

**Test Monitoring Center** 

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### ASTM D02.B0.07 Semi-Annual Report Bench Test Monitoring

# D6417, D5133 (GI), D5800, D6335 (TEOST), D7097(MTEOS), D6082, D874 and D7528 (ROBO)

April 2014

### D6417 (Volatility by GC)

- Degraded precision for a second consecutive period
  - Less precise than target precision
  - Mostly due, this period, to repeat fails on one instrument
- Performance 0.42 s severe





### D5800 (Volatility by Noack)

- New reference oils, replacing old oils, introduced coincident with start of this TMC report period
- Precision improved compared to prior period and comparable to NEW target precision
- Long-term severe trend with increase in severity since 01JUL06
  - Period severity on new oils is still severe (0.37 s), but less severe than prior periods using old (replaced) reference oils
  - Period severity on two of the three new oils is nearly 0.5 s severe (overall performance on third new oil is on-target)





### D5133 (Gelation Index)

- Degraded precision
  - More precise than target precision
- Performance –0.18 s severe
- Severe performing Oil 62 more than 1 s mild of oil target





- D6335 (TEOST-33C)
  - Precision (Pooled s) is more precise than prior period
    - Less precise than the updated target precision
  - Performance (Mean  $\Delta/s$ ) is nearly on target at -0.14 s mild
- D7097 (MHT-4 TEOST)
- Precision (Pooled s) is less precise than prior periods
  - Significantly less precise than target precision
- Performance (Mean  $\Delta$ /s) is on target
  - Significant lab performance differences noted
  - Catalyst affects on oil severity are also indicated
  - Overall severe performance of oil 432 (0.49 s, n=36) is nearly offset by overall mild performance of oil 434 (-0.34 s, n=35)



#### D6082 (High Temperature Foam)

- More precise than target precision
- Performance -0.39 s mild
- All operationally valid discrimination runs demonstrated acceptable discrimination

### D874 (Sulfated Ash)

- More precise than target precision
- Performance on target (0.00 s)
- No issues



- <u>D7528</u> (ROBO)
- Precision (Pooled s) is less precise than prior period
  - Less precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.43 s mild
  - Two labs on target, overall (Labs AM & G)
  - Two labs more than 1 s mild, overall (Labs AQ & B)
  - Precision on Oil 438 is degraded significantly
    - Partly due to two fails 4 s or more severe, reported as operationally valid
- CUSUM Severity Plot shows an ongoing overall mild trend since the 01APR11 timeline



## **Calibrated Labs and Stands\***

Test	Labs	Stands
D6417	5	7
D5800	8	17
D5133 (GI)	4	5
D6335 (TEOST)	4	6
D7097 (MTEOS)	7	31
D6082	4	5
D874	3	
D7528 (ROBO)	6	15

\*As of 3/31/2014

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# D02.B0.07 **TMC** Monitored Tests

### >>> October 1, 2013 –

March 31, 2014



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	13
Failed Calibration Test	OC	2
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Donated Screener Tests	AG	8
Total		23

Number of Labs Reporting Data: 5 Fail Rate of Operationally Valid Tests: 13%



Statistically Unacceptable Tests (OC)	No. Of Tests
Volatility Loss Mild	1
Volatility Loss Severe	1

- Both the mild and severe failing results were on same instrument (B1) and significantly affect the overall precision this period.
- No TMC technical updates issued this period





Period Precision and Severity Estimates

				Mean
Area % Volatized @ 371°C	n	df	Pooled s	$\Delta/s$
Initial Selected Oils from RR	54	51	0.39	
10/1/10 through 3/31/11	20	17	0.38	0.06
4/1/11 through 9/30/11	16	13	0.37	0.21
10/1/11 through 3/31/12	14	11	0.24	0.17
4/1/12 through 9/30/12	15	12	0.28	-0.19
10/1/12 through 3/31/13	14	11	0.22	0.05
4/1/13 through 9/30/13	17	14	0.56	0.17
10/1/13 through 3/31/14	15	12	0.66	0.42



### D6417 Precision Estimates

#### Area % Volatized @ 371°C Pooled s





## D6417 Severity Estimates





Current Period Severity Estimates by Lab Area % Volatized @ 371°C

	n	Mean $\Delta$ /s
Lab A	4	0.78
Lab B	3	0.86
Lab D	4	-0.34
Lab G	2	0.11
Lab S	2	0.86



