Rapid Analysis of Bituminous Material

Lab Test Reference:
British Standard Ref
BS 598 : Part 102 : 1989

Principal Apparatus as follows:-

(i) Measuring Flasks 2000ml, 1000ml, 500ml and 250ml capacity with a current calibration certificate. (Grade B Specified)
On the Central Bench in the BTL. Class A
Inventory No's XXXX.

(ii) Metal Extraction Bottle of 2500ml, > 6000ml and 7000ml capacity.
Under Central Bench in BTL.

(iii) Rubber bungs to fit Bottles in good condition.
With Bottles.

(iv) Roller Bottle Apparatus with current calibration certificate.
Speed of between 10 and 30 rev/min.
Within Fume Cupboard No.2.
Inventory No. XXXX

(v) Calibrated Timing device for the roller bottles.
Fitted within the fume cupboard No.x. Inventory No. XXXX
Refrigerated Denly Centrifuge on bench near swing doors. Inventory No. xxx.

vii) Pressure Filter Apparatus. In Fume Cupboard No. x.

viii) No. 1 and No. 5 Whatman Filter Papers 270mm diameter with 30mm central hole.
Stored in cupboard beneath fume cupboard No.xx.

ix) Torque wrench for tightening down top of pressure filter onto base near pressure filter.

x) Wide Necked funnel for introducing sample into extraction bottles.

xi) Pressure Filter Funnel to take 200mm dia sieves with collar to sit on entry hole in top of pressure filter.

xii) Two 200mm diameter 75um sieve with calibration certificate.
Take from sieve store each day. (BS410) Inventory No. xxx

xiii) 2 sets 200mm diameter 2.36mm, 425, 150, 75um sieves without certificates to be used to protect the above sieves. (near pressure filter)

xiv) Clean air supply to pressure filter.

xv) Grade A 50ml burette with current calibration certificate.
In Fume Cupboard No. 4 Inventory No. Xxx

xvi) Burette Stand, Clamps and bosshead assembly.
In Fume Cupboard No. 4.

xvii) Screw topped conical 250ml capacity storage flasks.
In Fume Cupboard No.4. and in lower cupboard for spares.

xviii) Extraction flasks wide necked flat bottomed 250ml capacity.
In Fume Cupboard No. 5 and in lower cupboard for spares.

ix) Desiccators with fresh indicating silica gel crystals.

xx) Boiling off Apparatus with current vacuum gauge certificate.
In Fume Cupboard No. 6 Inventory No. xxx

xxi) Fume Cupboard for drying out sand and filler.
Fume Cupboard No. 7 + 8 infrared lamps.

xxii) Electronic Balance to weigh at least 12kg to 0.1 gram with current calibration certificate.The Electronic Balance is on the bench in the Bituminous Testing Area. Inventory No. xxx.

xxiii) Electronic Balance to weigh to four decimal places with current calibration certificate.
The balance on the Plinth. Inventory No. xxx

xxiv) The BS test sieves will be signed out from the sieve store on request and will be selected from the following list depending on the type of material being tested.
Inventory No. xxx (BS410)

Sieves to be used in Particle Size Analysis
Nominal Aperture Size

Square Hole Perf. Plate 300mm Diameter	Wire Cloth, 300 or 200mm Diameter
mm	mm
75.0	3.35
63.0	2.36
50.0	1.70
37.5	1.18
28.0	
20.0	um
14.0	600
10.0	425
6.3	300
5.0	212
	150
	75*

xxv) A Mechanical Sieve Shaker.
In the sound proof cupboard in the Bituminous Testing Laboratory. Inventory No. xxx

xxvi) Square Trays sufficiently large to completely contain the sample and their portions.
General purpose laboratory equipment in BTL.

xxvii) Stiff bristled sieve brushes. General purpose laboratory ware in BTL.

xxiii) Screw topped 50ml polypropylene centrifuge tubes. General purpose laboratory ware in BTL.

xxix) A 15mm Riffle Box for quartering down the sand fraction. On the side bench. Inventory No. xxx

xxx) Hot air dryer.

