



# Measurement of Phosphorus Volatility Using TEOST MHT-4

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INDUSTRIAL MINERALS AND CHEMICALS

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# Agenda

- Background: TEOST MHT-4 Protocol
  - adapted for P Volatility measurements
- Description of Test Oils
- P Volatility Calculations
  - % Phosphorus Retention (%PR)
  - Phosphorus Emissions Index (PEI)
- Results & Conclusions

# Phosphorus Volatility via TEOST

## Background

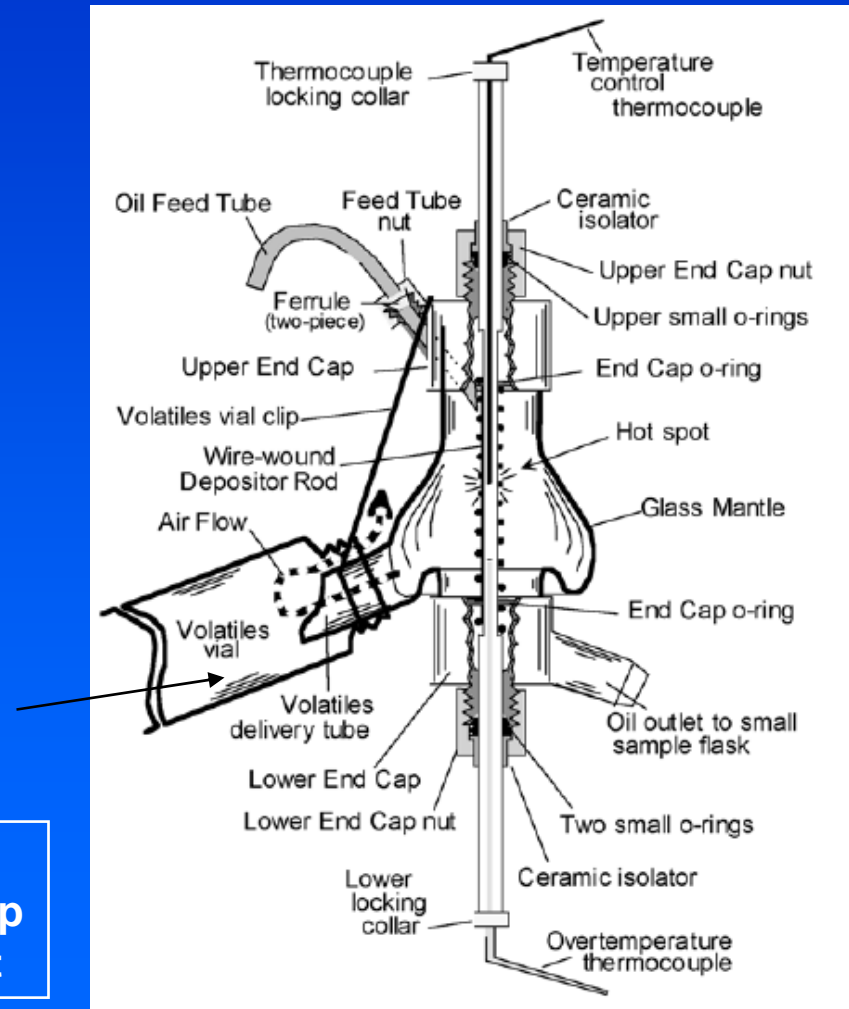
- TEOST MHT-4 study conducted during 2006-2007 comprising over 30 runs
  - primary purpose to assess high temperature deposit performance of engine oils treated with antioxidant boosters (not originally intended for P volatility work)
- Tests conducted in-house per ASTM D 7097
- Data and samples collected during study allowed for post-assessment of phosphorus volatility

# Phosphorus Volatility via TEOST

## TEOST MHT-4 test overview

- Oil sample size: ~8.4 g
- Oil flow rate: 0.25 g/min
- Air flow rate: 10 mL/min
- Organo-metallic catalyst: 0.1 g (Fe/Pb/Sn)
- Rod temperature: 285°C
- Test length: 24 hours
- Volatiles collected, weighed and retained

MHT-4 is good model of crankcase oil being pumped into piston ring zone. Sump is turned over nearly 50 times during test