## D6417 Lab Severity Estimates

Area % Volatized @ 371°C

Mean  $\Delta/s$ 



- Precision (Pooled s) is degraded for second consecutive period
  - Less precise than the target precision
  - Due to two consecutive fails on same instrument (B1), one -3.7 s mild and one 5.6 s severe, both on oil 58, both reported as operationally valid; lab passed on third calibration attempt.
- Performance (Mean  $\Delta/s$ ) is severe (0.42 s)
- Cusum plot shows variability and overall severe performance for this period.





- Eight non-calibrating tests were donated by two labs to screen oils for potential use as replacement reference oils.
  - The performance of the screened oils was unsuitable for our needs and further scouting for replacement D6417 reference oils continues.





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#### D6417 VOLATILITY BY GC INDUSTRY OPERATIONALLY VALID DATA



#### SAMPLE AREA % VOLATIZED



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#### Area % Volatized @ 371°C Performance by Oil

		Targets		10/1/12 - 3/31/13		4/1/13 - 9/30/13			10/1/13 - 3/31/14						
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
52	18	6.97	0.31	5	6.8	0.07	-0.55	4	6.8	0.34	-0.47	4	6.9	0.22	-0.23
55	18	11.68	0.51	4	12.0	0.36	0.63	8	11.9	0.74	0.36	5	11.9	0.51	0.47
58	18	5.61	0.30	5	5.7	0.17	0.19	5	5.7	0.23	0.37	6	5.8	0.90	0.80



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## D6417 Performance by Oil

#### Area % Volatized @ 371°C

Mean



## D6417 Performance by Oil

#### Area % Volatized @ 371°C

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## D6417 Performance by Oil

Area % Volatized @ 371°C

Mean  $\Delta/s$ 



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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	33
Failed Calibration Test	OC	5
Operationally Invalidated by Lab	LC, XC	1
Operationally Invalidated After Initially Reported as Valid	RC	0
Decoded for Shakedown	NN	2
Excluded For Other Reasons	NN	1
Total		42

Number of Labs Reporting Data: 8 Fail Rate of Operationally Valid Tests: 13%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Evaporation Loss Mild	1
Evaporation Loss Severe	4

- Failing results are across labs, instruments and oils.
- Reason for operationally invalid result:
  Daily calibration was not run in prior 48 hours

 One test (NN) on a new instrument (A10) reported as operationally valid but failed statistically, held out of statistics because instrument had not demonstrated a passing calibration. Subsequent run passed calibration.

 Two tests (NN) were run on decoded samples to troubleshoot instrument problems.
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- One TMC Technical Update was issued this report period:
  - TMC Memo 13–049, Issued October 4, 2013
    - New Reference Oils, Effective September 17, 2013
    - Replaced referenced oils 52, 55 and 58 with oils VOLC12, VOLD12, VOLE12, with all new performance targets and acceptance bands
- One operationally and statistically valid test this period was run on oil 58, all others are on the new reference oils
  - The single oil 58 result is included in this period's overall statistics, but excluded from 'by oil' statistics





Period Precision and Severity Estimates

Sample Evaporation Loss, mass %	n	df	Pooled s	Mean ∆/s
NEW Targets Effective 10/1/2013	78	75	0.50	
4/1/11 through 9/30/11	39	36	0.59	0.77
10/1/11 through 3/31/12	32	29	0.78	0.54
4/1/12 through 9/30/12	33	30	0.67	0.56
10/1/12 through 3/31/13	33	30	0.79	0.43
4/1/13 through 9/30/13*	30	27	0.72	0.58
4/1/13 through 9/30/13*	27	24	0.46	0.31
10/1/13 through 3/31/14	38	34	0.59	0.37

\*Period statistics with 3 severe results on same instrument included and excluded



### Performance Comparison by Procedure Sample Evaporation Loss, Mass %

	n	df	Pooled s	Mean ∆/s
Procedure B	32	28	0.61	0.46
Procedure C	6	3	0.56	-0.12





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### **D5800 Precision Estimates**

