance regarding this minimum. Selection of recirculators with smaller volumes will effectively limit the measurement range available to the analyst. It will be necessary to reduce the 'top size' of the particles introduced into the recirculator.

In general, samples submitted for this method are limited to between 40 and 140 grams due to the physical limitation of the extraction thimbles.

Properties: Samples containing bitumen must be cleaned prior to PSD measurement to avoid contaminating the flow path of the laser system or altering the performance of the sieve. All solids submitted with the original sample must be representatively subsampled for subsequent analysis – no solids may be arbitrarily removed from the sample.

8 SAFETY CONSIDERATIONS

This Method does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this Method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Waste from these procedures should be handled in accordance with relevant regulations.

9 WATER QUALITY

Water used in all procedures described herein must meet the minimum water quality specifications given here. Any source of water is suitable if the water meets the specification.

Table 2. Water Quality Specification for PSD

Parameter	Units	Limits	Importance
Filtration	microns	≤0.2	Remove particulates
Temperature	°C	25 to 40	Warm to the touch, to avoid condensation on the cell windows of laser systems
Degassed	--	According to recirculator manufacturer's recommendations	To avoid air bubbles in cell of laser systems
pH	--	6.5 to 8.5	Dispersion may be effected by pH
Conductivity (Calculated TDS ¹)	µS/cm (mg/L)	10 to 500 (5 to 300)	Some ionic strength is necessary for good dispersion
Calcium (Ca)	mg/L	<50	
Iron (Fe)	mg/L	<0.5	

¹ Calculated total dissolved solids (TDS):

Total dissolved solids calculated (mg/L) =

$$0.6*(\text{alkalinity}) + \text{Na} + \text{K} + \text{Ca} + \text{Mg} + \text{Cl} + \text{SO}_4 + \text{SiO}_3 + (\text{NO}_3\text{-N}) + \text{F}$$

[all concentrations in mg/L, in accordance with SM 1030 F]

OR

$$\text{Total dissolved solids calculated (mg/L)} = 0.64*(\text{conductivity in } \mu\text{S/cm})$$

10 PROCEDURES

The procedures necessary to generate data are detailed below. The method consists of 5 steps. Some steps are clearly specified while others provide the analyst with a choice of one of two alternate procedures. The analyst should consider carefully before beginning which choice will be selected at each stage so that appropriate amounts of sample may be prepared and necessary apparatus is available.

Flow diagram of each of five steps of the Method is illustrated in Figure 1. Flow Diagram of Choices.

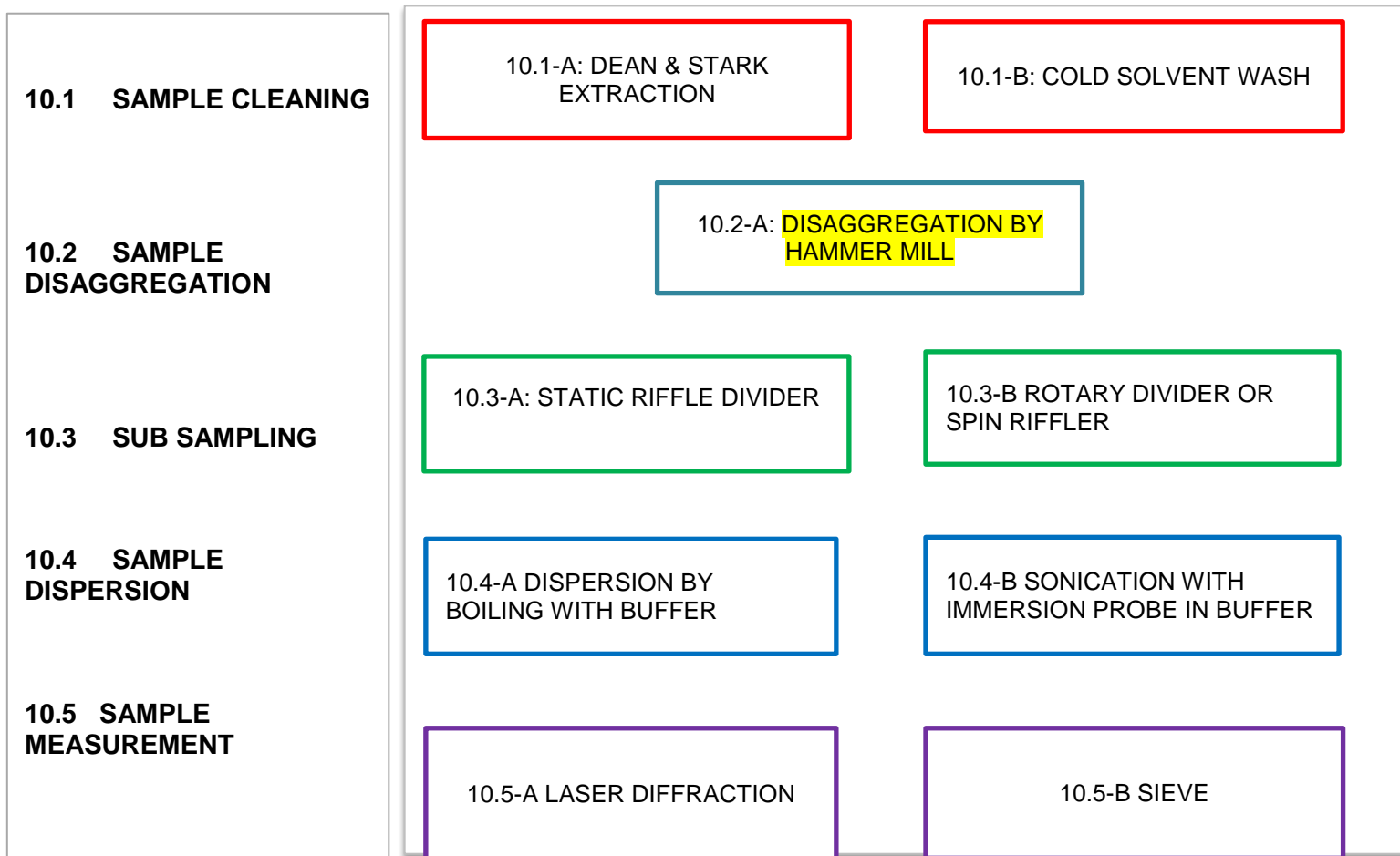


Figure 1. Flow Diagram of Choices

10.1 SAMPLE CLEANING

Samples must be cleaned one of two ways prior to analysis. Sample size for these cleaning procedures is limited to between 40 and 140 grams.

10.1-A DEAN & STARK EXTRACTION

This procedure is fully described in Appendix A: Determination of Bitumen, Water and Solids Content of Oil Sand Process Samples by Extraction. The purpose of this step is to remove any bitumen associated with the sample and to allow the determination of weight % solids in the as received sample.

1. Carry out a Dean Stark extraction as described in Appendix A: Dean and Stark Method. **Ensure that sufficient extractions times are used to completely remove the bitumen from the solids.**

Cleaning times are dependent upon sample type and vary widely. Certain tailings type samples may require cleaning times of 16 hours.

2. Record the weight % solids of the as received sample.
3. Proceed to 10.2 SAMPLE DISAGGREGATION with the clean dry solids.

10.1-B COLD SOLVENT WASH

Details for this choice are provided below. The purpose of this step is to remove any bitumen associated with the sample and to allow the determination of weight % solids in the as received sample.

10.1-B-1 APPARATUS, REAGENTS, MATERIALS

- Centrifuge
- translucent Nalgene Sample bottles to fit in centrifuge head
- Balance $\pm 0.05\text{g}$
- Paint shaker; (e.g. Red Devil Model 5400), or wrist-action shaker
- Evaporating dishes, porcelain, 525 mL
- Toluene/IPA lab solvent: 76 Vol % Toluene, 24 Vol.% IPA (isopropyl alcohol)
- Acetone

10.1-B-2 PROCEDURE

1. Weigh the individual sample in its container and record the mass as sample plus container.
2. Quantitatively transfer the sample using the IPA/toluene mixture from its container into a 250 mL Nalgene centrifuge bottle.
3. Reweigh the empty container to allow determination of the as received sample weight.
4. Fill the 250-mL Nalgene centrifuge bottle to about an inch below the neck with IPA/ Toluene. The IPA/Toluene is used to extract the bitumen from the sample.
5. Balance the bottles to within ± 0.2 grams using a top loading balance by adding IPA/Toluene.
6. Shake the samples for 6 minutes in the shaker.
7. Centrifuge the samples with a relative centrifugal force of 1250 for about 20 minutes.
Note: $RCF = 1.12R (RPM/1000)^2$, where RCF = relative centrifugal force, R is the radius of rotation measured in millimeters and RPM is the number of rotations per minute.
8. Remove the centrifuge bottles from the centrifuge when it has completely stopped.
9. Remove the solvent containing dissolved bitumen from the bottles using suction. Use caution. Remove only the solvent layer, retaining about 1 inch of solvent above the solids layer. This is done to ensure that no solids are lost from the sample.
10. Add toluene/IPA solvent mixture, shake, spin and remove solvent. Repeat until the liquid that is decanted after centrifuging is very clear and colourless indicating that all of the bitumen has been removed from the solids.
11. Quantitatively transfer the sample from the 250-mL Nalgene centrifuge bottles to an evaporating dish using acetone to rinse all the solids from the bottle.
12. Leave the evaporating dishes, containing the solids, in a fume hood until all the solvent has evaporated and the sample reaches constant weight. Ensure that the solids are protected from accidental loss while drying.
13. Record the mass of the dry solids. Calculate the mass % solids in the as received sample.

14. Proceed to **10.2 SAMPLE DISAGGREGATION** with the clean dry solids.

10.2 SAMPLE DISAGGREGATION

Visible/removable coal may be weighed and estimated as a percent by mass of the solids but must be returned to sample solids and carried through the entire analysis. Due to its friable nature some of the coal will report as sub 44 micron solids.

Disaggregation of the clean dried solids is needed since during the cleaning step the solids are typically agglomerated.

Disaggregation of the clean solids is handled the following procedure:

10.2-A DISAGGREGATION BY HAMMER MILL (Removed Soil Grinder)

10.2-A-1 APPARATUS, REAGENTS, MATERIALS

- Hammer Mill: general specifications:
 - With a general configuration such that the feed hopper is vertically positioned above the mill chamber with the exit screen positioned at the bottom of the mill chamber
 - 1000W; 60Hz 110V direct drive motor
 - ~3200 rpm rotating speed
 - fitted with a 2 mm screen/sieve
 - fitted with a Hammer Strike grinding attachment; smooth stator (blade grinding stator); AISI 316L Stainless Steel
 - hinged-arm Hammer-rotor (not blade-grinding rotor); AISI 420 Stainless Steel(hardened)
 - 2 mm clearance in grinding chamber.
 - ~300 mL hopper volume

- Mortar and pestle
- Sample collection jars
- 2000 micron sieve
- 200X microscope(for examination of questionable solid clumps)

10.2-A-2 PROCEDURE

1. Fit the hammer mill exit with a rubber stopper of suitable diameter for the receiving vessel selected. The rubber stopper is necessary to avoid excessive loss of fine particles into the fume hood. The rubber stopper should have a thin, ~2 mm wide, vertical notch cut into the side of the stopper. This is needed to allow a small amount of air flow out of the receiving container as solids fall into the container from the hammer mill. This notch also helps to prevent the hold-up of fine particles within the hammer mill. Solids may be collected into the original thimble or another sample container
2. Weigh the individual sample of clean and dried solids in its container (i.e., thimble from Dean and Stark or the evaporating dish from Cold Wash) and record the mass as sample plus container.
3. Ensure a good seal between the mill/grinder exit and the receiving container.
4. Turn on the mill/grinder. Set the rotational speed at 3200 rpm.

5. Quantitatively transfer the clean dry solids from the container to the mill/grinder hopper. A mortar and pestle may be used to break up any agglomerates that do not fit inside the feed hopper, **nominal lumps of 1.5 cm or less**. If the sample contains **rocks** that are greater than 2000 microns in size they must be retained and weighed separately, **use caution not to remove agglomerates**. This weight will be used in the calculations.

Note: It is important to ensure that as much of the solids as possible are removed from the thimble and transferred for disaggregation when Dean and Stark has been used for sample cleaning. Fine clays can form a hard layer on the surface of the thimble and be lost. This could result in erroneous results. There should be no visible clumps of material bound to the thimble.

6. Close the mill/grinder hopper lid to avoid fines loss.
7. Ensure all material has passed through the mill/grinder and has not been retained in the chute or mill. Care must be taken to examine the internals of the mill/grinder to ensure the entire sample has exited the unit.
8. Rocks which are larger than 2 mm may be retained on the exit screen of the mill/grinder. These must be recovered by opening the unit and subsequently adding these rocks to those recovered in **step 5** above. Ensure that there are no agglomerates in this material.
9. Weigh the empty container that held the sample and record. Subtract this weight from the sample plus container mass. Determine the total sample mass. Calculated sample mass must include any rocks that may have been previously removed. The total mass should correspond closely with the total mass of clean dry solids produced in the cleaning step.
10. Calculate the mass % ROCKS in the clean dry solids if any such material was recovered in **steps 5 or 8 above**.

$$\frac{\text{mass ROCKS}}{\text{Total clean dry sample mass}} \times 100$$

11. Proceed to 10.3 SUB SAMPLING using the disaggregated solids.

10.2-B DISAGGREGATION WITH MORTAR AND PESTLE (Removed)

10.2-B-1 APPARATUS, REAGENTS, MATERIALS (Removed)

10.2-B-2 PROCEDURE (Removed)

10.3 SUB SAMPLING

Once the solids have been disaggregated by either approach described above it is necessary to prepare a sub sample of the solids that is the appropriate mass for the subsequent analysis. The analyst must decide what the final measurement step will be and prepare a sub sample accordingly. **Guidance is given in Table 3.**

Cautionary note: Table 3 provides guidance only. **The specific subsample mass appropriate for a laser diffraction system will be dependent upon the specific system being used and upon the particle size distribution of the subsample being assayed. The final arbitrator of the appropriate subsample mass to be introduced into the laser diffraction systems is the obscuration or transmittance reading achieved for each subsample assayed. This reading must be within the permitted range for each measurement of an appropriately obtained representative subsample. It is possible that the obscuration/transmittance criteria will limit the mass of the subsample to a mass less than the minimum representative subsample mass required to fulfill the fundamental error considerations; thus requiring the analyst to remove particles from the subsample according to their size. This is termed "top sizing". If required, "top sizing" must be done after the sample is properly dispersed (section**

10.4) and prior to adding the subsample into the laser diffraction system. See APPENDIX B: Minimum Sample Mass Discussion for guidance on how to proceed.

The lab sample must be generated by proper sample division and not scoop sampled at any point. Grab or scoop sub-sampling does not meet the criteria of a correct sampling procedure for heterogeneous particles because it does not give each particle the same probability of being sampled. Sample increments once obtained shall be entirely utilized at all stages of sample division, preparation and measurement. No material may be added to or taken from the increment.

