Sample: 25wax:75lt hc @ 1:4 crude oil ratio Temperature : 40°F Spindle: Vane Model: RV			Sample: 25wax:75lt hc @ 1:4 crude oil ratio Temperature : 20°F Spindle: Vane Model: HB			Sample: 25wax:75lt hc @ 1:4 crude oil ratio Temperature : 9°F Spindle: Vane Model: HB		
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
53.33	1.7	122.2						
53.50	1.7	122.2						
53.67	1.7	122.2						
53.83	1.7	122.2						
54.00	1.7	122.2						
54.17	1.7	122.2						
54.33	1.7	122.2						
54.50	1.7	122.2						
54.67	1.7	122.2						
54.83	1.7	122.2						
55.00	1.7	122.2						
55.17	1.7	122.2						
55.33	1.7	122.2						
55.50	1.7	122.2						
55.67	1.7	122.2						
55.83	1.8	129.4						
56.00	1.7	122.2						
56.17	1.7	122.2						
56.33	1.7	122.2						
56.50	1.7	122.2						
56.67	1.7	122.2						
56.83	1.7	122.2						
57.00	1.7	122.2						
57.17	1.7	122.2						
57.33	1.7	122.2						
57.50	1.7	122.2						
57.67	1.7	122.2						
57.83	1.7	122.2						
58.00	1.7	122.2						
58.17	1.6	115.0						
58.33	1.7	122.2						
58.50	1.6	115.0						
58.67	1.7	122.2						
58.83	1.7	122.2						
59.00	1.7	122.2						
59.17	1.7	122.2						
59.33	1.7	122.2						
59.50	1.7	122.2						
59.67	1.7	122.2						
59.83	1.7	122.2						
60.00	1.7	122.2						
60.17	1.7	122.2						

Sample (ii)

