ATT-12/22, EXTRACTION, Part II, Centrifuge Method

Method "A" Filterless Extraction Method "B" Bowl Extraction

1.0 SCOPE

This method covers the procedures for determining the asphalt content of asphalt mix using the filterless extraction and filterless centrifuge method, or the bowl type filtered centrifuge and filterless centrifuge method.

2.0 EQUIPMENT

extraction apparatus (if applicable) bowl centrifuge apparatus (if applicable) electric hot plate or gas stove 25,000 µm minus sieve analysis equipment (see ATT-26, Section 2.0, Equipment)

Electronic Balance - capable of reading to 0.1 g and having an accuracy of at least 0.01% of the sample mass, e.g. for a 2000 g sample weight, the balance must be accurate to 0.2 g. The balance must be operated and calibrated as per manufacturer's recommendations.

drying pans	spoon (stainless steel, 12" long)	metal pail
3 core trimmers	large mixing pan	thermal gloves
detergent	plastic wash bottle	putty knife
respirators	extraction solvent	eye protection
grocer scoops, 1-large and 1-small	solvent resistant gloves	

Data Sheets: Core Density, Extraction and Sieve Analysis, such as MAT 6-79 Mix Asphalt Content and Sieve Analysis, MAT 6-101 (for QC testing only)

3.0 PROCEDURE

This test is performed on samples of at least **2000 g** for Quality Assurance Testing and Quality Control Testing where a sieve analysis is required. Smaller samples may be used for Quality Control tests where quick results are required, however the results cannot be used to show cause for an Appeal.

3.1 **Preparing Samples for Extraction Test**

3.1.1 Cores

Coring degrades the aggregate. Therefore, for accurate gradation results, the core outside cut rock must be removed with core trimmers. The trimming procedure is described later in this section.

If the cores are for quality assurance testing, additional cores(s) may have to be obtained adjacent to the segment core so that after sawing and trimming, the sample meets the minimum 2000 g extraction test requirement. After the density of the segment core is determined as described in ATT-7, the uncut rock portion of the core mix is combined with the adjacent core(s) uncut rock mix portions and the entire trimmed sample is extracted.

TABLE 1

For appeal testing, more than one core may be required for each location. The set of cores for each location is considered one sample. The uncut rock mix portions of the trimmed cores are combined and processed.

Use the following table to determine the minimum number of cores required to obtain a minimum 2 000 gram extraction sample using the 127, 114, and 100 mm inside diameter core trimmers. A similar table may be required if trimmers of different diameters are used.

TRIMMED CORE REDUCTION TABLE

Core Thickness	Core Estimated Thickness Core Weight (mm) (g)		ed Trimmed (g)	No.	Total Estimated	
(mm)			114mm Trimmer	100mm Trimmer	Cores	Weight (g)
100	4060		2235		1	2235
90	3655	2375			1	2375
80	3250	2110			1	2110
70	2840			1280	2	2560
60	2435			1095	2	2190
50	2030		1115		2	2230
40	1625	1055			2	2110
30	1220	795			3	2385
20	810	525			4	2100
Estimated weight will vary depending upon the actual core density.						

1. For each core, use a masonry saw to saw off the lift required for testing. Saw right at the tack line to eliminate any tack and/or seal coat chip contamination. Avoid cutting excess material as it contributes to the degradation of the sample.

If a core is obtained from a lower lift which was tacked, sawing may be required at the top and bottom of the core(s) to eliminate the tack coat.

2. Label and tare two hot drying pans (at 130°C) and record the tare pan number and weight in lines "G" and "I" of Form MAT 6-79, as shown in Figure 1.

If additional material is needed to meet the required minimum sample size of 2 000 grams, number and tare another pan and record the number and weight in line "R".

- 3. After the wet density determination (ATT-7 Density), place the segment core in the tare pan recorded in line "I".
- 4. Heat the core sample in the oven, set at $130^{\circ}C \pm 5^{\circ}C$, for about 20 minutes. This time may vary, but use the minimum time requirement which allows the specimen to be easily trimmed using a heated core trimmer. **Overheated** samples may **stick** to the pan, trimmers, putty knife and mixing spoon.
- 5. When additional material is needed to obtain the trimmed minimum sample size, heat the additional core(s) in separate pan(s) for about 20 minutes. Use the tare pan from line "R" as one of the pans, because after trimming, all the uncut rock mix from the additional core(s) will be combined in this tared pan.
- 6. Select a core trimmer as shown in Table 1. Heat the core trimmer be centering the base of the required core trimmer on the stove burner flame for a few minutes, or keep the core trimmers in the oven at 130°C.
- 7. Centre the heated core trimmer on the heated core (allow equal clearance on all sides of the core).
- 8. Hold the core trimmer vertically by its handle, and then press it down through the core until it reaches the bottom of the pan. Apply a slight twist if the heated core is difficult to penetrate. Leave the trimmer in the core.
- 9. Use a heated putty knife or mixing spoon to remove. from the pan, all the outside cut rock core mix. If the core dry density is required, place this outside cut rock material in the tared pan recorded in line "G" and follow ATT-7 to determine the "Moisture Content" and "Dry Density" of the core. Discard this outside cut rock material only if asphalt content and gradation tests are required.
- 10. Scrape off the mix adhering to the core trimmer, after each use, into the appropriate tare pan.
- 11. When additional core(s) are required to obtain the minimum 2000 g sample for asphalt content and gradation:
 - a) Using the core trimmer, remove from each additional core sample(s) the cut rock mix portion as described in steps 6 to 10.
 - b) Discard the outside cut rock mix portions from these additional cores.
- 12. Use a putty knife to break up the uncut rock core mix portion of each sample. Clean any material adhering to the putty knife back into the pan(s).
- 13. Place the pan(s) back in the oven and dry the core mix to a constant weight. This is verified as follows:
 - a) Oven dry the sample for at least two hours then weigh.