# Phosphorus Volatility via TEOST

## TEOST MHT-4 P Volatility protocol

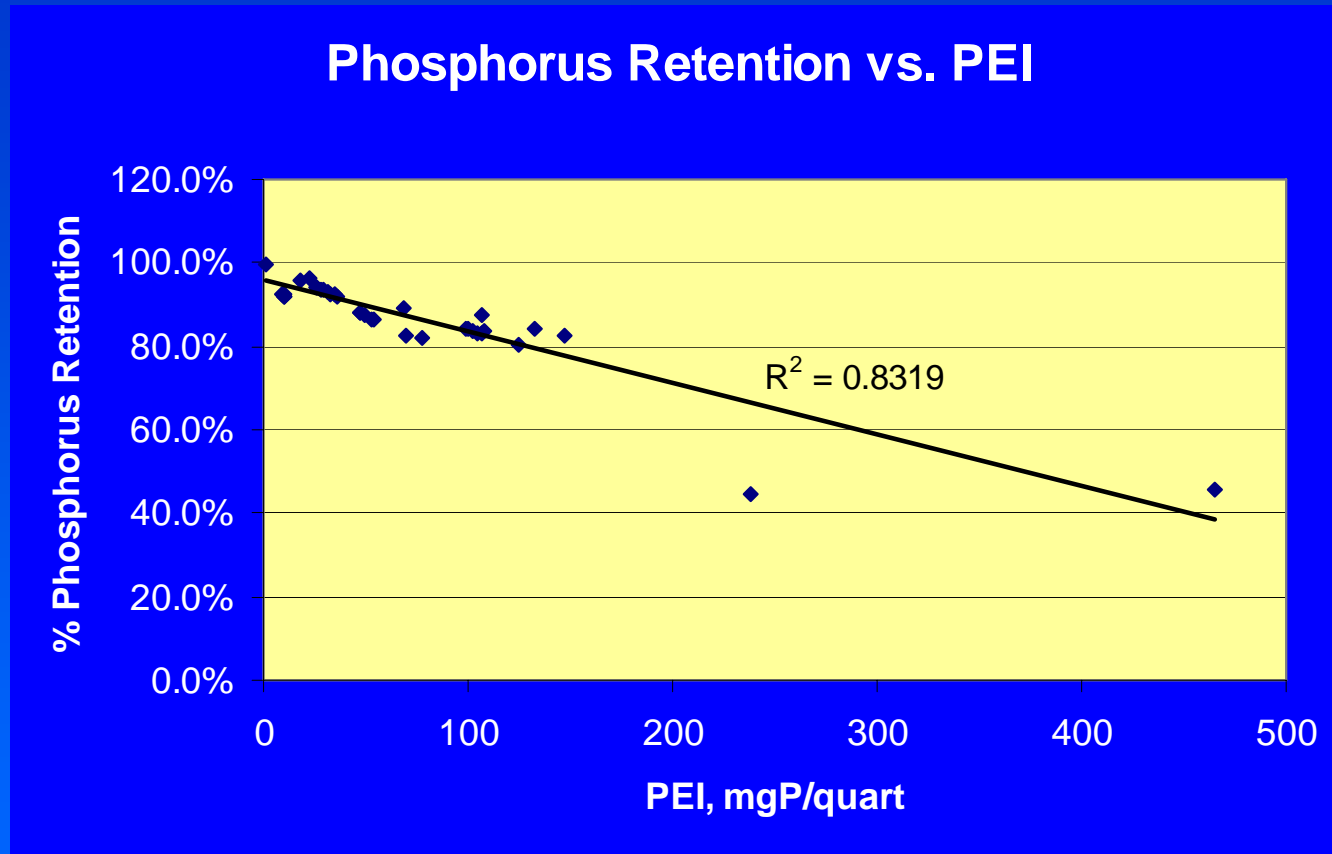
- Weight of sample and volatiles (g) determined ( $W_s$ ,  $W_v$ )
  - Volatiles collected ranged from 2.2g to 5.0g (26% to 59% of fresh oil), with average of 3.4g (40% of fresh oil)
- P content (ppm) of sample and volatiles measured by ICP ( $P_s$ ,  $P_v$ )
- Calculations:
  - % P volatilized =  $100 \times (P_v W_v) / (P_s W_s)$
  - % P retention = (100 - %P volatilized)
    - assumes all P that is not collected as volatiles is retained
  - PEI (mg P per quart of oil) =  $0.85 (P_v W_v / W_s)$ 
    - Phosphorus Emissions Index