25% Wax : 75% GTL + Crude Oil in the Ratio of 1:3

Sample: 25wax:75GTL @ 1:3 crude oil ratio Temperature : 40°F Spindle: Vane Model: LV			Sample: 25wax:75GTL @ 1:3 crude oil ratio Temperature : 40°F Spindle: Vane Model: RV			Sample: 25wax:75GTL @ 1:3 crude oil ratio Temperature : 20°F Spindle: Vane Model: HB			Sample: 25wax:75GTL @ 1:3 crude oil ratio Temperature : 9°F Spindle: Vane Model: HB		
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
0.00	0	0	0.00	0.0	0.0	0.00	0.1	57	0.00	0.0	0
7.00	30.5	205	0.17	0.0	0.0	0.17	0.2	115	0.17	0.4	230
7.17	31.2	210	0.33	0.0	0.0	0.33	0.2	115	0.33	0.4	230
7.33	32.1	216	0.50	0.0	0.0	0.50	0.2	115	0.50	0.5	287
7.50	32.9	222	0.67	0.0	0.0	0.67	0.2	115	0.67	0.4	230
7.67	33.7	227	0.83	0.0	0.0	0.83	0.2	115	0.83	0.4	230
7.83	34.5	232	1.00	0.0	0.0	1.00	0.2	115	1.00	0.5	287
8.00	35.2	237	1.17	0.1	7.2	1.17	0.2	115	1.17	0.5	287
8.17	36.0	243	1.33	0.2	14.4	1.33	0.2	115	1.33	0.4	230
8.33	36.9	249	1.50	0.4	28.7	1.50	0.2	115	1.50	0.4	230
8.50	37.6	253	1.67	0.5	35.9	1.67	0.2	115	1.67	0.5	287
8.67	38.4	259	1.83	0.6	43.1	1.83	0.2	115	1.83	0.5	287
8.83	39.2	264	2.00	0.8	57.5	2.00	0.2	115	2.00	0.5	287
9.00	40.1	270	2.17	1.0	71.9	2.17	0.2	115	2.17	0.5	287
9.17	40.8	275	2.33	1.1	79.1	2.33	0.3	172	2.33	0.5	287
9.33	41.7	281	2.50	1.3	93.4	2.50	0.3	172	2.50	0.5	287
9.50	42.4	286	2.67	2.1	150.9	2.67	0.2	115	2.67	0.5	287
9.67	43.2	291	2.83	2.7	194.0	2.83	0.3	172	2.83	0.5	287
9.83	44.0	296	3.00	3.3	237.2	3.00	0.3	172	3.00	0.4	230
10.00	44.8	302	3.17	4.0	287.5	3.17	0.3	172	3.17	0.5	287
10.17	45.6	307	3.33	4.6	330.6	3.33	0.3	172	3.33	0.5	287
10.33	46.4	313	3.50	5.2	373.7	3.50	0.3	172	3.50	0.5	287
10.50	47.2	318	3.67	5.8	416.8	3.67	0.3	172	3.67	0.5	287
10.67	48.0	323	3.83	6.4	460.0	3.83	0.3	172	3.83	0.5	287
10.83	48.8	329	4.00	6.9	495.9	4.00	0.3	172	4.00	0.5	287
11.00	49.6	334	4.17	7.5	539.0	4.17	0.3	172	4.17	0.6	345
11.17	50.4	340	4.33	8.0	575.0	4.33	0.3	172	4.33	0.5	287
11.33	51.2	345	4.50	8.4	603.7	4.50	0.3	172	4.50	0.6	345
11.50	52.0	350	4.67	8.8	632.5	4.67	0.3	172	4.67	0.6	345
11.67	52.8	356	4.83	9.1	654.0	4.83	0.3	172	4.83	0.6	345
11.83	53.6	361	5.00	8.9	639.6	5.00	0.3	172	5.00	0.6	345
12.00	54.5	367	5.17	8.4	603.7	5.17	0.3	172	5.17	0.6	345
12.17	55.1	371	5.33	8.3	596.5	5.33	0.3	172	5.33	0.6	345
12.33	55.9	377	5.50	8.4	603.7	5.50	0.3	172	5.50	0.7	402
12.50	56.8	383	5.67	8.4	603.7	5.67	0.3	172	5.67	0.8	460
12.67	57.5	387	5.83	8.6	618.1	5.83	0.3	172	5.83	1.1	632
12.83	58.4	393	6.00	9.1	654.0	6.00	0.3	172	6.00	1.3	747
13.00	59.2	399	6.17	9.7	697.1	6.17	0.3	172	6.17	1.4	805
13.17	59.9	404	6.33	10.3	740.3	6.33	0.4	230	6.33	1.4	805
13.33	60.7	409	6.50	10.8	776.2	6.50	0.6	345	6.50	1.6	920
13.50	61.6	415	6.67	11.2	804.9	6.67	0.7	402	6.67	1.7	977
13.67	62.3	420	6.83	11.4	819.3	6.83	0.8	460	6.83	1.7	977
13.83	63.1	425	7.00	11.7	840.9	7.00	0.9	517	7.00	2.2	1265
14.00	64.0	431	7.17	12.1	869.6	7.17	1.5	862	7.17	2.7	1552
14.17	64.7	436	7.33	12.7	912.7	7.33	1.8	1035	7.33	2.9	1667
14.33	65.5	441	7.50	13.1	941.5	7.50	1.9	1092	7.50	3.3	1897
14.50	66.3	447	7.67	13.6	977.4	7.67	2.1	1207	7.67	3.5	2012
14.67	67.1	452	7.83	14.1	1013.4	7.83	2.3	1322	7.83	3.6	2070
14.83	67.9	457	8.00	14.5	1042.1	8.00	2.5	1437	8.00	3.9	2242
15.00	68.7	463	8.17	14.9	1070.9	8.17	2.5	1437	8.17	4.2	2415
15.17	69.5	468	8.33	15.3	1099.6	8.33	2.8	1610	8.33	4.4	2530
15.33	70.3	474	8.50	15.6	1121.2	8.50	2.9	1667	8.50	4.6	2645
15.50	71.1	479	8.67	15.9	1142.7	8.67	3.0	1725	8.67	4.9	2817
15.67	71.9	484	8.83	16.3	1171.5	8.83	3.0	1725	8.83	5.1	2932
15.83	72.6	489	9.00	16.7	1200.2	9.00	3.0	1725	9.00	5.4	3105
16.00	73.4	494	9.17	17.1	1229.0	9.17	3.2	1840	9.17	5.8	3335
16.17	74.2	500	9.33	17.7	1272.1	9.33	3.2	1840	9.33	5.9	3392
16.33	75.0	505	9.50	18.0	1293.7	9.50	3.2	1840	9.50	6.4	3680
16.50	75.8	511	9.67	18.5	1329.6	9.67	3.2	1840	9.67	6.8	3910
16.67	76.6	516	9.83	19.0	1365.5	9.83	3.3	1897	9.83	7.0	4025
16.83	77.3	521	10.00	19.3	1387.1	10.00	3.1	1782	10.00	7.3	4197
17.00	78.1	526	10.17	19.6	1408.7	10.17	3.1	1782	10.17	7.6	4370
17.17	78.9	532	10.33	20.1	1444.6	10.33	3.2	1840	10.33	7.8	4485
17.33	79.7	537	10.50	20.6	1480.5	10.50	3.2	1840	10.50	8.2	4715
17.50	80.4	542	10.67	21.0	1509.3	10.67	3.2	1840	10.67	8.3	4772
17.67	81.3	548	10.83	21.5	1545.2	10.83	3.2	1840	10.83	8.5	4887
17.83	82.0	552	11.00	22.0	1581.1	11.00	3.3	1897	11.00	8.9	5117
18.00	82.8	558	11.17	22.3	1602.7	11.17	3.2	1840	11.17	9.2	5290
18.17	83.6	563	11.33	22.8	1638.6	11.33	3.3	1897	11.33	9.6	5520
18.33	84.3	568	11.50	23.2	1667.4	11.50	3.3	1897	11.50	9.8	5635
18.50	85.1	573	11.67	23.7	1703.3	11.67	3.4	1955	11.67	10.1	5807
18.67	86.0	579	11.83	24.1	1732.1	11.83	3.5	2012	11.83	10.5	6037
18.83	86.7	584	12.00	24.4	1753.6	12.00	3.7	2127	12.00	10.8	6210
19.00	87.4	589	12.17	24.7	1775.2	12.17	3.7	2127	12.17	11.0	6325
19.17	88.3	595	12.33	24.9	1789.6	12.33	3.8	2185	12.33	11.3	6497
19.33	89.0	600	12.50	25.4	1825.5	12.50	3.8	2185	12.50	11.8	6785
19.50	89.7	604	12.67	25.7	1847.1	12.67	3.8	2185	12.67	12.0	6900
19.67	90.6	610	12.83	26.2	1883.0	12.83	3.6	2070	12.83	12.4	7130
19.83	91.3	615	13.00	26.5	1904.6	13.00	3.7	2127	13.00	12.6	7244

