

WHITEHOUSE SCIENTIFIC BCR 'MIRROR' STANDARDS
(Project MATI-CT92-0023)

Methodology Details for Electroformed Sieve Analysis - Dr H Mercus

Sieving procedure for certification of 150-650 μ m BCR – CRM's (Dry sieving)

1. Equipment

1.1. Sieves

- o Electroformed precision sieves
- o Aperture sizes: 180; 212 or 210; 250; 300; 355; 425; 500; and 600 μ m
- o Square apertures
- o Maximum tolerance on aperture size $\pm 2\mu$ m (acc. to ISO 3310-3 (1990))
- o Bottom container
- o Sieve diameter 70-200mm.

1.2. Others

- o Drying oven (70°C)
- o Calibrated balance
- o Certified calibration weight, having a mass about equal to that of a sieve
- o Microscope or macroscope with magnification factors in the range of 20 to 100, having an exit tube for a camera and X, Y scale divisions in ocular
- o Camera for making photographs of microscopic images
- o If available, image analyser to be used in sieve calibration
- o Certified line width standard, applicable in the size range 100 to 600 μ m (if available)
- o Certified NPL reference stage graticule, which will be circulated amongst the participants.

2. Procedure

2.1 Prior sieve calibration: (see also ISO 3310-3 (1990), section 4.4)

2.1.1 Set the total magnification of the microscope at about 100 times and calibrate the scale divisions of the ocular of the microscope or the image analyser against a certified line width standard. If such a standard is not available, use the 100, 200 and 400 μ m grids of the NPL reference stage graticule instead.

2.1.2 Measure the width and height (including the width of one side) of one 100 μ m, one 200 μ m and the 400 μ m square of the grid of the NPL reference stage graticule to the nearest 0.1 μ m (as a check for traceability) (squares and measurement method are indicated in attached figure).

2.1.3 Measure for each sieve the width (W_x) and height (W_y) of 5 apertures in each of 9 measurement fields to the nearest 0.1 μ m.

2.1.4 Calculate for each sieve from these data the mean value of the aperture size, the minimum and maximum aperture size, the standard deviation of the total population of measurements and the variation coefficient.

2.1.5 Set the total magnification of the microscope at about 25 times and make a photograph of the microscopic image of each sieve medium.

2.1.6 Determine the mass of each sieve and the bottom container to the nearest 0.5mg after - if necessary cleaning and - drying in an oven at 70°C until constant mass (i.e. difference in mass after 20min. prolonged drying is less than 3mg).

2.2 Sieving: apply dry sieving with the following steps:

2.2.1 Weigh the complete contents of a bottle of CRM sample (about 10g) to the nearest 0.5mg on a calibrated analytical balance.

2.2.2 Transfer the material to the top sieve of a stack containing the sieves mentioned under 1a) (upper sieve 600 μ m, following sieves in order of decreasing aperture size).

2.2.3 Sieve by means of a machine or by hand-sieving until relatively few particles are passing through the sieves; use the guidelines of ISO 2591-1 (1988) for the sieving technique.

2.2.4 Continue the sieving by hand-sieving of each individual sieve starting with the one with the largest apertures until less than 5mg/min passes through the sieves; collect the particles passing through the apertures quantitatively and add them to the residue on the sieve with the next smaller aperture size.

2.2.5 Weigh the residue on each sieve and the bottom container to the nearest 0.5mg on a calibrated analytical balance. **Note:** alternatively, transfer the residue of each sieve quantitatively to a tared lightweight container and determine its mass to the nearest 0.5mg.

2.2.6 Make (for one sample CRM) a photograph of the microscopic image of a representative portion of each sieve fraction at a total magnification of about 25 times (see also 2.1.4).

2.2.7 Make a mass balance on the sieving process by comparing the sum of the masses of all fractions (from 5) with the mass of the charge (from 1); the difference between the two values should be less than 0.5% of the charge (i.e. 50mg).

2.2.8 Make at least five repeat analyses per CRM (i.e. at least one analysis per bottle of CRM).

2.3. Reporting: report the results in the following way: (a floppy disk with the tables prepared in Quattro Pro will be provided):

2.3.1 Sieves: (see also Table 1)

- o Sieve markings, including indication of aperture shape
- o Frame diameter
- o Nominal aperture size
- o Calibration graticule used
- o Microscopic magnification factor applied
- o Measured width and height of one 100, one 200 and on 400µm square of the NPL graticule
- o All results of the microscopic measurements of the sieve apertures and their photographs (2.1).