Mbertan CORE DENSITY, EXTRACTION AND SIEVE ANALYSIS										
Transportation	PROJECT :		:	HWY 40	40 DATE		LAID :	4-A	4-Apr-2012	
		STATION :		13+48	3	LOCA	TION :	3.8m Rt &		
MAT 6-79/13		LOT NO.	:	7		SEGME	ENT NO.		3	
SEGMENT D (see ATT-7, I	ENSITY Density)			ADDITIONAL UNCUT ROCK CORE MIX FOR EXTRACTION						
A. CORE THICKNESS		mm 43			(see	e ATT-12 Part	II, Extraction)			
B. SAWED CORE WEIGHT		g 1797.8	Q.	DRY WT. OF A	DDITIONAL UNC	UT ROCK CORE	MIX + PAN @ 1	g	1849.7	
C. SATURATED SURFACE DRY WEIGHT		g 1801.7	R.	WT. OF TARE P	AN @ 130°C		Pan No. AAA	g	798.5	
D. VOLUME OF CORE		cm ³ 784.4	S.	DRY WT. OF AI	DITIONAL UNC	UT ROCK CORE	MIX @ 130°C	(Q-R) g	1051.2	
E. CORE WET DENSITY	1000 (B / D)	kg/m ³ 2292			E)	XTRACTIC				
F. DRY WT. OF CUT ROCK CORE MIX + P/	AN	g 1580.0	_		(586					
G. WT. OF TARE PAN @ 130°C	Pan No. AA	g 830.9	Т.	TOTAL DRY W	F. OF UNCUT RO		S+H-I	g	2085.6	
	PAN @ 130°C	g 1908.0	U.		Y WI. OF AGG	GREGATE + PAN	Dan Na O	g	2785.7	
I. WI. OF TARE PAN @ 130°C	$\frac{Pan No. A}{(E - C) + (H - I)}$	g 8/3.6	v. w			PECATE	Pan No. 3	g	1050.9	
	(P-G) + (H-I) (B-J)	g 1703.5	vv. x				0- V	g	1950.8	
	100 (K / J)	9 14.3 % 0.8	<u>х.</u> У.			Beaker	No. 22	9 %	141.6 *	
	1000 (J / D)	ka/m ³ 2274	z.	WT. OF CENTRI	FUGED DRY FIN	JES	X - Y	%	25.3	
	1000 (0 / 2)	% 7.6		TOTAL WT. OF	DRY AGGREGA	ATE	W+Z	%	1976 1	
0. LOT AVERAGE MARSHALL DENSIT	Y	ka/m ³ 2333	BB.	WT. OF EXTRA	CTED ASPHALT		T - AA	a	109.5	
P. PERCENT COMPACTION	100 (M / O)	% 97.5	cc.	EXTRACTION A	SPHALT CONTE	ENT (uncorrected)	100 (BB / AA)	%	5.54	
TIME CORE(S) PLACED IN OV EN	ł	h : min 9:15					0 ATT 12 Porti		0.21	
TIME SAMPLES TAKEN OUT OF OVEN	OF OVEN hh : min 13:30 bh : min 4:15		DD.	EXTRACTION C	URRECTION PA	CIUR S	e ATT-12, Parti	11 70	0.31	
TIME EXTRACTION STARTED	hh : min 13:50		EE.	CORRECTED	ASPHALT CO	NTENT	CC + DD	%	5.85	
TIME EXTRACTION COMPLETED EXTRACTION TIME	hh:min 16:40			If more than 5	i0 g in the bea leaker and ch	eaker has fine in extraction	s up to the r & centrifuge	im, e screens		
	SIEV									
	(see ATT	-26, Sieve Analys	is)							
WT. OF DRY AGGREGATE	OF DRY AGGREGATE (AA) 1976.1 g.								6	
SIEVE SIZES	WEIGHT RETAINED	WEIGHT PASSI	NG	PERCENT PASSING	JOB MIX FORMULA	TOLERANCE				
(μm) 25.000	(g)	(g)		(%)	100			no problem	e	
20 000	0.0	1970.1		100	100	+5		no problem	3	
16 000	4.1	1970.1		100	100	+5				
12 500	193.5	1778 5		90	87	+5				
10 000	375.5	1403.0		71	74	+5				
5 000	395.2	1007.8		51	53	±5				
2 500										
1 250	217.4	790.4		40	42	±3				
630	296.4	494.0		25	26	±2				
315	177.8	316.2		16	17	±2				
160	108.7	207.5		10.5	10.0	±1.5				
80	94.9	112.6		5.7	6.1	±1.5				
SIEVE PAN	4.3			!						
gg. Total Weight	1867.8	% PASSING	∋=(W	= (WT. PASSING / WT. OF DRY AGG) * 100			DATE TESTE	D:	5-Apr-2012	
FF1. DRY WASHWT. + PAN	2705.1						TECHNOLOG	GIST :	B. GOOD	
FF2. TARE OF PAN	834.9							· · · ·]	2. 2002	
FF. DRY WASH WT. (FF1-FF2)	1870.2									
HH. DIFFERENCE (FF - GG) g	2.4	% DIFFEREN	CE = (DIFFERENCE / DF	YWASHWT)	* 100				
% DIFFERENCE (HH / FF) x 100 %	0.13 MAXIMUM % I			ERENCE IS 0.		enter data into shaded areas				