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 40°F
Spindle: Vane
Model: LV

Cum. Time minutes	Torque %	Stress (dyne/cm)
20.00	92.1	620
20.17	92.9	626
20.33	93.7	631
20.50	94.4	636
20.67	95.2	641
20.83	96.1	647
21.00	96.8	652
21.17	97.6	658
21.33	98.4	663
21.50	99.1	668
21.67	99.9	673
21.83	100.0	674

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 40°F
Spindle: Vane
Model: RV

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	26.7	1918.9
13.33	27.1	1947.7
13.50	27.5	1976.4
13.67	27.8	1998.0
13.83	28.1	2019.5
14.00	28.4	2041.1
14.17	28.7	2062.7
14.33	29.1	2091.4
14.50	29.4	2113.0
14.67	29.6	2127.4
14.83	29.9	2148.9
15.00	30.2	2170.5
15.17	30.4	2184.8
15.33	30.0	2156.1
15.50	30.1	2163.3
15.67	30.5	2192.0
15.83	30.8	2213.6
16.00	31.1	2235.2
16.17	31.4	2256.7
16.33	31.6	2271.1
16.50	31.8	2285.5
16.67	31.9	2292.7
16.83	31.9	2292.7
17.00	32.0	2299.8
17.17	32.0	2299.8
17.33	32.0	2299.8
17.50	32.1	2307.0
17.67	32.1	2307.0
17.83	32.0	2299.8
18.00	31.9	2292.7
18.17	31.8	2285.5
18.33	31.5	2263.9
18.50	31.4	2256.7
18.67	31.2	2242.3
18.83	30.8	2213.6
19.00	30.5	2192.0
19.17	30.2	2170.5
19.33	29.7	2134.5
19.50	29.2	2098.6
19.67	28.7	2062.7
19.83	28.2	2026.7
20.00	27.6	1983.6
20.17	27.2	1954.9
20.33	27.1	1947.7
20.50	26.5	1904.6
20.67	25.8	1854.2
20.83	25.0	1796.8
21.00	24.2	1739.3
21.17	23.6	1696.1
21.33	22.8	1638.6
21.50	22.0	1581.1
21.67	21.4	1538.0
21.83	20.5	1473.3
22.00	19.5	1401.5
22.17	18.5	1329.6
22.33	17.7	1272.1
22.50	16.8	1207.4
22.67	16.2	1164.3

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 20°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	3.8	2185
13.33	3.5	2012
13.50	3.4	1955
13.67	3.5	2012
13.83	3.5	2012
14.00	3.5	2012
14.17	3.5	2012
14.33	3.6	2070
14.50	3.8	2185
14.67	3.8	2185
14.83	3.6	2070
15.00	3.6	2070
15.17	3.8	2185
15.33	3.8	2185
15.50	3.8	2185
15.67	3.8	2185
15.83	3.8	2185
16.00	3.5	2012
16.17	3.7	2127
16.33	3.8	2185
16.50	4.0	2300
16.67	4.1	2357
16.83	4.2	2415
17.00	4.3	2472
17.17	4.4	2530
17.33	4.4	2530
17.50	4.1	2357
17.67	3.9	2242
17.83	3.9	2242
18.00	4.1	2357
18.17	4.0	2300
18.33	4.0	2300
18.50	4.0	2300
18.67	3.8	2185
18.83	3.9	2242
19.00	4.1	2357
19.17	4.2	2415
19.33	3.9	2242
19.50	3.9	2242
19.67	4.0	2300
19.83	4.0	2300
20.00	4.1	2357
20.17	4.3	2472
20.33	4.4	2530
20.50	4.4	2530
20.67	3.8	2185
20.83	3.7	2127
21.00	3.7	2127
21.17	3.9	2242
21.33	3.8	2185
21.50	3.7	2127
21.67	3.6	2070
21.83	3.8	2185
22.00	3.9	2242
22.17	4.0	2300
22.33	4.4	2530
22.50	4.5	2587
22.67	4.6	2645
22.83	4.7	2702
23.00	4.6	2645
23.17	4.8	2760
23.33	4.8	2760
23.50	4.3	2472
23.67	4.3	2472
23.83	4.4	2530
24.00	4.2	2415
24.17	4.2	2415
24.33	4.2	2415
24.50	4.3	2472
24.67	4.5	2587
24.83	4.7	2702
25.00	4.8	2760
25.17	5.0	2875
25.33	4.8	2760
25.50	4.7	2702
25.67	5.0	2875
25.83	4.6	2645
26.00	4.9	2817
26.17	5.1	2932