Two method options for sub sampling are provided: Static Riffle Divider and Rotary Divider.

Table 3. Approximate Sample Size Requirements

Measurement System	Approximate Subsample Size (grams)	Approximate Maximum Particle Size(microns)
LD with 400 mL recirculator	0.3	500
LD with 1000 mL recirculator	1	1000
LD with 4000 mL recirculator	5	1700
3 inch sieve (325 mesh)	11	
8 inch sieve (325 mesh)	80	

10.3-A STATIC RIFFLE DIVIDER

Riffle dividers should be calibrated regularly, at least annually, to confirm they are properly dividing the samples, by mixing and dividing a sample containing known quantities of particles such as glass beads. The riffle divider should always be used in a level position and be in good repair without any rough edges or deformations that may bias the mixing and dividing of the sample. Do not clean the divider or the collection pans by banging together or by using tools such as a mallet, hammer or knife. Wherever possible clean the divider and collection containers with compressed air. If the divider or collection pans are dirty or oily, they can be washed with warm water and mild detergent. The divider and the pans should be dried thoroughly with a soft lint free cloth that will not leave any residue and left to dry at least overnight.

10.3-A-1 APPARATUS, REAGENTS, MATERIALS

- Chute riffle with minimum 6 millimeter openings(3 times 2mm – the largest agglomerate loaded into the riffle) and maximum 10 mm openings. Select the riffle of appropriate size to handle the mass of material being subsampled. It will be necessary to have a number of static riffle dividers available to allow for the ever decreasing mass of subsample as the process proceeds.
- Assorted collection containers

10.3-A-2 PROCEDURE

1. Distribute the disaggregated solids evenly into a hopper or one of the collection bins provided with the riffle.
2. Pour the solids quickly over the top of the riffle using one smooth single motion.

3. Collect the solids which reported to each of the collection bins located on one side of the riffle ($\frac{1}{2}$ of the sample) from the underside of the riffle.
4. Remove and replace each of the collection bins with clean and empty bins.
5. Pour the collected sample half from step 3 over the riffle again.
6. Repeat steps 2 through 5 until the sample is reduced to the required mass for measurement. This may be difficult if extremely small sample masses are required, a rotary riffle might be more suitable.
7. Proceed to 10.4 SAMPLE DISPERSION with the entire contents of a single bin or with the entire contents of multiple bins.

10.3-B ROTARY DIVIDER OR SPIN RIFFLER

Selection of a rotary divider or spin riffle should be made to ensure that the size of the equipment is suitable to the volume of sample being sub-sampled. The goal should be to minimize the loss of fine particles during riffling by minimizing the amount of contact between the sample and the surface of the equipment and by minimizing the exposure to strong air currents. Typically 80-100 gram particle sample masses may require a 'micro' spinning riffle. The sectors and collection trays or vials should be symmetrical and of equal size.

The best results are obtained when operating the rotary splitter with a constant rotational velocity and feeding it at a constant slow rate. Slow feed rates increase the number of increments and help to minimize the grouping and segregation errors. The riffle should rotate a minimum of 20 times while the sample is being divided. Sample losses must be minimized. Careful alignment of the riffle parts and reasonable speeds help to minimize losses.

10.3-B-1 APPARATUS, REAGENTS, MATERIALS

- Appropriate spin riffle
- Assorted sample containers

10.3-B-2 PROCEDURE

1. Turn on the rotating collector plate at a constant speed. The speed selection should be based on the manufacturer's specifications for appropriate use of the riffle.
2. Pour the sample into the hopper or vibratory feed chute at a slow and even rate.
3. Turn on the vibration amplifier to a slow but constant rate so that the material begins to flow and feed into the collection trays or tubes.
4. Increase the vibration amplifier slightly, if necessary, so that any slower moving finer material moves to feed the collection trays or tubes. Continue to increase the intensity of the vibration until all material is fed into the rotating collection trays or tubes. Cautionary note; avoid creation of dust clouds with judicious selection of rotational and vibrational speeds.
5. Remove the collection bins and replace them with empty collection bins.
6. Empty the entire contents of one or more of the collection containers into the main feed tray and repeat the procedure until the desired sample size is achieved. If the sample size is unreasonable for the size of the rotary divider at this point, feed one of the divisions into a smaller scaled rotary divider.
7. Weigh each portion to make sure subsamples are of nearly equal mass.
8. Proceed to 10.4 SAMPLE DISPERSION with the entire contents of one or more bins combined

10.4 SAMPLE DISPERSION

Dispersion is one of the most critical steps in sample preparation. It is important, especially for samples containing fine minerals and hydrophobic surfaces. It is imperative that the samples be fully dispersed before the measurement of the particle size is attempted. Any and all of the material produced as the sub-sample in the preceding sub-sampling section must be utilized to prevent the representative sample from being compromised. Either of the listed dispersion processes below may be chosen.

10.4-A DISPERSION BY BOILING WITH CARBONATE/BICARBONATE BUFFER

10.4-A-1 APPARATUS, REAGENTS, MATERIALS

- Shallow stainless steel bowl with 500 mL capacity
- Small beakers
- Parafilm or plastic wrap
- Small spatula
- 500 mL graduated cylinder
- 600 mL beakers
- Isopropyl Alcohol (IPA)
- Hot plate/stirrer combination
- Heat resistant gloves
- 100 mm diameter watch glass
- Cooling rack or pan
- Clip on thermometer
- Timer set to 15 minutes with alarm.
- Reagent Grade Sodium Bicarbonate NaHCO_3
- Laboratory Grade 1.0 M Sodium Bicarbonate Solution
- Laboratory Grade 10% Sodium Hydroxide Solution

10.4-A-2 PROCEDURE

1. Prepare a carbonate/bicarbonate buffer solution. Two suggested methods are:
 - a. Buffer Option 1: 1.26g NaHCO_3 diluted to 900 mL and 10% NaOH added until pH = 9.6OR
 - b. Buffer Option 2: 15 mL of 1.0M NaHCO_3 (8.40g/100mL) diluted to 1000 mL and 10% NaOH added until pH = 9.6

Note: This solution will have a shelf life of four (4) weeks. Date of preparation should be marked on the bottle.

2. Transfer the weighed sample of the disaggregated dry solids into a shallow stainless steel bowl or **other suitable container**. Add 1-2mL of IPA, in drop-wise increments to the dried solids. If dispersing only a small mass of solids a small beaker may be utilized.
3. Mix the IPA into the sample by using a folding action with a spatula. **Encourage agglomerates to breakdown by pressing lightly on lumps**.
4. Repeat step 2 and 3 until all solids are visibly wet with no excess liquid. Consult **Table 4** for typical volumes.

Table 4. Typical amounts of IPA and BUFFER solutions

Sample weight (g)	IPA volume (mL)		Volume of BUFFER Solution (mL)
	Sample contains predominantly sand	Sample contains predominantly clay	
0-1	2	4	100
1-10	3	6	100
10-40	5	10	300
40-80	10	20	300
80-120	15	30	400

Note: These are suggested volumes. In particular, the volume of the BUFFER solution needs to be adjusted to ensure an appropriate boiling action is observed during the dispersion step.

5. Fill a graduated cylinder up to the appropriate mark with the BUFFER solution.
6. Add the BUFFER solution slowly to the stainless steel bowl/ beaker by pouring over the spatula used for mixing.
7. Cover the bowl/beaker with parafilm or plastic wrap.
8. Leave the sample to soak overnight for a minimum of 12 hours.
9. Quantitatively transfer the slurry to an appropriately sized beaker using a limited amount of water.
10. Place the beaker, with sample, on a stirrer hot plate and insert a stir bar for mixing.
11. Set the temperature on the hot plate to a setting ~med-low.
12. Turn on the stirrer. Increase both the heating and stirring until a gentle boil and mixing of the slurry occurs.
13. Start the timer when boiling state is reached.
14. Boil for 15 minutes. While boiling, push any material off the sidewalls with a rubber policeman on glass rod, rinsing the stirring rod and the sides of the beaker with water. This is needed to avoid loss

of sample and to make sure the entire sample is wetted and being boiled. Where necessary apply force to the agglomerated lumps with the rubber policeman to aid disaggregation.

15. Turn the hot plate off.
16. Place a watch glass on top of the beaker to avoid sample contamination.
17. Move the beaker to a cooling rack or pan.
18. Start a timer.
19. Measure and record the temperature at regular intervals until the temperature reaches 25°C.
20. Record the time at which the sample slurry reaches temperature of 25°C and use this for guidance on the cooling time requirements for future samples. DO NOT allow the samples to sit for more than 30 minutes prior to measurement, as time can affect the dispersion.

Note: An ice bath or cooler can reduce the cool down time to less than 20 minutes.

Proceed to 10.5 SAMPLE MEASUREMENTS. SAMPLE MEASUREMENT of the dispersed subsample. If the Analyst has selected laser diffraction as a measurement step then it is necessary at this point to determine the need for “top sizing” of the sample prior to measurement.

10.4-B SONICATION WITH IMMERSION PROBE

This dispersion technique provides high dispersion energy. It has been shown that the presence of various process aids (e.g. polyacrylamides, gypsum, sodium silicates, demulsifier) in the original sample does not significantly affect the PSD results. The process described below uses a sonication probe as specified. Sonication systems ‘on board’ laser diffraction optical benches do not supply the dispersive energy supplied by the specified probe and are not adequate for the purpose of creating an oil sands solids dispersion.

The use of a sonication bath is currently outside of the method. This exclusion is related to the amount of energy input into each sample dispersion container. The specific geometry of the energy sources in the bath will render some spots within the bath as higher energy environments than others. This will result in different amounts of energy input into the slurry processed in different spots in the bath hence different degrees of dispersion and higher variances in the data generated. The method development studies carried out did suggest that the degree of dispersion reached a plateau for long sonication times with an immersion probe. It is likely that this plateau could be reached with the use of a bath but each bath type would need to be evaluated for each ‘spot’ within that bath to ensure equivalent energy input. This has not been done.

10.4-B-1 APPARATUS, REAGENTS, MATERIALS

- Assorted beakers
- Ultrasonic processor and probe general specifications:
 - Power of the sonifier: **750 W**
 - Frequency and maximum amplitude of the sonification: 20 kHz frequency at 58 micrometers amplitude when the power is set to 100% automatic tuning and frequency control and automatic amplitude compensation. The actual power delivered is increased

and decreased automatically to compensate for viscosity changes in the dispersion medium in order to maintain frequency and amplitude.

- Sonifying power setting: 90%
- Probe dimensions: 3/4 inch (19 mm) diameter solid probe with a 5 inch (127 mm) length. Avoid sonifying probes where threaded portions of the probe are immersed in the liquid (e.g. replaceable tips) as fine particles may accumulate in the threads and reduce the effectiveness of sonification
- Probe composition: high grade titanium alloy Ti-6Al-4V

10.4-B-2PROCEDURE

1. Examine the sonication probe, at the beginning of each analysis day, to determine if erosion of the probe tip has become unacceptable (e.g. signs of pitting near the rim are just becoming visible). Performance of the sonication probe decreases at the earliest signs of pitting. **Pitted probes should be replaced.**
2. Switch on the ultrasonic processors. Set the timer as listed in Table 5 and set the amplitude at 90%.
3. Determine the correct placement of the sonication probe within the beaker to be used in the experiment. Place the sonication probe into the beaker, centered in the beaker and at a height specified by the manufacturer. Set the system so that this placement can be re-established readily for each use of the sonifier. Typically, this will result in the probe tip being immersed below the surface 1.5 x the probe tip diameter. For example, for a 3/4 inch probe tip the tip will be 1.125 inches below the sample surface.
4. Prepare a carbonate/bicarbonate buffer solution. Two suggested methods are:

Buffer Option 1: 1.2602g NaHCO₃ diluted to 900 mL and 10% NaOH added until pH = 9.6

OR

Buffer Option 2: 15 mL of 1.0M NaHCO₃ (8.4011g/100mL) diluted to 1000 mL and 10% NaOH added until pH = 9.6

Note: This solution will have a shelf life of four (4) weeks. Date of preparation should be marked on the bottle.

Table 5. Sonication Time and Volume

Sample weight, g	IPA Volume, mL	IPA Volume, mL	BUFFER Volume mL	BUFFE R Volume mL	Size of Beaker, mL	Diameter of Beaker, cm	Sonification time (min)
	Largely Sand	Largely Clay	Largely Sand	Largely Clay			
0-1	2	2	60	60	100	4.75	3
1-10	3	3	60	60	100	4.75	3

Sample weight, g	IPA Volume, mL	IPA Volume, mL	BUFFER Volume mL	BUFFE R Volume mL	Size of Beaker, mL	Diameter of Beaker, cm	Sonification time (min)
10 - 40	5-10	7-20	80-100	60-105	150	5.5	10
40 - 80	10-15	15-40	200-260	200-240	600	8.75	30
80 - 120	15-25	25-60	350-390	240-320	1000	10.25	60

1. Transfer a weighed sub-sample obtained from the riffle to a beaker, sized as specified in Table 5.
2. Wet the transferred solids with a minimum amount of isopropanol (IPA) (typical amounts are suggested in **(Table 5)** and manipulate the sample until the solids are completely wetted (no lumps).

Caution: Using excessive IPA can lead to bubble issues if using a laser diffraction system in the measurement step.

3. Add BUFFER to the beaker following the guidelines in **Table 5**.
4. Sonicate the sample for the time specified.
5. Inspect the probe for any solids that have traveled above the liquid surface and adhered onto the probe stem. Rinse any solids on the probe tip back into the beaker. A wash bottle containing water works well for this. Remove the probe from the suspension.
6. Do not let the sonified sample sit for more than 30 minutes before analysis; re-sonify as in step 7 if necessary.

Proceed to 10.5 SAMPLE MEASUREMENT with the dispersed subsample

Note: it may be necessary to screen the dispersed sample through a sieve of a specific size to reduce the maximum size of the particles prior to introduction to the recirculation system of a laser diffraction system if the volume of the recirculator is small. See the discussion in Appendix B on minimum representative sample size.

10.5 SAMPLE MEASUREMENT

There are two dispersed sub-sample measurement options: Laser Diffraction and Sieve methods.

10.5-A LASER DIFFRACTION

10.5-A-1 APPARATUS, REAGENTS, MATERIALS

- Laser diffraction instrument with capability of measuring particles between 0.1 microns and 2000 microns in size. At the 44 micron level the repeatability should be as stipulated previously in this method – see ACCURACY AND PRECISION.
- Recirculation system for Laser Diffraction system capable of recirculating water with a volume **of not less than 800 milliliters**. Larger volume recirculators minimize concerns regarding minimum representative sample masses (see APPENDIX B: Minimum Sample Mass Discussion)

- Recirculators must be connected to a tempered water source that is heated to 25°C. This is to avoid condensation on the flow cell windows
- Computer and instrument specific software
- Water to meet the water quality specified - see WATER QUALITY.
- Canned air duster.
- Micro-90 Concentrated Cleaning Solution
- Triton X-100 diluted for “as needed” cleaning

10.5-A-2 SELECTION OF THE OPTICAL MODEL

Most instruments currently available provide the analyst with a choice of optical models.