NOTE: TRANSFER NECESSARY DATA TO THE DAILY LOT PAVING REPORT

- b) Replace the sample in the oven for another ½ hour, then re-weigh.
- c) Repeat step (b) until two consecutive weights are the same.

You may have 3 tare pans for each segment; one with the cut rock core mix, one with the uncut rock core mix, and one with the additional uncut rock core mix. With 5 segments, this could make for a maximum of 15 tare pans in the oven drying to a constant weight.

- Record these core mix constant weights in the appropriate lines on MAT 6-79 (Figure 1). Line "F", "Dry Weight of *Cut Rock* Core Mix + Pan"
 Line "H", "Dry Wt. of *Uncut Rock* Core Mix + Pan"
 Line "Q", "Dry Weight of *Additional Uncut Rock* Core Mix + Pan, (if applicable).
- 15. For the **"ADDITIONAL UNCUT ROCK CORE MIX FOR EXTRACTION"** section, calculate the "Dry Wt. of Additional Uncut Rock Core Mix" (line "S") of the additional cores using the formula: *Line* "S" = *Line* "Q" – *Line* "R"

Line "S" = Dry Wt. of Additional Uncut Rock Core Mix and Pan - Wt. of Tare Pan

16. For the *"EXTRACTION DATA"* section, calculate the "Total Dry Wt. of Uncut Rock Core Mix (line "T") using the formula: Line "S" + (Line "H" – Line "I")

Line "T" =

"Dry Wt. of Additional Uncut Rock Core Mix" + ("Dry Wt. of Uncut Rock Core Mix and Pan - "Wt. of Tare Pan")

17. Proceed to Section 3.2, Extraction.

3.1.2 Marshall Specimens

If the extraction asphalt content of field formed Marshall specimens is required, use the two briquettes from a test series. Prepare the specimens for the extraction test as follows:

- 1. If testing for quality control purposes, form MAT 6-101 is used:
 - a) Label and tare a drying pan. Record in line "H", in the "Wt. of Sample Using Oven Dried Method" section, the pan number and weight, as shown in Figure 2.
- 2. After the wet density determination of the marshall specimens, place the specimens in the tared pan and put the pan in the oven set at $130^{\circ}C \pm 5^{\circ}C$.
- 3. After the Marshall specimens have been in the oven for about half hour, remove the pan from the oven and break up the specimens with a putty knife, cleaning off any material adhering to the putty knife.
- 4. Repeat step 13 of Section 3.1.1 Cores, to obtain a constant dry weight.
- 5. Record this "Weight of Dry Mix and Pan" in line "H" of form MAT 6-101.
- 6. Proceed to Section 3.2, Extraction.

