MICROSCOPICAL METHODS FOR DETERMINING COAL CONCENTRATIONS IN SETTLED DUST

MICRO-SCIENCES, LTD. METHOD 02

PREPARED FOR

POTOMAC RIVER GENERATING STATION

MIRANT MID-ATLANTIC, LLC

28 APRIL 2005

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ATTN: MS. D. KNIGHT
PROJECT MANAGER

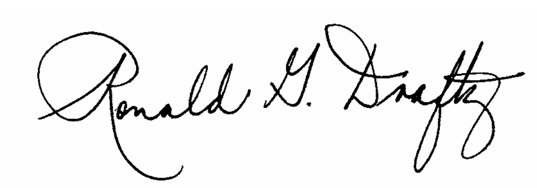
28 APRIL 2005

FOREWORD

The method described in this report was prepared for the Potomac River Generating Station, Mirant Mid-Atlantic, LLC, as part of a planned study of added emission controls for fugitive coal dust. The work was funded by Mirant Mid-Atlantic, LLC. The method details procedures for the microscopical determination of raw and pyrolyzed coal collected as settled dust.

Ms. Debra Knight of the Potomac River Generating Station serves as Project Manager for the Settled Dust Study.

Respectfully submitted,
Micro-Sciences, Ltd.
28 April 2005



Ronald G. Draftz
Principal Scientist

MICROSCOPICAL METHOD FOR DETERMINING COAL CONCENTRATIONS IN SETTLED DUST

MICRO-SCIENCES, LTD. PROJECT 04-01/METHOD 02

OVERVIEW FOR QUANTITATING RAW & PYROLYZED COAL DUST

Soiling complaints from settled dusts are usually due to black or dark-brown particles that adhere to light-colored surfaces, though the reverse also occurs. While there are no objective standards for what may produce a soiling event, complaints can be caused by episodic deposition of discrete, visible, large particles or by the accumulation of finer particles over a long time period.

Episodic soiling is usually attributable to large single particles or agglomerates that are often greater than 50µm in diameter and are therefore, seen as discrete particles. Cumulative soiling, typical of urban areas, is usually attributable to particles smaller than 50 µm in diameter. These smaller particles are not usually perceived as having caused soiling until they form into rings of particles following the evaporation of light rain or dew.

Prior to forming rings of deposits from evaporated dew, these smaller particles occur as a somewhat evenly distributed layer of dust that reduces surface reflectivity uniformly. The reduction in surface reflectivity isn't obvious because the particles are too small to be seen as discrete particles and form a uniform layer with no noticeable optical discontinuities. Therefore, the soiling effect of these particles isn't seen until the particles puddle into rings of dust after water droplets evaporate.

Particle soiling is due to obscuration, color contrast and/or reduced reflectivity. When there are multiple types of particles contributing to soiling it is necessary to measure the contribution of each type so its contribution can be attributed to its source, whether generic or specific. In this study the sole focus is on raw coal and on partially pyrolyzed coal associated with glassy flyash. Other soiling particles such as tire fragments, molds and assorted other dark particles common to urban areas will not be counted. The focus of this study is to determine the sources and concentrations of coal dust and their frequency of occurrence.

Particle soiling is a function of the surface area of the particle not its mass. Therefore, thin and thick particles of the same particle type would have the same soiling impact if they covered identical areas of a light colored surface. One of the most accurate methods for measuring the soiling contribution by particle type is microscopical counting and areal sizing. This is usually done with a graticule such as the British Standard graticule shown in Figure 1.

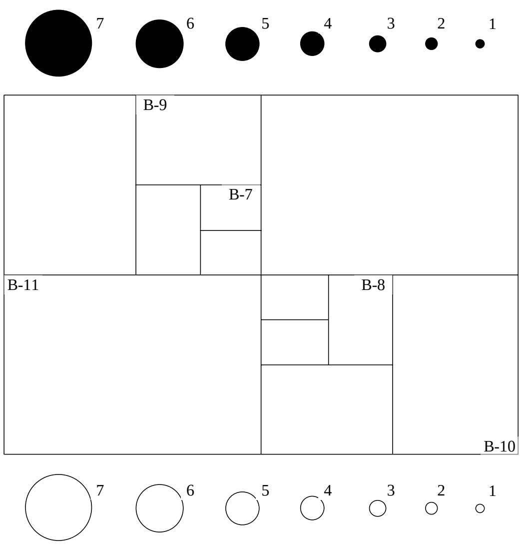


Figure 1. British Standard Graticule Layout

The rectangular boxes are used to define the field of view in which particles are counted. One box is pre-selected so that there are approximately ten particles of interest to be counted in that box at a given magnification. This target concentration is used so that the counting areas are spread over a larger area of the sample to minimize errors due to non-uniformity such as those caused by clustering of particles from dew. Typically, a hundred fields of are counted for every magnification and every particle type. For example, the counting done for this project necessitates using a 4x and a 10x objective. Therefore, one hundred fields of view are counted for each objective magnification. The process is repeated for each type of particle. In this study, there are two particle types: raw coal and partially pyrolyzed coal which means a total of four hundred fields of view, are counted for each sample.