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 9°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	12.8	7359
13.33	13.3	7647
13.50	13.6	7819
13.67	13.7	7877
13.83	14.1	8107
14.00	14.2	8164
14.17	14.4	8279
14.33	14.8	8509
14.50	14.9	8567
14.67	15.3	8797
14.83	15.5	8912
15.00	15.6	8969
15.17	16.0	9199
15.33	16.4	9429
15.50	16.7	9602
15.67	17.2	9889
15.83	17.6	10119
16.00	18.0	10349
16.17	18.2	10464
16.33	18.7	10752
16.50	19.1	10982
16.67	19.6	11269
16.83	20.4	11729
17.00	20.9	12017
17.17	21.1	12132
17.33	21.7	12477
17.50	22.4	12879
17.67	22.7	13052
17.83	23.4	13454
18.00	23.9	13742
18.17	24.1	13857
18.33	24.5	14087
18.50	25.0	14374
18.67	25.5	14661
18.83	26.3	15121
19.00	26.7	15351
19.17	27.2	15639
19.33	27.9	16041
19.50	28.6	16444
19.67	29.1	16731
19.83	29.7	17076
20.00	30.4	17479
20.17	30.9	17766
20.33	31.5	18111
20.50	32.2	18514
20.67	32.8	18859
20.83	33.5	19261
21.00	34.2	19664
21.17	34.8	20009
21.33	35.5	20411
21.50	36.4	20929
21.67	37.0	21274
21.83	37.8	21733
22.00	38.6	22193
22.17	39.2	22538
22.33	39.9	22941
22.50	40.6	23343
22.67	41.4	23803
22.83	42.0	24148
23.00	42.8	24608
23.17	43.5	25011
23.33	44.1	25356
23.50	45.0	25873
23.67	45.7	26276
23.83	46.3	26621
24.00	47.2	27138
24.17	47.9	27541
24.33	48.6	27943
24.50	49.3	28346
24.67	50.1	28805
24.83	50.6	29093
25.00	51.4	29553
25.17	52.2	30013
25.33	52.8	30358
25.50	53.6	30818
25.67	54.3	31220
25.83	54.9	31565
26.00	55.7	32025
26.17	56.6	32543

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 40°F
Spindle: Vane
Model: LV

Cum. Time minutes	Torque %	Stress (dyne/cm)

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 40°F
Spindle: Vane
Model: RV

Cum. Time minutes	Torque %	Stress (dyne/cm)

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 20°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
26.33	5.4	3105
26.50	5.7	3277
26.67	5.9	3392
26.83	6.1	3507
27.00	6.3	3622
27.17	6.5	3737
27.33	6.7	3852
27.50	6.8	3910
27.67	6.9	3967
27.83	6.8	3910
28.00	6.8	3910
28.17	6.8	3910
28.33	7.0	4025
28.50	7.3	4197
28.67	7.3	4197
28.83	7.4	4255
29.00	7.6	4370
29.17	7.7	4427
29.33	8.0	4600
29.50	8.2	4715
29.67	8.4	4830
29.83	8.7	5002
30.00	9.1	5232
30.17	9.1	5232
30.33	9.4	5405
30.50	9.8	5635
30.67	10.0	5750
30.83	10.2	5865
31.00	10.6	6095
31.17	10.8	6210
31.33	11.2	6440
31.50	11.7	6727
31.67	12.1	6957
31.83	12.6	7244
32.00	13.2	7589
32.17	13.7	7877
32.33	14.1	8107
32.50	14.6	8394
32.67	15.1	8682
32.83	15.7	9027
33.00	16.4	9429
33.17	16.9	9717
33.33	17.3	9947
33.50	17.9	10292
33.67	18.4	10579
33.83	18.9	10867
34.00	19.6	11269
34.17	20.2	11614
34.33	20.6	11844
34.50	21.3	12247
34.67	22.0	12649
34.83	22.4	12879
35.00	23.1	13282
35.17	23.9	13742
35.33	24.4	14029
35.50	25.2	14489
35.67	26.0	14949
35.83	26.6	15294
36.00	27.4	15754
36.17	28.1	16156
36.33	28.7	16501
36.50	29.5	16961
36.67	30.3	17421
36.83	30.9	17766
37.00	31.7	18226
37.17	32.4	18629
37.33	33.1	19031
37.50	33.8	19434
37.67	34.6	19894
37.83	35.3	20296
38.00	35.9	20641
38.17	36.7	21101
38.33	37.4	21504
38.50	38.0	21848
38.67	38.8	22308
38.83	39.5	22711
39.00	40.0	22998
39.17	40.7	23401
39.33	41.4	23803

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature : 9°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
26.33	57.2	32888
26.50	57.9	33290
26.67	58.8	33808
26.83	59.4	34153
27.00	60.1	34555
27.17	61.0	35073
27.33	61.7	35475
27.50	62.3	35820
27.67	63.2	36337
27.83	63.9	36740
28.00	64.6	37142
28.17	65.5	37660
28.33	66.2	38062
28.50	66.9	38465
28.67	67.8	38982
28.83	68.5	39385
29.00	69.2	39787
29.17	70.0	40247
29.33	70.8	40707
29.50	71.4	41052
29.67	72.2	41512
29.83	73.0	41972
30.00	73.6	42317
30.17	74.4	42777
30.33	75.1	43179
30.50	75.7	43524
30.67	76.5	43984
30.83	77.2	44387
31.00	77.8	44732
31.17	78.6	45192
31.33	79.3	45594
31.50	79.9	45939
31.67	80.7	46399
31.83	81.4	46802
32.00	81.9	47089
32.17	82.6	47492
32.33	83.3	47894
32.50	83.7	48124
32.67	84.1	48354
32.83	84.8	48757
33.00	85.5	49159
33.17	86.1	49504
33.33	87.0	50022
33.50	87.8	50481
33.67	88.5	50884
33.83	89.3	51344
34.00	90.1	51804
34.17	90.8	52206
34.33	91.6	52666
34.50	92.5	53184
34.67	93.1	53529
34.83	93.9	53989
35.00	94.8	54506
35.17	95.4	54851
35.33	96.3	55369
35.50	97.1	55829
35.67	97.6	56116
35.83	98.4	56576
36.00	99.3	57094
36.17	99.9	57439
36.33	100.0	57496