Albertan	MIX ASPHALT CONTENT AND SIEVE ANALYSIS EXTRACTION TEST									
Transportation		PROJECT :			HWY 40	:40	I	DISTRICT :	CE	NTRAL
		CON	TRACT NO. :		12345	5	DATE S	AMPLED :	12-D	ec-2012
MAT 6-101/13			LOT NO. :		1		SEGN	IENT NO. :		5
							EXTRACTIO	ON DATA		
(see ATT-15, Part V, I	loisture Conten	ıt)				(see	ATT-12, Part	t II, Extractio	on)	
A. WT. OF MOIST SAMPLE + PAN		g	2820.9	ĸ١	wt. of dry Mi	Х	Lir	ne Eor Line J	g	2208.8
B. WT. OF DRY SAMPLE + PAN		g	2819.0	L. 1	EXTRACTED D	ry wt. of a	GG. + PAN @ 1	30°C	g	3304.1
C. WT. OF WATER	A-B	g	1.9	м. \	M. WT. OF TARE PAN @ 130° C Pan No. YY			g	1242.7	
D. WT. OF PAN @ 130°C Pan No. 2	<u>ix</u>	g	798.5	N. 1	EXTRACTED DR	ry wt. of ac	GREGATE	L - M	g	2061.4
E WT. OF DRY SAMPLE	B-D	g	2020.5	O. \	WT.OF CENTRIF	FUGE DRY FIN	IES AND BEAKE	ER @130°C	g	201.2
F. MIX MOISTURE CONTENT	100 (C/E)	g	0.09	P. \	WT. OF BEAKE	R @ 130°C	Beaker No.	YY	g	165.8
T1 TIME SAMPLE PLACED IN OVEN	hr:min		3:03	Q. 1	WT. OF CENTRI	FUGED FINES		0 - P	g	35.4
T2 TIME SAMPLE TAKEN OUT OF OVEN	hr:min		5:05	R. ⁻	TOTAL WT. OF	DRY AGG.		N+Q	g	2096.8
TOTAL DRYING TIME T2 - T1	hr:min		2:02	S. \	WT. OF ASPHA	LT		K-R	g	112.0
T3 TIME EXTRACTION STARTED	hr:min		3:00	т. /	ASPHALT CON	TENT (uncorre	ected)	100 (S/R)	%	5.34
T4 TIME EXTRACTION COMPLETED	hr:min		6:00	U. /	ASPHALT CON	TENT CORREC	CTION FACTOR	(see ATT-12	, Part III) %	0.10
TOTAL EXTRACTION TIME T4 - T3	hrimin		3:00				T+U	%	5.44	
			5.00						3.44	
WT. OF SAMPLE USING CALCULATED SAMPLE DRY WT METHOD			OD	WT. OF SAMPLE USING OVEN DRIED METHOD						
G. WT. OF MOIST MIX + PAN g 2830			2830.4	B1. WT. OF DRY MIX + PAN g					2828.3	
H. WT. OF PAN @ 130°C Pan No. /	A	g	619.5	н١	WT. OF PAN @	2 130°C	Pan No. AA		g	619.5
I. WT. OF MOIST MIX	G-H	g	2210.9							
J. WT. OF DRY MIX	l / (100 + F)) x 10	0 (g)	2208.8	J8.8 J. WT. OF DRY MIX B ₁ - H		g	2208.8			
	SIEVE ANALYSIS									
(see ATT-26, Sie WT. OF DRY AGGREGATE (R)			2096.8	, g.				REMARKS		
SIEVE SIZE (µm)			WEIGHT		PERCENT		TOLERANCE		KEWARN	.5
	(g)		(g)		(%)	TONWOLA				
25 000	0.0		2096.8		100					
20 000	0.0		2096.8		100					
16 000	0.0		2096.8		100	100				
12 500	281.6	_	1815.2		87	87	±5			
10 000	260.0	_	1555.2		74	74	±5			
5 000	428.5	-	1126.7		54	53	±5			
1 250	280.0	-	846 7		40	42	+5			
630	310.5		536.2		26	26	±3			
315	179.6		356.6		17	17	±2			
160	167.6		189.0		9.0	10.0	±1.5			
80	72.1		116.9		5.6	6.1	±1.5			
SIEVE PAN	3.5									
W. TOTAL WEIGHT 1983.4 % PASSING =			= (W1	T. PASSING / W	T. OF DRY A	GG) * 100	DATE TES	TED :	5-Apr-2012	
X. DRY WASH WT. + PAN	3226.6							TECHNOL	OGIST :	B. GOOD
Y. TARE OF PAN	1243.0									
	1983.6	-		ICE		DRY WARLIN	VT) * 100			
% Difference	Maximum Activity Maximum Activity<				S 0.5 %	enter d	ata into sha	aded areas		

NOTE: TRANSFER NECESSARY DATA TO THE DAILY LOT PAVING REPORT

FIGURE 2

3.1.3 Loose Mix Samples

- 1. Obtain a representative sample of the mix, 20 kg (3/4 full metal pail), as directed in ATT-37, SAMPLING MIXES.
- 2. Dump the mix into a large heated mixing pan and use the heated large grocer scoop to thoroughly mix it. Use the grocer scoop to obtain the required size of sample for testing.
- 3. Proceed to 3.1.3.1 to use the "Oven Dried Sample Method", or go to 3.1.3.2 for the "Calculated Sample Dry Weight Method".