1. **Preliminaries**

1.0 The area designated as the Bituminous Laboratory will be used to perform this test. It must be ensured that the areas allotted to this test are tidy before testing proceeds.

1.1 Ensure that the Sample Number and the Test Schedule correspond.

1.2 Obtain a Test Worksheet No. 110/1/2/5.

1.3 Record the following details on the test sheet:-

i) Operatives name.

ii) Date of test.

iii) Laboratory sample no.

iv) Advice No.

v) Type of material.

vi) Assumed initial binder content.

vii) Any unusual features; smell, dry or fatty appearance etc.

viii) Standard or non-standard sample.

1.4 Ensure that all the equipment to be used in this test has the necessary calibration certificates and/or is clean and in good working order.

1.5 Ensure that the balances are reading accurately.

1.6 Check the sieves as required on receipt. If any dents, marks or splits are present the sieves shall be taken out of service.

1.7 The appropriate log will then be signed accepting the equipment is in a satisfactory condition before testing begins.

2. Standard Test Method

2.0 As a rule the sample to be tested will have been prepared in accordance with SP1 in the sample reception area and will be of the correct weight.

2.1 The white sample ticket will be removed from the bag and stapled on to the test work sheet. The blue copy remains in the retest sample bag.

Paragraphs 2.2 to 2.4 not used.

2.5 The first sub-sample will be weighed to the earnest 0.1 grams and the weight will be recorded on the work test sheet as (M)grams.

2.6 If for some reason the weights fall outside the following range for a specific material the sample will be returned to the reception area for further attention.

Insert Table of recommended weights

2.7 The sample will be introduced into an extraction bottle of the correct volume for the sample size through the wide necked funnel with care being taken not to loose any of the material.

2.8 The amount of solvent required will be calculated:-

Volume of Solvent (m1) = Mass of sample * Estimated binder content

To give a 3% solution.

2.9 The volume of solvent to be used will then be recorded on the work test sheet rounded to the nearest 250ml as (V)ml.

2.10 The solvent will be measured into the various volumetric flasks from the dispenser in fume cupboard No. 1 and carefully added to the extraction bottle containing the pre-weighed sample.

2.11 If for some reason it is suspected that the sample may contain moisture then 10 grams of pre-dried passing 75um silica gel will be added to the extraction bottle. Record the weight of the silica gel used on the work test sheet as (sg)grams.

2.12 Measure the temperature of the solution in the bottle and record as t, on the sheet.

2.13 A rubber bung of the correct size will be tightly inserted into the top of the extraction bottle.

2.14 The bottle and the contents will be placed horizontally on to a bottle roller apparatus in fume cupboard No.2, the timer will be set to the required time of 30 minute unless otherwise instructed, and the roller bottle set in motion. The timers are connected to the roller bottles and will switch them off automatically at the end of the period.

2.15 While the roller bottle is running set up the pressure filter in preparation.

2.16 Take a No. 1 and No. 5 filter paper and place them on to the pressure filter bed making sure that the pressure filter is clean and the gaskets are sound.

2.17 Place the pressure filter body over the central screw and clamp down using the preset torque wrench to give the final tightening.

2.18 Fit in the special filter funnel together with the certified 75um sieve on the bottom and the uncertified 75um and 2.36mm sieves on top.

2.19 Weigh two extraction flasks on the micro-balance to the 0.0001 grams and record their weights and numbers on the work test sheet.

2.20 When the timer has switched off the bottle roller remove the bottle and place upright on the bench. Leave for a few minutes to allow the filler to settle.

2.21 Remove the bung and decant the solution through the 2.36mm sieve into the pressure filter until the pressure filter is about ¾ full. If the pressure filter is not to be used then transfer 500ml of the solution directly to a conical flask and proceed to para 2.30.

2.22 Remove the funnel, placing the end into the current bottle, to redrain.

2.23 The following operations will then be performed:

i) Fit the hole clamp into place on the pressure filter.
ii) Close off both of the outlet tap.
iii) Prepare two Conical screw top flasks.
iv) Fit the air line on to the pressure filter.
v) Increase the pressure to about 100psi.
vi) Open the tap to the swan necked pipe and take two flasks full of solution.
vii) Close off the tap to the swan neck and open the tap that allows the rest of the solution to run to waste.
viii) Replace the screw caps onto the conical flask and place them into fume cupboard No. x.