Appendix B

Standard Laboratory Procedure

(SLP-307)

"Crude Oil Yield Point Determination By Vane Viscometry"

Subject: **Crude Oil Yield Point Determination by Vane Viscometry**	Document No: SLP-307
Date: July 31, 2000	Revision 3	Page 1 of 13

Prepared by: Neal Magri
Applicable to: Westport Technology Center International

Technical Review by: Bayram Kalpakci	**Date: July 31, 2000**
Safety Review by: Robert Jaros	**Date: July 31, 2000**
Quality Assurance Review: John Shillinglaw	**Date: July 31, 2000**

Scope: This test method describes the use of the Brookfield viscometer for the determination of the yield point of crude oils.

Safety Precautions: Approved safety glasses with side shield and protective clothing must be worn at all times in the laboratory. Protective gloves are required to be worn when handling crude oil and solvents. Keep away heat, sparks and open flame. Use only with adequate ventilation, (i.e. sampling performed within a fume hood if possible). Avoid contact with skin, eyes and clothing. Avoid breathing mist or vapor. Keep containers closed. Open containers with caution.

Important: Crude oil and container will be hot after the initial heating during the beneficiation process. Handle and dispose of syringes and needles properly. Empty containers may contain toxic, flammable/combustible or explosive residue or vapors. Do not cut, grind, drill, weld, reuse or dispose containers unless adequate precautions are taken against these hazards. Observe ALL PRECAUTIONARY LABELING.

Hazard: **CRUDE OIL**, Vapors may be harmful. Possible aspiration hazard if swallowed, can enter lungs and cause damage. May be irritating to the skin, eyes and respiratory tract. May release toxic hydrogen sulfide vapors. Skin cancer hazard based on tests with laboratory animals. Contains **BENZENE**—a cancer hazard. Extremely flammable liquid. Vapor may cause flash fire.

Reference Documents: ASTM D2983 Standard Test Method for Low-Temperature Viscosity of Automotive Fluid Lubricants; Westport Standard Procedure SLP-305 Rheological Properties of Crude Oils by Rotational Viscometer; Brookfield Digital Viscometer Model DV-II+ Version 2.0, Operating Instructions, Manual No. M/92-161-F1193; Brookfield WinGather Software, Operating Instructions, Manual No. M/95-320-C398; Operations and Programming Manual for Sigma Systems M26-C3 Environmental Chamber with Programmable Temperature Controller / Model CC-3. Dzuy, N.Q., Boger, D.V., "Yield Stress Measurement for Concentrated Suspensions", J. Rheology, 27 (4), 321-9 (1983).

Summary of Test Method

This test method consists of determining the yield point of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom). The crude oil is initially heated to 150°F to destroy all temperature and shear histories and then cooled to 90°F at which point it is loaded into the cup apparatus. The cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the sample is cooled in an environmental chamber at a controlled rate to -20°F. The cooling rate mimics the expected rate of cooling of the pipeline oil in the case of shut-in.

Samples are withdrawn from the environmental chamber at 10 test temperatures (approximately 80, 65, 50, 40, 20, 10, 0, -10, -15 and -20°F) and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle is attached to the Brookfield viscometer before the spindle clamping mechanism is released. The clamping mechanism is released and the viscometer is started at 0.01 rpm and torque as a function of time is measured, at least until a maximum reading is obtained. The maximum torque obtained is divided by a vane parameter constant K to obtain the yield stress. The constant K is calculated based on the dimensions of the vanes. ($K=36.19\ cm^3$, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm))

Significance and Use

The test method is used for determining the yield point of a cooled crude oil, with or without aging. This determination will be made with vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. The method will determine the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent viscosity of the fluid after initiation of flow. These data can be directly used in modeling of crude oil behavior in pipelines, during start-up conditions.

Equipment Required

- Certified rotational-type viscometers capable of a minimum rotational speed of 0.01 rpm such as: the Brookfield Viscometer, Model LV DVII+, RV DVII+ or HB DVII+ having the capability of 20 speeds when programmed accordingly.
- Thermometer, Fluke digital thermometer which has been certified to ±0.3°C from –40°C to 100°C and ±2.0°C from –50°C to –100°C, using standards traceable to the National Institute of Standards and Technology or the National Physical Laboratory or using natural physical constants or ratio calibration techniques.
- Westport vane viscometry cup apparatus and vane spindle for the Brookfield viscometer.
- 100 ml glass syringe, and syringe needle equipped with valve.
- Sigma System M26-CC3 environmental chamber, equipped with a liquid nitrogen supply for cooling.
- Temperature controlled refrigerated circulator bath, such as a Julabo FP-50 series.

PROCEDURES

A. Calibration of Apparatus

Calibration as it is normally understood, carrying out an experimental measurement with a standard material, in the same manner as for the unknown sample, does not apply to vane viscometry: there are no standards. However, as received, from the factory the Brookfield viscometers are certified to give accurate speed and percent torque readings, and it is these two measurements which are critical to vane viscometry.

Tests of consistent viscometer response will be carried out as verification that the viscometer is in good working order. Testing will be carried out for each viscometer when used in each experimental set (an experimental set entails the testing of all samples cooled in one set in an environmental chamber, generally a population size of 24).