#### Sample Evaporation Loss, mass % Pooled s





### **D5800 Severity Estimates**

# Sample Evaporation Loss, mass % Mean $\Delta/s$



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Current Period Severity Estimates by Lab Sample Evaporation Loss, mass %

	n	Mean $\Delta$ /s
Lab A	10	1.03
Lab B	11	0.43
Lab D	2	0.90
Lab F	4	-0.04
Lab G	3	-0.38
Lab I	3	1.59
Lab J	4	-1.27
Lab V	1	-1.22



## **D5800 Lab Severity Estimates**

Sample Evaporation Loss, mass %

Mean  $\Delta/s$ 



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Test

- Precision (Pooled s) is more precise than prior period but slightly less precise than new target precision.
- Performance (Mean  $\Delta/s$ ) is 0.37 s severe using new oil targets.
  - Improved over past performance on old targets
  - However, period severity on two of the three new oils is nearly 0.5 s severe (overall performance on third new oil is on-target)
- Severity plot shows unexplained long-term severe trend since 01JUL06 timeline
  - Appears to have some improvement since new targets were set; slight lag as new oils were still being shipped to labs after new targets effective date.





#### D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



#### **EVAPORATION LOSS, MASS%**



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#### D5800 VOLATILITY BY NOACK INDUSTRY OPERATIONALLY VALID DATA



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#### Sample Evaporation Loss, mass % Performance by Oil

	Targets			10/1/12 - 3/31/13				4/1/13 – 9/30/13				10/1/13 – 3/31/14			
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
52	33	13.75	0.61	12	14.7	0.92	1.49	9	14.3	0.70	0.96				
55	32	17.09	0.76	10	17.0	0.76	-0.12	7	17.6	0.83	0.61				
58	37	15.20	0.72	11	15.0	0.65	-0.23	14	15.4	0.67	0.32				
VOLC12	24	14.19	0.40									14	14.4	0.54	0.42
VOLD12	27	12.52	0.52									11	12.9	0.57	0.59
VOLE12	27	16.74	0.55									12	16.7	0.66	0.00


### D5800 Performance by Oil

#### Sample Evaporation Loss, mass %

Mean



# D5800 Performance by Oil

#### Sample Evaporation Loss, mass %

**S**<sub>R</sub>



## D5800 Performance by Oil

#### Sample Evaporation Loss, mass %

Mean  $\Delta/s$ 



APR '14

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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	13
Failed Calibration Test	OC	1
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		14

Number of Labs Reporting Data: 4 Fail Rate of Operationally Valid Tests: 8%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Gelation Index Mild	1
Gelation Index Severe	0

- No operationally invalid tests reported this period
- No TMC technical updates issued this period



**Period Precision and Severity Estimates** 

				Mean
Gelation Index	n	df	Pooled s	$\Delta/s$
Current Targets 7/15/2003	68	65	2.86	
10/1/10 through 3/31/11	33	30	3.17	-0.53
4/1/11 through 9/30/11	23	20	1.70	-0.25
10/1/11 through 3/31/12	24	21	1.36	0.06
4/1/12 through 9/30/12	24	21	1.88	-0.89
10/1/12 through 3/31/13	22	19	1.86	-0.48
4/1/13 through 9/30/13	19	16	1.15	0.17
10/1/13 through 3/31/14	14	11	1.47	-0.18



#### **D5133 Precision Estimates**

#### Gelation Index

#### Pooled s







### **D5133 Severity Estimates**

#### Gelation Index Mean $\Delta/s$



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#### Current Period Severity Estimates by Lab Gelation Index

	n	Mean ∆/s
Lab A	7	-0.23
Lab B	3	0.27
Lab I	1	-1.21
Lab S	3	-0.16

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### D5133 Lab Severity Estimates

#### Gelation Index Mean $\Delta/s$



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- Precision (Pooled s) is less precise than prior period More precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.18 s mild
- Severe performing Oil 62 is more than 1 S mild of target this period





#### D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA



#### **GELATION INDEX**

CUSUM Severity Analysis



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D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA







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#### Gelation Index Performance by Oil