2.3.2 Sieving: (see also Table 2)

- o CRM – code/type
- o Balance:
 - brand/type
 - nominal value of precision balance
 - certified and measured mass of calibration weight
- o Method of sieving: hand and/or machine
- o Brand of sieving machine and operating conditions
- o Duration of sieving
- o Table with all individual sieving results:
 - Nominal aperture size of sieve
 - Average microscopic aperture size of sieve
 - Mass of each sieve fraction (2.2.5)
 - The mass of the charge, the total mass of the fractions and the sieving loss (mass and percentage based on mass of charge)
 - Percentage of each sieve fraction with respect to total mass of fractions
 - Cumulative percentages undersize for each sieve size.

2.3.3 Photographs: for validation of sieves and sieving process, i.e. photographs of all sieves used and all fraction of the sieving process of one CRM sample from 2.1.5 and 2.1.6 respectively).

Note 1: Please provide all numerical data both in written form and on floppy disk using a spreadsheet program. (Quattro Pro, Lotus 123 or Microsoft Excel). A floppy disk with the tables set up in Quattro Pro will be provided.

Note 2: Please store fractions obtained and any left-over samples for eventual checking in case of problems.

Sieving procedure for certification of 10-100 μ m BCR-CRM's (dry sieving)

1. Equipment**1.1. Sieves**

- Electroformed precision sieves
- Aperture sizes: 20; 25; 32 (or 30); 36 (or 35); 45; 63 (or 65) and 90 μ m
- Square apertures
- Maximum tolerance on aperture size $\pm 2\mu$ m (acc. To ISO3310-3 (1990))
- Bottom container
- Sieve diameter 65-200mm.

1.2. Others

- Drying ovens (70°C)
- Appropriate sieving machine, type Sonic Sifter of alike
- Calibrated balance
- Certified calibration weight, having a mass about equal to that of a sieve
- Microscope with magnification factors in the range of 200-500, having an exit tube for a camera and X.Y scale divisions in ocular
- Camera for making photographs of microscopic images.
- If available, image analyser to be used in sieve calibration
- Certified line width standard, applicable in the size range 20 to 100 μ m (if available)
- Certified NPL reference stage graticule, which will be circulated amongst the participants.

2. Procedure**2.1. Prior sieve calibration: (see also ISO 3310-3 (1990), section 4.4)**

2.1.1 Set the magnification of the microscope at about 500 times.

2.1.2 Calibrate the scale divisions of the ocular of the microscope or the image analyser against a certified line width standard. If such a standard is not available, use the 25, 50 and 100 μ m grids of the NPL reference stage graticule instead.

2.1.3 Measure the width and height (including the width of one side) of one 25 μ m, one 50 μ m and one 100 μ m square of the grid and the horizontal and vertical diameter of the 48 μ m spot of the root-2 array of spots (squares and measurement method are indicated in attached figure).

2.1.4 Measure for each sieve the width (W_x) and height (W_y) of 5 apertures in each of 9 measurement fields to the nearest 0.1 μ m.

2.1.5 Calculate from these data the mean value of the aperture size, the minimum and maximum aperture size, the standard deviation and the variation coefficient of the total population of measurements.

2.1.6 Set the total magnification of the microscope at about 200 times and make a photograph of the microscopic image of each sieve medium.

2.1.7 Determine the mass of each sieve and the bottom container after-if necessary cleaning and-drying in an oven at 70°C until constant mass (i.e. difference in mass after 20min. prolonged drying less than 3mg).

2.2. Sample splitting: Divide, if necessary, the complete contents of a bottle of CRM into separate quantities of about 1-1.5 gram, by using a spinning riffler.

2.3. Sieving:

2.3.1 Weigh about 1-1.5 gram of CRM (see 2.2) to the nearest 0.5mg on a calibrated analytical balance.

2.3.2 Transfer the material to the top sieve of a stack containing the sieves and the bottom container mentioned under 1.1 (upper sieve 90 μ m, following sieves in order of decreasing aperture size).

