SOP
Air-void analysis with flatbed scanner

Updated: October 19, 2015 by Karl Peterson

Safety Notes:

Safety Equipment Required:

Eye protection and safety shoes/boots at all times during sample preparation, N95 respirator and ear protection when cutting samples with block saw in GB22, nitrile gloves when working with acetone in exhaust hood.

Principle:

Air-voids in concrete samples exposed on a cut and polished surface are contrast-enhanced with black ink and white powder, scanned, and air-void parameters measured.

Safety Notes and Operational Concerns:
- The block saw is loud and dangerous, do not make personal contact with the rotating diamond blade. Before starting, ensure you do not have any loose clothing or similar (e.g. drawstrings from hooded sweatshirt) that could get caught in moving parts. The mist generated during cutting contains respirable crystalline silica dust; wear an N95 respirator. Mist can be minimized by proper adjustment of water flow cooling the blade.
- A combination of compressed air and water spray is used when cleaning the sample surfaces, do not direct the compressed air nozzle towards yourself. Although a rare occurrence, compressed air can enter the blood stream through a break in the skin. Air bubbles in the blood stream (an embolism) can cause paralysis or death.
- The silicon carbide abrasives used during lapping should not be inhaled. Take care when scooping and transferring so as not to generate airborne particulates.
- The white wollastonite powder used to fill air-voids should not be inhaled. Take care when scooping and transferring so as not to generate airborne particulates.
- Perform all work involving acetone (matrix stabilization, and removal of nitrocellulose) under the exhaust hood.
- Perform initial inking of surface with black solvent marker under exhaust hood.

Equipment and Materials:
Block saw
Concrete
Grinding/lapping wheel

Diamond embedded platen
SiC paper
Lapping machine
Loose SiC grit
Solvent black marker
Zoom stereo microscope
Flatbed scanner

Method Outline:
(Detailed descriptions attached)
- Cutting and labeling
- Initial surface grinding
- Lapping
- Final polish
- Sticker application
- Contrast enhancement
- Darkening voids in aggregate
- Sample Scanning

Waste Disposal and Clean-up:

- Ensure block saw, grinding/polishing wheel, lapping machine drain hoses empty into 19 L buckets. Allow fines to settle in the drainage buckets before decanting. Discard fines in GB22 waste collection barrel. Rinse and wipe down machines and countertops after use.
- Waste acetone from matrix stabilization step and final polished-surface cleaning to be collected in acetone waste bottle, and stored in GB10 flammable cabinet for pick up by Environmental Health & Safety.
- Use wet paper towels to clean up white wollastonite powder spills, and discard in trash.

U of T automated flatbed scanner air-void analysis protocol

This guide is specific to equipment currently available at U of T, and it is not intended as a definitive guide to sample preparation and analysis. There are alternative methods that can accomplish the same objective (e.g. grinding/polishing on a rotating disc flat lap equipped with a series of magnetic backed diamond discs, darkening the surface with an ink pad, filling the voids with a zinc oxide paste, and analysis performed with a Rapid Air 457). Although an effort has been made here to describe the necessary steps for performing a contrast-enhanced automated air-void analysis, this guide is not comprehensive, and it does not replace the hands-on training required before the independent execution of the procedures described herein.

1) Cutting and labeling (5-10 minutes per core/cylinder)
- Use eye protection, ear protection, safety shoes, an N95 respirator, and water-proof apron.
- Ensure you do not have any loose clothing or similar (e.g. drawstrings from hooded sweatshirt) that could get caught in moving parts. Do not make personal contact with the rotating diamond blade.
- Ensure that the block saw drain hose empties into a 19 L bucket, and that there is sufficient water flow to cool the diamond blade. Use just enough water to cool the saw; too much water creates excess mist.
- Slice the 100 mm core/cylinder in half along the long axis using the sliding tray feed, and then cut two 75 mm segments from one core half as shown in Figure 1. Each pair of segments is considered one sample for air-void analysis. We are limited to a cross-sectional area of 100 × 75 mm due to the diameters of our grinding, lapping, and polishing equipment.
- Rinse the sliding tray with hose when finished. Collect all water in 19 L buckets, and allow fines to settle before decanting. Discard fines in sludge waste collection barrel.
- Rinse and dry the segments, and label the bottom face of each segment as shown in Figure 1.
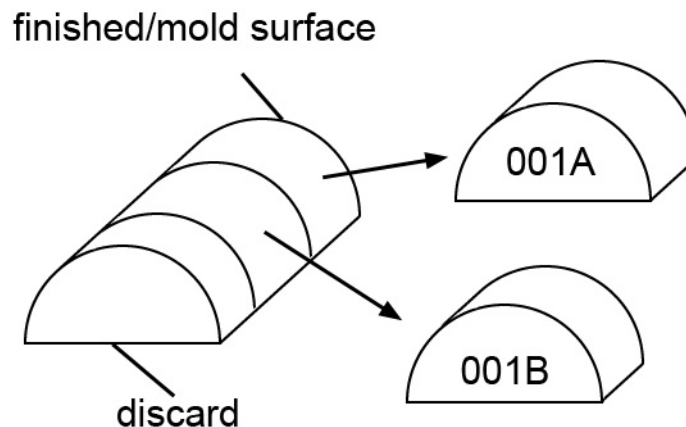

Figure 1: Cutting and labeling of core segments.