The numbers shown in the boxes, B7, B8, etc. do not appear in the graticule but are shown here to mark the sequence of box sizes. Each box has twice the area of the next smaller box. The two largest boxes are not marked but would be B13 for the largest and B12 for its two halves.

The projected areas of the particles are sized by comparison to the opaque circles above the boxes; the clear circles are used for transparent particles. The area of each circle is twice as large as the next smallest circle following the same progression as for the boxes.

The counting procedure is to select a particle type such as rubber tire fragments and then count the number by size in the selected box. The sample is then moved to another field of view for counting. Counting is continued until a predetermined number of particles or fields of view have been tallied. This process is then repeated at some other magnification until the full range of desired sizes for the selected particle type is completed. The process is repeated for the next type of particle.

There is no need to determine the mass of the soiling components because mass has no direct relationship to soiling unless the shape of the particle and its orientation on a surface is a constant. Thick and thin flakes with the same projected area have the same soiling contribution though one may be ten times as heavy.

The following sections describe methods for sample preparation, counting and computation of the areal concentrations of raw and pyrolyzed coal particles.

1. SAMPLE PREPARATION FOR COUNTING

1.1. Sample Inspection

Each sample is inspected upon receipt in the lab using the following steps.

1.1.1. Examine the outer shipping container, usually a cardboard box, for damage. Record all observed damage and note any corresponding damage to the cardboard sample containers.

1.1.2. Note whether the packing material is intact and whether it may have produced particles that could have entered the sample containers. Materials such as polystyrene foam pellets can be a cause of sample contamination.

1.1.3. Record the date samples were received. Complete and return the chain of custody form, if any, to the sender.

1.1.4. Examine and note damage to any of the sample containers, especially if the shipping container was damaged.

1.1.5. Open and inspect each sample container. The sampling substrate should be taped to the bottom of the container at each corner to prevent the substrate from making contact with the container cover.

1.1.6. Examine the surface of each sample for smudges or streak marks. Mark those areas with a felt tip pen so that they are not included in the sections used for analysis.

1.1.7. Cut the tape at each corner using a stainless steel scalpel. Cut alongside the edge of the substrate. This will leave part of the tape on the bottom of the sample container and the mating part of the tape on the substrate. The tape is used to orient the substrate for sectioning to eliminate bias in sampling.

1.1.8. Photograph each substrate to record the visual appearance of the entire substrate and the general loading of particles and any pattern or displacement of particles caused by dew, rain or physical action.

1.1.9. Take a closeup photograph of each sample to record the appearance of the particles and their loading.

1.2. Sample Sectioning

Sectioning is done to remove a strip of sample from the substrate. Four subsections are cut from this strip for the mounts used for microscopical counting.

1.2.1. Orient the substrate so that the bottom edge is identified from its position in the sample container. The bottom edge is that edge opposite the hinged cover of the sample container. This position is used to ensure that the sectioning is not biased and assumes the substrate has been placed randomly in the sample container by the sampling team.

1.2.2. Cut a strip from each substrate using scissors cleaned with isopropanol or glass cleaner. Cut the strip starting in the middle of the bottom edge of the substrate up to near the center of the sample substrate. The strip should be approximately ¾” wide so it will fit without overlap on the 1” wide glass microscope slide.

1.2.3. Place the strip on a clean surface and then cut four sections approximately 1½” long starting at the end of the strip that was at the middle of the substrate. Discard the second and fourth sections in order from the middle of the NACE sheet. Mount the first and third on glass slides.

1.2.4. Place the substrate back in its container and re-tape it so that is has the original orientation.

1.2.5. Discard the remainder of the strip once the sections have been mounted.

1.3. Sample Mounting

The two sections will be mounted on a cleaned glass slide and left uncovered. This improves the ability to see surface texture details and luster of the raw and pyrolyzed coal particles in reflected light. Immersion oil is placed between the section and glass slide to keep the section flat for microscopy.

