METHOD OF TEST FOR THEORETICAL MAXIMUM RELATIVE DENSITY OF BITUMINOUS PAVING MIXTURES

1. SCOPE

1.1 This method covers the determination of the theoretical maximum relative density of bituminous paving mixtures containing aggregates of low-absorption, as well as mixtures containing porous aggregates. Included are mixtures prepared for mix design purposes, uncompacted paving mixtures, and samples prepared from compacted pavement such as cores.

1.2 This method uses mercury manometers. Mercury has been designated as a hazardous material. Caution should be taken when handling mercury or mercury containing products. Refer to the Material Safety Data Sheet or the manufacturer's recommendations regarding the proper use of this apparatus.

2. RELEVANT DOCUMENTS

2.1	ASTM D 2041	Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures
2.2	Asphalt Institute MS-2	Mix Design Methods for Asphalt Concrete and Other Hot Mix Types

3. APPARATUS

3.1 BEAKERS OR VESSELS: Stainless steel vessel, 4000 ml capacity, no spout.

3.2 BALANCE: 4000 g capacity with an accuracy of ± 0.1 g.

3.3 RIFFLE SPLITTER: For splitting hot mix samples. Recommended width of the individual chutes is approximately 38 mm for all types of paving mix.

3.4 WIRE BASKET: Made from copper, stainless steel, or brass wire mesh, 160 ± 5 mm diameter x 180 ± 5 mm high to contain sample and beaker when weighing in water.

3.5 WIRE: Thin as possible to suspend the basket from the balance. The use of fishing line leaders has been found to be satisfactory.

3.6 VACUUM CELL: Stainless steel beaker or vessel fitted with a plexiglass top.

3.7 PUMP: Vacuum - capable of maintaining a residual pressure in the vacuum cell of at least 30 mm of Hg.

3.8 FILTERING FLASKS: Three 1000 ml Erlenmeyer filtering flasks with rubber stoppers.

3.9 RESIDUAL PRESSURE MANOMETER: To be connected directly to the vacuum vessel and which should be capable of measuring residual pressure below 30 mm of Hg (see Note 1).

Note 1: The use of a non-mercury device for the purpose of measuring the residual pressure is permitted if its accuracy is certified by the manufacturer or verified through the use of a traceable standard.

3.10 MANOMETER OR VACUUM GAUGE: Suitable for measuring the vacuum being applied in close proximity of the pump. This is required to check the reading given by the residual pressure manometer.

3.11 MANIFOLD: Standard 19 mm (0.75 in) I.D. galvanized water pipe fitted with up to 4 quick action valves and a bleeder valve.

3.12 BLEEDER VALVE: Needle valve to control flow of air into the system for pressure control.

3.13 SILICA GEL: Tell tale (3 mm – 1mm size).

3.14 TUBING: Rubber, vacuum, minimum 6.4 mm (0.25 in) I.D.

3.15 TROWEL: Metal, garden type.

3.16 CONSTANT TEMPERATURE WATER BATH: Capable of maintaining a temperature of 25 ± 1°C.

Note 2: Residual pressure in the vacuum vessel, measured in millimetres of mercury, is the difference in the height of mercury in the Torricellian leg of the manometer and the height of mercury in the other leg of the manometer that is attached to the vacuum vessel.

Note 3: One residual manometer may serve two vacuum vessels through the use of a two position three port valve (show in Figure 3 so that the residual pressure in each vessel may be measured alternately and independently of the other during the test).

Note 4: The Torricellian vacuum leg of the manometer occasionally acquires one or more bubbles of air that introduce error into the residual pressure reading. By the addition of the vacuum gauge this error can often be quickly detected by the differences between two vacuum measurements. Other potential problems such as partial restriction of the vacuum line in the silica gel can be detected in this way.

Note 5: If all 4 quick action valves are to be used at the same time the bleeder valve or an additional valve which opens to atmosphere may be used as a vacuum breaker. The latter is preferred because it minimizes the time required to adjust the needle valve for successive tests.

Note 6: The foregoing does not include apparatus required for the preparation of samples (both field and mix design). Reference can be made to LS-261 if this is required.

