

ASTM HDEOCP Meeting - Orlando, FL  
May 10, 2001

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# **UPDATE OF ASTM TASK FORCE ON LOW TEMPERATURE RHEOLOGY OF USED ENGINE OILS (LOTRUO)**

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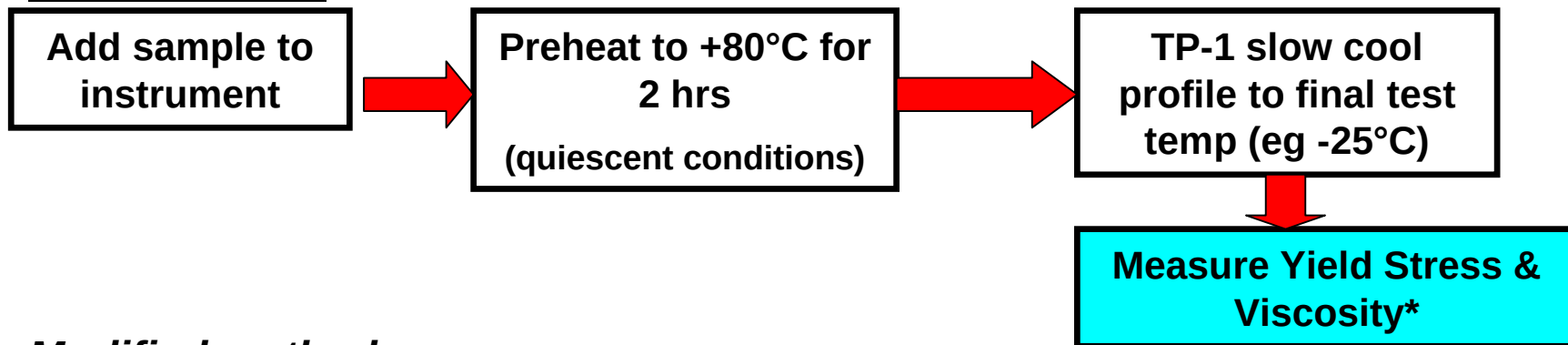
# Discussion Topics

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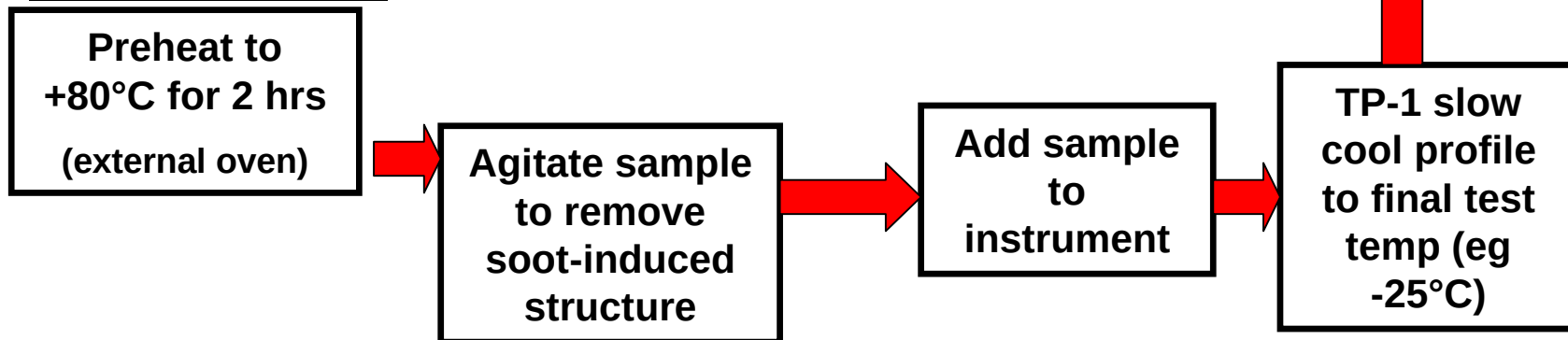
- **MRV Used Oil Round Robin**
  - Round robin and analysis completed on 9 samples at two temperatures (-20/-25°C) using two methods (D4684 or modified preheat); results will be previewed with HDECOP today (to be discussed in detail with the LOTRUO task force membership in June)
- **Review information on E-O-T oil samples from the PC-9 precision matrix received by IOL for potential LOTRUO work**