1.0 Procedure

1.1 Level the Brookfield viscometer, and the plastic bracket to hold the vane apparatus using the spirit levels attached to them.
1.2 Take an empty vane apparatus, with the top removed but with the spindle locked, and place the apparatus in the refrigerated circulator, temperature is not critical at this step.
1.3 The vane apparatus is held in place by a plastic bracket; make certain the apparatus is placed as far to the right as possible and as far towards the back of the bath as possible.
1.4 Tighten the clamp that holds the vane apparatus in the bracket.
1.5 Attach the alignment rod to the Brookfield viscometer. Move the Brookfield viscometer vertically and horizontally until the alignment rod fits into the top of the vane spindle (without lateral motion).
1.6 Remove the alignment rod and screw on the S hook attachment to the Brookfield viscometer.
1.7 Loosen the clamp that holds the vane apparatus in the bracket.
1.8 Attach the vane spindle to the Brookfield viscometer as shown in Figure 1.
1.9 Raise the Brookfield viscometer until all slack is taken out of the connections to the vane spindle but the vane spindle is not lifted off its plastic support bracket.
1.10 Rotate the vane apparatus until the torque reading is less than 0.05%. It is best not to start with negative torque readings as these are not recorded by the WinGather program.
1.11 Tighten bracket that holds the vane apparatus in place, using the knob in the top right corner of the bracket.
1.12 Start the WinGather program for timed torque readings. Time between readings will be 10 seconds for a speed of 0.01 rpm.

As the Brookfield software does not recognize the vane spindle, spindle input is not necessary. Time, torque and temperature readings, which are independent of spindle, are the only data that will be used.

The WinGather program is Version 1.1 from Brookfield Engineering Laboratories, Inc., located at 11 Commerce Blvd., Middleboro, MA 02346. See referenced manual for further WinGather information. The manual and a copy of the software will be archived at Westport Technology Center.

1.13 Start the Brookfield motor with the locked spindle mechanism in place.

1.14 Continue the test run until the torque reading goes off scale.
1.15 Save the WinGather data file and record the data file name.

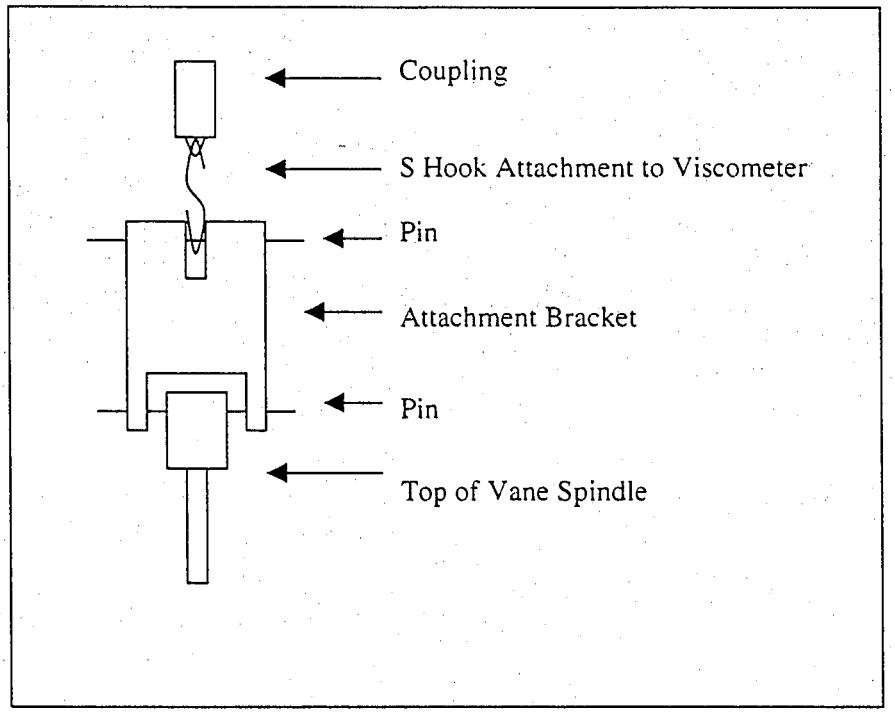

Figure 1. Attachment of Vane Apparatus to Viscometer

2.0 Acceptable Calibration

Differences in torque versus time measurements will include variations in viscometer speed and torque measurement. For purposes of measuring yield stress, variances in viscometer speed are not critical. Speed is important however for determining slippage of the sample at the yield point. A torque versus time run will be acceptable if it varies no more than 10% of the maximum torque of the viscometer, at any point in the experimental run.

B. Verification of Cooling Rate

1.0 Data Recording

Temperatures will be measured by the environmental chamber thermocouple and recorded by a YEW Model 3088 Hybrid Recorder (chart recorder). The chart recorder will print time and temperature at 0:00 and 12:00 each day, in addition to a continuous trace of the temperature. This temperature recording procedure has been verified against a certified Fluke thermometer and found to be within

±1°F. The temperature recording of the environmental chamber and Yew recorder will be re-verified each time the Fluke digital thermocouple meters are re-certified.

1.1 At the end of each vane experimental run, when all samples originally placed in the environmental chamber have been tested or disposed of, remove the chart recorded paper from the recorder. This data is to be saved, and archived with other items for this test method, such as laboratory notebooks.

1.2 The chart recording should be inspected to ascertain if there are any significant anomalies, such as rapid, transitory increases or decreases in temperature.