2.24 After the solution in the pressure filter has been blown through, the hole clamp will be removed the filter funnel will be replaced and the rest of the solution will be passed through the pressure filter.

The above process will be repeated with the sample being washed clean with at least two measures of 1000ml of methylene chloride.

2.25 All of the sample in the bottle will be washed out onto the top 2.36mm sieve. The bottle will be allowed to dry in the fume cupboard and any remaining particles will be added to the sample tray before sieving.

2.26 Remove the top 2.36mm mesh sieve and place the material retained on an awaiting tray. Add the material retained on both of the 75um sieves. Place the tray beneath the infra-red lights in fume cupboard No. x.

2.27. When all of the solution has been blown through, the top of the pressure filter will be taken off, the filter papers removed, placed on a second tray which will then be placed under the infra-red lights in fume cupboard No. x

2.28 Tickets with the sample number will be placed with the aggregate samples to prevent error should more than one sample be tested at one time.

2.29 The fume cupboard front will be lowered and the sample left to dry.

2.30 Four clean centifuge tubes and their tops will be weighed individually to the nearest 1.0 gram. The weights will be recorded.

2.31 Solution will be poured from the conical storage flasks into each of the centrifuge tubes until about 50ml has been added.

2.32 Each of the flasks will be weighed to the nearest 1.0 gram and the amounts contained in the tubes will be adjusted until there is 50 grams +/-1.0 gram. The side of the tube may then be marked and this may be used as a datum for further tests.

2.33 The caps will then be screwed into place and the tube fitted into the centrifuge at diametrically opposite sides.

2.34 The timer on the centrifuge will then be set for 20 minutes and the speed control turned up to maximum.

2.35 After the sample has been centrifuged the tubes will be carefully removed and placed into the stand next to the burette.

2.36 The amount of aliquot to be taken may be calculated as follows:

Aliquot portion (ml) = Total volume of solvent (ml)*100

Mass of sample(g) *Estimated binder content%

The amount of aliquot to be taken will be recorded on the work test sheet.

2.37 The tap on the burette will be closed and just over 25ml will be added from one of the centrifuge tubes. A further 25ml or so will be added from a second tube to just above the 50ml mark on the burette. Run the excess solution from the burette down to the 50ml meniscus mark. One of the pre-weighed wide necked flasks will be placed beneath the burette and the pre-defined amount of solution will be run in. This amount is usually 34ml. Repeat the above with the second wide necked flask and the other centrifuge tubes. Record the temperature of the solution in the flask and record as t2. If the difference between t1 and t2 is greater than 3^{o}, a further proportion of solvent shall be re-centrifuged at a lower temperature.

2.38 The bungs on the boiling off apparatus will be fitted into the top of each of the two extraction flasks and the bodies of the flasks will be immersed to about half their depth into the boiling water.

2.39 While the solution is being distilled the flasks will be agitated with a rotary motion so that the binder is deposited in a thin layer on the sides of the flask.

2.40 A vacuum will be applied to the flasks during the initial boiling off of not less than 600 mbar until frothing just occurs then.

i) For bitumens with penetration >100 the pressure shall be further reduced to 180 to 220mbar in 1.5 minutes and maintained at this pressure for a further 3 to 4 minutes. (N.B. Penetration is stated on ticket).

ii) For cut-back bitumens with penetrations <100 the pressure shall be allowed to increase to approximately atmospheric pressure then be reduced to 550 to 600 mbars in 1.5 minutes and maintained at this pressure for a further 3 to 4 minutes.

2.41 The flask will then be removed from the water bath and the pressure allowed to increase to atmospheric.

2.42 The outside of the flasks will then be wiped dry and the bungs removed while the flasks are held upside down. Remove all traces of solvent by use of hand held driers pointing into the flask. Do no set these for hot air.

2.43 The flasks will then be placed into the desiccator and allowed to cool.

2.44 When completely cool the flasks will be weighed to 0.0001 gram on the micro-balance and the weights recorded on the work test sheet.