2) Initial surface grinding (5-10 minutes per segment-pair sample)
- Use eye protection, safety shoes, and water-proof apron.
- Check the grinding wheel drain for blockages and clear if necessary. Ensure that the drain hose empties into a 19 L bucket.

- Mount the 200 mm dia. coarse 100 grit (180 µm) diamond embedded platen on the grinding wheel. Rotate the platen clockwise until it locks into place.
- Fill the water reservoir (Figure 2).

Figure 2: Surface grinding with water and diamond embedded platen.

- Grind the surface flat by applying evenly distributed hand pressure to the sample (~15 kPa) on the rotating (~300 rpm) diamond embedded platen. Use plenty of water to prevent grinding residues from building up. Maintain a consistent orientation; do not rotate or rock the sample, as this may introduce convexity to the surface[1]. Depending on the initial roughness of the surface, the hardness of the aggregate, the speed of rotation, and the pressure applied, this step should take between 1 and 2 minutes per sample.
- When finished, immediately clean the surface using a combination of water and compressed air (Figure 3). Do not direct the compressed air nozzle towards your body. Although a rare occurrence, compressed air can enter the blood stream through a break in the skin. Air bubbles in the blood stream (an embolism) can cause paralysis or death.

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[1] Technically speaking, a flat surface can be maintained through careful rotation of the sample, however, for the purposes of this manual (intended for new workers unfamiliar with the fine art of surface grinding/lapping) maintaining a consistent orientation is a much more straightforward and consistent approach.

Figure 3: Cleaning the surface with combined water and compressed air.

- Inspect the surface. If it is not flat, or does not appear to be uniformly ground (e.g. you can still see saw marks) then repeat the grinding process.
- When finished, rinse the tray and platen, remove the platen (use platen lifting tool if stuck), dry the grinding wheel and platen, and place the platen back in the storage rack.
- Allow fines to settle in the drainage bucket before decanting. Discard fines in waste collection barrel.

3) Lapping (25-30 minutes per segment-pair sample)

- Use eye protection, safety shoes.
- Ensure that the drain hose empties into a 19 L bucket. Fill the lapping machine slurry reservoir with water and 600 grit (12 μm) silicon carbide (SiC) abrasive. Take care when scooping and transferring the abrasive so as not to generate airborne particulates.
- Establish slurry flow, and center the retaining rings on the platen so that they extend an equal distance beyond the inner and outer edges of the platen. If beginning production for the day, run for three minutes, and then check the platen for flatness using the dial gage and reference flat plate (Figure 4). When checking for flatness ensure that the dial gage, the reference flat plate, and the radial sector of the platen under examination are meticulously clean. Platen flatness should be maintained to within ± 2 μm.
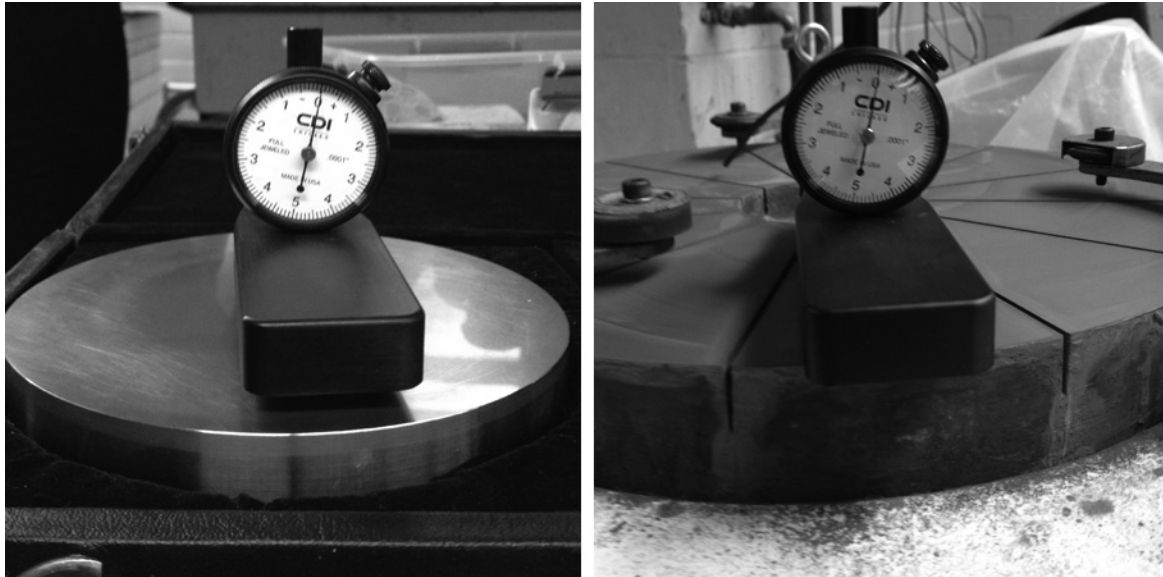
Figure 4: Dial gage zeroed out on flat reference plate (left) and placed on a flat platen (right). The smallest division on this gage is one "mil" or about 2 μm. If the platen were concave, the indicator would read to the left of the zero-point. If the platen were convex, the indicator would read to the right of the zero-point.