3.1.3.1 Oven Dried Sample Method (Fresh Mix, Reclaim or Liquid Asphalt Mixes)

- 1. Label and tare a drying pan. Record the weight and pan number, in the "Wt. of Sample Using Oven Dried Method" section, in line "H" of form MAT6-101 as shown in Figure 2.
- 2. Use a clean heated scoop to place **a minimum of 2000** g of the mix in the pan.
- 3. Use the putty knife to clean off any mix adhering to the scoop, into the tare pan.
- 4. Place the drying pan with the mix in the oven set at 130EC \forall 5EC and record the time the sample was placed in the oven.
- 5. Dry the sample to a constant weight. This is verified as follows:
 - a) Oven dry the mix sample for at least two hours, then weigh.
 - b) Replace the sample in the oven for another ½ hour, then re-weigh.
 - c) Repeat step (b) until two consecutive weights are the same.
- 7. Weigh the pan containing the mix, now at a constant weight, and record as "Wt. of Dry Mix + Pan" on line "B₁", in the "Wt. of Sample Using Oven Dried Method" section.
- 8. Subtract line "H" from line "B₁" and record as "Wt. of Dry Mix" on line "J".
- 9. Proceed to Section 3.2, Extraction.

3.1.3.2 Calculated Sample Dry Weight Method

This section is used when it is necessary to process the sample quickly, but it should be recognized that it may not be as accurate as drying the actual sample to a constant weight before beginning the extraction test. Here we will perform an extraction test on a mix sample that still has an as yet to be determined moisture content. To determine the moisture content of the mix, we are performing a mix moisture content test on a separate sample, and then back-calculating the actual weight of dry mix used for the extraction.

- 1. Label and tare a two drying pans. Record the weights and pan numbers in line "D" and line "H" of form MAT6-101 as shown in Figure 2. One tare pan will be used for the moisture content sample and one for the extraction sample.
- 2. Use the heated grocer scoop to place a minimum of 2000 g of mix in each of the tared pans. Ensure the mix is level and evenly distributed over the bottom of the pan.
- 3. While the scoop is still hot, use the putty knife to clean off the mix adhering to the scoop and place it in the appropriate pan.
- 4. Weigh the samples and record as "Wt. of Moist Sample + Pan" on line "A" in the Mix Moisture Content section and as "Wt. of Moist Mix + Pan" on line "G" in the Calculated Sample Dry Wt. Method section.
- 5. Calculate the Wt. of Moist Mix (line "I") for the extraction sample as follows: Line "I" = Line "G" – Line "H"
 Wt. of Moist Mix (g) = (Wt. of Moist Mix and Pan) - (Wt. of Pan)
- 6. Place the moisture content sample in the oven and dry it to a constant weight as described in Section 3.1.3.1 step 5.
- 7. Record the time the sample was last removed from the oven and calculate the drying time.
- 8. Weigh the hot sample and record as "Wt. of Dry Sample + Pan" on line "B".
- 9. Calculate the "Weight of Water" removed from the mix: Line "C" = Line "A" Line "C"
 Wt. of Water (g) = (Wt. of Moist Sample and Pan) (Wt. of Dry Sample and Pan)
- 10. Determine the "Wt. of Dry Sample", (line "E"), as follows: Line "E" = Line "B" Line "D" *Wt. of Dry Sample (g) = (Wt. of Dry Sample and Pan) (Wt. of Pan)*
- 11. Calculate the "Mix Moisture Content", to the nearest 0.01 % (line "F"), as follows:

Mix Moisture Content (%) =
$$\frac{Wt. \text{ of Water (line "C")}}{Wt. \text{ of Dry Sample (line "E")}} \times 100$$

12. Calculate the "Weight of Dry Mix", (line "J"), using the formula: Line "J" = (Line "I" / (100 + Line "F")) x 100

Wt. of Dry Mix (g) = $\frac{Wt. \text{ of Moist Mix}}{100 + \text{Mix Moisture Content (%)}} \times 100$

3.2 Extraction

3.2.1 Method "A" Filterless Extraction

- 1. Assemble the equipment as required.
- 2. Set the extraction basket in a clean drying pan. The drying pan is used to trap any spilled mix.
- 3. Set the pan containing the mix adjacent to the pan with the basket.
- 4. Use the tablespoon to fill the basket with the extraction sample.
- 5. Place the basket on top of the basket stand, in the battery jar.
- 6. Put on your respirator. In the fume cabinet, use the wash bottle containing chlorinated extraction solvent to rinse any mix residue on the spoon into the mix pan.
- 7. Use the wash bottle to flush the contents of the mix pan into the extraction basket.
- 8. Place the condenser on the battery jar.
- 9. Place a wire gauze on each burner. Centre the battery jar on the burner.
- 10. Ensure the condenser is on the battery jar, then light the burner.
- 11. Open the water tap to circulate a gentle steady flow of cold water through the condenser.
- 12. Record the "Time Extraction Started" on form MAT 6 79, or on form MAT 6-101.
- 13. Turn the burner to low heat until the battery jar and the chlorinated extraction solvent are hot. Rapid changes in jar temperature may crack battery jars.
- 14. Turn the heat on full under each jar until the chlorinated extraction solvent vapours start to condense and drip from the condenser. At this point, adjust the heat until there is a constant flow of chlorinated extraction solvent from the condenser. Take care to adjust the heat so that the filter baskets do not overflow.
- 15. Keep the sample in the basket continually saturated with chlorinated extraction solvent.
- 16. Adjust the water flow throughout the test to keep the condenser cool.