## 2 DIFFERENT METHODS COMPARED IN LOTRUO ROUND ROBIN

### D4684 method



### Modified method



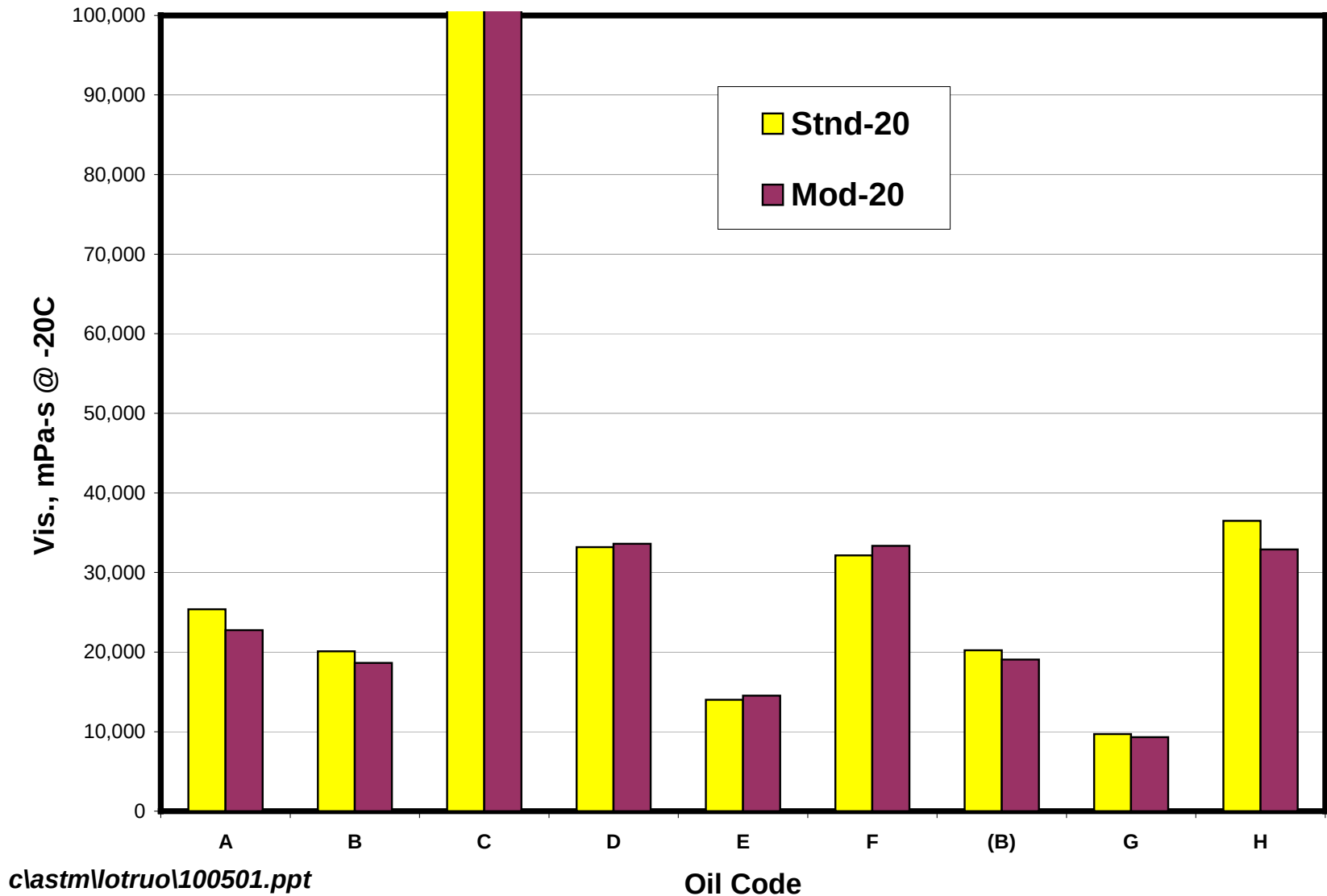
\* Note that MRV test evaluates both yield stress and viscosity; for >35 Pa yield stress (failing), viscosity not normally measured, but for round robin, labs asked to report both