- If the platen is out of tolerance in a concave sense, slide the retainer rings outward; if out of tolerance in a convex sense, slide the retainer rings inward (Figure 5). When positioning the retaining rings, ensure they extend beyond both edges (inner and outer) of the platen. For a concave correction, the ring will overlap more on the outer edge of the platen, and overlap just slightly on the inner edge of the platen. For a convex correction, the ring will overlap more on the inner edge of the platen, and overlap just slightly on the outer edge of the platen. If this rule is not followed, a "stair step" will be lapped into the platen (something you want to avoid). To perform the correction, run the lapping machine with just the retaining rings in place, and check for flatness periodically until concavity or convexity is corrected. The amount of time it will take to correct depends on the degree of concavity or convexity. Typically a deviation of ±3 μm will require about an hour to correct.

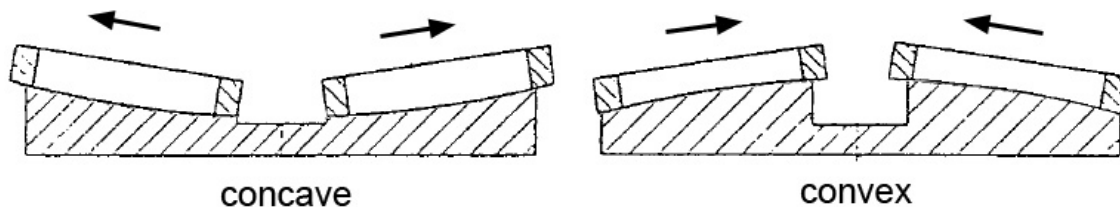

concave   convex

Figure 5: Positioning of retaining rings for platen flatness correction. Figure adapted from Stähli A. W., "The Technique of Lapping," a corporate publication of Stahli Lapping Technology Ltd., Pieterlen/Biel, Switzerland.

- If the platen is sufficiently flat, center the retaining rings, wet the sample surfaces, place them face-down inside the retaining rings, and lap the samples for 20 minutes (Figure 6).

- When finished, <u>immediately</u> clean the lapped surfaces using a combination of water and compressed air (Figure 3).
- Inspect each lapped surface under incident light for a uniform reflection (Figure 7). If the surface is not flat, repeat the lapping procedure. If the surface is flat, place the sample in the 35°C oven to dry.
- If finished for the day, remove the retaining rings and clear the platen grooves of abrasive and debris using a plastic credit card or similar. Wipe the machine and countertop clean. Allow fines to settle in the drainage bucket before decanting. Discard fines in waste collection barrel.
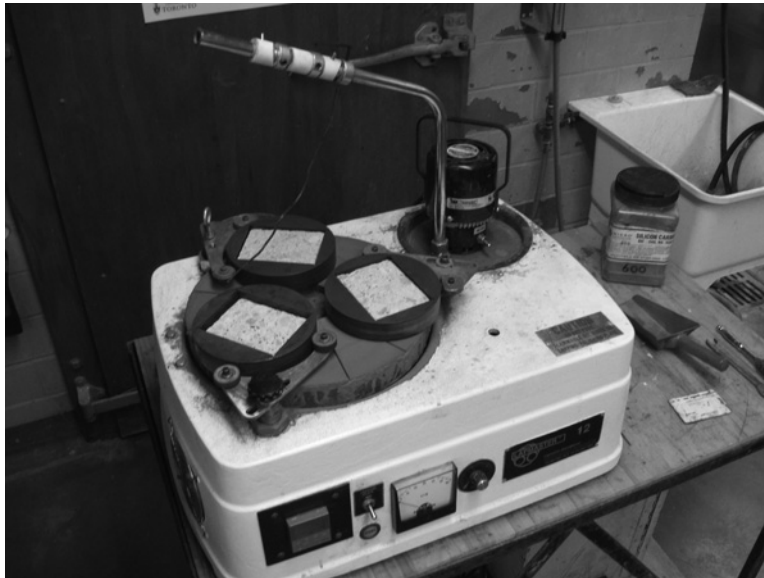

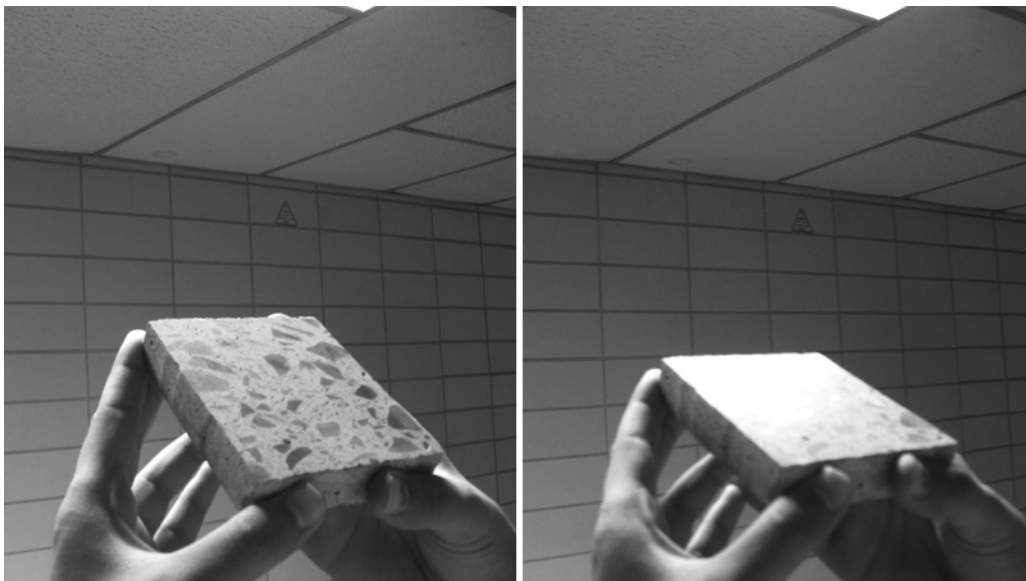Figure 6: Samples inside retaining rings on lapping platen.