- 17. Allow the refluxing action to continue in the jar until the wash from the basket is clear. At this point, turn off the heat, but **not** the condenser water.
 - **NOTE:** The extraction time depends on the asphalt content and gradation of the mix. It is approximately 2.0 to 2.5 hours for ACP plant mix. Reclaimed or recycled mixes require more time. Cutbacks and emulsified asphalt mixes may require less extraction time. Lime treated aggregates may need 4 hours. Check the colour of the chlorinated extraction solvent periodically until there is no noticeable change in colour. For very dirty aggregates, the flow of solvent from the basket may appear clear even though asphalt is still being extracted. This is caused by chlorinated extraction solvent condensing on the outside of the basket and flowing down to the bottom, diluting the stream and creating an apparent clear stream.
- 18. Allow the chlorinated extraction solvent to stop condensing, and the extract to cool to room temperature.
- 19. Record the "Time Extraction Completed" and calculate the total "Extraction Time" on form MAT 6-79.
- 20. Label and tare a drying pan and record the pan number and weight in line "V" of form MAT 6-79, or line "M" of form MAT 6-101.
- 21. Place the drying pan in the fume cabinet.
- 22. Put on solvent gloves and respirator, and remove the condenser from the jar.
- 23. Remove the extraction basket and basket stand from the battery jar.
- 24. Replace the condenser back onto the battery jar.
- 25. Invert the basket and basket stand in the drying pan, emptying the contents.
- 26. Brush all fines off the extraction basket and basket stand into the pan.
- 27. While the pan is in the fume cabinet, gently stir the aggregate in the drying pan to remove some of the chlorinated extraction solvent. Not all the aggregate may come out of the basket and stand, so gently place the basket and stand on top of the aggregate, to dry in the oven for about 1 hour, then brush off any leftover aggregate into the drying pan.
- 28. Ensure the oven is set at $130^{\circ}C \pm 5^{\circ}C$ and place the drying pan with the aggregate in the oven to dry to a constant weight.
- 29. Proceed to 3.3 Fines Correction, to catch any fines that passed through the basket into the extraction solvent.

3.2.2 Method "B" Bowl Extraction

- 1. Set the bowl in a clean drying pan. The drying pan traps spilled mix.
- 2. Set the pan containing the mix adjacent to the pan with the bowl.
- 3. Use the spoon to fill the bowl with the extraction sample.
- 4. Place the bowl into the centrifuge.
- 5. Put on your respirator. In the fume cabinet, use the wash bottle containing chlorinated extraction solvent to rinse the spoon (and the beaker, if required) into the mix pan.
- 6. Use the wash bottle to flush the contents of the mix pan into the extraction bowl.
- 7. Cover the sample with extraction solvent. (approx. 400 ml)
- 8. Place the lid on the bowl. A filter may be used but is not required. If a filter is used it should be dried in the oven and included with the pan tare in step 13 below.
- 9. Position a suitable container under the centrifuge effluent discharge spout to catch the effluent from the centrifuging operation.
- 10. Start the centrifuge at 250 rev/min. Once the flow has reduced significantly, increase the speed to 1000 rev/min., until the flow stops.
- 11. Stop the centrifuge, and then add an additional 200 ml of solvent into the top of the extractor.
- 12. Continue steps 10 and 11 until the solvent appearance is a light straw colour. Stop the bowl.
- 13. Label and tare a drying pan and record the pan number and weight in line "V" of form MAT 6 79, or line "M" of form MAT 6-101, to the nearest 0.1 gram.
- 14. Place the drying pan in the fume cabinet.
- 15. Put on the solvent resistant gloves and respirator and remove the bowl from the centrifuge.
- 16. Carefully transfer the aggregate from the bowl into the drying pan.
- 17. Brush all fines off the bowl into the drying pan.
- 18. While the pan is in the fume cabinet, gently stir the aggregate in the drying pan to remove some of the chlorinated extraction solvent.
- 19. Ensure the oven is set at $130^{\circ}C \pm 5^{\circ}C$ and place the drying pan with the aggregate in the oven.

3.3 Fines Correction using High Speed Centrifuge

The High Speed Centrifuge is used in the extraction of mineral fines from bitumen-laden solvents obtained from standard asphalt extraction tests. In operation, the solvent suspension is fed through the top funnel into a special aluminum beaker. Using a high centrifugal force, the liquid moves up the beaker wall and out the overflow tube while the solids remain in the beaker for easy removal at test completion. The system allows the continuous feeding of the suspension until the solids-retaining capacity of the beaker is reached.