# Phosphorus Volatility via TEOST

## Test Oils (34 total)

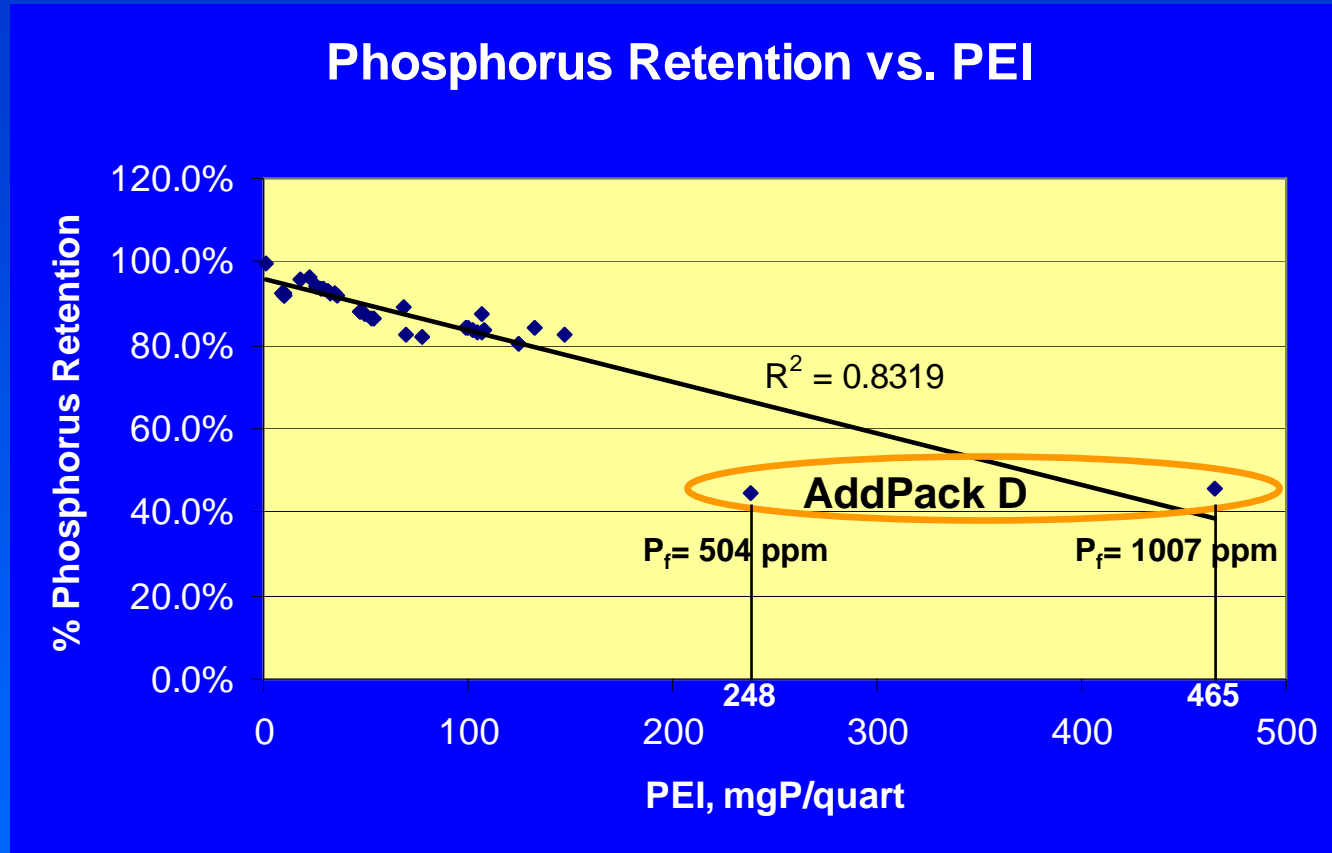
AddPack	SAE	Grade	#	Comment
A	5W-20	GF-4	9	~500 ppm P
B	15W-40	CD/SF	3	~1000 ppm P
C	5W-20	GF-4	11	~500 & ~750 ppm P
D	5W-20	GF-4	3	Short chain 1° ZDDP
E	5W-20	GF-5	5	~150 ppm P
PC-1	5W-20	GF-4	1	Commercial PCMO
PC-2	5W-30	GF-4	1	Commercial PCMO
PC-3	5W-20	GF-4	1	Commercial PCMO