Figure 7: Checking for a uniform flat reflective surface under reflected light.

4) Final polish (10-15 minutes per segment-pair sample)

- Use eye protection and safety shoes. When working with acetone use nitrile or latex gloves, and perform the work under an exhaust hood.
- After drying in the 35°C oven, apply a thin coat of a dilute nitrocellulose solution (typically a 5:1 solution of acetone:clear finger nail polish) to the surface with a clean applicator brush. Perform this step under an exhaust hood or in a well-ventilated area (Figure 8). Clean brush with fresh acetone. Transfer waste acetone to hazardous waste container for pick up by Environmental Health & Safety.
- Place the sample in the vented 35°C oven to dry.
- Mount pressure-sensitive-adhesive (PSA) backed 600 grit (12 μm) SiC paper onto the grinding wheel platen (Figure 9) and mount the platen on the grinding wheel (Figure 9).

Figure 8: Application of nitrocellulose solution to strengthen paste matrix.

Figure 9: Mounting PSA SiC paper to grinding wheel platen.

- Check the grinding wheel drain for blockages, and clear if necessary. Ensure that the drain hose empties into a 19 L bucket.
- Mount the platen on the grinding wheel. Rotate the platen clockwise until it locks into place.
- Fill the water reservoir (Figure 2).
- Polish the surface by applying evenly distributed hand pressure to the sample (~15 kPa) on the rotating (~300 rpm) platen. Use plenty of water to prevent polishing residues from building up. Maintain a consistent orientation for the first 30 seconds. After this initial period, rotate the sample through the four quadrants (Figure 10), applying pressure for 5-10 seconds at each rotation.
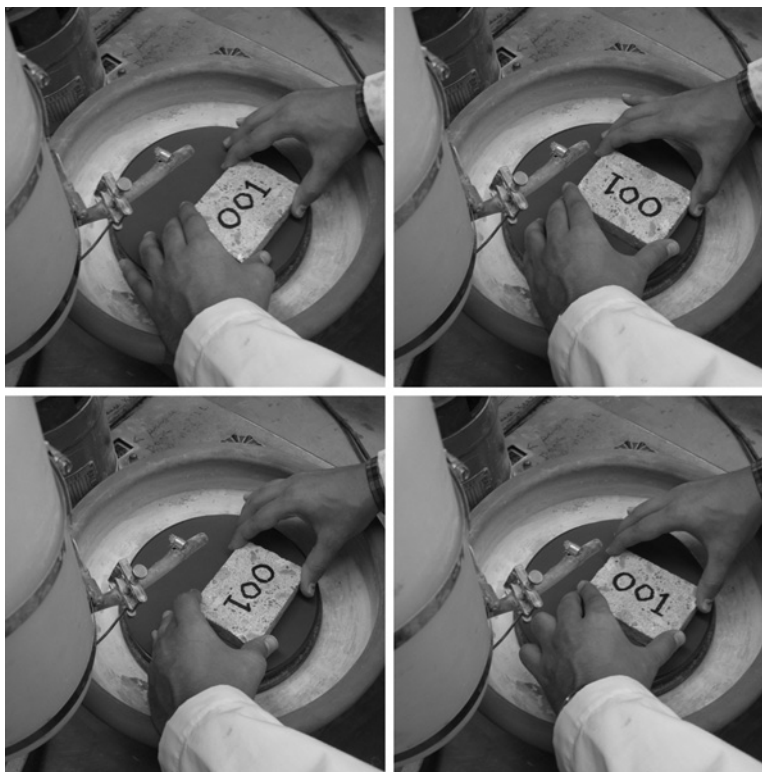

Figure 10: Rotation of sample during final polishing step.

- When finished, immediately clean the polished surface using a combination of water and compressed air (Figure 3).
- Inspect the surface under incident light (Figure 7), if it is not uniformly polished, repeat the polishing procedure.
- If the surface appears polished, then place the surface face down in a tray with acetone, followed by a brief rinse with fresh acetone. Perform this step under an exhaust hood. The acetone will remove any remaining nitrocellulose lining the air-voids. Transfer waste acetone to hazardous waste container for pick up by Environmental Health & Safety.
- When finished, rinse the tray and platen, remove the platen (use platen lifting tool if stuck), dry the grinding wheel and platen, and place the platen in the storage rack.
- Allow fines to settle in the drainage bucket before decanting. Discard fines in waste collection barrel.