4. SAMPLE SIZE

4.1 For mix design work and for loose field samples, the size of the test sample shall be governed by the 'Designated Large Sieve' size/mix type and shall conform to the requirements as shown in the following table:

Size of Test Sample

Mix Type	Designated Large Sieve Size (mm)	Minimum Mass of Sample (kg)
HL 2	2.36	0.5
SMA 9.5, Superpave 9.5	4.75	1.0
HL 1, HL 3, HL 3A, DFC, OFC, SMA 12.5, Superpave 12.5, 2.5FC 1, 12.5FC 2	9.5	1.5
HL 4 (Binder and Surface)	13.2	1.5
Superpave 19.0, SMA 19.0	12.5	2.0
HL 8, MDBC, HDBC	16.0	2.0
Superpave 25.0	19.0	3.0
Superpave 37.5	25.0	4.0

4.2 The Designated Large Sieve is a sieve size specifically designated for testing purposes.

5. PREPARATION OF MIX DESIGN SAMPLES

5.1 The following assumes that the desired blend of aggregates has been determined, and a weigh card prepared. Also, the aggregates have been dried, and split into sieve fractions.

5.2 Arrange the individually sized coarse aggregate(s) and fine aggregate(s) in bags or in suitable containers on a bench, in order of decreasing sieve sizes. The pass 2.36 mm from the coarse aggregate(s) and from the fine aggregate(s) shall be placed in separate flat-bottomed pans of appropriate size. Using the flat-bottom scoop, weigh up, into separate pans for each test specimen, a sufficient quantity of oven-dried sample of the selected aggregates, so that the total mass will yield a sample of the specified size. Place the aggregates in an oven maintained at a temperature not exceeding 28°C above the mixing temperature specified by the proprietor of the asphalt cement product. Heating time shall be a minimum of 16 hours, normally overnight.

5.3 Preheat the mixing bowl in the same oven as the aggregate.

5.4 Preheat the metal trowel and spatula on the hot plates or in an oven.

5.5 Heat the asphalt cement to the mixing temperature specified by the proprietor of the asphalt cement using either an oven or hot plate. The asphalt cement should not be held at the mixing temperatures for more than 1 hour prior to its use. Suitable shields, baffle plates, or sand pads shall be used on the surface of the hot plate to prevent localized overheating.

5.6 When the asphalt cement has reached the desired temperature, remove the container of aggregate from the oven, and quickly place it in the mixing bowl that has been previously tared on the balance. If the aggregate mass is not within ±1 g of the originally batched mass prior to overnight heating, adjust the batch by the addition of preheated pass 2.36 mm material from the primary fine aggregate. Using the mixer, dry mix the aggregate for 15 seconds.

5.7 After dry mixing, form a crater in the centre of the aggregate in the bowl, quickly add in the calculated amount of asphalt cement to give the desired asphalt cement content, and mix for 45 seconds or until the aggregate is coated. Do not mix longer than 1.5 minutes. Report any mixing problem such as coating of aggregate or balling of mix.

5.8 At the end of the wet mixing cycle, using the pre-heated spatula, scrape as much of the mix as possible from the mixing paddle and scraper blade.

5.9 Spread the mixture into the metal trough using a back and forth motion. Scrape all of the loose mixture in the bowl into a flat pan.

5.10 Place the mixture into a metal pan of sufficient size to yield a spread rate of 20-22 kg/m^2, cover with aluminum foil or other suitable cover, and place in an oven maintained at 135°C for two hours. Stir the mix occasionally during this period. For the design of Superpave mixes, the mixture shall not be covered with foil and the conditioning shall be in an oven maintained at the compaction temperature (see Note 7).

5.11 Following this conditioning period, allow the mix to cool to room temperature. During the cool down period, separate the particles by hand, taking care to avoid fracturing the aggregates, so that the particles of the fine aggregate portion are not larger than 6.3 mm.

Note 7: The mixing and compaction temperatures are typically supplied by suppliers or proprietors of the asphalt cement. See Figure 1 for details.

Note 8: While the determination of maximum theoretical relative density can be done for any asphalt cement content, the precision of the test is best when the mixture is close to the design asphalt content. Also, the test shall be done in duplicate or triplicate.

6. PREPARATION FROM LOOSE FIELD SAMPLES

6.1 Obtain loose field samples in accordance with specified procedures. Plate or loose mix samples shall be reduced to the proper size (See ASTM D2041, item 7.2). Two methods of reducing the field sample to the testing size are acceptable: quartering or splitting using a riffle splitter. Testing shall be done in duplicate.

Note 9: When the test sample is greater than 3000 g, each sample may be split into two halves placed in two separate beakers and vacuum cells. Results from the two halves of the sample shall be combined and recorded as one.

6.1.1 Quartering: Warm the field sample to achieve sufficient workability for quartering. A conventional oven maintained at 110°C or a microwave oven (see Note 8) may be used for this purpose. The surface upon which the sample is to be reduced should be flat, non-abrasive, non-absorptive and of sufficient area to provide for uniform quartering. Heat lamps may be used to keep the surface warm (see Note 9). The sample shall be mixed on the surface until uniform, then quartered, and opposite quarters removed. This process is to be repeated until the testing size is obtained.

