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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 2016

#### D6417 (Volatility by GC)

Precision (Pooled s) is more precise than last period
 More precise than target precision

- Performance (Mean  $\Delta/s$ ) is on target (0.04 s severe)
- CUSUM plot shows overall on-target performance this period.





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#### D5800 (Volatility by Noack)

- Precision (Pooled s), at 0.50 mass %, is more precise than prior period and matches the target precision.
- Performance (Mean  $\Delta/s$ ) is 1.08 s severe, comparable to last period and more severe than any period since at least April 2010
- Testing has moved substantially more severe over the last two report periods, but precision has improved over that same time.
- Fail rate of operationally valid tests (AC & OC) is 19% (fail rate of the three prior periods was 20%, 27% and 36%).
- Historical long-term severe trend continues with only a modest decrease in severity following the introduction of the new reference oils (3Q 2013), and a substantial increase in severity for the last two report periods.

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## D5800 (Volatility by Noack)

All three reference oils are again performing severe, with oil VOLC12 performing 1.5 s severe, and oil VOLE12 performing 0.92 s severe.





- D5133 (Gelation Index)
- Precision (Pooled s) is less precise than prior period
  More precise than target precision
- Performance (Mean  $\Delta/s$ ) is on target (0.03 s)
- Reference oil 62 inventory is down to 1.0 gallons remaining (but only 0.2 gallons shipped prior 12 months).





#### ▶ D6335 (TEOST-33C)

- Precision (Pooled s) is less precise than prior period
  - Less precise than target precision
  - Less precise than all periods since at least April 2013
- Performance (Mean  $\Delta/s$ ) is -0.43 s mild
  - Instrument G2 reported results 1.2 s, 4.8 s, -3.7 s and -1.3 s this period, with a mean severity of only 0.25 s, but contributing to the overall poor precision this period.
  - Instrument B5 had two consecutive fails, one 2 s and one -2 s, balancing out on severity, but also contributing to the poor precision estimate this period.
- All tests this period report using Rod Batches L or M





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#### B0.07 Bench Testing Executive Summary D7097 (MHT-4 TEOST)

- Precision (Pooled s) is more precise than prior period
  Less precise than target precision
- Performance (Mean  $\Delta$ /s) is 0.29 s severe
- All operationally valid tests this period report using Rod Batch L or M
- All operationally valid calibration tests this period report using Catalyst Batch 14AA or 15AA





#### B0.07 Bench Testing Executive Summary D7097 (MHT-4 TEOST)

- CUSUM severity plot shows slight severe trend this period
  - However, lab performance differences persist
  - Severe oil 432 overall performance is closer to target but is a result of performance differences between catalyst batches offsetting:
    - CATBATCH 14AA is 0.75 s severe (n=34)
    - CATBATCH 15AA is -0.86 s mild (n=10)
    - Catalyst batches have been observed to bias performance differently for different oils
      - · does not explain ongoing lab severity differences.

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#### D6082 (High Temperature Foam)

- Foam Tendency Precision (Pooled s) is more precise than prior period
  - More precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.45 s mild
- No non-zero occurrences of Foam Stability (as expected)
- All operationally valid discrimination runs demonstrated acceptable discrimination
- D874 (Sulfated Ash)
  - Precision (Pooled s) is more precise than the prior period
    - More precise than target precision
  - Performance (Mean  $\Delta/s$ ) is -0.41 s mild





#### • <u>D7528</u> (ROBO)

- Period overall precision and severity estimates shown with 9 s severe result included and excluded.
- Exceptionally high OC fail rate this period (32%), with individual rigs failing 3, 4 and 5 times, and 4 tests 5 s or more severe (5.0 s, 5.6 s, 5.6 s and 9.2 s)
- Precision (Pooled s) is less precise than prior period
  - Continues to be less precise than target precision
  - Even with extreme (9 s) result excluded, the worst period precision since at least April 2013
- Performance (Mean  $\Delta/s$ ) is -0.10 s mild (-0.20 s mild with 9 s result excluded)