Optical model based on Fraunhofer approximation

The Fraunhofer approximation was the first optical model used to determine particle size information from scattered laser light. This model is relatively simple and requires no knowledge about the optical properties of the material being sized. It assumes that:

- The particle interacts with a plane wave of light of known wavelength.
- The particle completely absorbs the light so that the only diffraction which occurs is at the edge of the particle.
- The particle is highly regular in shape (uniform in all orientations) and can thus be approximated by a sphere and adequately described by a single dimensional parameter.

The Fraunhofer approximation is most suited to particles which are opaque and much larger than the wavelength of the light used in the experiment.

Optical model based on Mie theory

Mie theory allows for the primary scattering from the surface of the particle predicting the intensity of this from the difference between the refractive index of the particles and the dispersion medium. Mie theory also predicts the secondary scattering caused by light refraction within the particle. Mie theory is the more rigorous and requires more knowledge of the system being measured. It does, however, account for light transmission through the particles and is thus suited to both transparent and opaque particles.

- The particle interacts with a plane wave of light of known wavelength.
- The particle is highly regular in shape (uniform in all orientations) and can thus be approximated by a sphere.
- The particle has no surface charges.
- The complex refractive index of the homogeneous particles is known.
- The refractive index of the dispersion medium is known.

Neither theory is ideal for the sizing of oil sand derived solids. **However, for the particle size of most interest the minus 44 micron fraction, Mie theory is more suitable.** Mie theory requires that the refractive index of the materials being measured is known. The refractive index of materials such as silica, illite, kaolinite, orthoclase feldspar, mica and quartz are all reported to lie in the range of 1.50 – 1.60. Unfortunately, some materials known to be present in oil sand solids such as rutile (RI: 2.56-2.90) and zircon (RI: 1.92-2.02) have refractive indexes very different from this range.

For the purposes of sizing oil sand solids by the method described herein the **choice of optical model** should be the **MIE model** employing a **refractive index for water at 25 °C equal to 1.333** and a **refractive index for the particulate matter of 1.55 with an imaginary component of 0.1*i***. It may be necessary to verify the refractive index of the circulating fluid if large amounts of IPA are added to the recirculation system. It is possible to achieve this by withdrawing about 10 mL of fluid and sample from the recirculator after the sample analysis but before draining. This withdrawn fluid should be filtered through a 0.1 micron filter and then the refractive index measured. The actual refractive index can then be employed to recalculate the data from the sample run.

10.5-A-3 DETERMINATION OF APPROPRIATE PARTICLE CONCENTRATION

Obscuration is a measure of the concentration of particles in the system. It is the percentage loss of laser light due to the number of particles in the system. Transmittance is another term used in laser diffraction instruments to measure concentration of particles in the system. It is the amount of light going directly through the cell without absorption and blockage due to particles.

It is important that the particle concentration in the measurement zone of the instrument be high enough to achieve adequate signal to noise ratio but low enough to ensure that multiple scattering effects are minimal. In general, multiple scattering effects generally result in larger angle shifts in the refracted light which in turn will be interpreted by the system as an increase in “fines” shifting the PSD to lower sizes.

The exact concentration appropriate for a specific measurement system is a function of the specific system configuration as well as the width of the particle size distribution. There are many more individual particles in a given mass of fine particulates than there are in a similar mass of coarse particulates. In general, the finer the particles and the narrower the distribution the lower the concentration needs to be to avoid multiple scattering.

Most instruments monitor either obscuration or transmission of the incident beam as a means of judging the likelihood of multiple scattering. The guidance given in an instrument manual with respect to the upper bound of the obscuration window is general in nature. An obscuration nearing 10%, or 90% transmittance, is typically used for oil sands solids measurements but the analyst may determine experimentally the concentration of particulates at which this multiple scattering affects the particle size distribution in a significant manner.

1. Select a sample of oil sand solids which has approximately 60% -75% of its particles sized as less than 44 microns.
2. Riffle the sample into a series of containers such that the contents of a combination of three single containers blended when dispersed and measured will result in an obscuration of ~3 or a transmittance of 97%.
3. Measure/record the obscuration (transmittance) of the blend of these first three containers and record the resultant PSD.
4. Make another measurement of a blend of four containers; noting both the resultant obscuration (transmittance) and the resultant PSD.

5. Continue making blends and measurements until you have recorded obscuration from 3 through 20, or 97 through 80% transmittance with the resultant PSD curves .
6. Plot determined %<44 microns as a function of obscuration; %<5 microns as a function of obscuration and %<0.5 microns as a function of obscuration.
7. Select an appropriate range of obscuration values such that the PSD determined is stable and does not show signs of either multiple scattering nor poor signal to noise ratios.

This acceptable range may be applied to the measurement of PSD of oil sand solids.

10.5-A-4 MINIMUM SUB SAMPLE MASS TO BE ASSAYED

The table below is the minimum sample mass to be measured based on the top nominal particle size in the sample. For detailed discussion and calculations, please refer to APPENDIX B: Minimum Sample Mass Discussion. Systems with small recirculators will restrict the sample mass due to obscuration or transmittance criteria. This restriction will result in the need to remove particles above the maximum allowable particle size given in Table 6. For example, if it is determined that to meet obscuration/transmittance limits the mass subsample mass is restricted to 0.27 grams or less, then the maximum particle size which can be introduced into and reported from the measurement system is 500 microns. A sieve should be used before the sample is introduced into the recirculator. The amount of material retained by this sieve will be required to correct the data obtained from the measurement system. This maximum allowable particle size limit is irrespective of the size limit quoted by the instrument manufacturer.

Table 6. Minimum Representative Sub Sample Mass Required

Minimum Representative Subsample Mass As per ISO 14488 considerations g	Maximum Allowable Particle Size, microns
0.27	500
0.73	710
1.88	1000
4.77	1400
8.13	1700
12.77	2000

10.5-A-5 INSTRUMENT SET-UP

- The instrument should be setup in accordance with manufacturer’s guidelines
- The instrument should be located on a stable, rigid bench in an area where temperature fluctuations are minimal (less than ± 1 °C).
- Consideration must be made for sufficient warm-up time, properly aligned optics, clean optics, lack of condensation, lack of bubbles, and no segregation of particles in the flow path.

- Ensure that the recirculation flow rate is sufficient to prevent settling of the larger particles in any part of the water flow path
- Set up desired user sizes within the instrument software.
- Create an internal instrument method with operational parameters that will be used with all oil sands derived solids. Automatic “Offsets” or “Align” functions should be set to run before start and at minimum every 90 minutes thereafter while running.
- Ensure that the instrument is correctly functioning as per instrument manufactures specifications when used to analyze a traceable spherical certified standard of beads in a size range between 1 micron and 100 microns.

10.5-A-6 RUNNING SAMPLES

1. Turn on the instrument and allow it to warm up according to instrument specification.
2. Turn on the computer and open the instrument software.
3. Optimize sample data acquisition (measurement) settings according to manufacturer’s instructions and set the background data acquisition (measurement) settings to the same value.
4. If the optical bench in use contains an in-line sonication system this should be set as per the instrument manufacturers guidelines and should remain at this setting for all runs of samples and blanks. This on board sonication system cannot be considered to take the place of the external sonication required in step 10.4-B Sonication with Immersion Probe.
5. Create an internal instrument method with operational parameters that will be used with all oil sands derived solids
6. Measure the background. If the background signal is too noisy take appropriate action to correct by either running micro90 solution through the system followed by sufficient rinses to avoid bubble formation or, by removing the cell windows for cleaning in accordance with the manufacturer’s instructions. Replace cell windows when necessary.

Note: The background is measured for each sample. Each instrument will have different inherent background signals based on the optical components. The background QC limits for each instrument must be chosen carefully such that a clean system will pass very often, but a system in poor condition will fail, such as when the following issues arise: (1) coarse solids from the previous sample were not fully rinsed away, (2) the recirculator water is dirty or contains fine bubbles, (3) the optical windows/lenses are dirty. Also, visually compare the background against a known acceptable reference background to help identify potential issues. Make sure that the background is within the established limits specific to each instrument before analyzing every sample.

7. Set the number of measurements per aliquot to 3. Remember to leave a wait time between sample addition and data collection to allow for the sample solids to thoroughly mix within the recirculating system.
8. Quantitatively transfer the dispersed subsample directly into the instrument recirculator water. All of the material in a single dispersion container selected for analysis must be added to the recirculator in order to ensure a representative sub-sample. **Ensure that the instrument does not reduce the volume in the recirculator automatically since this removal can bias the sample in the system and lead to erroneous results.**

Note: Do not pour from an elevated height above the water surface to avoid entraining air bubbles. Rinse the beaker with non-pressurized (de-gassed) water using a gentle spray to minimize splashing.

9. Observe that the obscuration/transmittance is in the acceptable range. If the obscuration is below acceptable range (transmittance above acceptable range) then a second sub-sample may be obtained and dispersed by methods described herein and added to the recirculator.
10. Abort the run if the obscuration/transmittance exceeds the concentration boundary.

Note: Do not add more water to the recirculator to bring the obscuration down to an acceptable level because of potential bubble entrainment, inadequate mixing, or automatic tank level drop.

11. Verify that there is no material being reported above the maximum particle size limit associated with the minimum representative subsample requirements arising from fundamental error considerations – see Appendix B. It may occur that due to the restriction in obscuration values the sample mass required to be used will be so small that the entire particle size range cannot be measured. The particles above the ‘top size’ limit must be removed. Runs which report particles above the maximum size must be aborted. New runs must be carried out that ‘top size’ the subsample first as discussed in Appendix B. Typically, this ‘top sizing’ is achieved by wet sieving the dispersed material through a 3 inch sieve of appropriate opening size into the recirculation system and then determining how much material was retained on the sieve to allow correction of the data generated.
12. Press the Drain button to automatically drain and fill the recirculator a minimum of 3 times to clean the system before the next background is run prior to analysis of the next sample.
13. Export the result into user friendly format such as an Excel file.
14. Account for any particles sizes removed via sieve or if lost in the Dean Stark cleaning process – see

CALCULATIONS

10.5-A-7 CARE AND MAINTENANCE

Note: Additional information is available in the instrument manual. All *manufacturers’* recommendations should be followed.

- Inspect the external recirculator filters for discoloration. Replace as needed.
- Do not exceed a water supply pressure of 28 psi.

- Verify and optimize the operation of the circulation pump, debubbling functions and cell rinsing and drainage operations on a routine basis using an acceptable QC sample (e.g. Fullers earth or traceable glass beads less than 40 microns in size)
- Qualified service personnel should service the instrument at regular intervals as specified in the manual.

10.5-A-8 CALCULATIONS

The required calculations are performed automatically by the instrument software. Consult the instrument manual for more details on these calculations. The following corrections must be accounted for:

1. Materials lost through the extraction thimble.

The material which passes through the extraction thimble is all less than nominally 11 microns. For the purposes of generating data regarding the amount of material present less than 44 microns the weight % of the solids which passed through the thimble need to be accounted for in the mass of material less than 44 microns.

$$CORRECTED \text{ wt\% fines in sample} = 100 \left(\frac{\text{corrected mass of fines in sample}}{\text{total mass of solids as determined by Dean Stark}} \right)$$

$$\begin{aligned} &CORRECTED \text{ mass of fines in sample} \\ &= (\text{mass of fines passing thimble}) \\ &+ (\text{mass fraction of fines as determined by LD on recovered solids}) (\text{mass of recovered solids}) \end{aligned}$$

2. Materials removed as oversize through the use of sieves.

Treat the whole sample before sieving or rock removal as 100
Subtract the percent of sieved out material.
Apply this to all % below user sizes selected:

$$\% \text{ below size} = \frac{(100 - \% \text{ removed by sieve}) \times LD \text{ report } \% \text{ below size}}{100}$$

10.5-B SIEVE MEASUREMENT OPTIONS

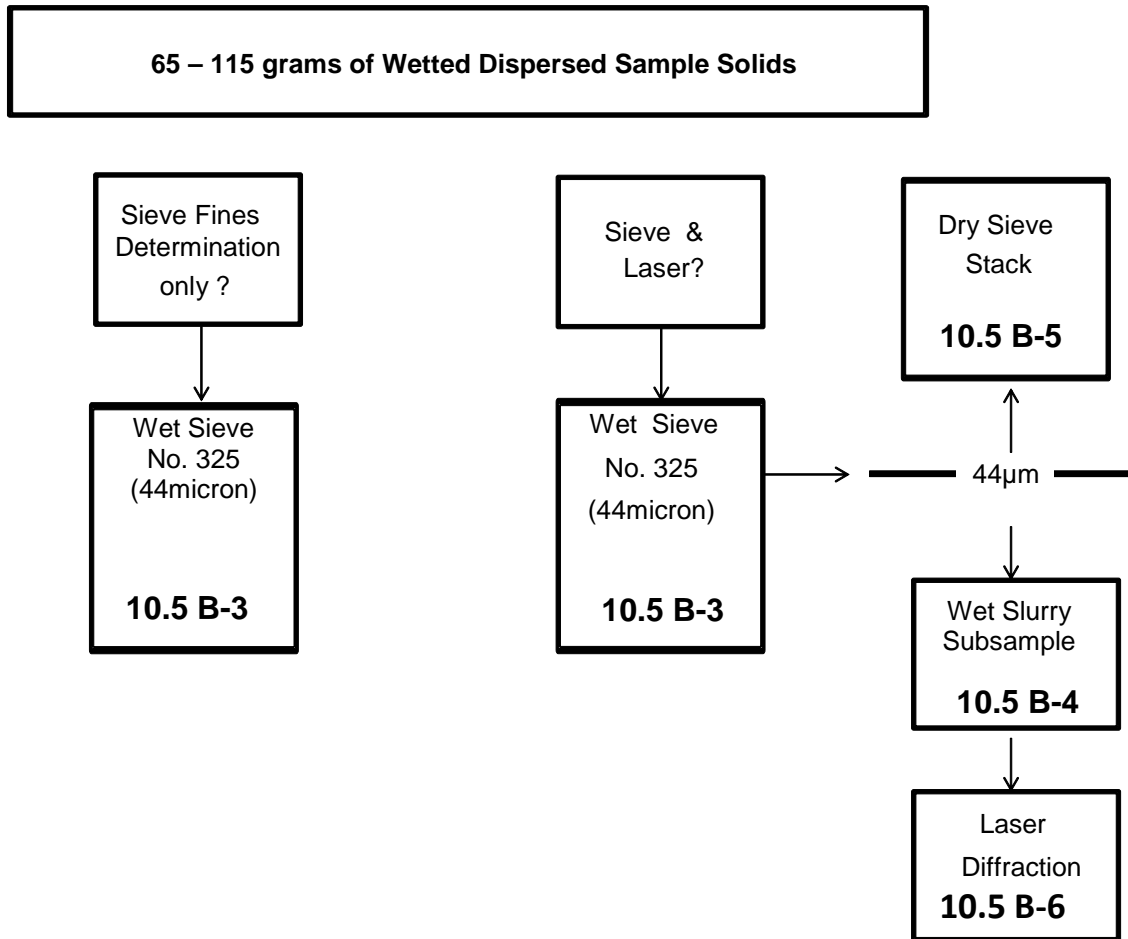
There are a few options with respect to performing the sieve measurements. The options provided are:

- If determining the weight percent of fines only, move to the wet sieve section below (WET SIEVE PROCEDURE). This procedure will enable an evaluation of the >44 and <44 micron particle sizes.
- If proceeding with sieve and laser under 44 micron, consult the following sections:
 - Wet sieve (10.5-B-3)
 - Slurry sampling of the less than 44 micron fraction (10.5-B-4)
 - Laser measurement section (10.5-A)
 - Dry sieve section (10.5-B-5).