		Targets			10/1/12 - 3/31/13			4/1/13 – 9/30/13				10/1/13 -	- 3/31/14		
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
58	17	5.8	0.69	7	5.5	0.62	-0.43	6	6.3	0.87	0.75	5	6.3	0.75	0.75
62	35	17.0	3.90	6	15.8	3.38	-0.30	5	14.7	1.78	-0.59	5	12.8	2.24	-1.08
1009	16	7.30	0.68	9	6.9	0.87	-0.64	8	7.4	0.81	0.20	4	7.2	0.68	-0.22

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#### D5133 Performance by Oil Gelation Index

Mean



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#### **Gelation Index**





**Gelation Index** 

Mean  $\Delta/s$ 



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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	14
Failed Calibration Test	OC	2
Operationally Invalidated by Lab	LC, XC	1
Operationally Invalidated After Initially Reported as Valid	RC	2
Decoded for Shakedown	NN	1
Total		20

Number of Labs Reporting Data: 5 Fail Rate of Operationally Valid Tests: 12%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Total Deposits Mild	2
Total Deposits Severe	0

- Three operationally invalid tests reported this period:
  Two invalidated (RC) due to incorrect PID setting after lab was informed of failing calibration result
  One aborted by lab (XC) because of temperature control problem noted during the run
- One decoded run (NN) to troubleshoot instrument following a mild fail (OC); lab subsequently calibrated successfully, but did not invalidate the mild run.

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No TMC technical updates issued this period





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#### **Period Precision and Severity Estimates**

				Mean
Total Deposits, mg	n	df	Pooled s	$\Delta/s$
Updated Targets 20130415	60	58	5.73	
10/1/10 through 3/31/11	14	10	6.25	0.14
4/1/11 through 9/30/11	19	15	6.52	-0.27
10/1/11 through 3/31/12	16	12	8.60	0.37
4/1/12 through 9/30/12	18	15	7.06	0.79
10/1/12 through 3/31/13	22	20	6.22	-1.00
4/1/13 through 9/30/13	17	15	8.38	-0.01
10/1/13 through 3/31/14	16	14	7.76	-0.14



#### **D6335 Precision Estimates**

#### Total Deposits, mg Pooled s







### D6335 Severity Estimates



#### Current Period Severity Estimates by Lab Total Deposits, mg

	n	Mean ∆/s
Lab A	6	-0.78
Lab B	4	1.14
Lab D	2	0.34
Lab G	2	-0.94
Lab V	2	-0.48





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### D6335 Lab Severity Estimates

Total deposits, mg

Mean  $\Delta/s$ 



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- Precision (Pooled s) is more precise than prior period
  - Less precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.14 s mild
- All tests this period report using Rod Batches K or L





#### TEOST-33C INDUSTRY OPERATIONALLY VALID DATA



#### TOTAL DEPOSITS MG

CUSUM Severity Analysis



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#### TEOST-33C INDUSTRY OPERATIONALLY VALID DATA





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#### Total Deposits, mg Performance by Oil

	Та	Targets 20130415			10/1/12 - 3/31/13			4/1/13 – 9/30/13				10/1/13 -	- 3/31/14		
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
435-2	30	26.71	4.76	11	25.1	4.32	-0.65	7	33.2	7.16	1.00	9	27.9	6.70	-0.17
75	30	53.66	6.56	11	47.5	7.66	-1.35	10	48.9	9.10	-0.72	7	53.0	9.00	-0.10





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#### Total Deposits, mg

Mean



#### Total Deposits, mg

S<sub>R</sub>

10.00 9.10 9.00 7.66 8.00 7.16 6.70 6.56 6.00 4.76 Oil 435-2 4.32 4.00 Oil 75 2.00 0.00 Target OCT '13 APR '13 APR '14

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#### Total Deposits, mg

Mean  $\Delta/s$ 



### D7097: Deposits by MHT TEOST

Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	64
Failed Calibration Test	OC	7
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Non-blind Shakedown Run	NN	2
Total		73

Number of Labs Reporting Data: 7 Fail Rate of Operationally Valid Tests: 10%

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### D7097: Deposits by MHT TEOST

Statistically Unacceptable Tests (OC)	No. Of Tests
Total Deposits Mild	0
Total Deposits Severe	7