#### ▶ <u>D7528</u> (ROBO)

- Oils 434-1 and 435-1 are especially imprecise (even with extreme 9 s result excluded on 435-1)
- CUSUM Severity Plot shows an overall mild trend since the 01APR11 timeline (following a 2011 ROBO workshop) with significant leveling coincident with the October 2015 ROBO workshop held in San Antonio, TX.
- One Information Letter Issued for ROBO This Period:
  - ROBO IL 16-1; March 11, 2016; Numerous Revisions to Test Method D7528





## Calibrated Labs and Stands\*

Test	Labs	Stands
D6417	5	6
D5800	9	24
D5133 (GI)	5	9
D6335 (TEOST)	6	9
D7097 (MTEOS)	8	37
D6082	4	4
D874	3	
D7528 (ROBO)	4	12

\*As of 9/30/2016

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

#### >>> October 1, 2015 –

March 31, 2016

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

Number of Labs Reporting Data: 5 Fail Rate of Operationally Valid Tests: 0%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Volatility Loss Mild	0
Volatility Loss Severe	0

 There were no technical memos issued this period for D6417.



**Period Precision and Severity Estimates** 

Area % Volatized @ 371°C	n	df	Pooled s	Mean ∆/s
Initial Selected Oils from RR	54	51	0.39	
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
4/1/14 through 9/30/14	15	12	0.34	-0.35
10/1/14 through 3/31/15	14	11	0.40	-0.01
4/1/15 through 9/30/15* 4/1/15 through 9/30/15*	16 15	13 12	0.57 0.42	-0.36 -0.04
10/1/15 through 3/31/16	13	10	0.19	0.04

\*Extreme OC result included and excluded





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#### D6417 Precision Estimates

#### Area % Volatized @ 371°C

#### Pooled s





## D6417 Severity Estimates



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

	n	Mean $\Delta$ /s
Lab A	4	0.48
Lab B	2	-0.38
Lab D	3	-0.27
Lab G	2	-0.12
Lab S	2	0.24

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## D6417 Lab Severity Estimates

Area % Volatized @ 371°C

#### Mean $\Delta$ /s



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- Precision (Pooled s) is more precise than last period More precise than target precision
- Performance (Mean  $\Delta/s$ ) is on target (0.04 s severe)
- CUSUM plot shows overall on-target performance this period.





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



#### SAMPLE AREA % VOLATIZED



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

		Targets		10/1/14 - 3/31/15			4/1/15 - 9/30/15			10/1/15 - 3/31/16					
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	2	6.8	0.35	-0.39	7	6.8	0.61	-0.69	4	6.9	0.24	-0.23
55	18	11.68	0.51	6	11.6	0.51	-0.09	4	11.6	0.76	-0.11	5	11.7	0.15	0.12
58	18	5.61	0.30	6	5.7	0.26	0.19	5	5.6	0.28	-0.10	4	5.7	0.19	0.22





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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  $\Delta/s$ 



Return to Executive Summary

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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	46
Failed Calibration Test	OC	11
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		57

Number of Labs Reporting Data: 9 Fail Rate of Operationally Valid Tests: 19%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Evaporation Loss Mild	0
Evaporation Loss Severe	11

Failing results are across multiple labs, instruments and oils.

 Apparatus J5 (model NCK25G) contributed three severe fails this period, the most from one instrument; one on VOLC12 and, later, two consecutive on VOLE12.

Number of operationally valid results reported by oil:

- VOLC12: 13 AC, 5 OC (severe)
- VOLD12: 13 AC, 3 OC (severe)
- VOLE12: 20 AC, 3 OC (severe)





There were no technical updates issued this report period.





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Period Precision and Severity Estimates

Sample Evaporation Loss,				
mass %	n	df	Pooled s	Mean ∆/s
Targets Effective 10/1/2013	78	75	0.50	
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
10/1/13 through 3/31/14	38	34	0.59	0.37
4/1/14 through 9/30/14	55	52	1.04	0.38
10/1/14 through 3/31/15	60	57	0.80	0.44
4/1/15 through 9/30/15*	55	52	0.67	1.04
4/1/15 through 9/30/15*	54	51	0.61	0.95
10/1/15 through 3/31/16	57	54	0.50	1.08