This procedure will enable a detailed assessment of the >44 and <44 micron particle size distribution.

Note: This procedure is preferred for small reservoir Laser Diffraction systems to help overcome possible issues with sample concentration within the Laser Diffraction instrument when a large distribution in particle sizes are to be measured. (See APPENDIX B: Minimum Sample Mass Discussion).

Figure 2. Diagram of Sieve Measurement Choices



10.5-B-1 APPARATUS, REAGENTS, MATERIALS:

- Analytical Balance
- Ultrasonic bath for cleaning
- Oven
- Mechanized sieve shaker
- Sieve series US standard:
 - No. 10 (200 microns)
 - No. 18 (1000 microns)
 - No. 20 (850 microns)
 - No. 35 (707 microns)
 - No. 40 (425 microns)
 - No. 45 (354 microns)
 - No. 60 (250 microns)
 - No. 80 (177 microns)
 - No. 120 (125 microns)

- No. 140 (106 microns)
- No. 200 (74 microns)
- No. 325 (44microns)
- Bottom sieve collection tray
- Beakers of appropriate size
- Turbidity meter
- Thermometer
- 1000ml graduated cylinder
- Timer
- Wash bottles
- Rubber Policeman on glass rod
- Overhead mixer
- Disposable syringe or pipette

10.5-B-2SIEVE LOADING CONSIDERATIONS

There are a number of considerations involved in the selection of an appropriate subsample mass for sieving. The charge for testing purposes depends upon size of the sieve apertures, the cross sectional area of the sieve, the particle size distribution of the charge and the bulk density of the material. In addition, There is a maximum size of particle which can be loaded onto a sieve cloth before damage to the cloth is probable.

Maximum particle size

It is recommended by ISO, ASTM and BS that the largest particle placed on a sieve cloth to avoid damage to the cloth is related to the aperture size A (size in millimeters) by the following:

$$\text{maximum particle size in millimeters} = 10(A)^{0.7}$$

Therefore for a screen with 44 micron openings the maximum size of a particle loaded onto the sieve should be 1200 microns.

Sample Loading Considerations

Table 7 shows the appropriate sample loading for sieves of various mesh openings and diameters. This loading is based on scaling the surface area of the sieve cloth in the various sieves. This is the maximum mass of material on any individual sieve to avoid overloading. Overloading will result in retention of material on the sieve which should have passed and thus result in an underestimation of the fines content.

Table 7. Appropriate Maximum Sample Loading for Sieves

Mesh Size (microns)	3 inch Diameter Sieve	8 inch Diameter Sieve	12 inch Diameter Sieve
	Maximum mass retained (g)	Maximum mass retained (g)	Maximum mass retained (g)
2000	25	180	410
850	16	115	260
425	10	75	170

Mesh Size (microns)	3 inch Diameter Sieve	8 inch Diameter Sieve	12 inch Diameter Sieve
	Maximum mass retained (g)	Maximum mass retained (g)	Maximum mass retained (g)
250	8	60	135
150	6	40	90
106	4	30	70
75	3	20	50
44	2	12	27

10.5-B-3WET SIEVE PROCEDURE

1. Inspect the 325-mesh sieve under magnifying lens to ensure that it is in good condition. If the mesh is stretched or otherwise damaged, replace this sieve with one that is in good condition.
2. Weigh the clean and dry 325-mesh sieve and record the mass.
3. Weigh the sieve collection pan and record the mass.
4. If there is material retained on the 2000 micron screen used in the disaggregation step, then a guard sieve is required. Use a 1190 micron (16 mesh) guard sieve stacked above the 44 micron (325-mesh) sieve. This is to protect the 325 mesh screen from being blocked and stretched by large particles. Record the mass of the guard sieve.
5. Stir the freshly dispersed subsample prepared in the preceding dispersion section. If boiling was the selected dispersion technique, the sample should be allowed to cool to < 30 °C but should not be allowed to re-agglomerate by sitting longer than ½ hour. A chiller or ice bath may be used to cool the sample.
6. Start a timer set at 2 minutes and let the sample settle.
7. Pre-wet the sieve by spraying a small amount of water across the surface of the sieve.
8. After the 2 min settling period, decant the fluid portion of the sample onto the sieve guiding the liquid with a rubber tipped policeman.
9. Add IPA over the material in the sieve to break the surface tension if the slurry pools on top of the sieve and allow the fines laden liquid to flow through. Up to 30 ml of IPA may be used.
10. Add 50 to 100 mL of water to the sample in the beaker and stir sample with the rubber policeman.
11. Start the timer and allow the sample to settle for 1 min.
12. Decant the remaining fluid part of the sample in the beaker over the sieve.
13. If the supernatant is still muddy, repeat the water addition and stirring using 30 seconds to settle. This step should be repeated until the supernatant is just cloudy. **Ensure that there are no agglomerated particles in the beaker by gently applying force to them with the rubber policeman.**
14. Quantitatively transfer the coarser settled material on to the sieve using an inverted wash bottle with the tube removed from the base of the cap.

15. Rinse the sieve with water. If a guard sieve was used rinse the guard sieve well and remove it from the sieve stack. The guard sieve must be dried and weighed so that the mass % of material retained on this guard sieve can be determined and used in the final calculations.
16. With the 325 mesh sieve held at a 20 – 30 degree angle over the receiving pan, wash the sieve by rotating it 180 degrees radially while adding water with a normally configured wash bottle (tube attached to cap) until the under washings are clear.
17. Collect the last 100 mL of washings from the 325 mesh sieve and determine the turbidity of the sample to confirm that the washings are “clear”. The solution coming through the 325 mesh to be less than 45 NTU.
18. Do not touch the sieve.
Note: If there is difficulty getting the sample to drain through the 325 mesh consider whether the sample has been adequately dispersed or if the sample loading on the sieve is appropriate.
19. Oven-dry the 325 mesh sieve (and the 16 mesh sieve if used) at 75 ± 5 °C for at least 3 hours to allow determination of the dry weight of solids retained on the top of the sieves.

Note: The drying temperature for test sieves should not exceed 80°C. At higher temperatures the fine metal gauze sieves in particular could become distorted, the gauze tension in the sieving frame could be reduced and the sieve could become less effective during the sieving process.
20. Remove the 325 sieve and its contents from the oven.
21. Cool the 325 sieve and solids to room temperature.
22. Weigh the 325 sieve (and the 16 mesh sieve if used) and record. *These values will allow calculation of the fraction of the sample solids greater than 44 microns. The weight of the -44 μm fraction is done by difference.*

After wet sieving, the sample fractions so isolated may be used for further analysis if desired. One option is sampling the resultant slurry of less than 44 micron material to allow analysis by Laser Diffraction for the purposes of determining particle size distribution of the below 44 micron fraction. The slurry sampling step is described below.

10.5-B-4SLURRY SAMPLING OF THE < 44 MICRON FRACTION (FOR THE PURPOSES OF LASER DIFFRACTION MEASUREMENT)

*Note: This slurry sub sampling step can only be done on slurries which contain **no material larger than 50 microns.** Method development studies have shown that this slurry subsampling step will bias results to higher fines levels if particles greater than 50 microns in size are present in the slurry.*

1. Transfer the material less than 44 micron that washed through the 325 sieve quantitatively to a suitably size beaker or other appropriate container.
2. Insert a 45°pitched blade impeller from an overhead stirrer and slowly ramp up the rpm until it reaches approximately 1725rpm \pm 25rpm. The rpm may be adjusted to create and maintain a 1½” – 2” vortex. An impeller mixer is preferred to ensure the particles are moving vertically as well as horizontally to ensure the sample aliquot is representative.

3. Remove a suitably small increment (e.g. 0.5 mL) of the slurry ~2 cm below the surface using a disposable pipette with a minimum 1 mm opening. Add this aliquot directly to the laser diffraction instrument reservoir.
4. Observe the obscuration or transmittance shown on the laser diffraction instrument display window.
5. Continue to remove small increments of the slurry while it is in motion; adding these to the recirculator until the obscuration or transmittance reaches the manufacturers specified optimum range. Remember that each aliquot so withdrawn must be added to the reservoir in its entirety.

Note: The volume of the slurry sampled will be related to the volume recovered from the wet sieving step. It is preferred that a minimum of 5 aliquots be slurry sampled to obtain the desired obscuration or transmittance value on the laser instrument

6. Start the measurement. Follow the instructions in the laser diffraction measurement section.
7. Abort the measurement if upon addition of the <44micron slurry if the obscuration exceeds the manufacturer recommended range.

10.5-B-5DRY SIEVE PROCEDURE

This procedure is applicable to the sieve sizing of greater than 44 micron sized material. Any resultant value for material of the less than 44 micron fraction obtained from this procedure should not be reported.

1. Disaggregate the oven dried solids retained on the 325 mesh sieve gently with a mortar and pestle.
2. Inspect standard sieves and receiving pan to ensure that they are not clogged, damaged or punctured. Clean or replace if necessary.
3. Weigh and record the mass of the receiving pan.
4. Weigh and record the mass of each sieve to be used.
5. Stack the desired sieves on top of the receiving pan ranging from the 325 mesh sieve at the bottom and moving up in size sequentially ending with the largest sieve at the top. The sieves in the stack should have openings which can be described with a regular mathematical relationship. The two most commonly used relationships are the $\sqrt{2}$ relationship and the $^4\sqrt{2}$ relationship.
6. Weigh and record the mass of the dried solids.
7. Pour the solids over the top of the stacked sieves carefully.
8. Place the sieve stack on the shaker and clamp the stack together with the sieve holder. Shake the appropriate time and settings such that an additional minute of sieving results in a change in weight reporting to any sieve of less than 1%
9. Remove the sieve stack (with pan) from the shaker.
10. Place the stack on the lab bench and rotate the stack 90 degrees, 4 times; each time slightly tapping the sieve with the hand from top to the bottom to ensure that all particles smaller than the openings of the mesh pass through.

11. Re-weigh each sieve (with retains) on the scale and record each of the weights.

10.5-B-6CARE AND MAINTENANCE

Fine mesh sieves are delicate and prone to stretching and damage which affects their accuracy. When using or cleaning sieves;

- Never push or force material through a sieve.
- When cleaning, only brush or wash from the bottom side; the opposite direction of solid flow.
- Weekly, any sieve seen to contain trapped solids, is to be inverted in an ultrasonic bath filled with water, non-foaming surfactant and sonicated for 5 minutes.

10.5-B-7CALCULATIONS

$$\text{Cumulative percent retained (sieve)} = \frac{\sum \text{mass.retained}(g)}{\text{total.mass}(g)} \times 100$$

Wt % less than 44 microns = 100- percent retained on 325 mesh sieve during the wet sieve step- percent retained on guard sieve if used.

The following corrections must be accounted for:

1. Materials lost through the extraction thimble.

The material which passes through the extraction thimble is all less than nominally 11 microns. For the purposes of generating data regarding the amount of material present less than 44 microns the weight % of the solids which passed through the thimble need to be accounted for in the mass of material less than 44 microns.

$$\text{CORRECTED wt\% fines in sample} = 100 \left(\frac{\text{corrected mass of fines in sample}}{\text{total wt of solids as determined by Dean Stark}} \right)$$

$$\begin{aligned} & \text{CORRECTED mass of fines in sample} \\ & = (\text{mass of fines passing thimble}) \\ & + (\text{mass fraction of fines as determined on recovered solids}) (\text{mass of recovered solids}) \end{aligned}$$

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APPENDIX A Dean and Stark Method

SCOPE

This method is used to determine the amount of bitumen (oil), water and solids (O/W/S) in a variety of oil sand processing streams. It may also be used to prepare solids originating from these samples for further analysis such as Particle Size Determination. Sample size for this method is limited to between 40 and 140 grams.

SUMMARY

A weighed sample is separated into bitumen, water and solids by refluxing toluene in a Dean & Stark Soxhlet extraction apparatus. Condensed solvent and co-distilled water are continuously separated in a trap designed to recycle the toluene through the extraction thimble, dissolving the bitumen present in the sample, while the water is retained in the trap for determination. This determination may be done either volumetrically or gravimetrically.

When all the bitumen has been extracted, the washed solids that are retained in the thimble are dried and weighed.

The resulting bitumen/solvent extract and non-filterable solids fraction is separated by centrifuging or by micro-filter filtration. An aliquot of bitumen/solvent extract without the solids fraction is dispersed on a filter paper allowing evaporation of the solvent and gravimetric determination of the bitumen content

BACKGROUND

The Dean & Stark extraction method has long been used to measure the oil, water, and solids content of oil-containing samples. Dean & Stark extraction is based on Soxhlet extraction, which was first proposed by Franz Ritter von Soxhlet in 1879 for the determination of milk fat, and has become the benchmark solid-liquid extraction technique (Soxhlet, 1879). In 1920, a water trap was described by Dean & Stark (Dean & Stark, 1920) for the co-distilled water to be separated from the boiling solvent and subsequent measurement. A combination of the extraction thimble and recycled hot solvent of the Soxhlet extractor with the water trap of Dean & Stark is the basis of the currently used extraction apparatus and method.

The Dean & Stark method was first evaluated for use by Cities Service Athabasca Inc. (now Syncrude Canada Ltd.) in 1962, and by 1964 it became the routine method for analyzing core samples.

Variations of the Dean & Stark method were developed to analyze other sample-types, including extraction froth and tailings streams (Syncrude Canada Ltd., 1979).

Over time, the Dean & Stark method has been refined and has become the industry standard reference method for measuring bitumen, water, and solids in a variety of oil sands samples, including ore, slurry, froth, middlings, tailings, and froth treatment streams.

A significant advantage of the Dean & Stark method is that the bitumen, water, and solids are physically separated and individually measured, typically through weighing. The weight of the measured bitumen + water + solids content typically agrees very well with the original sample weight (see Table A-1)

While the Dean & Stark method enjoys widespread acceptance, it is important to note that the method as practiced was optimized to analyze typical oil sands core, extraction, froth treatment and

tailings samples that contain primarily bitumen, water, solids, and sometimes naphtha. Consideration should be given as to where high concentrations of diluent, process aids or other additives would report. Caution is required when using this technique for samples which have additives or diluents present.