# Test Oils / Labs

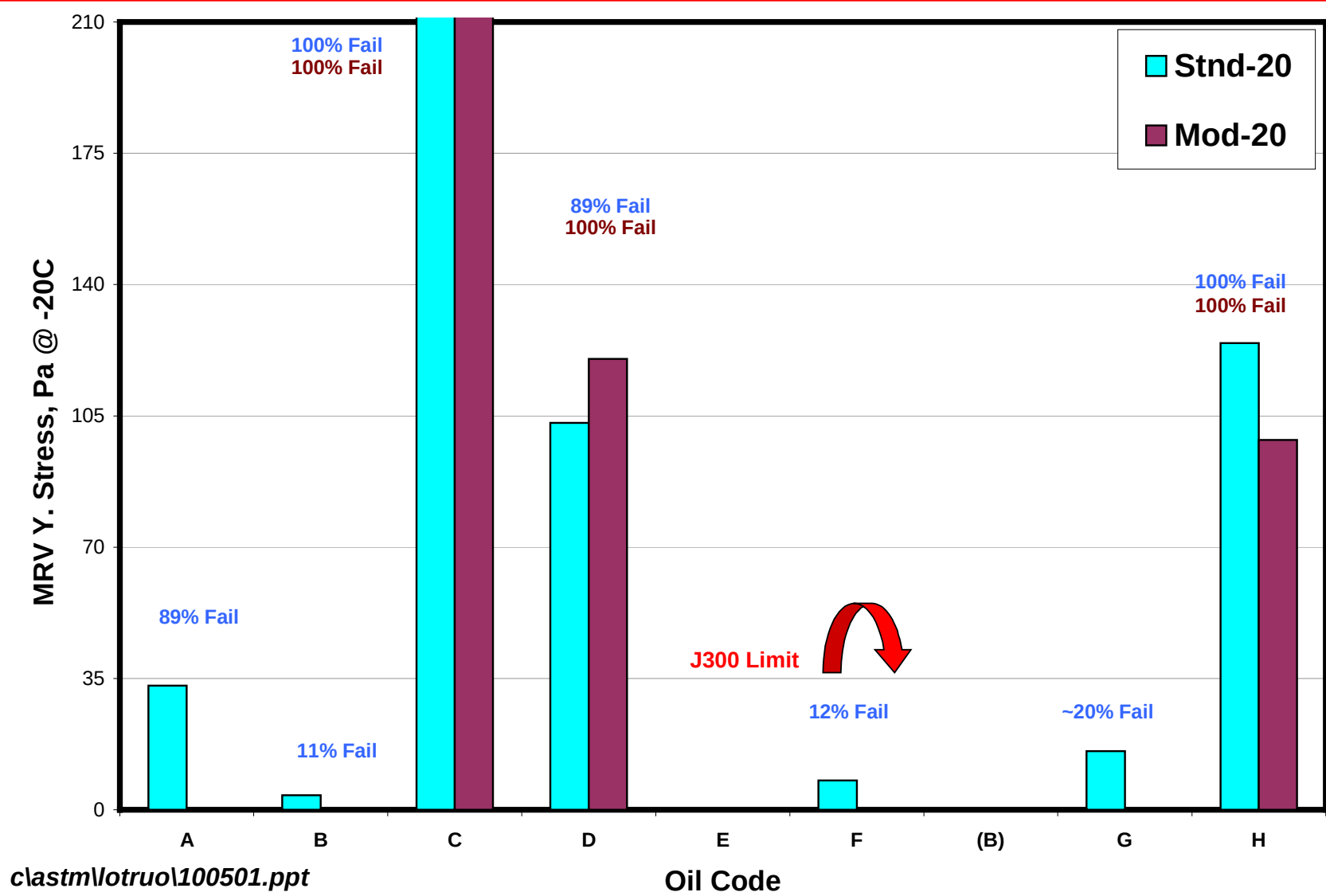
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- **Nine oil samples (including double-blind sample)**
  - **Four T8 drain samples (~5% soot)**
  - **One T8E drain sample (~9% soot)**
  - **Two M11 EGR drain samples (8-9% soot)**
  - **One Mack T10 drain sample (soot not measured)**
- **Oils screened to ensure a range of viscosity/yield stress values at -20/-25°C**
- **9 labs reported D4684 results, 6 labs reported modified MRV results**
  - **Cannon Inst., Chevron, Citgo, Conoco, Imperial Oil, Infineum, Lubrizol, Pennzoil-Quaker State, RohMax GmbH, RohMax USA**
  - **Data analyzed using ASTM D2PP software**