\*Extreme OC result included and excluded

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Performance Comparison by Procedure & Model Sample Evaporation Loss, Mass %

	n	df	Pooled s	Mean ∆/s
Procedure B	50	47	0.50	1.08
Procedure C	7	4	0.47	1.12
Model	n	df	Pooled s	Mean ∆/s
Model NCK2	n 11	df 8	Pooled s 0.32	Mean ∆/s 1.46
Model NCK2 NCK25G	n 11 39	df 8 36	Pooled s        0.32        0.54	Mean ∆/s 1.46 0.97





#### **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 ∆/s
Lab A	6	1.29
Lab B	12	0.73
Lab D	3	1.04
Lab E1	6	0.59
Lab F	6	1.54
Lab G	10	1.37
Lab I	4	1.10
Lab J	8	1.21
Lab V	2	0.80

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## **D5800 Lab Severity Estimates**

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



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- Precision (Pooled s), at 0.50 mass %, is more precise than prior period and matches the target precision.
- Performance (Mean  $\Delta/s$ ) is 1.08 s severe, comparable to last period and more severe than any period since at least April 2010
- Testing has moved substantially more severe over the last two report periods, but precision has improved over that same time.
- Fail rate of operationally valid tests (AC & OC) is 19% (fail rate of the three prior periods was 20%, 27% and 36% ).
- Historical long-term severe trend continues with only a modest decrease in severity following the introduction of the new reference oils (3Q 2013), and a substantial increase in severity for the last two report periods.




## D5800: Evaporation Loss of Lubricating Oil by Noack Method

- Breakdown of tests reported this period by severity of results:
  - 2 < and < 3 s severe of targets:
    - 11 tests (two pass on acceptance bands due to rounding)
    - models NCK2 and NCK25G.
  - 3 < and < 4 s severe of targets:
    - 2 tests
    - models NCK2 and SVT1
- All three reference oils are again performing severe, with oil VOLC12 performing 1.5 s severe, and oil VOLE12 performing 0.92 s severe.







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



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#### PRCDR='B'

**EVAPORATION LOSS, MASS%** 



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#### PRCDR='C' **EVAPORATION LOSS, MASS%**



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## D5800: Evaporation Loss of Lubricating Oil by Noack Method

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

	Targets			10/1/14 – 3/31/15			4/1/135– 9/30/15				10/1/15 – 3/31/16				
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
VOLC12	24	14.19	0.40	21	14.3	0.86	0.33	25	14.7	0.75	1.32	18	14.8	0.44	1.57
VOLD12	27	12.52	0.52	21	13.0	0.73	0.93	16	12.8	0.65	0.57	16	12.9	0.62	0.77
VOLE12	27	16.74	0.55	18	16.7	0.81	0.00	14	17.3	0.52	1.10	23	17.2	0.45	0.92



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# D5800 Performance by Oil

### Sample Evaporation Loss, mass %

Mean



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# D5800 Performance by Oil

#### Sample Evaporation Loss, mass %

**S**<sub>R</sub> 1.00 0.86 0.81 0.80 0.75 0.73 0.65 0.62 0.60 Oil VOLC12 0.55 0.52 0.52 0.44 0.45 Oil VOLD12 0.40 0.40 Oil VOLE12 0.20 0.00

Target APR '15 OCT '15 APR '16





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# D5800 Performance by Oil

Sample Evaporation Loss, mass %

Mean  $\Delta/s$ 



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	30
Failed Calibration Test	OC	1
Operationally Invalidated by Lab	LC, XC	2
Operationally Invalidated After Initially Reported as Valid	RC	0
Instrument Shakedown	NN	8
Total		41

Number of Labs Reporting Data: 8 Fail Rate of Operationally Valid Tests: 3%





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

- Two operationally invalid tests reported this period:
  - Temperature control failure (XC)
  - Software data collection failure (XC)

New instruments S4 and S5 reported eight shakedown runs (validity NN); lab has not yet reported any calibration runs on these rigs.