5) Sticker application (2 minutes per segment-pair sample)

- To help prevent scratches to the flatbed scanner glass plate, apply a small sticker to each of the four corners of the sample (Figure 11). Press the stickers flat; if a sticker does not rest flat (e.g. wrinkles or bent corners present), remove it and any remaining adhesive material and replace with a new sticker. If the presence of a large entrapped air-void precludes the possibility of corner placement, it is okay to adjust the position slightly away from the corner.

Figure 11: Application of stickers (cut from sheet) to four corners.

- If a manual point count for aggregate content is planned, the sample should be scanned at this point. A resolution of 8 µm per pixel (3,175 dpi) and 24-bit RGB color are recommended. Further details regarding scanning and point counting are included in Sections 8 and 9. Regardless of whether or not a point count is performed, a scanned image prior to contrast enhancement can be helpful when darkening voids in aggregate as discussed in Section 7.

6) Contrast enhancement (10-15 minutes per segment-pair sample)

- Inspect the polished surface. If not properly polished revisit Sections 2, 3, or 4 as necessary.
- Avoiding the stickers, use a permanent marker to cover the surface with overlapping parallel lines (Figure 12). If the marker is solvent-based, perform this step in a well-ventilated area or under an exhaust hood. If ink does get on a sticker, wipe away the ink immediately with a paper towel.

Figure 12: Darkening the surface with marker.

- Rotate the sample 90° and repeat the previous step.
- Place sample in ventilated 35°C oven to dry.
- After drying, place the sample face up, and distribute loose white powder (2 μm sized $BaSO_4$ barite, $CaSiO_3$ wollastonite, or similar) over the entire surface in an approximate 5 mm thick layer. Take care when scooping and distributing the powder so as not to generate airborne particulates.
- Compact the powder into the surface using a glass slide or similar flat and rigid surface.

Figure 13: Removal of excess powder with razor blade.

Figure 14: Wiping of excess powder with lightly-oiled fingertip.

- Remove excess powder by gently scraping with the edge of a new razor blade (Figure 13). If air-voids are not fully packed, re-apply powder as needed to specific areas and repeat the previous step. Razors present a cutting hazard, so use with care to avoid injury.
- Lift the sample and wipe excess powder from the counter top with a wet paper towel, and collect into waste container. Take care when cleaning up so as not to generate airborne particulates.
- With a <u>very</u> lightly oiled fingertip, wipe away excess powder from the sample surface (Figure 14) using gentle pressure (~10 kPa). Use a paper towel to clean your fingertip after each wipe. If too much oil is used, or too much pressure, powder retained in air-voids will be removed (something you want to avoid). Wash your hands when finished.
- Inspect the surface under a zoom stereo microscope at low magnification, and remove any remaining streaks of white powder by wiping with clean fingertip. Wash your hands when finished. If un-packed voids are common, re-apply powder as needed and repeat the previous four steps.

7) Darkening voids in aggregate (5-10 minutes per segment-pair sample)
- Open the color scanned image collected in Section 5. You can use this image as a guide to assist with the location of air voids in coarse aggregate, and then color them in with a marker (Figure 15).
- After coloring in voids in aggregate that are easily observed without magnification, move to the zoom stereo microscope, and color in any voids in aggregate missed in the previous step as well as any cracks observed in the mortar (Figure 16).

Figure 15: Darkening of macro-sized voids in aggregate using marker.

Figure 16: Darkening of micro-sized voids in aggregate using marker.

8) Sample scanning (5-10 minutes per segment-pair sample)

Different scanners have different software. What follows are instructions for an EPSON Perfection V500 Photo.

- Start the scanner and the EPSON Scan software.
- Use a soft brush to clear the scanner plate glass (Figure 17), and use lens paper to remove any remaining smudges or fingerprints.
- Clean the stickers at the four corners, and carefully place the sample face down on the scanner glass plate (Figure 18) next to the black/white reference block (e.g. a flat plate with black and white vinyl tape).
- Cover the scanner with the black box to exclude exterior light sources (Figure 19).

Figure 17: Removing dust from scanner plate glass using soft brush.

Figure 18: Gently placing sample face down on scanner plate glass.

Figure 19: Covering scanner with light-proof non-reflective black-interior box prior to image collection.