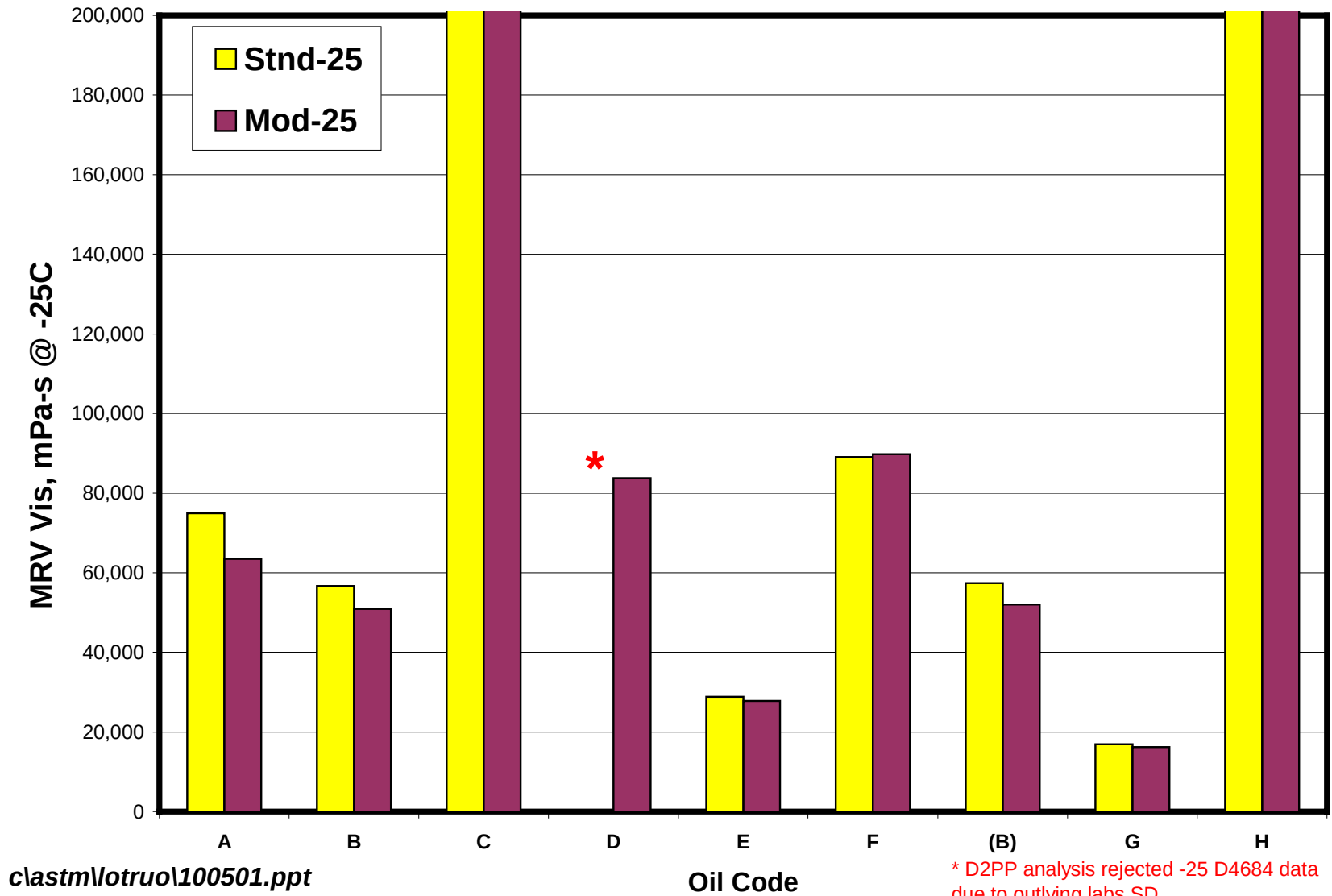
# Comparison of Viscosity Results @-20°C