No TMC technical updates issued this period

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**Period Precision and Severity Estimates** 

				Mean
Gelation Index	n	df	Pooled s	∆/s
Current Targets 7/15/2003	68	65	2.86	
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
4/1/14 through 9/30/14	24	21	2.46	-0.17
10/1/14 through 3/31/15	28	25	1.48	0.12
4/1/15 through 9/30/15	34	31	1.69	-0.17
10/1/15 through 3/31/16	31	28	2.24	0.03



## **D5133 Precision Estimates**

### Gelation Index Pooled s

#### 3.50 2.86 3.00 2.46 2.50 2.24 1.86 2.00 1.691.48 1.47 1.50 1.151.00 0.50 0.00 OCT OCT OCT APR Target APR APR APR '14 '14 '15 '16 n=68 '13 '13 '15





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# **D5133 Severity Estimates**

### Gelation Index Mean ∆/s



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### Current Period Severity Estimates by Lab Gelation Index

	n	Mean $\Delta/s$
Lab A	6	-0.85
Lab B	10	0.56
Lab D	4	-0.07
Lab E1	2	0.99
Lab G	3	-1.15
Lab I	3	0.10
Lab S	2	0.00
Lab V	1	1.88



# 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 on target (0.03 s)
- Reference oil 62 inventory is down to 1.0 gallons remaining (but only 0.2 gallons shipped prior 12 months).





#### D5133 GELATION INDEX INDUSTRY OPERATIONALLY VALID DATA



#### **GELATION INDEX**

CUSUM Severity Analysis



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### Gelation Index Performance by Oil

	Targets			10/1/14 – 3/31/15			4/1/145– 9/30/15				10/1/15 – 3/31/16				
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	9	6.2	1.15	0.58	11	5.8	1.13	-0.03	11	6.1	0.86	0.46
62	35	17.0	3.90	9	15.7	2.09	-0.34	11	16.0	2.61	-0.26	13	14.5	3.29	-0.64
1009	16	7.30	0.68	10	7.4	1.00	0.12	12	7.2	0.85	-0.22	7	7.7	0.69	0.61

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### **Gelation Index**

Mean



### **Gelation Index**







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### **Gelation Index**

Mean  $\Delta/s$ 



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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	17
Failed Calibration Test	OC	4
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Instrument Shakedown	NN	7
Total		28

Number of Labs Reporting Data: 6 Fail Rate of Operationally Valid Tests: 19%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Total Deposits Mild	3
Total Deposits Severe	1

- No operationally invalid tests reported this period
- Shakedown runs on instruments A1, B5 and G2
- No TMC technical updates issued this period



### Period Precision and Severity Estimates

				Mean
Total Deposits, mg	n	df	Pooled s	$\Delta/s$
Updated Targets 20130415	60	58	5.73	
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
4/1/14 through 9/30/14	15	13	7.14	0.15
10/1/14 through 3/31/15	15	13	5.28	-0.28
4/1/15 through 9/30/15	16	14	7.12	-0.11
10/1/15 through 3/31/16	21	19	8.93	-0.43



## **D6335 Precision Estimates**

## Total Deposits, mg Pooled s





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# D6335 Severity Estimates





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

	n	Mean ∆/s
Lab A	4	-0.73
Lab B	6	-0.51
Lab D	3	-1.38
Lab E1	2	0.44
Lab G	4	0.24
Lab V	2	-0.35

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# D6335 Lab Severity Estimates

Total deposits, mg

Mean  $\Delta$ /s





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- Precision (Pooled s) is less precise than prior period
  - Less precise than target precision
  - Less precise than all periods since at least April 2013
- Performance (Mean  $\Delta/s$ ) is -0.43 s mild
  - Instrument G2 reported results 1.2 s, 4.8 s, -3.7 s and -1.3 s this period, with a mean severity of only 0.25 s, but contributing to the overall poor precision this period.
  - Instrument B5 had two consecutive fails, one 2 s and one -2 s, balancing out on severity, but also contributing to the poor precision estimate this period.
- All tests this period report using Rod Batches L or M



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



#### TOTAL DEPOSITS MG

CUSUM Severity Analysis



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





COUNT IN COMPLETION DATE ORDER

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### Total Deposits, mg Performance by Oil

	Targets 20130415			10/1/14 – 3/31/15				4/1/15 – 9/30/15				10/1/15 – 3/31/16			
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	7	30.5	3.87	0.37	9	28.6	5.50	-0.01	13	25.8	9.8	-0.62
75	30	53.66	6.56	8	48.1	6.24	-0.85	7	52.2	8.84	-0.22	8	52.9	7.3	-0.11





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### Total Deposits, mg

Mean



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### Total Deposits, mg

S<sub>R</sub>

12.00 9.77 10.00 8.84 8.57 7.29 8.00 6.56 6.24 5.50 Oil 435–2 6.00 4.99 4.76 3.87 Oil 75 4.00 2.00 0.00