Note 10: Caution: Frequent mixing may be necessary to prevent localized overheating when using a microwave oven to heat paving mixtures. Also, the presence of metal particles in some mixtures may render the microwave oven unsafe for the heating of these mixtures. Mixes made with polymer modified asphalt cement will have to be heated to a temperature higher than 110°C to achieve sufficient workability. The actual temperature should be the minimum required to achieve this and should be determined by trial and error.

Note 11: If required, 'Pam' or equivalent used in minimal quantities has been found suitable in preventing the sample from adhering to the surface. Motor oils must not be used for this purpose.

6.1.2 Riffle Splitter: Warm the field sample to achieve sufficient workability for splitting. A conventional oven maintained at 110°C or a microwave oven (see Note 8 above) may be used for this purpose. The mix may agglomerate if it is too cold or stick to the splitter if too hot. A temperature range of 90°C to 110°C has been found to be generally satisfactory. Heat lamps may be used to keep the walls of the riffle splitter warm (see Note 10 above). Chutes shall be cleaned after each split. The use of a putty knife or a 25 mm diameter wire brush used to clean glassware has been found to be suitable.

6.2 Spread the mixture out on a rectangular pan, dry to constant mass in an oven maintained at a temperature of 110 ± 5°C and allow to cool to room temperature. Constant mass is defined as no change in mass in excess of 0.1% for each 30 minute drying period. During the cool down period, separate the particles by hand, taking care to avoid fracturing the aggregates, so that the particles of the fine aggregate portion are not larger than 6.3 mm.

7. PREPARATION FROM COMPACTED PAVEMENT SAMPLES

7.1 Dry the entire core or sawn sample to a constant mass by placing the pan containing the material in an oven maintained at a temperature of 110 ± 5°C. Constant mass is defined as no change in mass in excess of 0.1% for each 30 minute drying period.

Note 12: Pavement samples used in this test may also require the determination of their bulk relative density. Such samples may contain residual moisture from the sawing/coring operation. These samples should have their mass in water and their surface dry mass determined prior to drying (see LS-262).

7.2 After the core or sawn sample has been dried to constant mass, trim to a depth approximately equivalent to the "Designated Large Sieve" size of the particles to remove the particles which were cut during the coring/sawing operation.

7.3 Allow the material to cool down to room temperature. During this cool down period, separate the particles by hand, taking care to avoid fracturing the aggregates, so that the particles of the fine aggregate portion are not larger than 6.3 mm.

Note 13: If, for a mix with a "Designated Large Sieve" size of 16 mm, the mass of the pavement cored/sawn sample after trimming is less than 1000 g, the MRD shall not be determined. For cores, *this would represent a sample of less than 25.4 mm (1") thickness and would not be considered typical.*

8. PROCEDURE

8.1 Set up the equipment for this test as shown in Figures 2 or 3. Transfer the loose mixture into a vacuum cell which has been previously calibrated for its mass in air and water. Add sufficient water at approximately 25°C to completely cover the sample.

8.2 Place the plexiglass top on the vessel ensuring a tight seal, and gradually reduce the pressure to 30 ± 1 mm Hg. Maintain this pressure for 15 minutes with periodic (or continuous) agitation to ensure that all the air has been removed. At the end of the specified time, release the vacuum slowly.

8.3 Suspend the vessel and contents in the water bath and determine the mass after 10 ± 1 minute immersion. Record the temperature of the water in the water bath if different from 25°C.

Note 14: *For testing multiple samples, the vessels and their contents may be submerged in a water bath at about 25°C, so that the total time of immersion (in the water bath and during subsequent weighing) is 10 ± 1 minutes.*

8.4 For porous aggregates or aggregates that are not thoroughly sealed by a bituminous film, refer to ASTM D2041-95, Section 11 to conduct a supplemental procedure.