REPORT FORMAT AND REPORTING LIMITS

The results are reported as bitumen, water and solid content as weight % to the nearest 0.01% along with the mass balance achieved for the sample.

PRECISION, ACCURACY AND QUALITY CONTROL

Precision and Accuracy: The precision of this determination is illustrated below. Table A-1 contains typical mass balance ranges for a variety of sample types which may be assayed by this method. Typical values relating the precision of the determination for some example oil sand grades are tabulated in Table A-2 below. Each data set was derived from the analysis of 10 replicate subsamples following homogenization of a larger quantity of oil sand. Hence, precision values indicated include the variance due to subsampling.

Method Repeatability:

Table A-1: Mass Balance Limits for OWS Analyses

Sample Type	Acceptable Mass Balance Range
Middlings	99.00% - 101.00%
Oilsand	99.50% - 100.25%
Primary/Secondary Froth	98.00% - 101.00%
Reject	99.50% - 100.25%
Tailings	98.00% - 101.00%
Froth Treatment Feed/Product (contains diluent)	99.00% - 101.00%
Froth Treatment Tailing (contains diluent)	99.00% - 101.00%

Table A-2: Precision Estimates for Oil Sand Samples

	Mean Value	Standard Deviation	Relative Standard Deviation
	Weight %	Weight %	
Oil Sand 1			
Bitumen	9.00	0.11	1.23
Water	3.57	0.16	4.43
Solids	87.28	0.17	0.19
Oil Sand 2			
Bitumen	8.36	0.25	3.05
Water	4.80	0.16	3.37
Solids	86.68	0.20	0.23

Quality Control:

The mass balance is monitored and reported along with the results.

SAMPLE CONSIDERATIONS

Destructive:	Yes
Amount required:	40 to 140 g. Sample must fit entirely into extraction thimble.
Interferences:	Humidity can affect the mass of materials
Remaining Sample:	The isolated bitumen and water should be considered as hazardous waste. The isolated clean dry solids may be used for further analysis; otherwise they should be appropriately disposed of.

SAFETY CONSIDERATIONS

This method does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Below are some general safety considerations:

- Before handling any chemical you must read and understand the appropriate Material Safety Data Sheet (MSDS).
- Toluene - Flammable, flashpoint 4°C. Harmful vapor; irritates eyes and mucous membranes, may cause dizziness, headache, nausea and mental confusion. Liquid can be absorbed through skin. Liquid may cause dermatitis. All manipulations involving toluene solutions should be carried out in a fume hood.
- Bitumen – Flammable, flashpoint 166 °C (Cleveland Open Cup). May contain compounds exhibiting carcinogenic properties.
 - Work in fume hood/well-ventilated area.
- Glassware - Due to its nature, glassware is easily broken and poses a cut hazard. Handle glassware with respect avoiding excessive force and keeping your hands and body out of the “line-of-fire”. Replace chipped, scratched or damaged components as soon as possible. Consider cut resistant gloves (double glove with chemical resistant gloves when appropriate) for higher risk activities (e.g. applying higher than finger-tip force, handling wet or slippery glassware, etc.). In the event of breakage, please ensure that all chips and shards (big and small) are carefully and thoroughly removed and disposed of in a “broken glass bin”.
- Kettles may crack during the extraction - glassware should be checked before use.

APPARATUS, REAGENTS, MATERIALS

The assembled extraction apparatus is shown schematically in Figure 1 (modified from Bulmer and Starr (Syncrude Canada Ltd, 1979). All of the dimensions given are for use with 43 x 123 mm cellulose extraction thimbles. It is necessary to scale the equipment for use with other thimble sizes.

Apparatus

- Condenser - A straight tube condenser having a jacket at least 400 mm long with a 24/40 standard taper joint. (West type condenser specified in ASTM E123 (2007) is most suitable).
- Water traps* - Graduated traps of 10, 25 or 50 mL capacities or a drain-type trap, having 24/40 standard taper joints. Designs are similar to those described in ASTM E123(2007) See Appendix A-1 for specifications.
- Kettle* - A heat resistant glass vessel having a nominal capacity of 500 mL and a 250 mm minimum neck length (including a 55/50 standard taper female joint to accommodate adapter) for use with 43X123 mm extraction thimble. For use with the larger 60X180 mm thimbles the nominal capacity

must be increased to 1000 mL and a 250 mm minimum neck length (including a 103/60 standard taper female joint to accommodate adapter)

- Kettle-to-Trap Adaptor* - When used with the 43X123 mm thimble this has a 55/50 standard male joint with a perforated downcomer extending below the ground glass surface. The upper part of the adapter is fitted with a 24/40 standard taper female joint. When used with the 60X180 mm thimble this has a 103/60 standard male joint with a perforated downcomer extending below the ground glass surface. The upper part of the adapter is fitted with a 24/40 standard taper female joint.
 - Thimble Basket[#] - A corrosion resistant basket to support the thimble. Fabricated from 1.5 mm diameter nichrome or chromel wire using a silver solder bottom joint (see Figure 2).
 - Solvent Distributor[#] - A corrosion resistant screen (mesh 8) to snugly fit over the open end of the thimble (see Figure 3).
 - Heating mantle - 250 Watt, Glas-col, to fit either a 500 mL or a 1000 mL kettle
 - Variacs or temperature controllers.
 - Metal thimble holders and metal baskets.
 - Centrifuge – capable of generating a relative centrifugal force of 1250. (suitable for use with flammable hydrocarbons)
 - Drying oven –, set at 100°C (rated for use with flammable hydrocarbons)
 - Balances:
 - Top loading balance: 810 g capacity, ±0.001 g (1 mg)
 - Analytical balance: 220 g capacity, ±0.0001 g (0.1 mg)
- * denotes articles obtained from a glassblower
denotes custom made articles

Reagents

- Toluene: reagent grade

Materials

- Extraction thimble - 43 x 123 mm cellulose thimble, single thickness (1 mm wall thickness and 10.0 µm nominal particle retention; Whatman, Cat. No. 2800432) OR 60 x 180 mm cellulose thimble, single thickness (1 mm wall thickness and 10.0 µm nominal particle retention (Whatman Cat. No. 2800-608)
- Tissue wipes - 11 x 21 cm (VWR Cat. No. 82003-820)
- Centrifuge tubes (c-tubes) - 100 mL, conical, graduated (Fisher, Cat. No. 05493)
- Volumetric flasks - 250 mL, Class A
- Filter papers - Glass microfibre filters, 15.0 cm diameter, Whatman grade 934-AH
- Volumetric pipette and bulb - 5 mL
- Volumetric pipette and bulb - 100 mL
- Syringe, 50 mL
- Disposable Whatman syringe filters - 0.45 µm, 25 mm GD/X (Cat. No. 6875-2504)
- Erlenmeyer flasks - 25 mL with stoppers
- Metal funnel
- Tweezers
- Desiccator complete with desiccant
- Metal spatulas

- Watch glass – diameter 14 cm
- Timer with a second hand

PROCEDURE

The detailed procedural steps are based on the apparatus presented in Figure 1. It is acknowledged that other minor equipment variations may be acceptable.

PRELIMINARY PREPARATION:

1. Check all glassware for cracks.
2. Prior to use, insert four tissue wipes into each thimble. Only new, unused thimbles are to be used. Dry these for one hour at 100°C. Remove from oven and store in a desiccator. Place dried thimble with kimwipes into holders and weigh to the nearest 1 mg. Store in desiccator until needed.
3. Dry cleaned centrifuge tubes for one hour at 100°C in oven, store in a desiccator. Weigh to the nearest 0.1 mg prior to use.
4. Dry the glass fiber filters by placing an open box of filters in an oven at 100°C for two hours. Cool the filters in a desiccator. Number the filters with a pencil and weigh to the nearest 0.1 mg.
5. Fill the kettles with toluene to just below the heating mantle level, approximately 200 to 300 mL in the 500 mL kettle OR 300 to 450 mL in the 750 mL kettle.
6. Weigh the sample and bottle to the nearest 1 mg.

SAMPLE HANDLING:

Loading the Sample into the Extractor

1. Turn on the water to the condensers.
2. Remove the weighed thimble holders with thimbles and kimwipes from the desiccator and place in front of the appropriate extractor kettle number at most 10 minutes before loading begins.
3. Remove the tissue wipes and place the thimble in its support basket. .
4. Mount the thimble and basket in the neck of the kettle by spreading the basket's attaching wires.
5. Quantitatively transfer the sample to the thimble using a spatula and washing with small amounts of toluene, wipe the jar and lid dry with the tissue wipes. Ensure that there are no agglomerates in the sample greater than 0.25 inches in size since these may not properly extract during the assay process.
6. Place the tissue wipes in the thimble.
7. Cover the thimble with the “solvent distributor”, attach the thimble basket to the “kettle-to-trap adaptor” and lower the sample into place.
8. Select the proper trap from Appendix A and assemble apparatus as shown in Figure 1, ensuring that all connections are vapor and liquid tight. (**Do not use grease on the ground glass joints.**)
9. Record the weight of the empty sample jar. Determine the mass of the sample being extracted by difference.

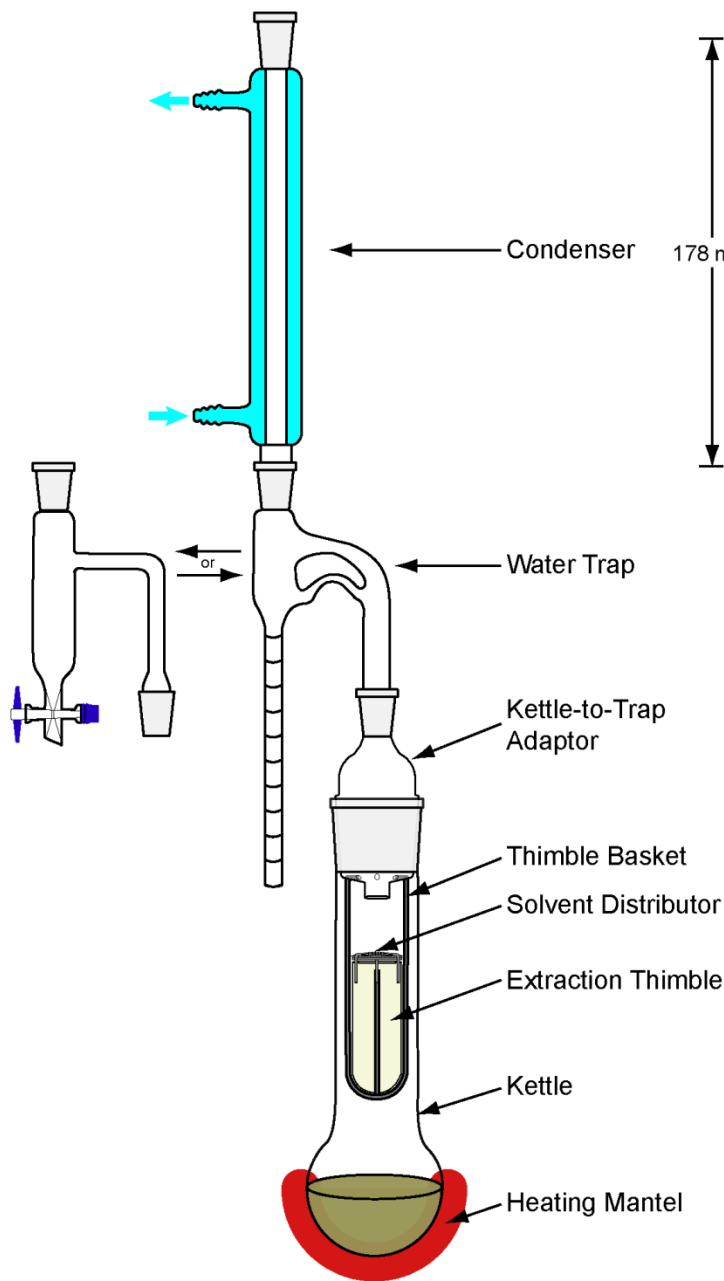


Figure 1. Extraction Apparatus

NOTE: Dimensions shown are for use with 43 X 123 mm thimbles. All equipment must be scaled for use with 60 X 180 mm thimbles.

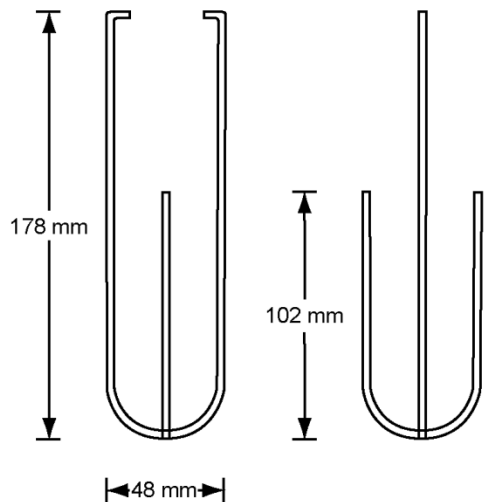


Figure 2. Thimble Basket

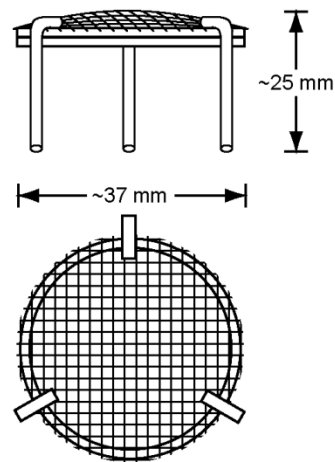


Figure 3. Solvent Distributor

EXTRACTING THE SAMPLE

1. Reflux the sample extract at a rate that does not overflow the thimble. Aluminum foil may be wrapped around the upper portion of the extraction apparatus to speed up the process and/or to ensure water does not condense in the side arm of the trap.

2. Continue refluxing for an additional 30 minutes after the water in the sample is completely removed from the sample and is collected in the trap. Before reading the water level or collecting water in a jar, add a small amount of toluene to the top of the condenser; thus washing any water droplets down into the trap. Check that solvent dripping from the thimble is **colourless**. Some samples may require more extraction time. Extraction times vary widely depending upon the amount of bitumen in the sample and upon the size of the agglomerates in the thimble. Typical extraction times are in the range of 3 to 10 hours. The smaller 43X123 thimbles require less extraction time than the 60X180 thimbles.
3. Drain the toluene from the traps until the level in the kettles is below the top of the heating mantle to ensure the volume is appropriate for easy transfer to a 250 mL volumetric flask in the case of use with the 43X123 mm thimbles OR a 500 mL volumetric flask in the case of the 60X180 mm thimbles. .
4. Turn off power to each individual heating mantle.
5. Check that solvent dripping from the thimble is colorless.