OCT '14 APR '15 OCT '15 Target APR '16

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# D6335 Performance by Oil

#### Total Deposits, mg

Mean  $\Delta/s$ 



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	75
Failed Calibration Test	OC	9
Operationally Invalidated by Lab	LC, XC	4
Operationally Invalidated After Initially Reported as Valid	RC	0
Donated Catalyst Screener Runs	AG	4
Non-blind Shakedown Run	NN	5
Excluded from Statistics (New Rig)	MC	2
Total		99

Number of Labs Reporting Data: 9 Fail Rate of Operationally Valid Tests: 11%

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Statistically Unacceptable Tests (OC)	No. Of Tests
Total Deposits Mild	3
Total Deposits Severe	6

- Four operationally invalid calibration tests this period:
  - Spilled reference sample at start of test, one test (XC)
  - Rod weight not recorded before test start, one test (LC)
  - Sample completely volatized in under 4 hours, two tests (XC) •Unexplained phenomena
    - Same lab, two different instruments (E1 1 & E1 2)
    - Both on TMC oil 434; TMC confirmed oil ID's by FTIR
    - Instrument E1 1 subsequently passed calibration, E1 2 failed severe on subsequent run (new rig)





- Two test excluded from statistics (MC), new rigs, reported as operationally valid but failed to calibrate.
- Five shakedown runs (NN), four to troubleshoot new instrument B13, which subsequently calibrated successfully.
- Four donated runs to screen catalyst batch 15AA (AG), eleven more donated runs were reported in prior report period.
- No TMC technical updates issued this period





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Period Precision and Severity Estimates

Total Deposits, mg	n	df	Pooled s	Mean ∆/s
Current Targets 7/31/2006	90	87	5.63	
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
4/1/14 through 9/30/14	76	74	7.16	-0.03
10/1/14 through 3/31/15*	94	92	6.60	0.19
10/1/14 through 3/31/15*	90	88	6.08	0.04
4/1/15 through 9/30/15	84	82	7.56	0.39
10/1/15 through 3/31/16	84	82	6.69	0.29

\*Four severe OC tests from instrument G1 included and excluded

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#### **D7097 Precision Estimates**

'13

'13

n=90



'14

'14

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'15

'15



'16

# **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	27	0.49
Lab AK	6	0.77
Lab B	19	-0.65
Lab D	10	-0.04
LAB E1	3	0.92
Lab G	11	0.58
Lab J	6	1.65
Lab V	2	0.06

Lab AU reported one result, but excluded from statistics (MC)

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# **D7097 Lab Severity Estimates**

Total Deposits, mg

Mean  $\Delta/s$ 





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



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





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



- Precision (Pooled s) is more precise than prior period
  Less precise than target precision
- Performance (Mean  $\Delta/s$ ) is 0.29 s severe
- All operationally valid tests this period report using Rod Batch L or M
- All operationally valid calibration tests this period report using Catalyst Batch 14AA or 15AA





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- CUSUM severity plot shows slight severe trend this period
  - However, lab performance differences persist
  - Severe oil 432 overall performance is closer to target but is a result of performance differences between catalyst batches offsetting:
    - CATBATCH 14AA is 0.75 s severe (n=34)
    - CATBATCH 15AA is -0.86 s mild (n=10)
    - Catalyst batches have been observed to bias performance differently for different oils
      - does not explain ongoing lab severity differences





#### 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			10/1/14 – 3/31/15 4/1/15 – 9/30/15 10/1/15 – 3/31/16			4/1/15 – 9/30/15							
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	50	48.7	5.55	0.38	40	50.7	5.60	0.81	44	48.2	4.84	0.27
434	30	27.37	6.57	40	24.8	6.68	-0.39	44	27.4	8.98	0.00	40	29.4	8.27	0.31



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# D7097 Performance by Oil

#### Total Deposits, mg

Mean



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#### Total Deposits, mg

S<sub>R</sub>



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#### Total Deposits, mg

Mean  $\Delta/s$ 



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	8
Acceptable Discrimination Test	AS	4
Failed Calibration Test	OC	0
Operationally Invalidated by Lab	LC, XC	1
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		13