# Phosphorus Volatility via TEOST



- There is a relationship between %PR and PEI
  - an engine oil with higher phosphorus retention gives lower PEI

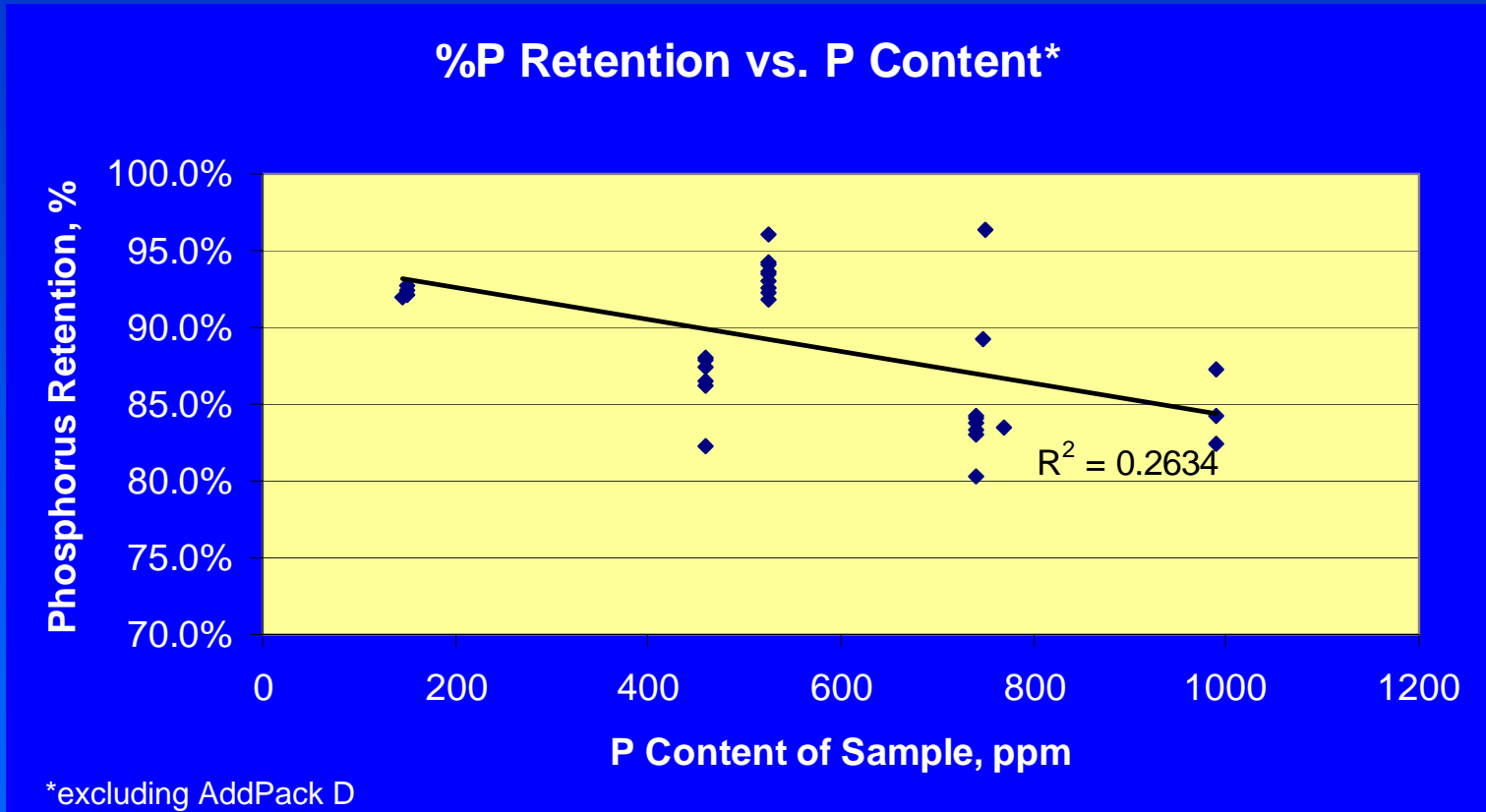
# Phosphorus Volatility via TEOST



- PEI appears to relate directly to fresh oil P content
- %PR appears to be independent of fresh oil content