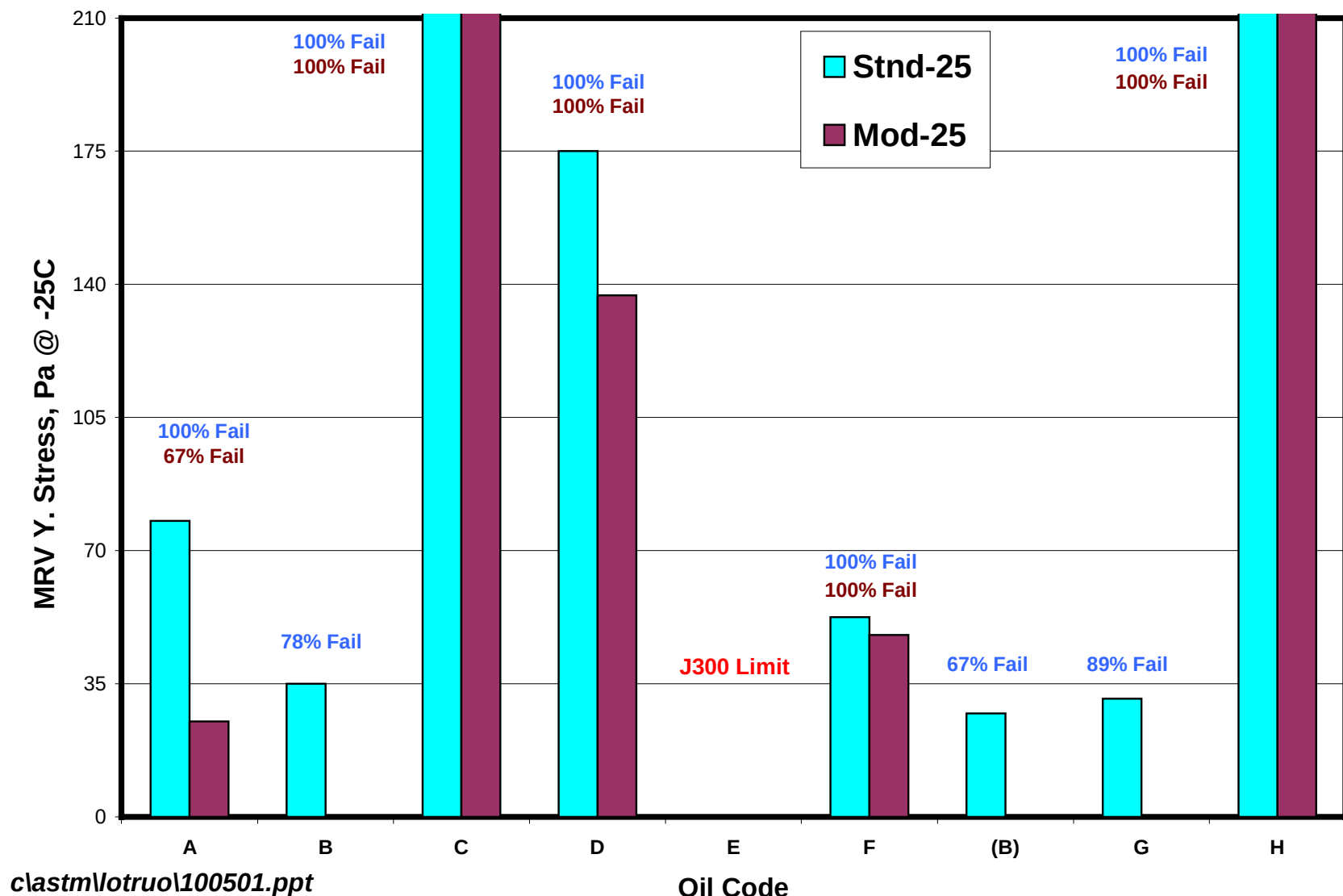
# Comparison of Yield Stress Results @-20°C