- No operationally invalid calibration tests this period
- Two decoded runs (NN) to troubleshoot instrument following a severe fail (OC); lab subsequently calibrated successfully, but did not invalidate the severe run.
- No TMC technical updates issued this period





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### D7097: Deposits by MHT TEOST

Period Precision and Severity Estimates

				Mean
Total Deposits, mg	n	df	Pooled s	$\Delta/s$
Current Targets 7/31/2006	90	87	5.63	
10/1/10 through 3/31/11	55	52	7.59	0.27
4/1/11 through 9/30/11	46	43	6.00	0.03
10/1/11 through 3/31/12	56	54	5.88	0.09
4/1/12 through 9/30/12	65	62	5.63	0.26
10/1/12 through 3/31/13	68	66	6.65	1.07
4/1/13 through 9/30/13	85	83	6.86	0.19
10/1/13 through 3/31/14	71	69	7.36	0.08



#### **D7097 Precision Estimates**

#### Total Deposits, mg Pooled s






### **D7097 Severity Estimates**

#### Total Deposits, mg Mean $\Delta/s$



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#### Current Period Severity Estimates by Lab Total Deposits, mg

	n	Mean $\Delta$ /s
Lab A	20	-0.41
Lab AK	4	-0.76
Lab B	17	-0.27
Lab D	9	0.43
Lab G	16	0.89
Lab J	3	1.10
Lab V	2	0.10

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## **D7097 Lab Severity Estimates**

Total Deposits, mg

Mean  $\Delta/s$ 





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Total Deposits, mg Mean  $\Delta$ /s Severity by CATBATCH and Period



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Precision (Pooled s) is less precise than prior period
 Significantly less precise than target precision

- Performance (Mean  $\Delta/s$ ) is nearly on-target at 0.08 s severe
- All completed tests this period report using Rod Batches K or L
- All completed tests this period report using Catalyst Batch 1307





- CUSUM severity plot shows OVERALL severity issues associated with prior catalyst batches seem to be resolved with significant leveling to nearly on target performance since the introduction of catalyst batch 1307
  - However, significant lab performance differences are also observed; overall precision is still quite poor compared to current target precision, and compared to prior periods
  - Overall severe performance of oil 432 (0.49 s, n=36) is nearly offset by overall mild performance of oil 434 (-0.34 s, n=35)
  - Difficult to separate lab differences from catalyst affect on each oil's severity, but catalyst batches have proven to bias performance, and may partially explain observed severity differences by catalyst batch and oil





#### MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA



#### TOTAL DEPOSITS MG

CUSUM Severity Analysis





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#### MHT-4 TEOST INDUSTRY OPERATIONALLY VALID DATA





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# D7097 Performance by Oil

#### Total Deposits, mg Performance by Oil

		Targets		10/1/12 - 3/31/13			4/1/13 – 9/30/13			10/1/13 – 3/31/14					
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
432	30	47.04	4.50	37	54.5	5.75	1.65	44	51.1	7.35	0.91	36	49.2	6.69	0.49
434	30	27.37	6.57	31	29.81	7.60	0.37	41	23.5	6.30	-0.58	35	25.2	8.00	-0.34



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# D7097 Performance by Oil

#### Total Deposits, mg

Mean



#### Total Deposits, mg

S<sub>R</sub>

10.00 8.00 7.60 8.00 7.35 6.69 6.57 6.30 5.75 6.00 Oil 432 4.50 4.00 Oil 434 2.00 0.00 Target **OCT** '13 APR '13 APR '14

#### Total Deposits, mg

Mean  $\Delta/s$ 



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	11
Acceptable Discrimination Test	AS	4
Failed Calibration Test	OC	0
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		15

Number of Labs Reporting Data: 4 Fail Rate of Operationally Valid Tests: 0%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Foam Tendency Mild	0
Foam Tendency Severe	0

No Operationally invalid test reported this period

All operationally valid discrimination runs reported this period could discriminate oil 66 as a GF-5/SN failing oil for Foam Tendency

No TMC technical updates issued this period





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Period Precision and Severity Estimates Oil 1007