9. CALCULATION

9.1 Calculate the theoretical maximum relative density (MRD) as follows:

$$\text{MRD} = \frac{(D - E)}{(D - E) - (G - H)}$$

where:

D = mass of beaker and mixture in air, g

E = mass of beaker in air, g

G = mass of beaker and mixture in water at test temperature, g

H = mass of beaker in water at test temperature, g

9.2 If the temperature varies from 25°C, a correction to the MRD will be made in accordance with the following:

MRD (@ 25°C) = MRD (at test temperature) x K

where: K = correction factor listed in Table 1 for the test temperature

10. REPORTING OF RESULTS

10.1 All results are entered on a Bituminous Mix Form Sheet (Figure 4).

10.2 For duplicate testing, the results are considered acceptable if their range is less than 0.011.

11. GENERAL NOTES AND PRECAUTIONS

11.1 Most aggregates will absorb water if extensive asphalt stripping occurs while the test sample is under vacuum.

11.2 If the vacuum pump cannot produce the residual pressure required (30 ± 1 mm Hg), check the pressure at the vacuum pump. If this pressure is less than 30 mm Hg and the pressure at the vacuum vessel is not, check for blockage of the system in the silica gel tube or flask. If pressure at the pump is higher than the 30 mm Hg check the cell lids, valves and all tubing and connections for air leaks. Use heavy vacuum grease and/or hose clamps to seal connections. Do NOT use grease on the lid seals.

11.3 Check the oil level in the vacuum pump regularly. If water is present, drain the old oil, remove all moisture, and add clean oil.

11.4 Dry silica gel has a deep blue colour. When the silica gel is pink in colour, it is wet, and should be removed from the flasks and dried in an oven overnight so that it can be re-used.

11.5 When determining the mass of the sample in water, the basket and beaker must be completely immersed.

11.6 After vacuuming, care must be taken to avoid exposing the mixture to air before the mass in water is measured.

11.7 When determining the mass in water, check that no air bubbles are adhering to the wire basket or beaker (particularly under the lip).

11.8 It is not necessary for the water to boil continuously to ensure that all the air has been removed. Table 2 shows that water at 25°C will not boil unless the residual pressure is 23.756 mm Hg or lower.

11.9 Increasing the temperature of the water in the vacuum cell above 30°C will give rise to higher boiling rates, which will adversely affect the ability to maintain the system at the required absolute pressure.

11.10 The water in the vessel should be maintained as close to 25°C as possible. Where more than one vessel is being used, vessel pressures will show slightly different pressures unless the temperature of the water is approximately the same in each vessel.

Determination of the Laboratory Mixing and Compaction Temperatures of Hot Mix for Design of Pavement Mixtures

The viscosity temperature relationship of an asphalt cement is used as a guide for establishing the mixing and compaction temperature in the laboratory. The temperature to which the asphalt cement must be heated to produce a viscosity of 0.17 ± 0.02 Pa.s is the mixing temperature. The temperature to which the asphalt cement must be heated to produce a viscosity of 0.28 ± 0.03 Pa.s is the compaction temperature. This has been the guide for Marshall mix design criteria and the relationship continues to be valid for laboratory purposes within Superpave. In Ontario, the mixing and compaction temperature are the temperatures where the asphalt cement has viscosities of 0.17 and 0.28 Pa.s respectively.

The Brookfield Viscometer, a rotational device (ASTM D4402), is used to determine the mixing, compaction, and pumping temperatures in the Superpave system. Viscosity measurements at 135°C and 165°C are plotted on a log viscosity vs. temperature chart as shown below and the mixing and compaction temperature interpolated.

The procedure for establishing the mixing and compaction temperatures for the laboratory is valid for refinery produced asphalt cements and may not be valid for some polymer modified asphalt cements. The supplier of these asphalt cements must be consulted for their mixing and compaction temperature recommendations.