# Comparison of Viscosity Results @-25°C



# Comparison of Yield Stress Results @-25°C





# Overall Results of Statistical Analysis

- For oils with measurable viscosity (<400,000 mPa-s)

| <u>Method</u>     | <u>Temp.,<br/>°C</u> | <u>This Study (used HDEO's)</u> |                     | <u>D4684 Published (fresh oils)</u> |                     |
|-------------------|----------------------|---------------------------------|---------------------|-------------------------------------|---------------------|
|                   |                      | <u>r, % of mean</u>             | <u>R, % of mean</u> | <u>r, % of mean</u>                 | <u>R, % of mean</u> |
| D4684             | -20                  | 14.3                            | 21.1                | 7.3                                 | 12.1                |
|                   | -25                  | 10.3                            | 20.8                | 11.7                                | 17.5                |
| Modified<br>D4684 | -20                  | 8.8                             | 18.6                | ---                                 | ---                 |
|                   | -25                  | 6.2                             | 20.9                | ---                                 | ---                 |

- All labs detected oils with gross viscosity failure (>400,000 mPa-s); at this point, test becomes go/no-go
- D4684 yield stress precision appears consistent with step-function nature of measurement

# Conclusions

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- **Used Diesel Engine Oil Round Robin successfully completed**
  - **Soot levels and viscosity/yield stress ranges expected to be relevant to PC-9 category development**
  - **D4684 MRV viscosity at -20°C has poorer precision than stated for fresh oils, but at -25°C, appears to be close to that stated for D4684**
  - **Modified D4684 viscosity measurements at -20/-25C (separate external preheat/agitation) have better repeatability but about the same reproducibility**
    - **the two methods give similar mean viscosities but significantly higher percentage of oils show yield stress with the standard D4684 procedure ⇒ evidence of soot agglomeration during sample preheat**

# Next Steps

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- **Review Round Robin results to be reviewed with working group/task force (June 18th)**
  - **completed statistical analysis (incl. Y. Stress)**
  - **research report on used oil R/R**
  - **potential to incorporate results into D4684 (or new method?) along with guidelines for handling of used oils**
- **Input/Guidance from HDEOCP?**