2.3.3 Sieve by means of the sieving machine until the mass of the residues on each sieve change less than 1mg/min.

2.3.4 Weigh the residue on each sieve and in the bottom container to the nearest 0.1mg on a calibrated analytical balance. Note: alternatively, transfer the residue of each sieve quantitatively to a tared, lightweight container and establish its mass.

2.3.5 Make a mass balance on the sieving process by comparing the sum of the masses of all fractions (from 4) with the mass of the charge (from 1). The difference between the two values should be less than 2% of the charge (i.e. 30mg).

2.3.4 Make at least five repeat analyses per CRM (i.e. at least one analysis per bottle of CRM).

2.4. Reporting: report the results in the following way: (a floppy disk with the tables prepared in Quattro Pro will be provided):

2.4.1 Sieves: (see also Table 3)

- o Sieve markings, including indication of aperture shape
- o Frame diameter
- o Nominal aperture size
- o Calibration graticule used
- o Microscopic magnification factor applied
- o Measured width and height of tone 25 μ m, one 50 μ m and one 100 μ m square of the grid and the horizontal and vertical diameter of the 48 μ m spot of the NPL reference stage graticule
- o All results of the microscopic measurements of the sieve apertures and their photographs (2.1).

2.4.2 Sieving: (see also Table 4a)

- o **CRM** – code/type
 - Balance: · brand/type
 - nominal value of precision
 - certified mass of calibration weight
 - measured mass of calibration weight
- o Brand of sieving machine and operating conditions
- o Duration of sieving
- o Table with all individual sieving results:
 - Nominal aperture size of sieve
 - Average microscopic aperture size of sieve (from 2.1)
 - Mass of each sieve fraction (2.3.4)
 - The mass of the charge (2.3.1), the total mass of the fractions and the sieving loss (mass and percentage based on mass of charge)
 - Percentage of each sieve fraction with respect to total mass of fractions
 - Cumulative percentages undersize for each sieve size.

Note 1: Please provide all these data both in written form and on floppy disk using a spreadsheet program. (Quattro Pro, Lotus 123 or Microsoft Excel). A floppy disk with the tables set up in Quattro Pro will be provided.

Note 2: Please store fractions obtained and any left-over samples for eventual checking in case of problems.

H.M. (040); 4.10.94.

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Sieving procedure for certification of 10-100 μ m BCR-CRM's (wet sieving)

1. Equipment

1.1. Sieves

- o Electroformed precision sieves
- o Aperture sizes: 20; 25; 32 (or 30); 36 (or 35); 45; 63 (or 65) and 90 μ m
- o Square apertures

- Maximum tolerance on aperture size $\pm 2\mu\text{m}$ (acc. To ISO 3310-3 (1990))
- Membrane filter (2mm or less) and holder
- Sieve diameter 65-200mm.

1.2. Equipment for wet sieving

- Bottom pan with exit for liquid
- Sonication bath (about 40 W, 40kHz).

1.3. Others

- Clean water (demineralised or distilled) and dispersion liquid (composition to be specified), filtered through membrane filter)
- Drying oven (70°C)
- Spinning riffler
- Calibrated balance
- Certified calibration weight, having a mass about equal to that of a sieve
- Microscope with magnification factors in the range of 200 to 500, having an exit tube for a camera and X, Y divisions in ocular
- Camera for making photographs of microscopic images
- If available, image analyser to be used in sieve calibration
- Certified line width standard, applicable in the size range 20-100 μm (if available)
- Certified NPL reference stage graticule, which will be circulated amongst the participants.

2. Procedure

2.1 Prior sieve calibration: (see also ISO 3310-3 (1990), section 4.4)

2.1.1 set the total magnification of the microscope at about 500 times.

2.1.2 Calibrate the scale divisions of the ocular of the microscope or the image analyser against a certified line width standard. If such a standard is not available, use the 25, 50 and 100 μm grids of the NPL reference stage graticule instead.

2.1.3 Measure the width and height (including the width of one side) of one 25 μm , one 50 μm and one 100 μm square of the grid and the horizontal and vertical diameter of the 48 μm spot of the root-2 array of spots (squares and measurement method are indicated in attached figure).