- Click the *Preview* button, and select the area for analysis, including the black/white reference block (Figures 20 and 21).
- Every scanner and scanner software package has its own peculiarities. The basic objective is to collect an 8 μm pixel (3,175 dpi) resolution image that is 24-bit RGB color if point counting aggregate, or 8-bit grayscale if performing an automated air-void analysis. All other scanner software image-enhancement options should be de-activated. Although this sounds simple it can sometimes be a challenge depending on the software. Figure 20 shows the window interface for EPSON scan. When collecting a grayscale image for air-void analysis, the settings shown in Figure 20 should be used. If collecting a color image, the only difference is that *Image Type* should be set to 24-bit color. In EPSON Scan it is possible to de-select all of the image adjustment features (Unsharp Mask, Descreening, Color Restoration, Backlight Correction, Dust Removal, DIGITAL ICE Technology) with the exception of *Auto Exposure Type*. Unfortunately, *Auto Exposure Type* is a mandatory setting, so just leave it as *Photo*. Fortunately, it is possible to undo the histogram stretching performed by the auto exposure feature by selecting the histogram button shown (Figure 20). In the *Histogram Adjustment* window set both the input and the output end points to 0 and 255, and the gamma setting to 1.00 to ensure a linear scan (Figure 20).
- Select the *Scan* button (Figure 20). Save the image in TIFF format. At U of T, we use the sample ID as the filename and append "_color" if it is a 24-bit RGB image, and append "_bw" if it is an 8-bit grayscale image. Note that Epson Scan also automatically appends a three digit number to the end of each filename. It is good practice to remove this appended number from the filename after the scan is finished to avoid any possible confusion during subsequent data management.
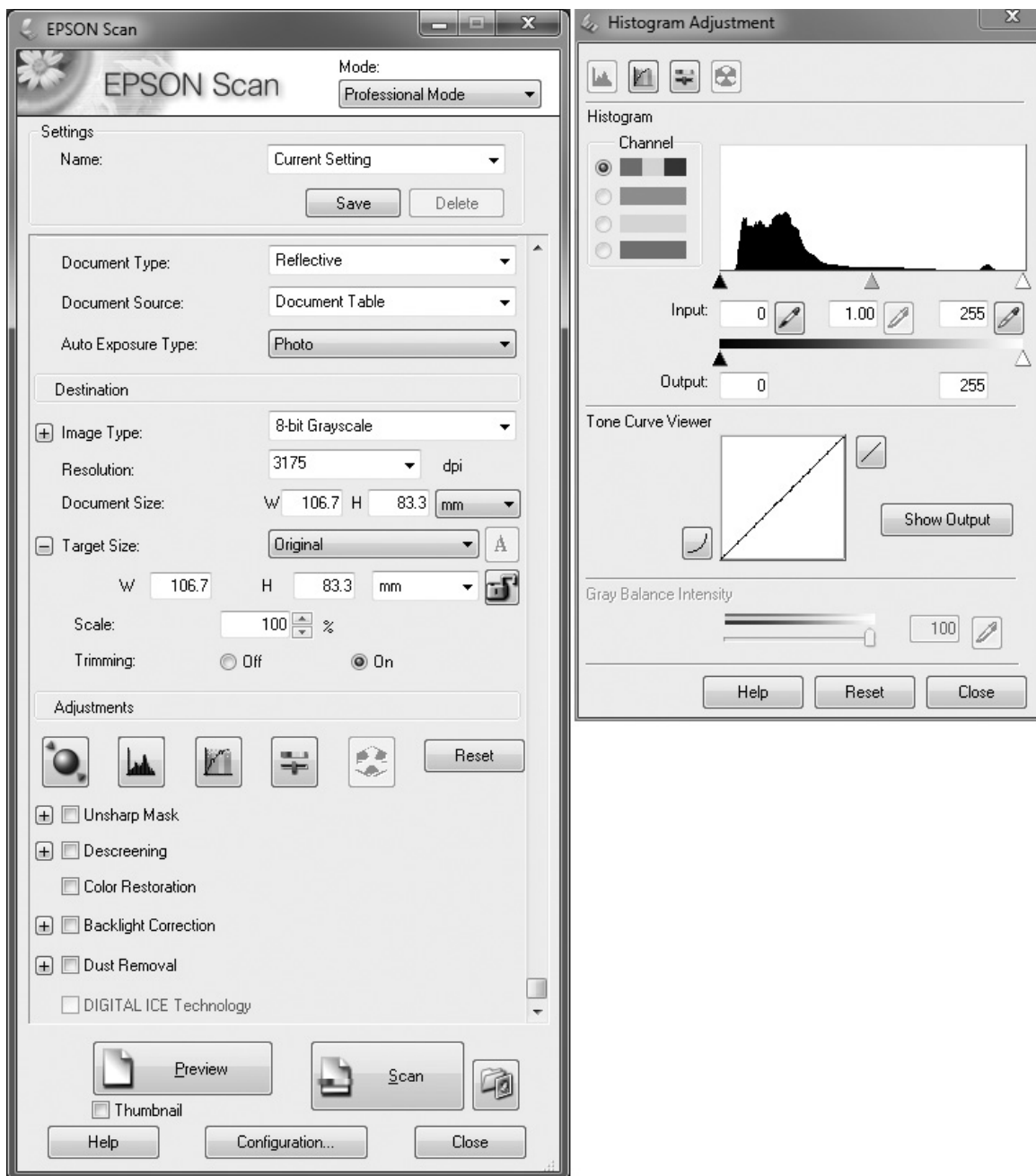
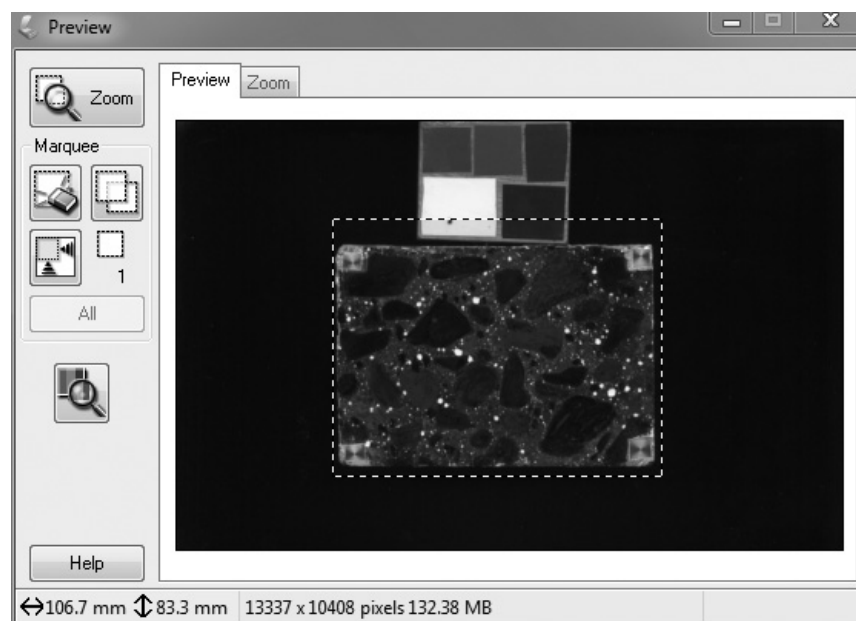Figure 20: Windows for scan collection settings.