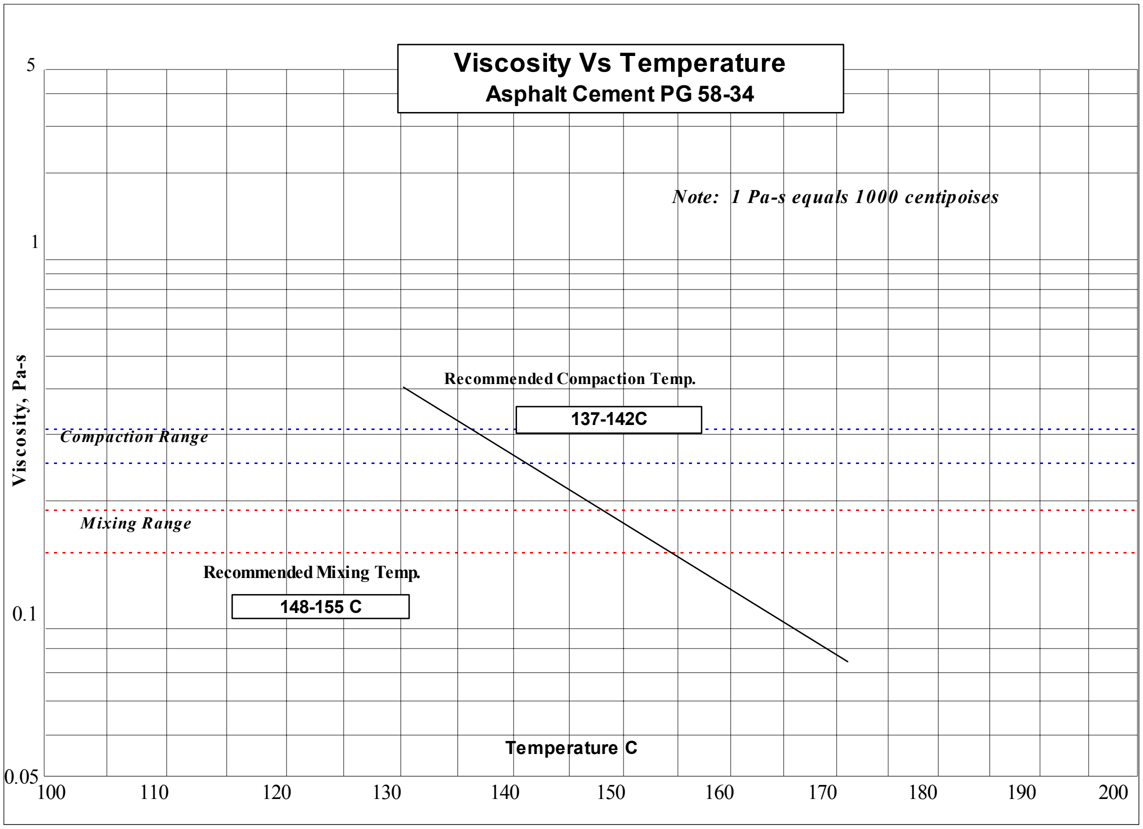

Figure 1
Sample Temperature Viscosity Chart

The relationship between temperature and viscosity in this figure shows the idealized laboratory mixing and compaction ranges. The asphalt cement is a “super lubricant” at the mixing temperature and a “super glue” somewhere around the compaction temperature range.