1.3 Printed temperatures from the chart recorder should be recorded and contrasted with target temperatures as part of final reporting.

Table 1. Times and Target Temperatures

Day (24 hrs)	Target Temperature (°F)
0	90
2.5	70
5.0	50
7.5	35
10	20
12.5	10
15	0
17.5	-10
21	-20

C. Preparation of Sample

The solubility of paraffins in crude oil decreases with decreasing temperature. As an oil cools past its wax appearance temperature, regardless of the rate at which the oil was cooled, significant amounts of paraffin may precipitate. However, the rheological properties of the precipitated wax are highly dependent upon the shear and temperature history of the oil above and below the wax appearance temperature. The initial step in determining yield stress of an oil is to heat the sample to 150°F and hold the oil at that temperature for at least 2 hours, to destroy all temperature and shear histories. It is important to make certain the oil container is tightly closed during this initial conditioning, as loss of light ends through evaporation may significantly increase yield stress.

Following the initial heating of the oil, allow the oil to cool to 90°F in a Sigma environmental chamber. At this point the oil containers, the vane apparatuses and the 100-ml glass syringe for oil transfer are thermally equilibrated in the environmental chamber at 90°F. Allow at least an hour for all materials to reach temperature.

D. Loading of Vane Apparatus

A 100-ml glass syringe equipped with needle and valve is used to load the oil sample into the vane apparatus.

19. Save the WinGather data file and record the data file name.
20. At test completion, insert certified Fluke thermocouple into vane apparatus and take a final sample temperature.
21. Repeat the test as stated:
    - Under test conditions where (possibly above the oil's WAT) observed torque readings are below 10% full scale on the LV viscometer (lowest torque spring viscometer) only test one sample.
    - If torque readings are above 10% full scale on the LV, then run a second sample for repeatability. If the two test results are not within 10%, run a third sample for precision purposes.
    - Three samples should always be tested at the lower temperatures if test samples are available. If necessary, for precision statements, repeat additional times.

G. Data Recording

Initiate Data Verification and Validation Checklists. Data recording, which will include quality assurance tests such as calibration and data checks, will be achieved using the enclosed vane viscometry data sheets.

Data Sheet 1 (DS1-307) includes recording of data when all the samples, for an entire tests, are prepared, transferred to their respective vane apparatuses, cooled to test temperature and aged.

The data to be recorded include:

- Test Number; Designate the test by number so that subsequent individual vane data sheets (DS2-307) can be traced to the parent data sheet. (i.e. IDC Test 1, Phase 1–Test 1...)
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Sample Benefication; Record temperature treatment information for the samples before transfer to the vane apparatuses.
- Transfer of Samples; Record start and stop times for transfer of all samples from original bottles to vane apparatuses to environmental chambers.
- Cooling/Aging of Sample; Record temperature of environmental chamber when samples are first introduced, also record target temperature (normally –20°F) and the target period of time for cooling samples to that temperature (normally 21 days). Temperature/time measurements will be recorded by the Yew hybrid chart recorder.

Data are to be recorded for each individual sample withdrawn and tested from the environmental chambers on vane viscometry Data Sheet 2 (DS2-307). Record data including:

- Test Number; the same number as used on DS1-307, establishes tracking of DS2-307 sheets.
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Testing; date and time of sample withdrawal, the temperature of the environmental chamber, the name of the WinGather data file where testing data is stored, the type of viscometer (LV, RV or HB) and the speed (rpm) at which the test was carried out (normally 0.01).

- Data Check; data for the vane testing will be recorded using the WinGather program. The data check section of the data sheet will be used for quality assurance of this WinGather data. During testing (at 5 to 10 minute intervals), record the percent full scale (torque) reading directly from the Brookfield viscometer display, and record the data point of the WinGather program where this data was recorded electronically.
- Maximum Torque; Record the observed highest percent full-scale reading (torque).
- Data Points of Maximum Torque; Record the approximate range of WinGather points during maximum torque (i.e. numbers 73-78).
- Final Oil Temperature; Record the sample temperature after completion of test.

H. Calculation and Interpretation of Results

The experimental test is designed to produce direct readings. temperature and %torque, as data.

Calculation of torque and yield stress.

1. Calculate torque readings from the percent of full-scale readings recorded by the WinGather software by multiplying the percent full-scale reading by 6.733 dyne-cm for the LVDV-II+ viscometer, or 71.87 dyne-cm for the RVDV-II+ viscometer, and 574.96 for the HBDV-II+ viscometer. Torque data may be interpreted by graphing the torque versus time readings obtained during testing. Determine a yield point from this data by observing where a maximum torque reading had been obtained, followed by a decrease in torque reading over time. Calculate the Yield Stress by the following equation;

Yield Stress = Maximum Torque Obtained (dyne-cm)/ K (36.19 cm^3)

I. Reporting

1. Report the following information:
   - 1.1 Completed and signed Data Verification and Validation Checklists,
   - 1.2 Date of test,
   - 1.3 Sample Identification,
   - 1.4 Cooling time for sample,
   - 1.5 Aging/testing temperature in degrees Fahrenheit (environmental chamber temp.),
   - 1.6 Final oil temperature at end of test (measured directly in cup),
   - 1.7 Viscometer speed,
   - 1.8 Maximum torque reading,
   - 1.9 Yield Strength versus Temperature,
   - 1.10 Locked Spindle Torque versus Time Curve,
   - 1.11 Time versus torque reading for each test will be reported graphically,
   - 1.12 Numerical data for the graph,
   - 1.13 Combined Plot of all Torque vs. Time Curves on one Log Scale Plot,
   - 1.14 When multiple oils are analyzed, plot all Yield Strength vs. Temperature curves on one Lop Scale Plot.