- 1. Label and tare a hot centrifuge beaker. Record the beaker weight and number in line "Y", "Weight of Beaker", on a form such as MAT 6-79, or line "P" on a form such as MAT 6-101.
- 2. Cool the beaker, and then insert it into the centrifuge holding head.
- 3. Place the fine sieve (80µm) in the sieve funnel.
- **NOTE:** It is very important that the fines sieve (80μm) be used as the effluent will contain some plus 80 μm material. This +80μm material will be put back into the dry aggregate sample for the sieve analysis. The sieve analysis procedure assumes only minus 80 μm material in the beaker and therefore the gradation of the sample will be effected if the beaker contains plus 80 μm material.
- 4. Place an empty uncovered plastic container underneath the centrifuge outlet and turn the centrifuge on.
- 5. With the unit rotating at full speed set the tap controlling the flow rate to the calibration mark. This will control the flow rate so that the output is 0.1 to 0.4 litres per minute, as set out in the centrifuge manual of operation, preventing the fines from being washed over the beaker wall and out the overflow tube into the effluent container.
- 6. Put on the respirator and pour the cool extract from the filterless extraction through the centrifuge sieve funnel. If the extract is not at room temperature, a high amount of chlorinated extraction solvent will be lost due to vaporization.
- 7. If Method "A" is being used, thoroughly flush all the extract from the battery jar into the sieve funnel using a wash bottle containing chlorinated extraction solvent. No fines should be left in the battery jar. If Method "B" is being used, thoroughly flush all the extract from the container holding the extract from the bowl outlet.
- 8. When the extract level falls below the fine sieve in the sieve funnel, thoroughly wash the fines collected on the screen with the solvent wash bottle.
- 9. Air dry the fine sieve and brush the retained material into the pan containing the oven dry aggregate from the extraction (step 25 of Section 3.2.1, Filterless Extraction or step 16 of Section 3.2.2, Bowl Extraction).
- 10. When all the extract is centrifuged, flush clean chlorinated extraction solvent through the sieve funnel until it flows clean into the plastic container underneath the centrifuge outlet.
- 11. Cover the plastic container to minimize vaporization of chlorinated extraction solvent.

- 12. Allow the centrifuge run for a few minutes to evaporate the chlorinated extraction solvent within the beaker.
- 13. Turn off the centrifuge and allow the beaker to coast to a stop. This will provide sufficient time for excess vapours to clear. The brake can be used to slow the centrifuge a maximum of 5 times per hour (refer to the centrifuge manual of operation).
- 14. Withdraw the beaker and place it in the drying pan containing the extracted aggregate.
- 15. Place the pan in the oven set at $130^{\circ}C \pm 5^{\circ}C$ and oven dry the aggregate and beaker to a constant dry weight.
- 16. Remove the beaker from the pan and weigh the beaker and its contents. Record as "Wt. of Centrifuged Dry Fines + Beaker" on line "X" of form MAT 6-79, or line "O" of form MAT 6-101.
- 17. Weigh the hot pan and its contents. Record as "Extracted Dry Wt. of Aggregate + Pan" on line "U" of form MAT 6-79, or line "L" of form MAT 6-101.
- 18. Calculate the "Extracted Dry Wt. of Aggregate" on line "W" of form MAT 6-79, or line "N" of a form such as MAT 6-101 as follows:
 Line "W" = Line "U" Line "V" (MAT 6-79)
 Line "N" = Line "L" Line "M" (MAT 6-101)

Extracted Dry Wt. of Agg. = Extracted Dry Wt. of Agg. and Pan - Wt. of Tare Pan

19. Calculate the "Wt. of Centrifuged Dry Fines" on line "Z" of form MAT 6-79, or line "Q" of a form such as MAT 6-101 as follows:
Line "Z" = Line "X" - Line "Y" (MAT 6-79)
Line "Q" = Line "O" - Line "P" (MAT 6-101)

Wt. of Centrifuged Fines = *Wt.* of Centrifuged Dry Fines and Beaker - *Wt.* of Beaker

20. At the beginning of a project or when results are suspect, the high speed centrifuge extract should be checked to ensure complete removal of fines. Run the extract through the centrifuge a second time, using a new beaker. Only traces of fines should be separated in the process.

Appreciable quantities of fines in the check beaker may be caused by the following:

- a) Speed of the centrifuge was below the minimum of 9000 rev/min.
- b) Output was too fast. Reduce the flow rate of the extract.
- c) A distorted beaker was used. Always check beaker before use.
- d) Excessive fines in the extract (greater than 50 g) ran through the beaker. To correct, distribute the extract over several beakers.
- 21. Clean out the fines from the inside of the beaker with a stiff bristle brush, discarding the material.