Figure 21: Preview window with scanning area selected. Selected area should include the four corner stickers, and a portion of the black/white reference block.

9) Point-counting aggregate (20-30 minutes per segment-pair sample)

- Open a 24-bit RGB, 8 μm (3,175 dpi) resolution image in ImageJ. ImageJ can be downloaded at http://imagej.nih.gov/ij/. Use the rectangular area selection tool to select the area you wish to cover with the point count. If the BubbleCounter macro package has been installed for ImageJ, select *Plugins>BubbleCounter>pointcount* (if not installed, visit http://www.appropedia.org/BubbleCounter and follow instructions for download and installation). The area of interest will be divided up into approximately 500 frames, and presented on the screen frame by frame with an accompanying dialog box asking if the point is on an aggregate particle (use the "Y" or "N" keys to answer yes or no). The aggregate fraction (a number between 0 and 1) can be used later as an input in Section 10 for the subsequent determination of paste content.

10) Automated air-void analysis (2 minutes per segment-pair sample)

- Open an 8-bit grayscale 8 μm (3,175 dpi) resolution image in ImageJ. Use the rectangular area selection tool to select an area of interest that covers the black/white reference block (Figure 22). Select *Plugins>BubbleCounter>whitebalance*. The whitebalance macro records the modal values of the black and white reference materials, and stores the values in a new file with the same name as the image, but with "_bcProps.txt" appended to the filename. The new file is stored in the same folder as the original image file.

Figure 22: Selection of area for *whitebalance* macro.

- Use the rectangular area selection tool to select the area you wish to analyze (Figure 23). Select *Plugins>BubbleCounter>bubblecounter*. A new window will appear prompting you to provide:
  a. The number of traverses. This controls the total length traversed.
  b. Paste content determination method. You have three options. If you do not enough information to select an option, it is recommended you select a paste volume fraction of 0.3. Later on, if more detailed information is available, it is a simple matter to recalculate the spacing factor after the fact using a spreadsheet.
     o *Paste volume fraction*. Enter a paste fraction directly (a value between 0 and 1). Most concrete has a paste content in the range of 25-35%. Small variations in paste content (±5%) have a limited influence on the resultant value for spacing factor.
     o *Aggregate volume fraction*. Enter an aggregate fraction directly (a value between 0 and 1). This value may be determined by performing a manual point count on a 24-bit RGB 8 um (3,175 dpi) resolution image using the *pointcount* macro.
     o *Paste/aggregate volume ratio*. If the mix design information is known, and the specific gravities of the constituents are known, the paste/aggregate volume ratio can be calculated and entered here.
  c. Analysis Type. The BubbleCounter macro relies on a threshold value to make the distinction between air-void (brighter pixels) and non air-void (darker pixels). After the image is partitioned between air-void and non air-void, the total length of air-void