Foam Tendency, ml	n	Mean	Pooled s	Mean $\Delta$ /s
Current Targets	28	65.71	19.28	
10/1/10 through 3/31/11	8	61	10	-0.25
4/1/11 through 9/30/11	9	80	26	0.74
10/1/11 through 3/31/12	8	65	13	-0.05
4/1/12 through 9/30/12	9	63	13	-0.14
10/1/12 through 3/31/13	8	58	10	-0.45
4/1/13 through 9/30/13	9	60	7	-0.32
10/1/13 through 3/31/14	11	59	8	-0.39





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Period Precision and Severity Estimates Oil 1007

Foam Stability @ 1 min, ml	n	Mean	S
Current Targets	28	0.00	0.00
10/1/10 through 3/31/11	8	No non-zero d	occurrences
4/1/11 through 9/30/11	9	No non-zero d	occurrences
10/1/11 through 3/31/12	8	No non-zero d	occurrences
4/1/12 through 9/30/12	9	No non-zero d	occurrences
10/1/12 through 3/31/13	8	No non-zero d	occurrences
4/1/13 through 9/30/13	9	No non-zero d	occurrences
10/1/13 through 3/31/14	11	No non-zero d	occurrences



#### Foam Tendency, ml Mean, Oil 1007







#### Foam Tendency, ml s<sub>R</sub>, Oil 1007





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#### Current Period Severity Estimates by Lab Foam Tendency, ml TMC Oil 1007

	n	Mean ∆/s
Lab A	3	-0.67
Lab B	4	-0.45
Lab G	2	-0.05
Lab V	2	-0.18

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Current Period Severity Estimates by Lab Foam Tendency, ml

TMC Oil 1007



- Foam Tendency Precision (Pooled s) is comparable to prior period
  - More precise than target precision
- Performance (Mean ∆/s) is -0.39 s mild
  Comparably mild for last three report periods
- No non-zero occurrences of Foam Stability
- All operationally valid discrimination runs demonstrated acceptable discrimination

Test Monitoring Center



#### D6082 HIGH TEMPERATURE FOAM INDUSTRY OPERATIONALLY VALID DATA



IND='1007' FOAM TENDENCY

CUSUM Severity Analysis



16MAY14:13:17



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#### Foam Tendency, ml Performance by Oil

		Targets			10/1/12 - 3/31/13			4/1/13 – 9/30/13				10/1/13 – 3/31/14			
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
1007	28	65	19	8	58	10	-0.45	9	60	7	-0.32	11	59	8	-0.39

Return to Executive Summary

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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	5
Failed Calibration Test	OC	0
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		5

Number of Labs Reporting Data: 3 Fail Rate of Operationally Valid Tests: 0%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Sulfated Ash Mild	0
Sulfated Ash Severe	0

 No operationally or statistically invalid tests reported this period

No TMC technical updates issued this period





#### Period Precision and Severity Estimates

Total Deposits, mg	n	df	Pooled s	Mean ∆/s
Current Targets	81	78	0.07	
10/1/10 through 3/31/11	6	3	0.05	0.11
4/1/11 through 9/30/11	6	3	0.01	-0.28
10/1/11 through 3/31/12	6	4	0.02	0.25
4/1/12 through 9/30/12*	7	4	0.37	-1.64
4/1/12 through 9/30/12*	6	3	0.04	0.01
10/1/12 through 3/31/13	7	4	0.03	0.14
4/1/13 through 9/30/13	6	3	0.05	-0.12
10/1/13 through 3/31/14	5	2	0.02	0.00

\*Period statistics with and without extreme result included

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#### Sulfated Ash, mass%

#### Pooled s





#### Sulfated Ash, mass%

#### Mean $\Delta/s$



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#### Current Period Severity Estimates by Lab Sulfated Ash, mass%

	n	Mean ∆/s
Lab A	2	0.19
Lab B	2	0.11
Lab G	1	-0.62



# Sulfated Ash, mass% Mean $\Delta/s$



Precision (Pooled s) is comparable to prior periods
 More precise than target precision

• Performance (Mean  $\Delta/s$ ) is on target





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#### D874 INDUSTRY OPERATIONALLY VALID DATA