# Imperial Oil has received 25 E-O-T Oil Samples for Potential LOTRUO use

| CMIR Code | Ind. Oil Code | Lab Code | Engine Source | Qty Given | TGA Soot, % |      | -20°C D4684       |                      | -25°C D4684       |                      |
|-----------|---------------|----------|---------------|-----------|-------------|------|-------------------|----------------------|-------------------|----------------------|
|           |               |          |               |           | TMC         | SRC  | -20C MRV Vis., cP | -20C MRV Y. Str., Pa | -25C MRV Vis., cP | -25C MRV Y. Str., Pa |
| 38811     | PC-9A         | D        | T-10          | 1 gal     | 5.5         | 5.1  | 19,900            | 0<Y<=35              | 43,900            | 0<Y<=35              |
| 38814     | PC-9A         | F        | T-10          | 1 gal     | 5.7         | 5.5  | 26,400            | 0<Y<=35              | 59,300            | 0<Y<=35              |
| 38810     | PC-9A         | A        | T-10          | 4L        | 6.0         |      | 24,500            | 0<Y<=35              | 53,100            | 0<Y<=35              |
| 38951     | PC-9A         | G        | T-10          | 4L        | 5.9         |      | 23,400            | 0<Y<=35              | 51,000            | 0<Y<=35              |
| 38967     | PC-9B         | A        | M11 EGR       | 8L        | 8.0         |      | 22,600            | 0<Y<=35              | 70,400            | 0<Y<=35              |
| 38939     | PC-9C         | A        | T-10          | 4L        | 5.4         |      | 23,200            | 0<Y<=35              | 61,300            | 0<Y<=35              |
| 38931     | PC-9E         | D        | M11 EGR       | 1 gal     | 8.1         | 7.7  | 28,200            | 0<Y<=35              | 214,100           | 105<Y<=140           |
| 38932     | PC-9E         | A        | M11 EGR       | 1 gal     | 8.7         | 8.5  | 38,900            | 0<Y<=35              | 305,400           | 175<Y<=210           |
| 38933     | PC-9E         | A        | M11 EGR       | 2 gal     | 7.7         | 7    | 24,000            | 0<Y<=35              | 135,000           | 140<Y<=175           |
| 38929     | PC-9E         | G        | M11 EGR       | 4L        | 8.8         |      | 29,400            | 0<Y<=35              | 208,500           | 140<Y<=175           |
| 38930     | PC-9E         | G        | M11 EGR       | 4L        | 8.6         |      | 31,200            | 0<Y<=35              | 343,100           | 140<Y<=175           |
| 38927     | PC-9E         | G        | M11 EGR       | 4L        | 9.1         |      | 26,300            | 0<Y<=35              |                   |                      |
| 38937     | PC-9E         | A        | T-10          | 4L        | 4.8         |      | 19,000            | 0<Y<=35              | 102,400           | 140<Y<=175           |
| 38962     | PC-9F         | G        | M11 EGR       | 4L        | 8.7         |      | 28,900            | 35<Y<=70             | 133,600           | 70<Y<=105            |
| 38945     | PC-9F         | D        | T-10          | 1 gal     | 5.3         | 5.09 | 17,300            | 0<Y<=35              | 76,100            | 35<Y<=70             |
| 38953     | PC-9H         | F        | T-10          | 1 gal     | NR          | 5.1  | 14,600            | 0<Y<=35              | 45,300            | 0<Y<=35              |
| 38947     | PC-9H         | G        | T-10          | 4L        | 7.1         |      | 19,900            | 0<Y<=35              | 57,100            | 0<Y<=35              |
| 38821     | ?             |          | 1Q            | 1 gal     |             | 2.6  | 20,800            | 0<Y<=35              | 50,600            | 0<Y<=35              |
| 38966     | ?             |          | M11 EGR       | 4L        |             |      | 31,800            | 105<Y<=140           | 77,900            | 140<Y<=175           |
| 38969     | ?             |          | M11 EGR       | 8L        |             |      | 66,000            | 175<Y<=210           | 683,700           | 315<Y<=350           |
| 38934     | ?             |          | M11 EGR       | 8L        |             |      | 31,000            | 0<Y<=35              | 262,300           | 140<Y<=175           |
| 38958     | ?             |          | M11 EGR       | 4L        |             |      | 20,800            | 0<Y<=35              | 55,100            | 0<Y<=35              |
| 38928     | ?             |          | M11 EGR       | 4L        |             |      | 22,300            | 0<Y<=35              |                   |                      |
| 38813     | ?             |          | T-10          | 1 gal     |             | 5.8  | 25,900            | 0<Y<=35              | 60,200            | 0<Y<=35              |
| 38942     | ?             |          | T-10          | 4L        |             |      | 19,100            | 0<Y<=35              | 42,100            | 0<Y<=35              |