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 statistically invalid tests reported this period
- One aborted test (XC), lab ran wrong test method.
- 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	
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
4/1/14 through 9/30/14	11	65	22	-0.05
10/1/14 through 3/31/15	10	61	12	-0.26
4/1/15 through 9/30/15	11	59	16	-0.36
10/1/15 through 3/31/16	8	58	10	-0.45



Period Precision and Severity Estimates Oil 1007

Foam Stability @ 1 min, ml	n	Mean	S
Current Targets	28	0.00	0.00
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
4/1/14 through 9/30/14	11	No non-zero d	occurrences
10/1/14 through 3/31/15	10	No non-zero d	occurrences
4/1/15 through 9/30/15	11	No non-zero d	occurrences
10/1/15 through 3/31/16	8	No non-zero d	occurrences





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





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#### 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	2	0.21
Lab B	3	-0.84
Lab G	2	-0.58
Lab V	1	-0.32



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



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- Foam Tendency Precision (Pooled s) is more precise than prior period
  - More precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.45 s mild
- No non-zero occurrences of Foam Stability
- All operationally valid discrimination runs demonstrated acceptable discrimination





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



IND='1007'

FOAM TENDENCY

CUSUM Severity Analysis



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Return to Executive Summary

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Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	7
Failed Calibration Test	OC	0
Operationally Invalidated by Lab	LC, XC	0
Operationally Invalidated After Initially Reported as Valid	RC	0
Total		7

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 Δ/s
Current Targets	81	78	0.07	
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
4/1/14 through 9/30/14	6	3	0.07	0.09
10/1/14 through 3/31/15	6	4	0.07	-0.25
4/1/15 through 9/30/15*	8	5	0.13	-1.36
4/1/15 through 9/30/15*	7	4	0.05	-0.36
10/1/15 through 3/31/16	7	4	0.03	-0.41

\*Period statistics with and without extreme result included

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#### Sulfated Ash, mass% Pooled s





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




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

	n	Mean ∆/s
Lab A	2	-0.64
Lab B	3	0.01
Lab G	2	-0.81



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



- Precision (Pooled s) is more precise than the prior period
  - More precise than target precision
- Performance (Mean  $\Delta/s$ ) is -0.41 s mild





#### D874 INDUSTRY OPERATIONALLY VALID DATA



#### TEST SAMPLE PERCENT SULFATED ASH



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06APR16:13:06





### Performance by Oil Sulfated Ash, mass%

		Targets			10/1/14	- 3/31/1	5	4/1/15 – 9/30/15			10/1/15 – 3/31/16				
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	3	1.57	0.10	-0.04	2	1.10	0.28	-5.88	3	1.52	0.03	-0.62
90	27	1.07	0.08	0				4	1.09	0.05	0.22	2	1.03	0.03	-0.50
91	27	0.82	0.05	3	0.80	0.03	-0.47	2	0.82	0.01	0.00	2	0.82	0.03	0.00





### Sulfated Ash, mass%

Mean



Test Monitoring Center



### Sulfated Ash, mass%

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



Target OCT '14 APR '15 OCT '15 APR '16

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### Sulfated Ash, mass%

### Mean $\Delta/s$



Test Status	Validity Code	No. Tests
Acceptable Calibration Test	AC	63
Failed Calibration Test	OC	29
Operationally Invalidated by Lab	LC, XC	5
Operationally Invalidated After Initially Reported as Valid	RC	1
Total		98

Number of Labs Reporting Data: 6 Fail Rate of Operationally Valid Tests: 32%

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**Operationally Invalid Tests** 

- I test unexplained high EOT volatiles (RC)
- 3 tests heater or heater control failure (LC, XC)
- 2 tests NO<sub>2</sub> flow problems (LC)





Statistically Unacceptable Tests (OC)	No. Of Tests
Natural Log (MRV Viscosity) Mild	15
Natural Log (MRV Viscosity) Severe	14

 One Information Letter Issued for ROBO This Period:
ROBO IL 16-1; March 11, 2016; Numerous Revisions to Test Method D7528