intercepts with the test line is divided by the total test line length (to get air content), and the total number of air-void intercepts is divided by the total test line length (to get void frequency). Due to the limitations of the resolution of the flatbed scanner, a relatively low threshold may be required to detect intercepts with the smallest of air-voids. But, at the same time, a lower threshold will also tend to overestimate the area of larger air-voids. For this reason, you have the option to set two separate threshold levels: one for the determination of air content (AC), and another for the determination of void frequency (VF).
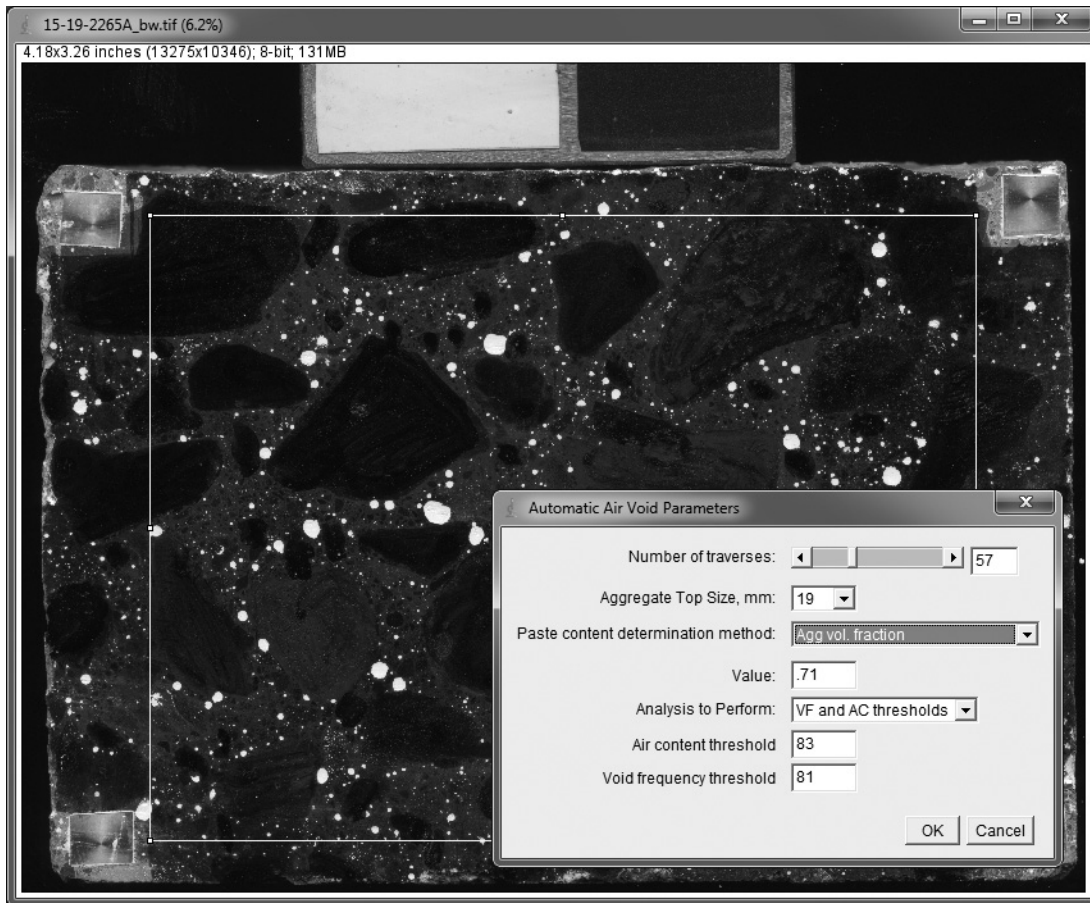
Figure 23: Selection of area for *bubblecounter* macro.

- After the selection of analysis options, the test lines are extracted, saved to a new file with "_bcTraverses.txt" appended to the original image filename, and the air-void parameter calculations are performed (Figure 24). The results are also written to the same "_bcProps.txt" file, and an abridged version is written to another file with "_bcResults.txt" appended to the original filename. All three files are stored in the same folder as the original image file.
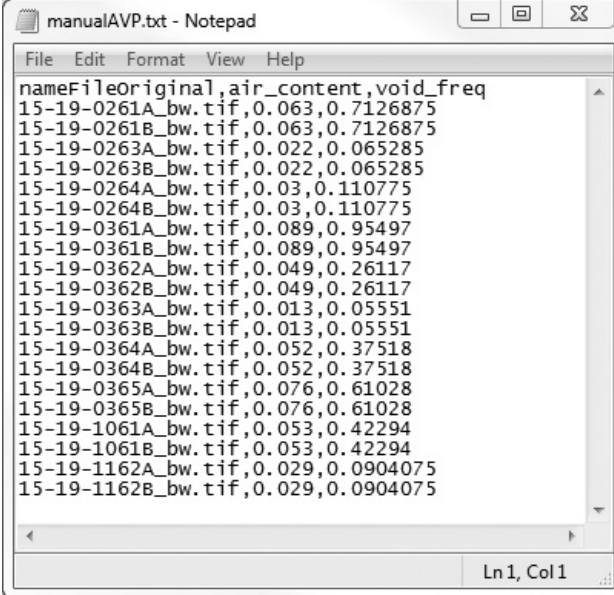
Figure 24: Results of analysis.

11) Determination of appropriate threshold level(s) and batch processing.

Two additional macros are provided with the BubbleCounter macro package: *optimize*, and *batch folder summary*.

- The *optimize* macro is used to help determine appropriate threshold levels based on a population of samples where manual ASTM C457 or LS-432 results are available. The same samples are prepared with contrast enhancement, scanned, and analyzed using the *bubblecounter* macro. The threshold levels used during the initial analyses are not important; just the "_bcTraverses.txt" test lines extracted from the original images are required in order to perform the threshold optimization calculations. A comma separated value (*.csv) text file is also required that contains the image filenames and the corresponding human-operator values for air content and void frequency. This file must be named "manualAVP.txt" and must be placed in the same directory as the scanned images. The format of the "manualAVP.txt file" is shown in Figure 25. Air content should be expressed as a fraction, and void frequency in units of voids/mm. The *optimize* macro finds the individual threshold levels for each image that yield the best agreement with the manual ASTM C457 or LS-432 test results. Based on this population of threshold levels, an informed decision can be made as to a fixed set of threshold levels (one for air content determination, and another for void frequency determination) for all subsequent analyses. The arithmetic mean of the threshold

population is the simplest approach, and has been shown to produce results for the flatbed scanner that are in general agreement with manual operator test results.

Figure 25: Example file with manual operator results (required for *optimization* macro).

- The *batch folder summary* macro is used to process all of the "_bcTraverse.txt" files contained in a directory. A single text file named "summaryResults.txt" is generated and contains all of the air-void parameters for all of the samples. More details regarding these two macros can be found at: http://www.appropedia.org/BubbleCounter.