WATER MEASUREMENT

1. Water measurements are taken after the collected water in the trap has reached a constant volume and the temperature of the collected water has equilibrated to room temperature. This may be up to 45 min before the extractors are taken down.
2. Ensure that the water in the sample has collected in the bottom of the water trap and that there are no droplets of water 'hung-up' on the glassware. Obvious water droplets clinging to the walls of the sidearm after draining signal that the glassware used has not been adequately cleaned.
3. Read the volume of water in the volumetric trap to within the maximum reading error (listed in Appendix A) and place it on the data sheet. If using a drain trap, drain water into the weighed empty sample bottle and when all water has been collected and cooled to room temperature, record the weight of the bottle and water. If desired, phase separating paper may be used to ensure that the solvent and water removed from the drain trap are adequately separated.

SOLIDS COLLECTION

Remove aluminum foil if used and allow the thimble to "drip dry" until no more toluene is dripping from the thimble.

Disassemble the apparatus.

Remove the thimble from its support basket and return it to its original metal weighing holder. Place thimble in the fume hood for a few minutes to allow most of the toluene to evaporate before placing the thimbles in an oven rated for use with flammable hydrocarbon.

Weigh the dry thimble containing the solids to the nearest 1 mg the following morning after the sample has cooled in a desiccator to room temperature.

If it is observed that the solids still contain bitumen, the extraction process is repeated until all of the bitumen has been removed.

BITUMEN SOLUTION

1. Transfer the bitumen/solvent solution (which may contain fine solids) to a 250 mL OR 500 mL volumetric flask, depending on the kettle size, rinsing with small portions of toluene. When cool to room temperature, dilute to volume. (NOTE: Volumetric glassware is calibrated at 20°C.)
CAUTION: Bitumen/solvent solution and glassware maybe hot; wait for glassware to cool before handling.

FINES CORRECTION

Small amounts of very fine solids are known to travel through the thimble and report to the hydrocarbon solution in the kettle. The mass of fines must be determined and added to the weight of the solids retained in the thimble when the mass of these fines is greater than ~0.5 grams. Appendix A-2 provides a discussion of these fines; it has been found experimentally that these fines are largely oil wet solids which are likely carried through the thimble by the hydrocarbon/solvent mixture. Samples containing more than ~15 grams of bitumen (as loaded into the thimble) have been found to allow enough solids to travel through the thimble to require a fines correction/centrifugation step (see Appendix A-2).

1. Invert the stoppered volumetric flask containing the bitumen/toluene solution several times to suspend the fines present and immediately transfer 100 mL to a clean, dry, pre-weighed 100 mL centrifuge tube.
2. Centrifuge with a relative centrifugal force of 1250 for 20 minutes to force the solids present to the base of the tube.
3. Carefully discard the remaining solution (supernatant) in the centrifuge tube so as not to dislodge any particles, gently rinse the tube with toluene to remove all of the bitumen solution.
4. Once rinsed, dry the centrifuge tubes and fine solids in the drying oven (rated for flammable hydrocarbon use) at 100°C for a minimum of one hour.
5. Cool in a desiccator to room temperature and reweigh.
6. Determine the weight % of 'fines' which permeated through the thimble.

BITUMEN DETERMINATION

1. Invert the stoppered volumetric flask containing the bitumen/toluene solution several times to ensure proper mixing.
2. Rinse a syringe with the sample extract before withdrawing approximately 20 mL from the volumetric flask.
3. Attach a 0.45 micron Whatman syringe filter to the syringe and filter the extract into a 25 mL Erlenmeyer flask and stopper the flask.
4. Support a weighed glass fiber filter on a watch glass and pipet a 5 mL aliquot of the extract solution (from the 25 mL Erlenmeyer flask) onto the filter paper's surface, evenly dispensing and

distributing the aliquot in a spiral pattern. This application process is needed to ensure the bitumen solution does not penetrate through the filter paper

5. Hang the bitumen containing glass fiber filter from a drying rack to evaporate the solvent. Repeat this for each sample. Remember to rinse the pipette to avoid any carry-over. Drying curves need to be determined according to each fume hood's optimal sash height and corresponding face velocity. Please refer to APPENDIX A-3 for instructions on the determination of an appropriate drying time. Ensure the drying time is adequate to remove all of the solvent but not so long as to cause evaporative losses of bitumen. Typical drying times are on the order of 15 to 20 minutes.
6. Immediately after the allotted drying time, weigh the filter + bitumen on the analytical balance.

CALCULATIONS

$$\% \text{ Water} = \frac{\text{Wt. Water}}{\text{Wt. Sample}} \times 100$$

Density of water is taken as 1 g/mL; hence weight of water (g) is equal to volume of water (mL).

when using a 250 mL volumetric flask

$$\% \text{ Solids} = \frac{\text{Wt. Solids} + (250/100) (\text{Wt. Fines})}{\text{Wt. Sample}} \times 100$$

$$\text{Wt. of Bitumen in Sample} = \frac{250}{5} \times [(\text{Wt. of Filter \& Bitumen}) - \text{Wt. Filter}]$$

$$\% \text{ Bitumen} = \frac{(\text{Wt. of Bitumen in sample})}{(\text{Wt. Sample})} \times 100$$

QUALITY CONTROL PROCESS

The mass balance is monitored and reported along with the results.

ROUTINE MAINTENANCE

- Replace all broken glassware.
- After fume hoods are serviced, the drying curve of the filter and bitumen samples should be checked as these will be dependent on current laboratory conditions (sash height and corresponding face velocity).

REFERENCES

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2. ASTM Standard D473, 2007, "Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method", ASTM International, West Conshohocken, PA, 2007, DOI: 10.1520/D0473-07, www.astm.org.
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4. ASTM Standard D95, 2005 (2010), "Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation", ASTM International, West Conshohocken, PA, 2005, DOI: 10.1520/D0095-05R10, www.astm.org.
5. Dean, E.W., Stark, D.D., "A Convenient Method for the Determination of Water in Petroleum and Other Organic Emulsions", *Journal of Industrial & Engineering Chemistry*, 1920, 12 (5), 486-490.
6. Soxhlet, F.R.v., "Die gewichtsanalytische Bestimmung des Milchfettes", *Dingler's Polytechnisches Journal*, 1879, 232 461-465.
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APPENDIX A-1: Water Trap Specifications

TABLE A-1-1: WATER TRAP SPECIFICATIONS

Volume of Trap (mL)	Scale Division (mL)	Maximum Scale Error (mL)
10	0.1	0.05
25	0.2	0.1
50	0.2	0.1
Drain	none	n/a

APPENDIX A-2: Fines Escaping from Thimble

It has been observed that fine solids may report through the thimble into the extract at the bottom of the extraction kettle. In the few cases where sufficient amounts of these solids passing the thimble were isolated, the size of these solids was found to be less than 11 microns. The mass of solids reported from the extraction assay should include both those solids retained in the thimble as well as the solids which report through the thimble into the extraction kettle. These solids have been quantitated for over 900 samples of varying types. It has been found that there are cases where the amount of solids which pass through the thimble are minimal while in other cases the 'lost' solids are found to have significant weight. A useful guideline is that masses of 'lost' solids greater than ~0.5 grams require correction.

The experimental data does not support an intuitive view that a greater quantity of <44 micron material in the thimble should lead to more fines passing through the thimble. Figure 1 compares these two factors. The chart shows data which is very scattered without any clear correlation between the two factors. For example, when the mass of 'fines' loaded into the thimble was between 30 and 40 grams virtually no 'fines' passed through the thimble. The largest mass of 'fines' passing through the thimble was observed to be ~6 grams which occurred when ~15 grams of fines were in the solids loaded into the thimble.

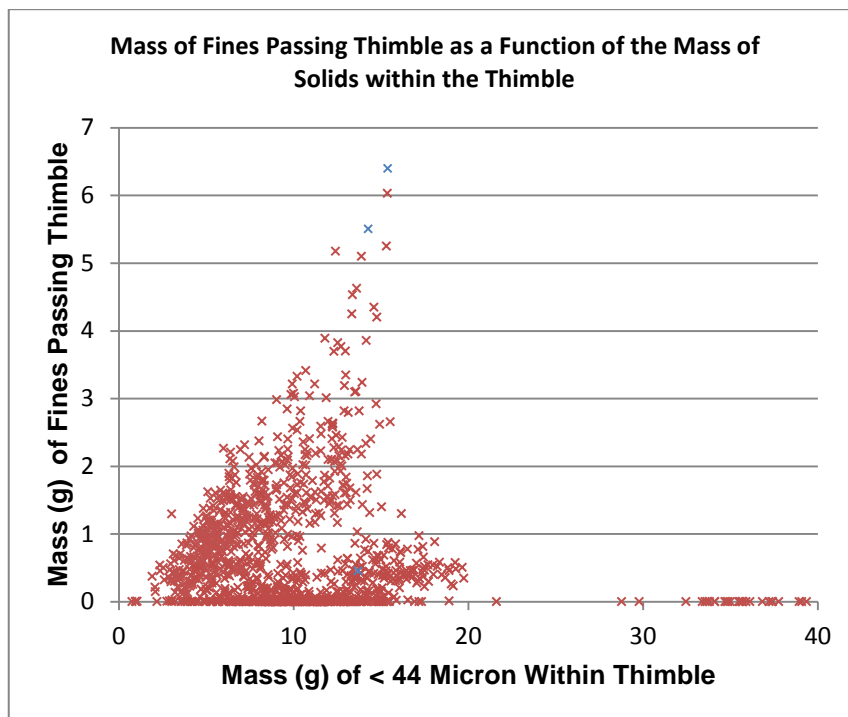


Figure A-2-1. Comparison of the mass of < 44 micron fines passing through the thimble with the mass of < 44 micron fines placed within the thimble

Figure A-2-2 plots the mass of 'fines' passing through the thimble as a function of the amount of bitumen loaded into the thimble for the same samples as shown in Figure A-2-1. This plot indicates that there is a strong relationship between the amount of bitumen loaded into the thimble and the amount of solid material which passes through the thimble. The working hypothesis is that the solids which pass through the thimble are oil wet and therefore move with the solvent/hydrocarbon through the thimble. This relationship can be used to allow the analyst to select samples most likely to require 'fines' correction to the solids mass based on the amount of bitumen present in the thimble. Samples which contain more than ~15 grams of bitumen present in the thimble are likely to lose more than ~0.5 grams of fines through the thimble. This then

suggests that 'fines' corrections should be carried out for these samples. Samples which contain almost no bitumen were observed to have very low losses of fines through the thimble. One can conclude that those samples which contain low levels of bitumen do not require 'fines' corrections to be carried out even though most of the 100 grams of solids loaded into the thimble are <44microns in size.

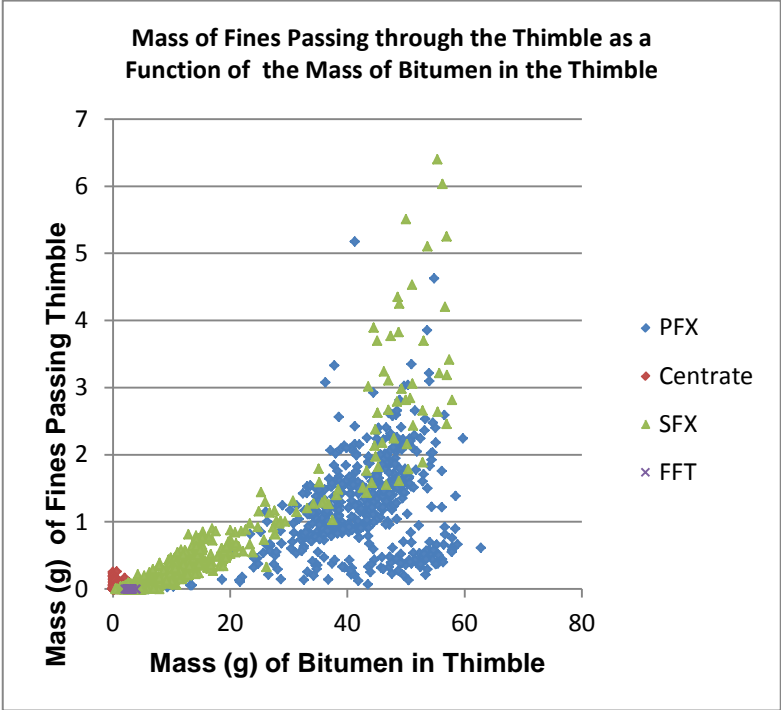


Figure A-2-2. Comparison of the mass of fines passing through the thimble as a function of the mass of bitumen in the thimble

Consideration for the amount of solids lost into the interstices of the thimble itself is more complex. (COSIA may consider future work in this area at a later date)

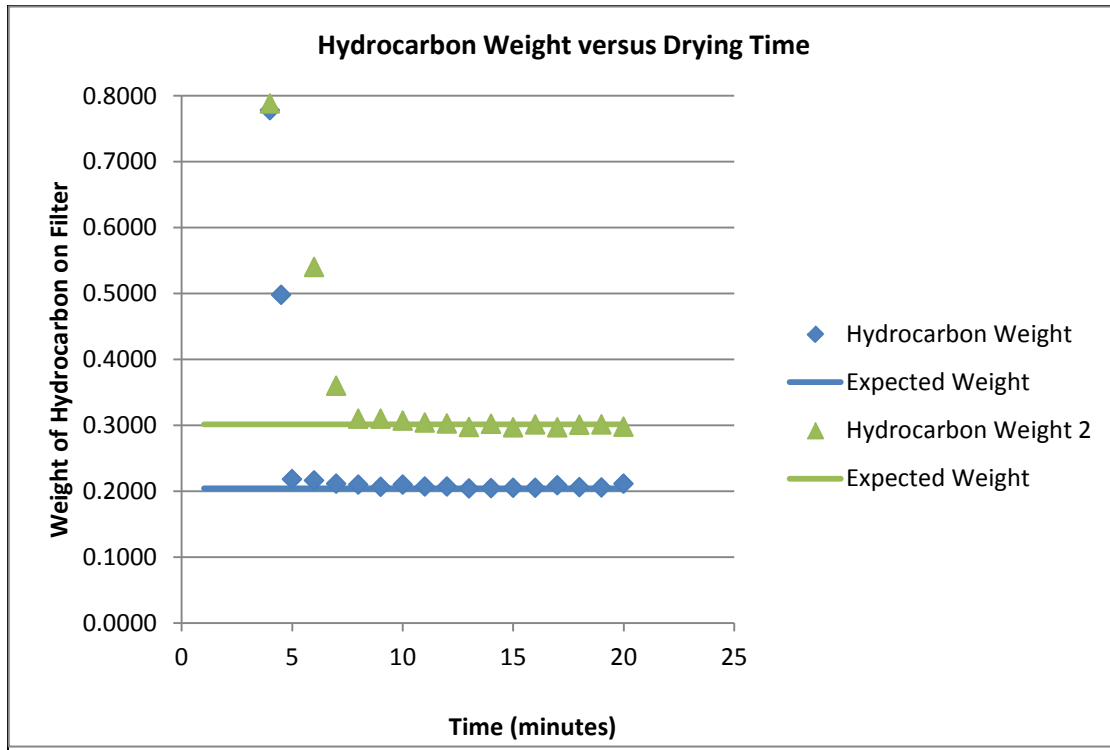
APPENDIX A-3: Establishing an appropriate drying time for Bitumen Determination:

The gravimetric determination of bitumen in the extract taken from the Dean Stark system requires dispensing an aliquot of the extract from a pipet onto a pre-weighed glass fiber filter paper followed by drying the filter paper to constant weight in a fumehood. Each individual laboratory will have a different configuration of drying system and thus will find it necessary to establish the appropriate drying time for the filters.