3.4 Corrected Extraction Asphalt Content

1. Determine the "Total Weight of Dry Aggregate" on line "AA" of form MAT 6-79, or line "R" of form MAT 6-101, using the formula:

= Extracted Dry Wt. of Aggregate + Wt. of Centrifuged Dry Fines

2. Determine the "Weight of Extracted Asphalt" on line "BB" of form MAT 6-79, or line "S" of form MAT 6-101, as follows:

= Total Dry Wt. of Uncut Rock Core Mix (Dry Mix) - Total Wt. of Dry Aggregate

3. Calculate the "Extraction Asphalt Content (uncorrected)" to the nearest 0.01% on line "CC" of form MAT 6-79, or line "T" of form MAT 6-101, using the formula:

Asphalt Content (uncorrected) = <u>Wt. of Extracted Asphalt</u> Total Wt. of Dry Aggregate x 100

- 4. Enter the "Extraction Correction Factor", as determined in ATT-12, Part III, on line "DD" of form MAT 6-79, or on line "U" of form MAT 6-101.
- 5. Calculate the "Corrected Asphalt Content" on line "EE" of form MAT 6-79, or line "V" of form MAT 6-101, as follows:

Corrected Asphalt Content = Extraction Asphalt Content + Extraction Correction Factor

3.5 Sieve Analysis

- 1. Transfer the "Total Weight of Dry Aggregate" on line "AA" of form MAT 6-79, or line "R" of form MAT 6-101, to the top of the "Sieve Analysis" section of the form.
- 2. Perform a wash sieve analysis on the extracted aggregate, as directed in ATT-26.

4.0 Hints and Precautions

- 1. Fumes from chlorinated extraction solvents are toxic. Perform the extraction test using an exhaust system in a well-ventilated area, since these are toxic substances. Use the lab fume cabinet for the filterless extractions and the centrifuge fume cabinet/case for centrifuge extractions.
- 2. Respirators and solvent resistant gloves should be worn when handling chlorinated extraction solvent (e.g., pouring the solvent). Each person should have a personal respirator, safety glasses, and a pair of gloves. The proper respirator for the hazard must be used. Proper use of dust and chemical respirators is discussed in the applicable manufacturers' equipment manuals.
- 3. Respirators must be stored in airtight containers and away from all fumes so that filters do not accumulate fumes from the air.
- 4. Chlorinated extraction and cleaning solvent containers must be sealed when not in use. Battery jars containing chlorinated extraction solvent must be kept covered and in a fume cabinet when not in use. This prevents loss due to evaporation and minimizes exposure to the solvent fumes.
- 5. The wire mesh of the extraction basket should be inspected before testing begins, to ensure that it is clean. A dirty basket may change the weight of aggregate obtained during the test.
- Chlorinated extraction solvent or an approved substitute must be used. Some of the approved substitutes are flammable.
 Solvent substitutes, such as varsol, <u>must not be used</u>, as they will explode.
- 7. Beakers must be handled with care. Do not tap the beakers when removing fines. Deformation of the beaker may cause a loose fit in the holding head, resulting in incomplete separation of fines.
- 8. Use a maximum of 5 brakes per hour to slow the centrifuge (or as suggested in the centrifuge operator manual). An excessive amount of braking will change the polarity of the motor and reverse the centrifuge's direction of rotation.
- 9. Keep the centrifuge upright when it is being moved and transported, as oil may leak out of the spindle if the machine is at an angle.
- 10. Use a bottle brush and hot soapy water to clean the beakers.
- 11. Ensure that lime-treated aggregate samples are identified as "lime treated" on the sample identification form.
- 12. In no event should the solvent be used to clean your hands.

13. Materials Safety Data Sheets (MSDS) should be provided by the supplier of the solvents when they are purchased. These sheets should be made readily available to all employees using these solvents. The safety precautions listed in these MSDS sheets should be adhered to at all times.

A summary of first aid, storage guidelines, and protective equipment for using chlorinated extraction solvents is as follows:

REACTIVITY AND STORAGE

INCOMPATIBLE WITH	Water, Oxidizing Material, chemically active metals and aluminum.
PRECAUTIONS	Keep container tightly closed. Store in a secure area. Store in a cool, well-ventilated area away from sources of heat, flame or ignition. <i>Do not smoke around this product.</i>

FIRST AID

INHALATION	Remove to fresh air. If not breathing, give artificial respiration. Have trained personnel give oxygen if breathing is difficult. Keep warm and at rest. Obtain immediate medical attention.
INGESTION	Give 2 or 3 glasses of milk or water. Keep warm and at rest. Obtain immediate medical attention.
EYES	Immediately flush with a gentle stream of water for at least 15 minutes, lifting upper and lower lids occasionally. Obtain medical attention if irritation persists after flushing.
SKIN	Wash exposed skin thoroughly with water while removing contaminated clothing. Obtain medical attention if irritation persists.

PERSONAL PROTECTIVE EQUIPMENT (PPE)

Use only NIOSH-approved respirators to prevent overexposure to vapor. Store respirators & cartridges in airtight containers to maintain "freshness".

Eye protection is mandatory. Use Splash-proof Safety Goggles. Full Face-shields provide the best protection.

Use a Lab coat. Wear appropriate impervious rubber gloves and protective clothing to prevent skin contact. Use an impervious apron when splashing is likely. Remove contaminated clothing promptly and launder before reuse.

PLEASE <u>READ</u> THE MATERIALS SAFETY DATA SHEET (MSDS) <u>BEFORE</u> YOU USE THE EXTRACTION SOLVENT.