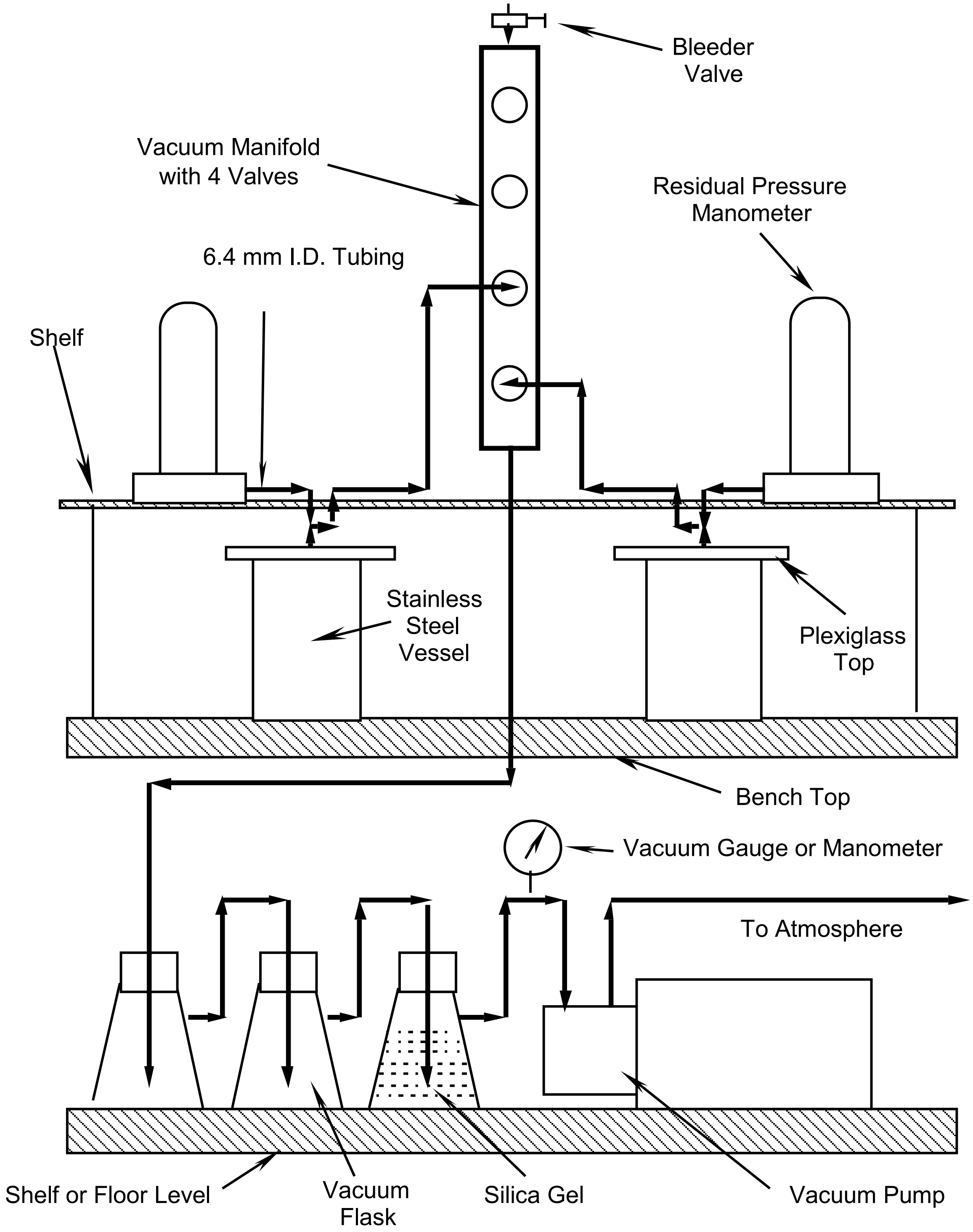

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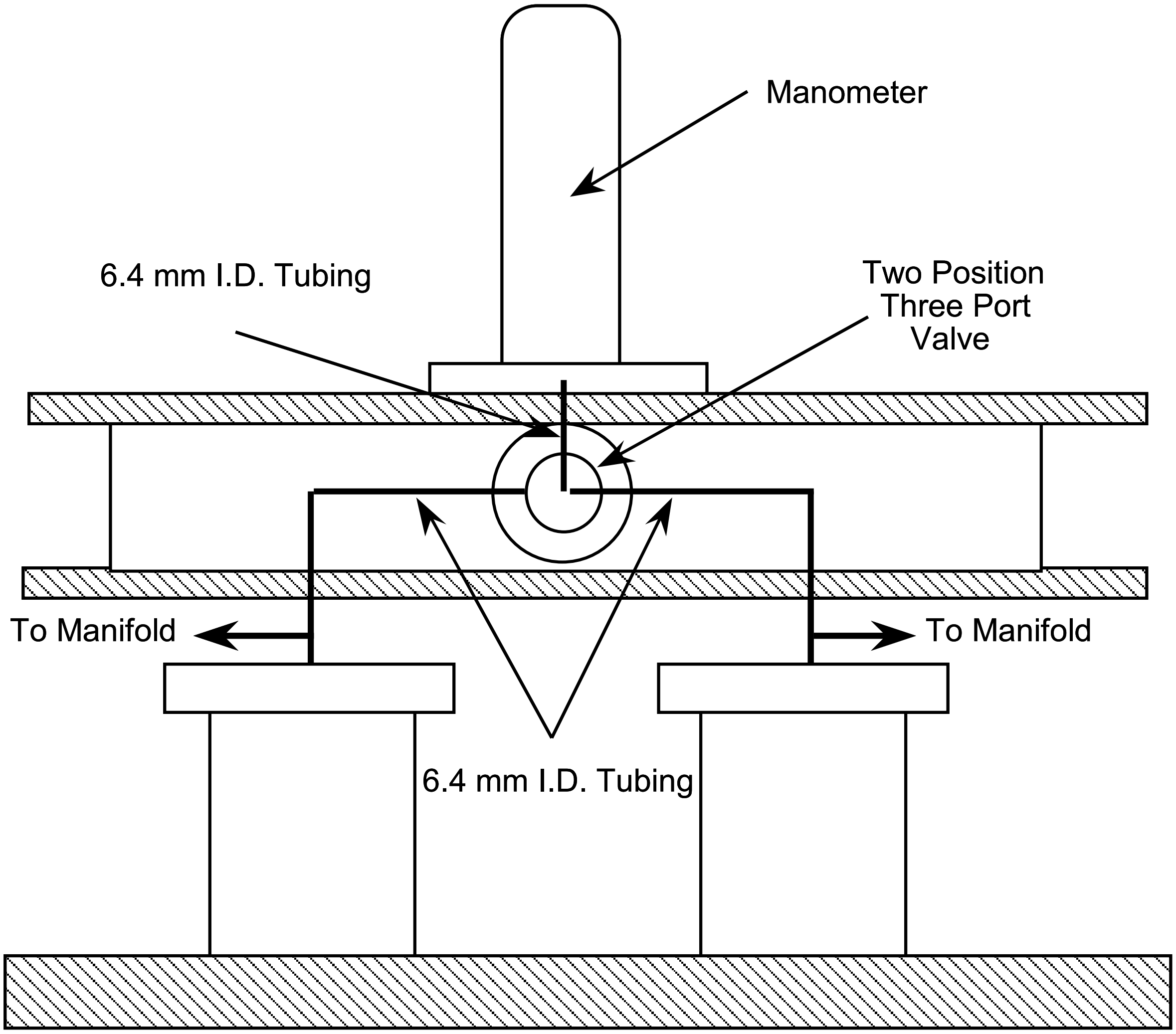
Figure 2
Vacuum Saturation System