This can be achieved by following the gravimetric procedure as given in the main body of the method but beginning at 5 minutes record the mass of the glass fiber paper at 1 minute intervals. Plotting the resultant data should lead to a chart like that shown in Figure A-3-1 below . The time at which the mass becomes reasonably constant should be chosen as the drying time.

When establishing the drying times be sure to spread the bitumen and toluene aliquot slowly and evenly in a spiral fashion over the filter ensuring that the liquid spreads and does not drip through the filter. Remember that the clip used to hang the paper to dry must not cover any of the solution applied to the filter.

Figure A-3-1: Hydrocarbon Weight depending on drying Time



APPENDIX B: Minimum Sample Mass Discussion

The total error of a method is made up of several cumulative errors, but in general, the total error equals fundamental error + sub-sampling or segregation error + analytical error. The representative minimum sample mass is set to satisfy a desired maximum fundamental error.

“The fundamental error (FE)...stems solely from the constitution heterogeneity CHL of the material being sampled and is the error that remains when a sampling operation is perfect. The fundamental error is the only error that never cancels out.”(Meyers, 1997, p.209) The larger the representative sample mass, the further the fundamental error will be reduced.

A minimum representative sample mass for measurement is calculated in order that all particles in that received sample have equal opportunity of being selected in the sub-sampling procedure.

There are several references illustrating the calculation of the fundamental error. One selected here is:

- ISO 14488:2007(E) “Particulate materials – Sampling and sample splitting for the determination of particulate properties”.

This reference relate the fundamental error to the top size of particles in the lot as well as the distribution. The goal is to achieve a minimum number of these larger particles in the subsample. This is important since large particles occupy more volume or mass than their smaller counterparts on a per particle basis. It is essential to properly account for these particles in the sub-sample if the original properties of the sample are to be represented.

ISO 14488:

ISO 14488 uses the D90 as the characteristic size where the fundamental error is controlled. When this upper tail is selected then the user may be confident that the incidental overall PSD is very well represented. “It is assumed that the variance of the fundamental error and the number of particles n_0 are related in accordance with Poisson statistics”:

$$CV(n_0) = 100/\sqrt{(n_0)}\%$$

If 10% coefficient of variance (representing fundamental error only) is chosen and the formula rearranged,

$$n_0 = \left(\frac{100}{V(n_0)} \right)^2 = \left(\frac{100}{10(n_0)} \right)^2 = 100 \text{ particles above D90 value}$$

From this point the required minimum sample mass is calculated empirically from previous PSD results. The bin sizes in the distribution are converted to average volume per particle (assuming spheres), then individual mass per particle (assuming 2.65 gm/cc), followed by number of particles within each bin, and finally the required mass to reach 100 particles in the bins above the 90th percentile. The table below shows an example of this process: Other worked examples are shown in Appendix B-1

Coarse Ore Sample											
	A	B	C	D	E	F	G	H	I	J	K
	x µm	Q ₃ % by volume	<x> µm	dQ ₃ % by mass	Volume of particle (m ³)	<M _i > g	n _i number in size class ; per 100g of sample	dQ ₀ % by number	Q ₀ % by number	Number if FE (amount >x90,3) to be 10%	Mass if FE (amount >x90,3) to be 10% g
1	1	0.15	1.4	0.5	1.48E-18	3.92E-12	1.32E+11	83.6	83.6	4.2E+09	1.65E-02
2	2	0.67	3.2	1.1	1.66E-17	4.39E-11	2.42E+10	15.2	98.9	7.7E+08	3.37E-02
3	5	1.73	10.0	2.4	5.24E-16	1.39E-09	1.74E+09	1.1	99.9	5.5E+07	7.65E-02
4	20	4.14	29.7	2.2	1.37E-14	3.62E-08	6.16E+07	0.0	100.0	2.0E+06	7.08E-02
5	44	6.37	51.4	1.2	7.10E-14	1.88E-07	6.22E+06	0.0	100.0	2.0E+05	3.72E-02
6	60	7.54	66.6	1.3	1.55E-13	4.11E-07	3.05E+06	0.0	100.0	9.7E+04	3.97E-02
7	74	8.79	86.0	3.2	3.33E-13	8.83E-07	3.60E+06	0.0	100.0	1.1E+05	1.01E-01
8	100	11.97	111.8	3.9	7.32E-13	1.94E-06	2.01E+06	0.0	100.0	6.4E+04	1.24E-01
9	125	15.86	136.9	4.3	1.34E-12	3.56E-06	1.20E+06	0.0	100.0	3.8E+04	1.36E-01
10	150	20.14	162.0	4.4	2.23E-12	5.90E-06	7.39E+05	0.0	100.0	2.3E+04	1.38E-01
11	175	24.5	187.1	4.3	3.43E-12	9.09E-06	4.72E+05	0.0	100.0	1.5E+04	1.36E-01
12	200	28.79	212.1	4.1	5.00E-12	1.32E-05	3.12E+05	0.0	100.0	9904.1	1.31E-01
13	225	32.92	237.2	3.9	6.99E-12	1.85E-05	2.12E+05	0.0	100.0	6743.6	1.25E-01
14	250	36.85	262.2	3.7	9.44E-12	2.50E-05	1.48E+05	0.0	100.0	4686.0	1.17E-01
15	275	40.54	287.2	3.5	1.24E-11	3.29E-05	1.06E+05	0.0	100.0	3371.5	1.11E-01
16	300	44.03	324.0	6.4	1.78E-11	4.72E-05	1.36E+05	0.0	100.0	4319.5	2.04E-01
17	350	50.45	385.7	8.1	3.00E-11	7.96E-05	1.02E+05	0.0	100.0	3244.1	2.58E-01
18	425	58.58	461.0	6.8	5.13E-11	1.36E-04	5.00E+04	0.0	100.0	1586.8	2.16E-01
19	500	65.37	591.6	12.7	1.08E-10	2.87E-04	4.41E+04	0.0	100.0	1400.7	4.02E-01
20	700	78.04	771.4	6.3	2.40E-10	6.37E-04	9.81E+03	0.0	100.0	311.7	1.99E-01
21	850	84.29	922.0	4.7	4.10E-10	1.09E-03	4.31E+03	0.0	100.0	137.0	1.49E-01
22	1000	88.98	1021.2	1.0	5.58E-10	1.48E-03	6.90E+02	0.0	100.0	21.9	3.24E-02
23	1042.78	90	1208.3	6.3	9.24E-10	2.45E-03	2.58E+03	0.0	100.0	82.0	2.01E-01
24	1400	96.32	1673.3	3.7	2.45E-09	6.50E-03	5.66E+02	0.0	100.0	18.0	1.17E-01
25	2000	100							100.0		
26	TOTAL			99.9			1.58E+11	100.0		5.03E+09	2.26
27	Total rows 23 & 24			10.0			3.15E+03		1.99E-08	100	

Columns A, B and D give the results of a preliminary analysis.

Column C is the calculated geometric mean size (the square root of the products of the size class limits: $\sqrt{\text{lower size bin} * \text{next upper size bin}}$, cell C1 = $\sqrt{B1*B2}$, cell C2 = $\sqrt{B2*B3}$, etc.

Column E is the individual particle volume in meter cubed: $1/6\pi(\langle x \rangle / 1000000)^3$

Column F is the individual particle mass in grams: volume of particle x 2650kg/m³ x 1000

Column G represents the number of particles within a size bin if the total sample size was 100g: $100/\text{individual mass} * \text{percent in bin} / 100 = 100/F1 * D1/100$

Columns H and I represent the distributions by number of particles and serve as checks for mass balance:

H: number in size class per 100g sample / total number of particles in 100g sample * 100

I: cumulative number of particles in 100g sample

Column J is the number of particles per size bin if the fundamental error is to be 10%:

cell I1/100 * cellJ26

Column K is the mass of particles per size bin if the fundamental error is to be 10%:

Cell J1 * cell F1 (number of required particles for 10% fundamental error times individual particle mass)

Cell K26 is the minimum sample mass.

**Table B1: Predicted Minimum
Representative Subsample Mass Required
per ISO 14488**

Minimum Representative Subsample Mass, g	Maximum Allowable Particle Size, microns
0.27	500
0.73	710
1.88	1000
4.77	1400
8.13	1700
12.77	2000

Minimum Sample Mass Recommendations

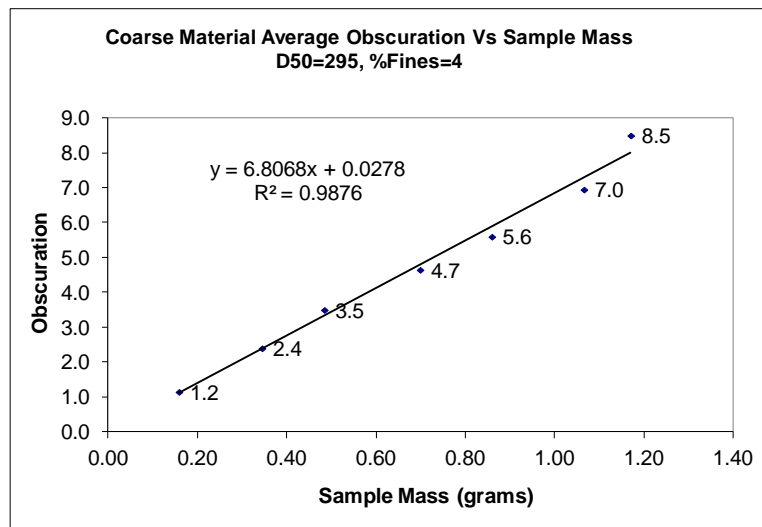
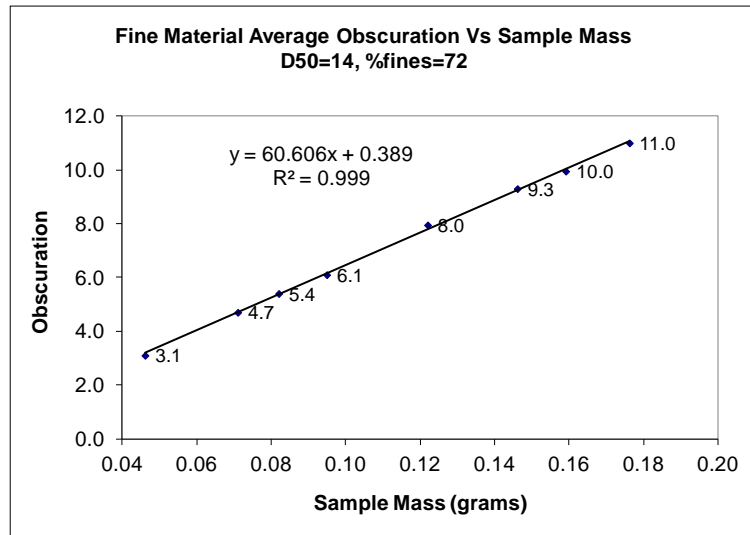
The ISO 14488 approach is preferred for the application of PSD measurement for oil sands and its process streams.

Since we are measuring the particle size distribution of a variety of samples and we do not have a good estimate of the properties of each sample beforehand, a ‘worst case scenario’ of basing our fundamental error on the possibility of a few particles in the upper end of the distribution will allow a blanket value to cover any samples analyzed.

The minimum sample size limitations arising from a consideration of fundamental error do not strongly impact the use of sieves in the range of particle sizes of interest since all 3 inch or 8 inch sieves will require subsample masses much larger than that associated with a consideration of fundamental error.

A limitation of most laser diffraction instruments is the amount of material that can be introduced into the system. The concentration of particles must be dilute enough to avoid multiple scattering. Many systems are supplied with recirculator volumes which restrict the amount of sample which may be assayed to a value much less than that associated with a consideration of fundamental error. Large volume recirculators address this problem. Comminution, or the reduction of average particle size to a smaller particle size, will also address this problem. In a PSD method, the particle size cannot be reduced by comminution as this would render the analysis void; however, the use of sieves to remove larger particles serves the same end result and allows these larger particles to be re-incorporated into the final distribution result after measurement by laser diffraction.

It is recommended to assess the specific instrument in use for the mass versus obscuration or transmittance to observe the maximum mass allowable to reach an obscuration/transmittance within the manufacturers recommended range. Below are example graphs generated on a Mastersizer 2000 showing that a fine sample may reach a mass of about 0.2 grams and a coarser sample may reach a mass of about 1.4 grams with a stable 10% obscuration. Using this information, and the example fundamental error tables attached it can be concluded that a 710 micron sieve cut will render representative sample masses for measurement on the Malvern 2000 with Hydro G recirculator at ~800ml volume. To keep resolution above this size, a series of sieves is needed (i.e. 2000 µm, 1700 µm, 1400 µm, 1000 µm, 710 µm). A larger micron sieve cut for a larger recirculator volume is granted but the imperial data must be used to determine the appropriate mesh size. Extreme caution must be used when carrying this ‘top cut’. Unfortunately, it is very difficult to assess whether aggregates are being retained on the sieve.