2.45 The weight of binder in the flasks will be calculated which should be between 0.75 and 1.25 grams with the difference between the two recoveries being less than 0.02 grams. If this is not the case then the test will be repeated on the second sample with the amounts of solvent and aliquot adjusted accordingly or on second aliquots if the flask differences are greater than 0.02 grams.

2.46 The average of the two weights will be taken and used to calculate the percentage binder content as follows:-

$$\text{Percentage Binder } S = \frac{10000\, zV}{vM(100-P)} \quad \frac{(1+z)}{(dv)}$$

M = total masss of sample (grams)
z = average mass of binder recovered from the two aliquotes (grams)
V = Volume of Solvent used (ml)
v = Volume of aliquotes taken
d =Relative density of binder (1.0 for bitumen and 1.15 for Tars)
P = Percentage Water Content.

2.47 When the mineral aggregate is dry the material retained on the 75 um sieve will be weighed and the weight recorded on the work test sheet as a check weight.

2.48 The filler passing the 75um sieve will also be weighed and the weight recorded on the work test sheet.

2.49 The material retinaed on the 75um sieve will then be placed in a nest of 300mm dia. Sieves specified for the material being tested.

2.50 After initial shaking in the nest of sieve each sieve will be removed and the sieve will be shaken over a tray to ensure that all the material has passed that sieve.

2.51 The weight retained on each sieve will be recorded on the works test sheet to the nearest 0.1 grams.

2.52 The material passing the 3.35mm sieve will weighed and the weight recorded on the work test sheet.

2.53 This material will then be riffled down and a portion of about 200 grams will be taken. The weight of the portion will be recorded on the work test sheet to the nearest 0.1 grams. Take care to ensure the fine sieves are not overloaded.

2.54 This portion will then be placed into a nest of 200mm sieves specified for the material being tested.

2.55 The lid of the nest will be fitted and the nest of sieves will be placed on the sieve shaker for 10 minutes.

2.56 Each sieve will then be removed from the nest in turn and shaken over a tray to ensure all the material has passed.

2.57 The material retained on each of the sieves will be weighed and the weights will be recorded to the nearest 0.1 on he work test sheet.

3 Calculations

3.1 The percentage Binder content is calculated as above.

3.2 The weight of material passing the 75 um sieve will be calculated by the following method:

Weight of passing 75um = Weight on filter paper - weight of filter papers - weight of silica gel + weight of material passing the 75 um sieve from the sieve analysis.

3.3 The grading of the mineral aggregate is the percentage passing each sieve calculated on the total mass of mineral aggregate.

Mass of Mineral Aggregate = Mass of Sample - Mass of Binder in Sample

Actual mass passing 200mm dia sieves = recorded mass*riffle factor

Where, Riffle factor = $\frac{\text{Mass passing 3.35mm sieve}}{\text{Mass after riffling}}$

3.4 Percentage retained each sieve = $\frac{\text{Mass retained on sieve}}{\text{Total mass of mineral aggregate}}$ *100%

Percentage passing each sieve = $\frac{\text{\% passing previous sieve}}{\text{less \% retained present sieve}}$

3.5 As a rule the above calculations are performed on the computer work station where the results of the tests are also recorded into a database.

4. Reporting Results

4.1 As a rule the report is generated by the computer work station where the following facts are recorded:

i) The Laboratory Test Number.

ii) The Site Advice Number.

iii) The Client Name.

iv) The Scheme Name.

v) The Site Location.

vi) The Manufacture or supplier of the Product.

vii) The Plant or Quarry from which the material was supplied.

viii) The date received, sample description and test method.

ix) The date tested.

x) The binder content to the nearest 0.1%.

xi) The grading of the aggregate coarse to the nearest 1.0% fine to the nearest 0.1%.

xii) Filler content where required to the nearest 0.1%.

xiii) Passing 2.36 retained 600um when required to the nearest 0.1%.

xiv) Traffic category where applicable.

xv) Anything unusual about the sample.

xvi) Compliance with the specified requirement.

xvii) Date Reported.

Xviii) Signature.

Xviv) Unique Report No. and Name and Address of Laboratory

xvv) Sampling Certificate.