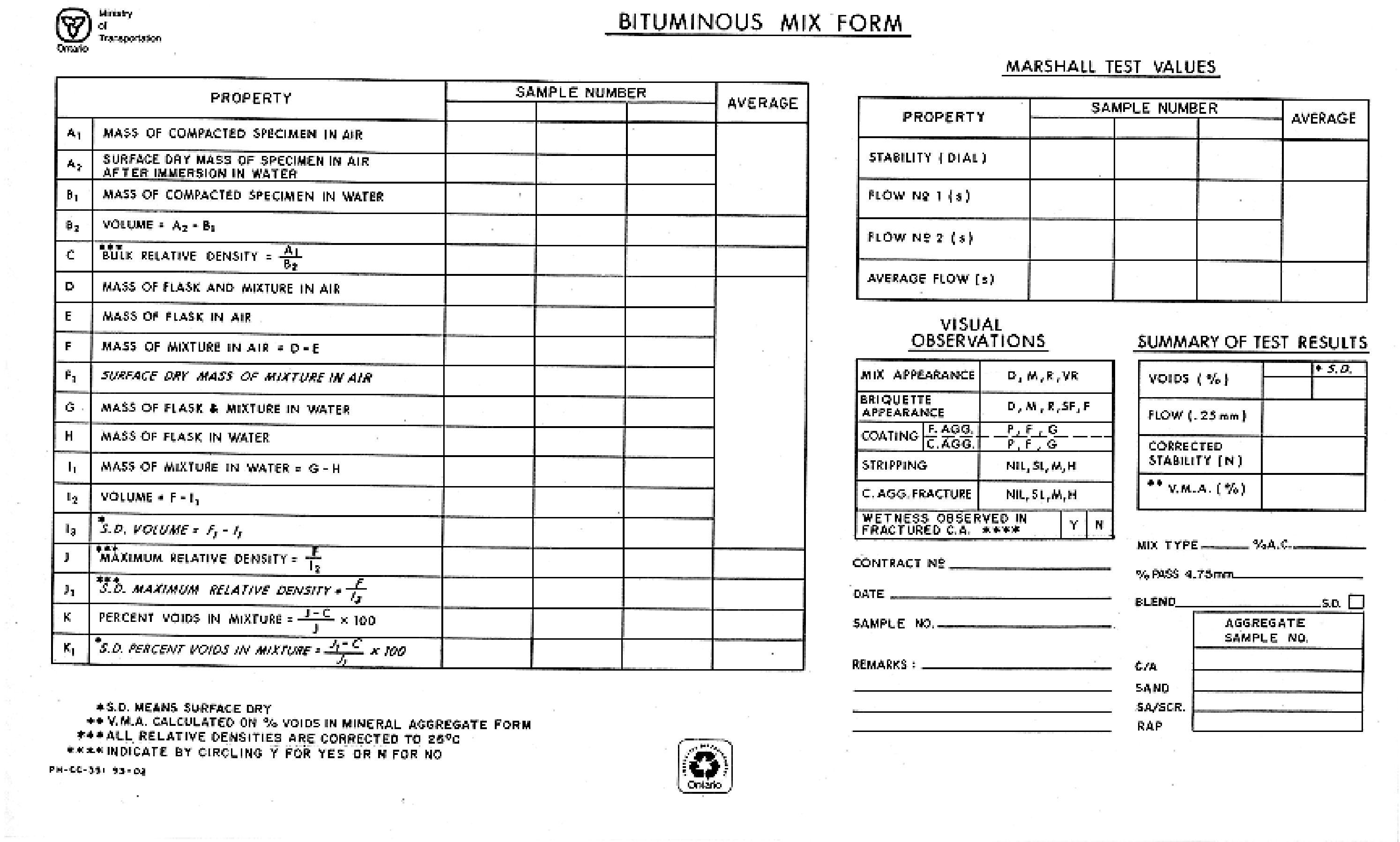


MRD equipment using one manometer for two vessels

Note: Tubing between the manometer and the vessels must be kept as short as possible to enable accurate pressure readings.