APPENDIX B-1 Example Calculations

Fine Ore Sample D90 of 90µm											
	A	B	C	D	E	F	G	H	I	J	K
	x µm	Q ₃ % by volume	<x> µm	dQ ₃ % by mass	Volume of particle (m ³)	<M _i > g	n _i number in size class i per 100g of sample	dQ ₀ % by number	Q ₀ % by number	Number if FE (amount >x90,3) to be 10% g	Mass if FE (amount >x90,3) to be 10% g
1	1	5.27	1.4	9.2	1.48E-18	3.92E-12	2.35E+12	83.8	83.8	7.9E+07	3.09E-04
2	2	14.5	3.2	18.7	1.66E-17	4.39E-11	4.26E+11	15.2	99.0	1.4E+07	6.26E-04
3	5	33.21	10.0	36.7	5.24E-16	1.39E-09	2.65E+10	0.9	100.0	8.8E+05	1.23E-03
4	20	69.92	29.7	12.5	1.37E-14	3.62E-08	3.44E+08	0.0	100.0	1.2E+04	4.17E-04
5	44	82.39	51.4	3.4	7.10E-14	1.88E-07	1.80E+07	0.0	100.0	6.0E+02	1.13E-04
6	60	85.77	66.6	2.2	1.55E-13	4.11E-07	5.33E+06	0.0	100.0	1.8E+02	7.32E-05
7	74	87.96	81.6	2.0	2.85E-13	7.54E-07	2.70E+06	0.0	100.0	9.0E+01	6.82E-05
8	90	90	94.9	1.1	4.47E-13	1.19E-06	9.11E+05	0.0	100.0	3.0E+01	3.61E-05
9	100	91.08	111.8	2.3	7.32E-13	1.94E-06	1.16E+06	0.0	100.0	3.9E+01	7.52E-05
10	125	93.33	136.9	1.7	1.34E-12	3.56E-06	4.86E+05	0.0	100.0	1.6E+01	5.79E-05
11	150	95.06	162.0	1.3	2.23E-12	5.90E-06	2.27E+05	0.0	100.0	7.6E+00	4.48E-05
12	175	96.4	187.1	1.0	3.43E-12	9.09E-06	1.10E+05	0.0	100.0	3.7E+00	3.34E-05
13	200	97.4	212.1	0.7	5.00E-12	1.32E-05	5.36E+04	0.0	100.0	1.8E+00	2.37E-05
14	225	98.11	237.2	0.5	6.99E-12	1.85E-05	2.65E+04	0.0	100.0	8.9E-01	1.64E-05
15	250	98.6	262.2	0.3	9.44E-12	2.50E-05	1.04E+04	0.0	100.0	3.5E-01	8.69E-06
16	275	98.86	287.2	0.1	1.24E-11	3.29E-05	3.95E+03	0.0	100.0	1.3E-01	4.35E-06
17	300	98.99	324.0	0.1	1.78E-11	4.72E-05	1.27E+03	0.0	100.0	4.3E-02	2.01E-06
18	350	99.05	385.7	0.0	3.00E-11	7.96E-05	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
19	425	99.05	461.0	0.0	5.13E-11	1.36E-04	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
20	500	99.05	591.6	0.0	1.08E-10	2.87E-04	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
21	700	99.05	771.4	0.0	2.40E-10	6.37E-04	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
22	850	99.05	922.0	0.0	4.10E-10	1.09E-03	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
23	1000	99.05	1183.2	0.0	8.67E-10	2.30E-03	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
24	1400	99.05	1673.3	1.0	2.45E-09	6.50E-03	1.46E+02	0.0	100.0	4.9E-03	3.18E-05
25	2000	100							100.0		
26	TOTAL			94.7			2.81E+12	100.0		9.38E+07	3.17E-03
27	Total rows 8-24			10.0			2.99E+06		1.07E-06	100	

Minimum sample mass

Ore Sample with D90 ~700											
	A	B	C	D	E	F	G	H	I	J	K
	x µm	Q ₃ % by volume	<x> µm	dQ ₃ % by mass	Volume of particle (m ³)	<M _i > g	n _i number in size class i per 100g of sample	dQ ₀ % by number	Q ₀ % by number	Number if FE (amount >x90,3) to be 10% g	Mass if FE (amount >x90,3) to be 10% g
1	1	0.34	1.4	0.9	1.48E-18	3.92E-12	2.29E+11	85.3	85.3	2.1E+09	8.43E-03
2	2	1.24	3.2	1.6	1.66E-17	4.39E-11	3.71E+10	13.8	99.1	3.5E+08	1.53E-02
3	5	2.87	10.0	3.1	5.24E-16	1.39E-09	2.20E+09	0.8	100.0	2.1E+07	2.86E-02
4	20	5.92	29.7	3.3	1.37E-14	3.62E-08	9.08E+07	0.0	100.0	8.5E+05	3.08E-02
5	44	9.21	51.4	2.1	7.10E-14	1.88E-07	1.14E+07	0.0	100.0	1.1E+05	2.00E-02
6	60	11.35	66.6	1.5	1.55E-13	4.11E-07	3.56E+06	0.0	100.0	3.3E+04	1.37E-02
7	74	12.81	86.0	1.9	3.33E-13	8.83E-07	2.16E+06	0.0	100.0	2.0E+04	1.79E-02
8	100	14.72	111.8	1.8	7.32E-13	1.94E-06	9.13E+05	0.0	100.0	8.6E+03	1.66E-02
9	125	16.49	136.9	2.5	1.34E-12	3.56E-06	7.05E+05	0.0	100.0	6.6E+03	2.35E-02
10	150	19	162.0	3.4	2.23E-12	5.90E-06	5.73E+05	0.0	100.0	5.4E+03	3.17E-02
11	175	22.38	187.1	4.2	3.43E-12	9.09E-06	4.63E+05	0.0	100.0	4.3E+03	3.94E-02
12	200	26.59	212.1	4.9	5.00E-12	1.32E-05	3.67E+05	0.0	100.0	3.4E+03	4.55E-02
13	225	31.45	237.2	5.2	6.99E-12	1.85E-05	2.81E+05	0.0	100.0	2.6E+03	4.87E-02
14	250	36.65	262.2	5.3	9.44E-12	2.50E-05	2.12E+05	0.0	100.0	2.0E+03	4.97E-02
15	275	41.96	287.2	5.2	1.24E-11	3.29E-05	1.58E+05	0.0	100.0	1.5E+03	4.86E-02
16	300	47.15	324.0	9.8	1.78E-11	4.72E-05	2.07E+05	0.0	100.0	1.9E+03	9.17E-02
17	350	56.94	385.7	12.0	3.00E-11	7.96E-05	1.51E+05	0.0	100.0	1.4E+03	1.12E-01
18	425	68.93	461.0	9.1	5.13E-11	1.36E-04	6.68E+04	0.0	100.0	6.3E+02	8.51E-02
19	500	78.01	584.0	12.0	1.04E-10	2.76E-04	4.34E+04	0.0	100.0	4.1E+02	1.12E-01
20	682	90	690.9	0.8	1.73E-10	4.58E-04	1.68E+03	0.0	100.0	1.6E+01	7.21E-03
21	700	90.77	771.4	3.9	2.40E-10	6.37E-04	6.17E+03	0.0	100.0	5.8E+01	3.68E-02
22	850	94.7	922.0	2.0	4.10E-10	1.09E-03	1.88E+03	0.0	100.0	1.8E+01	1.91E-02
23	1000	96.74	1183.2	1.9	8.67E-10	2.30E-03	8.40E+02	0.0	100.0	7.9E+00	1.81E-02
24	1400	98.67	1673.3	0.7	2.45E-09	6.50E-03	1.05E+02	0.0	100.0	9.8E-01	6.37E-03
25	2000	99.35							100.0		
26	TOTAL			99.0			2.69E+11	100.0		2.52E+09	0.93
27	Total rows 20-24			84.6			1.07E+04		3.97E-08	100	

Minimum sample mass

Ore Sample with D90 ~600											
	A	B	C	D	E	F	G	H	I	J	K
	x µm	Q ₃ % by volume	<x> µm	dQ ₃ % by mass	Volume of particle (m ³)	<M> g	n _i number in size class, per 100g of sample	dQ ₀ % by number	Q ₀ % by number	Number if FE (amount >x90,3) to be 10%	Mass if FE (amount >x90,3) to be 10% g
1	1	0.46	1.4	1.2	1.48E-18	3.92E-12	2.93E+11	81.4	81.4	1.5E+09	6.01E-03
2	2	1.61	3.2	2.8	1.66E-17	4.39E-11	6.31E+10	17.5	98.9	3.3E+08	1.45E-02
3	5	4.38	10.0	5.2	5.24E-16	1.39E-09	3.76E+09	1.0	100.0	2.0E+07	2.73E-02
4	20	9.6	29.7	3.5	1.37E-14	3.62E-08	9.52E+07	0.0	100.0	5.0E+05	1.80E-02
5	44	13.05	51.4	2.0	7.10E-14	1.88E-07	1.08E+07	0.0	100.0	5.6E+04	1.06E-02
6	60	15.08	66.6	1.8	1.55E-13	4.11E-07	4.46E+06	0.0	100.0	2.3E+04	9.56E-03
7	74	16.91	86.0	3.9	3.33E-13	8.83E-07	4.36E+06	0.0	100.0	2.3E+04	2.01E-02
8	100	20.76	111.8	4.5	7.32E-13	1.94E-06	2.34E+06	0.0	100.0	1.2E+04	2.37E-02
9	125	25.29	136.9	5.2	1.34E-12	3.56E-06	1.47E+06	0.0	100.0	7.7E+03	2.73E-02
10	150	30.51	162.0	5.6	2.23E-12	5.90E-06	9.47E+05	0.0	100.0	4.9E+03	2.92E-02
11	175	36.1	187.1	5.7	3.43E-12	9.09E-06	6.30E+05	0.0	100.0	3.3E+03	2.99E-02
12	200	41.82	212.1	5.6	5.00E-12	1.32E-05	4.24E+05	0.0	100.0	2.2E+03	2.93E-02
13	225	47.43	237.2	5.3	6.99E-12	1.85E-05	2.88E+05	0.0	100.0	1.5E+03	2.78E-02
14	250	52.76	262.2	4.9	9.44E-12	2.50E-05	1.97E+05	0.0	100.0	1.0E+03	2.57E-02
15	275	57.68	287.2	4.5	1.24E-11	3.29E-05	1.37E+05	0.0	100.0	7.2E+02	2.36E-02
16	300	62.19	324.0	7.9	1.78E-11	4.72E-05	1.67E+05	0.0	100.0	8.7E+02	4.12E-02
17	350	70.07	385.7	8.7	3.00E-11	7.96E-05	1.10E+05	0.0	100.0	5.7E+02	4.57E-02
18	425	78.81	461.0	6.2	5.13E-11	1.36E-04	4.55E+04	0.0	100.0	2.4E+02	3.23E-02
19	500	85	547.3	5.0	8.58E-11	2.27E-04	2.20E+04	0.0	100.0	1.1E+02	2.61E-02
20	599.02	90	647.5	5.7	1.42E-10	3.77E-04	1.50E+04	0.0	100.0	7.8E+01	2.96E-02
21	700	95.66	771.4	1.4	2.40E-10	6.37E-04	2.18E+03	0.0	100.0	1.1E+01	7.26E-03
22	850	97.05	922.0	1.7	4.10E-10	1.09E-03	1.52E+03	0.0	100.0	7.9E+00	8.62E-03
23	1000	98.7	1183.2	0.8	8.67E-10	2.30E-03	3.44E+02	0.0	100.0	1.8E+00	4.13E-03
24	1400	99.49	1673.3	0.5	2.45E-09	6.50E-03	7.84E+01	0.0	100.0	4.1E-01	2.66E-03
25	2000	100							100.0		
26	TOTAL			99.5			3.60E+11	100.0		1.88E+09	0.52
27	Total rows 20-24			79.2			1.91E+04		5.32E-08	100	

Minimum sample mass

Ore Sample with D90 ~300											
	A	B	C	D	E	F	G	H	I	J	K
	x µm	Q ₃ % by volume	<x> µm	dQ ₃ % by mass	Volume of particle (m ³)	<M> g	n _i number in size class, per 100g of sample	dQ ₀ % by number	Q ₀ % by number	Number if FE (amount >x90,3) to be 10%	Mass if FE (amount >x90,3) to be 10% g
1	1	0.57	1.4	1.2	1.48E-18	3.92E-12	3.11E+11	84.2	84.2	2.0E+08	7.72E-04
2	2	1.79	3.2	2.4	1.66E-17	4.39E-11	5.47E+10	14.8	99.0	3.5E+07	1.52E-03
3	5	4.19	10.0	4.7	5.24E-16	1.39E-09	3.38E+09	0.9	100.0	2.1E+06	2.97E-03
4	20	8.88	29.7	4.4	1.37E-14	3.62E-08	1.22E+08	0.0	100.0	7.7E+04	2.80E-03
5	44	13.31	51.4	1.4	7.10E-14	1.88E-07	7.39E+06	0.0	100.0	4.7E+03	8.80E-04
6	60	14.7	66.6	2.0	1.55E-13	4.11E-07	4.90E+06	0.0	100.0	3.1E+03	1.27E-03
7	74	16.71	86.0	7.2	3.33E-13	8.83E-07	8.15E+06	0.0	100.0	5.2E+03	4.56E-03
8	100	23.91	111.8	10.3	7.32E-13	1.94E-06	5.33E+06	0.0	100.0	3.4E+03	6.55E-03
9	125	34.25	136.9	11.4	1.34E-12	3.56E-06	3.20E+06	0.0	100.0	2.0E+03	7.22E-03
10	150	45.65	162.0	11.0	2.23E-12	5.90E-06	1.86E+06	0.0	100.0	1.2E+03	6.94E-03
11	175	56.61	187.1	9.7	3.43E-12	9.09E-06	1.07E+06	0.0	100.0	6.8E+02	6.15E-03
12	200	66.32	212.1	8.1	5.00E-12	1.32E-05	6.09E+05	0.0	100.0	3.9E+02	5.10E-03
13	225	74.38	237.2	6.5	6.99E-12	1.85E-05	3.50E+05	0.0	100.0	2.2E+02	4.10E-03
14	250	80.86	262.2	5.0	9.44E-12	2.50E-05	2.01E+05	0.0	100.0	1.3E+02	3.18E-03
15	275	85.88	287.2	3.9	1.24E-11	3.29E-05	1.19E+05	0.0	100.0	7.5E+01	2.48E-03
16	300	89.8	300.7	0.2	1.42E-11	3.77E-05	5.30E+03	0.0	100.0	3.4E+00	1.27E-04
17	301.45	90	324.8	5.2	1.79E-11	4.76E-05	1.09E+05	0.0	100.0	6.9E+01	3.29E-03
18	350	95.2	385.7	3.6	3.00E-11	7.96E-05	4.51E+04	0.0	100.0	2.9E+01	2.27E-03
19	425	98.79	461.0	0.5	5.13E-11	1.36E-04	3.38E+03	0.0	100.0	2.1E+00	2.91E-04
20	500	99.25	591.6	0.0	1.08E-10	2.87E-04	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
21	700	99.25	771.4	0.0	2.40E-10	6.37E-04	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
22	850	99.25	922.0	0.0	4.10E-10	1.09E-03	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
23	1000	99.25	1183.2	0.0	8.67E-10	2.30E-03	0.00E+00	0.0	100.0	0.0E+00	0.00E+00
24	1400	99.25	1673.3	0.8	2.45E-09	6.50E-03	1.15E+02	0.0	100.0	7.3E-02	4.75E-04
25	2000	100							100.0		
26	TOTAL			99.4			3.69E+11	100.0		2.34E+08	0.06
27	Total rows 20-24			76.1			1.58E+05		4.28E-07	100	

Minimum sample mass

APPENDIX C Change process

This unified fines measurement method will be refined as industry practice evolves including data results from subsequent studies, laboratory experience and evolving sampling methodology. It is understood that after the Inter-Laboratory Study is completed, COSIA will provide a structured and transparent process to update the method in the future, where industry practitioners and others can propose improvements to the method. It is expected this process will follow other methods currently in place with other standard organizations.

At a later date, COSIA will evaluate the need to have this method incorporated into an existing standards organization such as CGSB, ASTM, ISO or other organization.