2.1.4 Measure for each sieve the width (W_x) and height (W_y) of 5 apertures in each of 9 measurement fields to the nearest 0.1 μm .

2.1.5 Calculate from these data the mean value of the aperture size, the minimum and maximum aperture size, the standard deviation and the variation coefficient of the total population of measurements.

2.1.6 Set the total magnification of the microscope at about 200 times and make a photograph of the microscopic image of each sieve medium.

2.2. Sample splitting

Divide, if necessary, the complete contents of a bottle of CRM into separate quantities of about 0.5-1 gram (for ϕ 65 μm sieves), by using a spinning riffler.

2.3. Sample dispersion

2.3.1 Weigh about 0.5-1 gram of CRM to the nearest 0.5mg on a calibrated analytical balance.

2.3.2 Disperse it well in about 25cm³ (liquid and dispersion conditions to be specified).

2.4. Sieve instalment:

2.4.1 Wash the sieves and the membrane filter used for collection of the sub-20 μm fraction with clean water, dry them in an oven at 70°C until constant mass (i.e. difference in mass after 20 min. prolonged drying less than 3mg) and register this tared mass.

2.4.2 Combine one or more of the above sieves in order of decreasing aperture size with the bottom pan and a liquid exit tube.

2.4.3 Place the bottom part of the sieve stack in the Sonication bath.

2.4.4 Fill the sieve stack through the exit tube (i.e. in reverse direction) with clear dispersion liquid, avoiding air bubbles underneath the sieving medium.

2.4.5 Check that the Sonication is still effective at the upper sieve of the stack; if this is not the case, remove the lowest sieve from the stack and check again.

2.5. Sieving: apply wet sieving with the following steps:

2.5.1 Transfer the dispersed material (see 2.3) to the upper sieve of the installed sieve stack (see 2.4), starting with the sieve having the largest aperture of the ones mentioned under 1.

2.5.2 Start the sieving process by switching on the Sonication bath and selecting a flow of dispersion liquid through the sieve(s) of at least 50cm³/min; collect the exiting liquid from the bottom pan in a clean beaker.

2.5.3 Proceed with the sieving, while adding sufficient dispersion liquid to the top sieve in order to avoid drying up, until no more particles are passing through the sieves, resp. no more particles are contained in the exiting liquid from the bottom pan. Generally, a volume equal to about three times that of the stack is required. Note: it should be checked that there is sufficient movement of particles on all sieves by the Sonication and that none of the sieves is overloaded; in case of overloading the charge of sample should be decreased.

2.5.4 Finalise the sieving by letting all the liquid exit from the stack into the beaker (liquid is no longer added to the top sieve).

2.5.5 Collect the sub-20µm particles from the beaker on the membrane filter (2µm or less). Note: in the case that more than one sieve stack is applied, use liquid plus undersize particles from one stack as a feed for the one with the next smaller sized apertures; to that end, let the particles settle to the bottom, separate the clear upper liquid from the dispersion at the bottom, use the latter dispersion as feed and the former upper liquid as primary washing liquid; proceed as above 2.5.4, until all sieves have been used in the order of aperture size.

2.5.6 Wash the residues on the sieves and the membrane filter with clean water and dry them in an oven at 70°C until constant mass (i.e. difference in mass after 20 min. prolonged drying less than 3mg). If this is impossible, transfer the residue of the sieves with clean water to appropriate, tared filters and dry filters plus residue in an oven at 70°C until constant mass (again differences in mass at prolonged drying less than 3mg).

2.5.7 Weigh the residues to the nearest 0.1mg on a calibrated analytical balance.

2.5.8 Make a mass balance on the sieving process by comparing the sum of the masses of all fractions (from 7) with the mass of the charge (from 2.3.1); the difference between the two values should be less than 2% of the charge (i.e. 20mg).

2.5.9 Make at least five repeat analyses per CRM (i.e. at least one analysis per bottle of CRM).

2.6. Reporting: report the results in the following way: (a floppy disk with the tables prepared in Quattro Pro will be provided):

2.6.1 Sieves: (see also Table 3)

- o Sieve markings, including indication of aperture shape
- o Frame diameter
- o Nominal aperture size
- o Calibration graticule used
- o Microscopic magnification factor applied
- o Measured width and height of one 25µm, one 50µm and one 100µm square of the grid and the horizontal and vertical diameter of the 48µm spot of the NPL reference stage graticule
- o All results of the microscopic measurements of the sieve apertures (2.1).