Figure 3
Vacuum Saturation System - Alternative Setup

Figure 4



Ministry of Transportation Ontario

BITUMINOUS MIX FORM

	PROPERTY	SAMPLE NUMBER			AVERAGE
A_1	MASS OF COMPACTED SPECIMEN IN AIR				
A_2	SURFACE DRY MASS OF SPECIMEN IN AIR AFTER IMMERSION IN WATER				
B_1	MASS OF COMPACTED SPECIMEN IN WATER				
B_2	VOLUME = $A_2 - B_1$				
C	*** BULK RELATIVE DENSITY = $\frac{A_1}{B_2}$				
D	MASS OF FLASK AND MIXTURE IN AIR				
E	MASS OF FLASK IN AIR				
F	MASS OF MIXTURE IN AIR = D − E				
F_1	*SURFACE DRY MASS OF MIXTURE IN AIR*				
G	MASS OF FLASK & MIXTURE IN WATER				
H	MASS OF FLASK IN WATER				
I_1	MASS OF MIXTURE IN WATER = G − H				
I_2	VOLUME = $F - I_1$				
I_3	* *S.D. VOLUME* = $F_1 - I_1$				
J	*** MAXIMUM RELATIVE DENSITY = $\frac{F}{I_2}$				
J_1	*** *S.D. MAXIMUM RELATIVE DENSITY* = $\frac{F}{I_3}$				
K	PERCENT VOIDS IN MIXTURE = $\frac{J-C}{J} \times 100$				
K_1	* *S.D. PERCENT VOIDS IN MIXTURE* = $\frac{J_1-C}{J_1} \times 100$				

*S.D. MEANS SURFACE DRY
** V.M.A. CALCULATED ON % VOIDS IN MINERAL AGGREGATE FORM
*** ALL RELATIVE DENSITIES ARE CORRECTED TO 25°C
**** INDICATE BY CIRCLING Y FOR YES OR N FOR NO

PH-CC-351 93-02

MARSHALL TEST VALUES

PROPERTY	SAMPLE NUMBER			AVERAGE
STABILITY (DIAL)				
FLOW № 1 (s)				
FLOW № 2 (s)				
AVERAGE FLOW (s)				

VISUAL OBSERVATIONS

MIX APPEARANCE		D, M, R, VR
BRIQUETTE APPEARANCE		D, M, R, SF, F
COATING	F. AGG.	P, F, G
	C. AGG.	P, F, G
STRIPPING		NIL, SL, M, H
C. AGG. FRACTURE		NIL, SL, M, H
WETNESS OBSERVED IN FRACTURED C.A. ****		Y N

SUMMARY OF TEST RESULTS

VOIDS (%)		* S.D.
FLOW (.25 mm)		
CORRECTED STABILITY (N)		
** V.M.A. (%)		

CONTRACT № ________

DATE ________

SAMPLE NO. ________

REMARKS : ________

MIX TYPE ____ %A.C. ____

% PASS 4.75mm ________

BLEND ________ S.D. ☐

	AGGREGATE SAMPLE NO.
C/A	
SAND	
SA/SCR.	
RAP	

Table 1

MRD Correction Factors

TEMPERATURE °C	CORRECTION FACTOR K
20	1.001162
21	1.000950
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467
28	0.999187
29	0.998898
30	0.998599

Table 2

Influence Of Water Temperature On Pressure*

TEMPERATURE OF WATER	PRESSURE AT WHICH BOILING OCCURS		CORRESPONDING VACUUM AT WHICH BOILING OCCURS	
°C	mm of Hg	Inches of Hg	mm of Hg	Inches of Hg
20	17.535	0.69	742.47	29.23
21	18.650	0.73	741.35	29.19
22	19.827	0.78	740.17	29.14
23	21.068	0.83	738.93	29.09
24	22.377	0.88	737.62	29.04
25	23.756	0.94	736.24	28.98
26	25.209	0.99	734.79	28.93
27	26.739	1.05	733.26	28.87
28	28.349	1.12	731.65	28.80
29	30.043	1.18	729.96	28.74
30	31.824	1.25	728.18	28.67
31	33.695	1.33	726.31	28.59
32	35.663	1.40	724.34	28.52
33	37.729	1.49	722.27	28.43
34	39.898	1.57	720.10	28.35
35	42.175	1.66	717.83	28.26

*Note: Data taken from 55th Edition Handbook of Chemistry and Physics, Page D159.