#### TEST SAMPLE PERCENT SULFATED ASH



16MAY 14:13:19





#### Performance by Oil Sulfated Ash, mass%

	Targets			10/1/12 - 3/31/13				4/1/13 – 9/30/13				10/1/13 – 3/31/14			
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
820-2	27	1.57	0.08	2	1.56	0.02	-0.19	2	1.56	0.09	-0.06	2	1.54	0.03	-0.38
90	27	1.07	0.08	3	1.08	0.03	0.12	1	1.10		0.38	2	1.12	0.01	0.56
91	27	0.82	0.05	2	0.84	0.02	0.50	3	0.80	0.01	-0.33	1	0.80		-0.40

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#### Sulfated Ash, mass%

Mean



#### Sulfated Ash, mass%




### D874: Sulfated Ash

### Sulfated Ash, mass%

#### Mean $\Delta/s$



Return to Executive Summary

Test Monitoring Center



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	73
Failed Calibration Test	OC	12
Operationally Invalidated by Lab	LC, XC	10
Operationally Invalidated After Initially Reported as Valid	RC	1
Non-reference shakedown, excluded from statistics	NN	3
Total		99

Number of Labs Reporting Data: 10 Fail Rate of Operationally Valid Tests: 14%

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**Operationally Invalid Tests** 

- 3 tests vacuum system failure (LC)
- 3 tests NO<sub>2</sub> leak or flow problem (XC, LC)
- 3 tests heater failure (XC)
- > 2 tests unexplained high EOT volatiles (XC, RC)
- Also had 3 pre-calibration shakedown runs reported by new lab, new rig, as required (NN)





Statistically Unacceptable Tests (OC)	No. Of Tests
Natural Log (MRV Viscosity) Mild	8
Natural Log (MRV Viscosity) Severe	4

No TMC technical updates issued this period.





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Period Precision and Severity Estimates

Natural Log (MRV Viscosity)	n	df	Pooled s	Mean ∆/s
Current Targets	49	46	0.1945	
10/1/10 through 3/31/11*	121	118	0.7092	0.29
10/1/10 through 3/31/11*	120	117	0.4628	0.05
4/1/11 through 9/30/11	96	92	0.2593	-0.69
10/1/11 through 3/31/12	93	90	0.2068	-0.39
4/1/12 through 9/30/12	86	83	0.2975	-0.29
10/1/12 through 3/31/13	109	106	0.2684	-0.58
4/1/13 through 9/30/13	90	87	0.2368	-0.94
10/1/13 through 3/31/14	85	82	0.2715	-0.43

\*Period statistics with and without extreme result included



### Natural Log (MRV Viscosity) Pooled s





### Natural Log (MRV Viscosity) Mean $\Delta/s$







### Current Period Severity Estimates by Lab Natural Log (MRV Viscosity)

	n	Mean ∆/s
Lab A	25	-0.37
Lab AM	6	-0.02
Lab AN	4	-0.86
Lab AP	3	-0.87
Lab AQ	2	-1.42
Lab AS	2	0.45
Lab AT	2	-0.24
Lab B	11	-1.34
Lab D	4	-0.63
Lab G	26	-0.06

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### Natural Log (MRV Viscosity)

Mean  $\Delta/s$ 



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- Precision (Pooled s) is less precise than prior period
  - Less precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.43 s mild
  - Two labs on target, overall (Labs AM & G)
  - Two labs more than 1 s mild, overall (Labs AQ & B)
  - Five tests reported as operationally valid are more than 3 s from target (all fail to calibrate but included in statistics):
    - Rig G4 4.6 s severe, Oil 438
    - Rig G7 -3.5 s mild, Oil 435-1
    - Rig G1 4.0 s severe, Oil 438
    - Rig A3 -4.2 s Mild, Oil 434-1
    - Rig AQ1 -3.7 s mild, Oil 435-1





- Precision on Oil 438 is degraded significantly
  - Partly due to two severe fails noted above

 CUSUM Severity Plot shows an ongoing overall mild trend since the 01APR11 timeline (following the ROBO workshop) with only brief periods of leveling (on-target) performance.