1.3.1. Clean two glass slides with glass cleaner to remove surfactant and soap residues used during manufacturing. Wipe the slide with a low-lint tissue.

1.3.2. Place a drop of immersion oil (Zeiss n_d = 1.515, or equivalent) on the center of each slide.

1.3.3. Place each section on top of the oil drop and allow the drop to flow under the section until the section lies flat.

1.3.4. Add preprinted labels to each slide. The labels should contain: slide number, sample number, immersion medium, preparer and date prepared, as a minimum.

1.3.5. Store the slides flat in a slide cabinet so that the immersion oil does not leak off the slide.

2. COUNTING PROCEDURE

The counting procedure requires the British Standard graticule or similar that will provide field-defining areas (the Fields Of View, FOV) for counting and a regular array of circles for sizing the projected areas of the particles.

The graticule must be calibrated for each objective magnification and any additional magnification produced by a supplemental intermediate lens in the microscope. The supplemental lens described in this procedure is the OPTOVAR™ of the Carl Zeiss Company.

The method for calibrating the graticule using a calibrated stage micrometer is not part of this procedure but is assumed to be known to any microscopist using this method.

Both the raw coal and pyrolyzed coal are counted and sized by the same procedure.

Oblique, reflected, darkfield illumination provides better reflected light images and detail of surface morphology than samples covered with a coverslip. Coverslips reflect much of the oblique reflected light.

The counting strategy used for these samples cover a particle size range from approximately 10 µm and larger in diameter, equivalent circular area. This requires the use of four counting size ranges with a 10x objective and four with a 4x objective. This range extends from 11.3 to 152 μm in diameter. The size range can be easily adjusted by addition of a fifth size range, if larger particles are encountered in the samples.

2.1.1. Place one of the uncovered sample mounts into a mechanical stage on the microscope.

2.1.2. Mentally divide the sample mount into three horizontal sections. Move the mount to the left (or right) edge and middle of the top horizontal section to begin counting.

2.1.3. Select the largest field of view box for counting that will produce a total count of approximately 10 particles for each field of view. The outer box in Figure 1, is typically the one used for defining the counting area.

2.1.4. Record the number of fields of view and particle sizes using a five button tally counter.

2.1.5. Count all particles by size of raw coal that fall within the size ranges corresponding to the selected magnification. The particles must be either entirely within the FOV box selected for sizing or touching the left or top line of the box. Any particle that touches the bottom or right line of the box must not be counted, even if just the edge of the particle rests on those lines. Correspondingly, the particles should be counted even if just a tip of the particle is superimposed on the left or top lines.

2.1.6. If the particle touches the left and bottom lines simultaneously, the particle should be counted. If the particle touches the upper and right lines, it should not. This is a simple statistical strategy to randomize the counting.

2.1.7. Particles are sized by comparison to the area of the opaque circles. Actually, particles are matched with two adjacent circles. If the particle's projected area falls between the two circles then it is counted as a particle in that size range. If the particle closely matches the area of the smaller circle of the pair it is consider in the size range; if it matches the larger circle of the pair, it is consider to be in the next larger size range.

2.1.8. After the first FOV is counted, the sample mount is moved a small lateral distance towards the opposite edge of the slide using the mechanical stage. It is usually sufficient to move the slide enough so that the next FOV does not contain any particles seen in the previous field of view.

2.1.9. Continue to move the slide, recording the number of fields of view and particles by size range until reaching the opposite edge of the slide. Move the slide vertically to occupy the center third of the strip. Continue counting and sizing by moving the slide back to the opposite edge of the slide.

2.1.10. A total of at least 50 fields of view should be counted and sized on the first slide. This will take approximately one to two horizontal passes along the entire length of the sample section.

2.1.11. Record the number of fields of view and numbers of particles in each size range.

2.1.12. Repeat steps 2.1.2 to 2.1.11 for the second slide of the sample.

2.1.13. Repeat the counting and sizing procedure in steps 2.1.1 to 2.1.12 for the next objective magnification and the same type of particle.

2.1.14. Repeat steps 2.1.1 to 2.1.13 for the next particle type. This completes the counting and sizing for a sample. This procedure is continued until all the samples have been counted.

3. CALCULATIONS OF AREAL CONCENTRATIONS

The area covered by the raw and by the pyrolyzed coal can now be calculated from the count data to determine the percent coverage of the NACE surface. These percentages are the concentrations that will be used with wind directional persistence to to determine whether specific source controls are adequate to minimize soiling.