J. Precision

Precision – See Section A2 for precision during QC/QA calibration checks. Initial Demonstration of Capability for test resulted in the following:

Determinability (d) – Measurements are performed on individual oil samples, taken at selected temperatures, through a maximum shear condition which destroys any wax structure present. No attempt was made to duplicate measurements on the same oil sample within the test cell; the results would be misleading. Therefore, no statement of determinability can be made on.

Repeatability (r) - The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of this method, have a relative standard deviation at or below 15%.

Reproducibility (R) - The Brookfield viscometers are a very common apparatus for measuring rheological properties. However, with the Vane cup and spindle apparatus design and modifications for low temperature testing of the TAPS/COS samples, no statement of reproducibility by other independent laboratories can be made.

Data Sheet 1 for Vane Viscometry

Form: DS1-307

Heating, Transfer, Cooling, and Aging for Sample Sets

Test Number:	
Sample Description:	

Sample Beneficiation			Transfer of Samples
Date:	Start Time:		Start Time:
Temp.:	Stop Time:		Stop Time:

Cooling/Aging of Samples

Chamber Temp.:		Target Temp.:	
Ramp Start Time		Target Time:	

Sample Identification

Chamber 1		Chamber 2	
Vane No.	Sample ID	Vane No.	Sample ID

Analyst Signature:_____

Subject:	QA Procedure for Crude Oil Quantification by Capillary Gas Chromatography
Date:	January 4, 2001

Prepared by: Ray Collins	Date: January 4, 2001

Safety Review by: Robert Jaros	Date: January 4, 2001

Scope: This procedure gives quantitative compositions of crude oils and condensates utilizing capillary gas chromatography (CGC).

Safety Precautions: Approved safety glasses with side shield and protective clothing must be worn at all times in the laboratory. Protective gloves must be worn when handling crude oil and solvents. Keep away heat, sparks, and open flame. Use only with adequate ventilation, (i.e. sampling performed within a fume hood if possible). Avoid contact with skin, eyes, and clothing. Avoid breathing mist or vapor. Keep containers closed. Open containers with caution.

Calibration Standards: Prudhoe Bay oil, Identifier: Reference "C".
Colombian oil, Identifier: Reference "W".
D-2887 Reference Gas Oil, Identifier: RGO.

PROCEDURES

Sample Preparation

The sample used in CGC analysis must be free of water and solids. One to 1.3 grams of sample is weighed into a 2ml auto-sampler vial and then a fixed known amount of internal standard is weighed into the sample. The sample is then thoroughly mixed to ensure sufficient mixing of sample and internal standard. Carbon disulfide is added to viscous samples that are too thick to be drawn into a syringe.

Gas Chromatograph Preparation

Prior to analyzing samples, the gas chromatograph (GC) is heated to the columns upper temperature limit to remove contaminants and stabilize the baseline. The inlet septum is changed weekly to reduce septum bleeding.

Calibration of GC Apparatus

The GC is a computer controlled, method-driven gas chromatograph. Internal testing of the instrument functions is carried out upon startup. The standard calibration curve is determined by repeated analyses of Prudhoe Bay (Ref "C") crude oil standard. Accepted values (+/- 3 percent

per component – upper and lower control limits) are plotted versus the results of the Ref "C" analysis that was treated as an unknown sample (see Attachment-1). Similar plots of the D-2887 Reference Gas Oil, Reference "W", and Ref "C" with carbon disulfide standard are plotted when used. If two or more points of the results from the analysis are outside of the control limits, the GC must be undergo maintenance to restore acceptable capabilities.

GC Sequences

A sequence of samples run on the GC consists of a suite of standard blanks and at least one unknown sample. The order of an extended sequence is given on the right. The standards analyzed at the beginning of the sequence are treated as unknowns. If the resultant CGC calculation agrees with the results in Attachment-1, the instrument is deemed to be within calibration specifications and the remainder of the sequence can proceed. The intermediate standards run at the end of each subsequence are similarly treated as unknowns. If they are out of calibration, the sequence is terminated and the preceding subsequence is re-run. If the intermediate standard is acceptable (results within the criteria specified), the sequence proceeds to the next subsequence. The ending standard is treated in the same way as an intermediate standard (as an unknown sample).

Table 2: Test Sequence

Blank
Blank
Standard
Subsequence 1
- Sample 1
- Sample 2
- ...
- Sample 10

Standard
Subsequence 2
- Sample 11
- Sample 12
- ...

Ending Standard

The ending standard is a discrimination standard, which consists of normal paraffins ranging from C-13 through C-30; this standard is used to verify the GC's suitability for the next sequence of samples by comparing the relative response factors of the normal paraffins that must be between 0.95 and 1.05. If the factors fall out of acceptable region, then corrective maintenance must be performed on the injector system and the GC must be re-tested with the discrimination standard.

Data Acquisition

Agilent Technology's Chemstation is used to control and acquire the gas chromatographic data. The Chemstation software provides accurate pressure control to ensure repeatable retention times from sequence to sequence. To provide additional retention time repeatability, Retention Time Locking software is used to lock the retention time base on a known compound. These capabilities ensure correct identification of compounds.

Attachment 1