M11 EGR 13  
T-10 11  
1Q 1

All data generated by IOL to date

# IOL D4684 Data Comparing Same Oil, Different Runs

|                      |            |            |            |            |            |         |
|----------------------|------------|------------|------------|------------|------------|---------|
| CMIR Code            | 38931      | 38932      | 38933      | 38929      | 38930      | 38927   |
| Ind. Oil Code        | PC-9E      | PC-9E      | PC-9E      | PC-9E      | PC-9E      | PC-9E   |
| Lab Code             | D          | A          | A          | G          | G          | G       |
| Engine Source        | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR |
| % Soot Reported      | 8.1        | 8.7        | 7.7        | 8.8        | 8.6        | 9.1     |
| -20C MRV Vis., cP    | 28,200     | 38,900     | 24,000     | 29,400     | 31,200     | 26,300  |
| -20C MRV Y. Str., Pa | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35 |
| -25C MRV Vis., cP    | 214,100    | 305,400    | 135,000    | 208,500    | 343,100    |         |
| -25C MRV Y. Str., Pa | 105<Y<=140 | 175<Y<=210 | 140<Y<=175 | 140<Y<=175 | 140<Y<=175 |         |

|                      |         |         |         |         |         |         |
|----------------------|---------|---------|---------|---------|---------|---------|
| CMIR Code            | 38811   | 38814   | 38810   | 38951   | 38953   | 38947   |
| Ind. Oil Code        | PC-9A   | PC-9A   | PC-9A   | PC-9A   | PC-9H   | PC-9H   |
| Lab Code             | D       | F       | A       | G       | F       | G       |
| Engine Source        | T-10    | T-10    | T-10    | T-10    | T-10    | T-10    |
| % Soot Reported      | 5.5     | 5.7     | 6.0     | 5.9     | NR      | 7.1     |
| -20C MRV Vis., cP    | 19,900  | 26,400  | 24,500  | 23,400  | 14,600  | 19,900  |
| -20C MRV Y. Str., Pa | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 |
| -25C MRV Vis., cP    | 43,900  | 59,300  | 53,100  | 51,000  | 45,300  | 57,100  |
| -25C MRV Y. Str., Pa | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 | 0<Y<=35 |

# IOL D4684 Data Comparing Same Oil, T10 vs M11

|                      |            |            |            |            |            |         |            |
|----------------------|------------|------------|------------|------------|------------|---------|------------|
| CMIR Code            | 38931      | 38932      | 38933      | 38929      | 38930      | 38927   | 38937      |
| Ind. Oil Code        | PC-9E      | PC-9E      | PC-9E      | PC-9E      | PC-9E      | PC-9E   | PC-9E      |
| Lab Code             | D          | A          | A          | G          | G          | G       | A          |
| Engine Source        | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR    | M11 EGR | T10        |
| % Soot Reported      | 8.1        | 8.7        | 7.7        | 8.8        | 8.6        | 9.1     | 4.8        |
| -20C MRV Vis., cP    | 28,200     | 38,900     | 24,000     | 29,400     | 31,200     | 26,300  | 19,000     |
| -20C MRV Y. Str., Pa | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35    | 0<Y<=35 | 0<Y<=35    |
| -25C MRV Vis., cP    | 214,100    | 305,400    | 135,000    | 208,500    | 343,100    |         | 102,400    |
| -25C MRV Y. Str., Pa | 105<Y<=140 | 175<Y<=210 | 140<Y<=175 | 140<Y<=175 | 140<Y<=175 |         | 140<Y<=175 |

|                      |           |          |
|----------------------|-----------|----------|
| CMIR Code            | 38962     | 38945    |
| Ind. Oil Code        | PC-9F     | PC-9F    |
| Lab Code             | G         | D        |
| Engine Source        | M11 EGR   | T10      |
| % Soot Reported      | 8.7       | 5.3      |
| -20C MRV Vis., cP    | 28,900    | 17,300   |
| -20C MRV Y. Str., Pa | 35<Y<=70  | 0<Y<=35  |
| -25C MRV Vis., cP    | 133,600   | 76,100   |
| -25C MRV Y. Str., Pa | 70<Y<=105 | 35<Y<=70 |