2.6.2 Sieving: (see also Table 4.2)

- o CRM – code/type
- o Balance:
 - brand/type
 - nominal value of precision
 - certified mass of calibration weight
 - measured mass of calibration weight
- o Brand of sieving machine and operating conditions

- o Duration of sieving
- o Table with all individual sieving results:
 - Nominal aperture size of sieve
 - Average microscopic aperture size of sieve (from 2.1)
 - Mass of each sieve fraction (2.5.7)
 - The mass of the charge (2.3.1), the total mass of the fractions and the sieving loss (mass and percentage based on mass of charge)
 - Percentage of each sieve fraction with respect to total mass of fractions
 - Cumulative percentages undersize for each sieve size.

Note 1: Please provide all these data both in written form and on floppy disk using a spreadsheet program. (Quattro Pro, Lotus 123 or Microsoft Excel). A floppy disk with the tables set up in Quattro Pro will be provided.

Note 2: Please store fractions obtained and any left-over samples for eventual checking in case of problems.

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Overview NPL graticule

Appendix - Whitehouse Scientific Procedure

Electroformed Sieve Analysis

(i) Sieve Calibration

A high quality microscope with planar lenses of magnification for 1.3 to 60 is essential for this work. 75mm Gilson electroformed sieves were viewed with transmitted light and the image reversed so that the clear sieve apertures became black squares which were then analysed

When calibrating a sieve, the maximum magnification was used to optimise the accuracy of the measurement, even though only 4-6 holes per microscope frame could be analysed. Measurements were taken from at least 5 different fields across the sieve. The X and Y dimension was measured at the centre of each aperture and the sieve size as the minimum dimension as it is this dimension that prevents spherical beads from passing.

(ii) Sieve Shakers

An electromagnetic sieve shaker made made by Fritsch was used for the 150-650 micron reference standard. In the case of the 10-100 micron sample, the Fritsch A3 was also used down to 45 microns, while from 32 to 20 microns, the Gilsonic Autosiever was used. Excellent sample recovery was achieved in both cases; greater than 99.5%, while the reproducibility for the 5 samples was less than 1.5%.

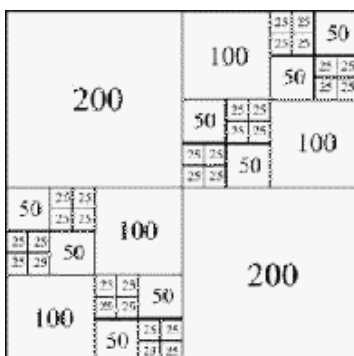


figure 1: Grid (400µm square)

To calibrate the image analyzer and check for non-squareness and optical distortion

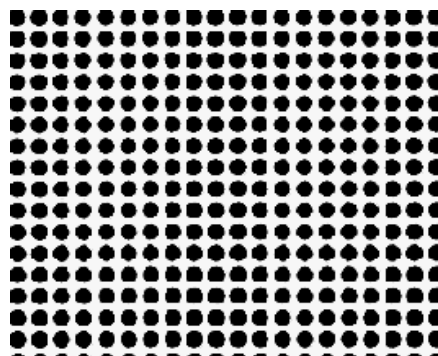


figure 2: Monosize Array (15µm spots)

To look for localized distortion and find the usable area of the screen for accurate measurements

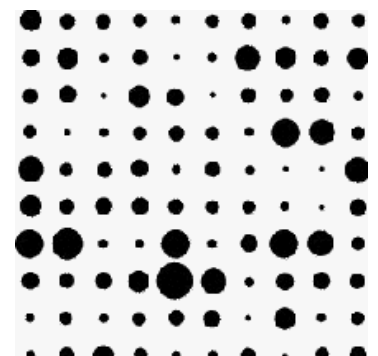


figure 4: Log-Normal Distribution (4.5µm-27µm)

To look for localized distortion and find the usable area of the screen for accurate measurements



figure 3: Root-2 Progression (3µm-48µm)

To measure the effect of grey level thresholds and evaluate edge location routines