#### **ROBO TEST INDUSTRY OPERATIONALLY VALID DATA**



#### AGED OIL MRV APPARENT VISCOSITY



COUNT IN COMPLETION DATE ORDER

30APR14:16:37

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#### ROBO TEST INDUSTRY OPERATIONALLY VALID DATA



#### AGED OIL MRV APPARENT VISCOSITY



30APR14:16:38

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### Performance by Oil Natural Log (MRV Viscosity)

	Targets 10/1/12 - 3/31/13		4/1/13 - 9/30/13			10/1/13 - 3/31/14									
Oil Code	n	Mean	s <sub>R</sub>	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s	n	Mean	s <sub>R</sub>	Mean ∆/s
434-1	13	10.6599	0.1672	39	10.5821	0.2831	-0.47	27	10.4663	0.2154	-1.16	33	10.5212	0.2227	-0.83
435-1	22	11.0416	0.2030	50	10.9221	0.2721	-0.59	40	10.8727	0.2749	-0.83	33	10.9540	0.2530	-0.43
438	14	10.2676	0.2037	20	10.1095	0.2250	-0.78	23	10.0881	0.1818	-0.88	19	10.3213	0.3658	0.26

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#### Natural Log (MRV Viscosity)

Mean





#### Natural Log (MRV Viscosity)

 $\mathbf{S}_{\mathsf{R}}$ 





#### Natural Log (MRV Viscosity)

#### Mean $\Delta/s$



### Non-monitored Bench Tests

### D6922 Homogeneity and Miscibility

- The TMC distributes six D6922 reference oils.
- The TMC does not collect reference data or monitor test results for this test at this time.
- Oils rec'd by TMC 2002 2003
  - Formulations are at least 12 years old now
  - Should section or panel consider updating?

### D7563 Emulsification

- The TMC distributes two D7563 reference oils.
- The TMC does not collect reference data or monitor test results for this test at this time.





### **Reference** Oil Inventory >>> As of 3/31/2014

**Test Monitoring Center** 



#### D5800, D6417, GI

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
VOLC12	2013	D5800	52.9	0.7
VOLD12	2013	D5800	52.9	0.7
VOLE12	2013	D5800	53.0	0.7
52	1995	D6417	62.1	0.1
55	1995	D6417	66.2	1.2
58	1998	D6417, GI	110.3	0.8
62	1996	GI	1.4	0.1
1009*	2002	GI	46.9	

\*Multi-test oil; estimated aliquot reserved for bench testing.

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#### **TEOST, MTEOS & ROBO**

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
432	1998	MTEOS	113.0	1.5
434	2003	MTEOS	4.5	0.5
75	2010	TEOST	5.7	0.7
435-2*	2010	TEOST	45.7	
434-1*	2008	ROBO	7.8	
435-1*	2008	ROBO	34.8	
438*	2003	ROBO	20.1	

\*Multi-test oil; estimated aliquot reserved for bench testing.

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#### D6082 & D874

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
1007	1998	D6082	19.9	2.9
66	2002	D6082	90.9	1.1
820-2	2001	D874	10.3	0.0
90	2005	D874	31.7	2.4
91	2006	D874	4.1	0.0



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#### D6922 Homogeneity & Miscibility Oils

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
HMA	2002	H&M	150.0	7.7
НМВ	2002	H&M	153.7	7.7
HMC	2003	H&M	140.0	7.7
HMD	2002	H&M	147.7	7.7
HME	2002	H&M	133.7	7.7
HMF	2002	H&M	156.2	7.7



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#### **D7563 Emulsion Retention Oils**

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
EM2	2011	Emulsion	7.9	0.8
EM2-1	2011	Emulsion	25.0	0.0
EM5	2011	Emulsion	7.9	0.8
EM5-1	2011	Emulsion	25.0	0.0





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# **Reference Oil Shipping Aliquots**

Test	Quantity
D6417	1 ml
D6417QC	118 ml
D5800	100 ml
GI	25 ml
MTEOS	17 ml
TEOST	125 ml
D6082	525 ml
D874	32 ml
D874QC	1000 ml
ROBO	300 ml
ROBOQC	1000 ml
H&M	1000 ml
D7563	1000 ml

Test Monitoring Center



### **Additional Information**

**Test Monitoring Center** 



## **Additional Information**

- Available on the TMC's Website:
  - CUSUM Severity Plots
  - Reference Data, Period Statistics and Timelines
  - Information Letters and Technical Memos
  - Report Forms & Data Dictionaries
  - Online Store, and more...

www.astmtmc.cmu.edu





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