Period Precision and Severity Estimates

Natural Log (MRV Viscosity)	n	df	Pooled s	Mean $\Delta$ /s
Current Targets	49	46	0.1945	
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
4/1/14 through 9/30/14	83	80	0.2535	-0.78
10/1/14 through 3/31/15	97	94	0.3069	-0.69
4/1/15 through 9/30/15	85	82	0.2363	-0.90
10/1/15 through 3/31/16*	92	89	0.4115	-0.10
10/1/15 through 3/31/16*	91	88	0.3661	-0.20

\*Period statistics with and without extreme result included

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### Natural Log (MRV Viscosity) Pooled s







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



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

	n	Mean $\Delta$ /s
Lab A	34	-0.85
Lab AM	16	1.21
Lab AQ	3	-0.62
Lab B	11	0.44
Lab D	4	-0.87
Lab G	24	0.04

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Natural Log (MRV Viscosity)

Mean  $\Delta/s$ 



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- Lab B, Instrument 4A, reported a result more than 9 s severe (MRV >400,000 cP). Period overall precision and severity estimates shown with this result included and excluded.
- Exceptionally high OC fail rate this period (32%), with individual rigs failing 3, 4 and 5 times, and 4 tests 5 s or more severe (5.0 s, 5.6 s, 5.6 s and 9.2 s)
- Precision (Pooled s) is less precise than prior period
  - Continues to be less precise than target precision
  - Even with extreme (9 s) result excluded, the worst period precision since at least April 2013
- > Performance (Mean  $\Delta/s$ ) is -0.10 s mild (-0.20 s mild with 9 s result excluded)



- Oils 434-1 and 435-1 are especially imprecise (even with extreme 9 s result excluded on 435-1)
- CUSUM Severity Plot shows an overall mild trend since the 01APR11 timeline (following a 2011 ROBO workshop) with significant leveling coincident with the October 2015 ROBO workshop held in San Antonio, TX.





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



#### AGED OIL MRV APPARENT VISCOSITY



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#### ROBO TEST INDUSTRY OPERATIONALLY VALID DATA



#### AGED OIL MRV APPARENT VISCOSITY



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

		Targets			10/1/14	- 3/31/1	5	4/1/15 - 9/30/15			10/1/15 - 3/31/16				
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	30	10.4826	0.2055	-1.06	26	10.4390	0.1991	-1.32	31	10.5730	0.3303	-0.52
435-1	22	11.0416	0.2030	44	10.9258	0.2345	-0.58	41	10.8794	0.2220	-0.80	40*	11.0667	0.4304	0.12
438	14	10.2676	0.2037	23	10.1817	0.4891	-0.42	18	10.1619	0.3085	-0.52	20	10.1948	0.2612	-0.36

\*Extreme (9 s) result excluded





### Natural Log (MRV Viscosity)

Mean



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### Natural Log (MRV Viscosity)





### 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 13 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 4/1/2016

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### D5800, D6417, GI

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
VOLC12	2013	D5800	47.8	0.9
VOLD12	2013	D5800	49.7	0.6
VOLE12	2013	D5800	48.6	0.7
VOLD14	2014	D5800QC	312	97.4
52	1995	D6417	59.1	0.0
55	1995	D6417	66.2	0.0
58	1998	D6417, GI	116.4	0.4
62	1996	GI	1.0	0.2
1009*	2002	GI	46.7	

\*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	108.7	0.8
434	2003	MTEOS	3.2	0.7
75	2010	TEOST	3.8	0.9
435-2*	2010	TEOST	44.9	
434-1*	2008	ROBO	1.6	
435-1	2008	ROBO	454.1	15.3
438*	2003	ROBO	10.9	

\*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	13.1	2.6
66	2002	D6082	88.0	1.1
820-2	2001	D874	10.2	0.1
90	2005	D874	25.5	3.8
91	2006	D874	4.0	0.1





### D6922 Homogeneity & Miscibility Oils

Oil	Year Rec'd By TMC	Tests	TMC Inventory, gallons	Gallons Shipped last 12 months
HMA	2002	H&M	133.6	6.1
HMB	2002	H&M	137.1	6.1
HMC	2003	H&M	123.9	6.1
HMD	2002	H&M	131.4	6.1
HME	2002	H&M	117.6	6.1
HMF	2002	H&M	140.1	6.1



### **D7563 Emulsion Retention Oils**

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

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### **Additional Information**

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## **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