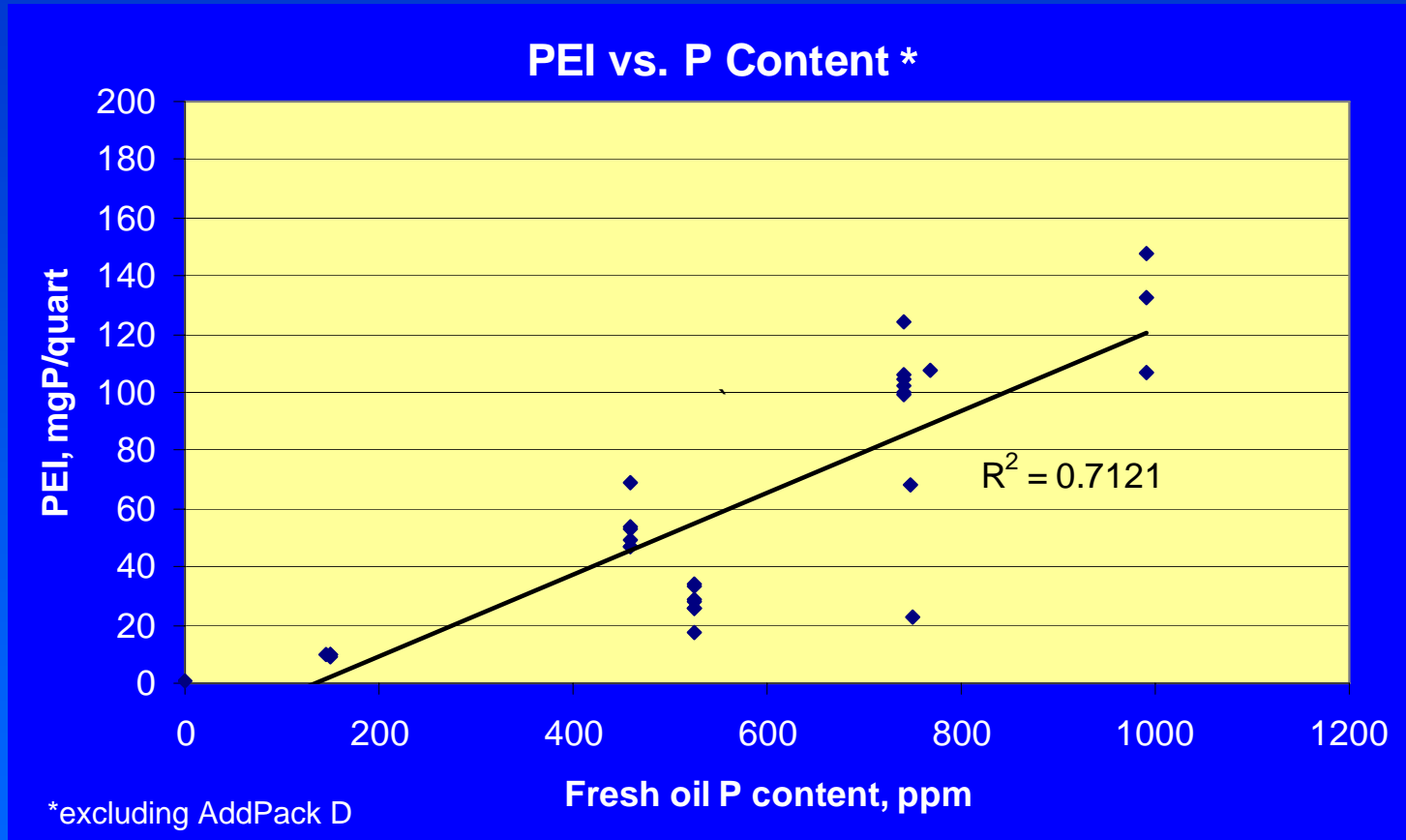


# Phosphorus Volatility via TEOST



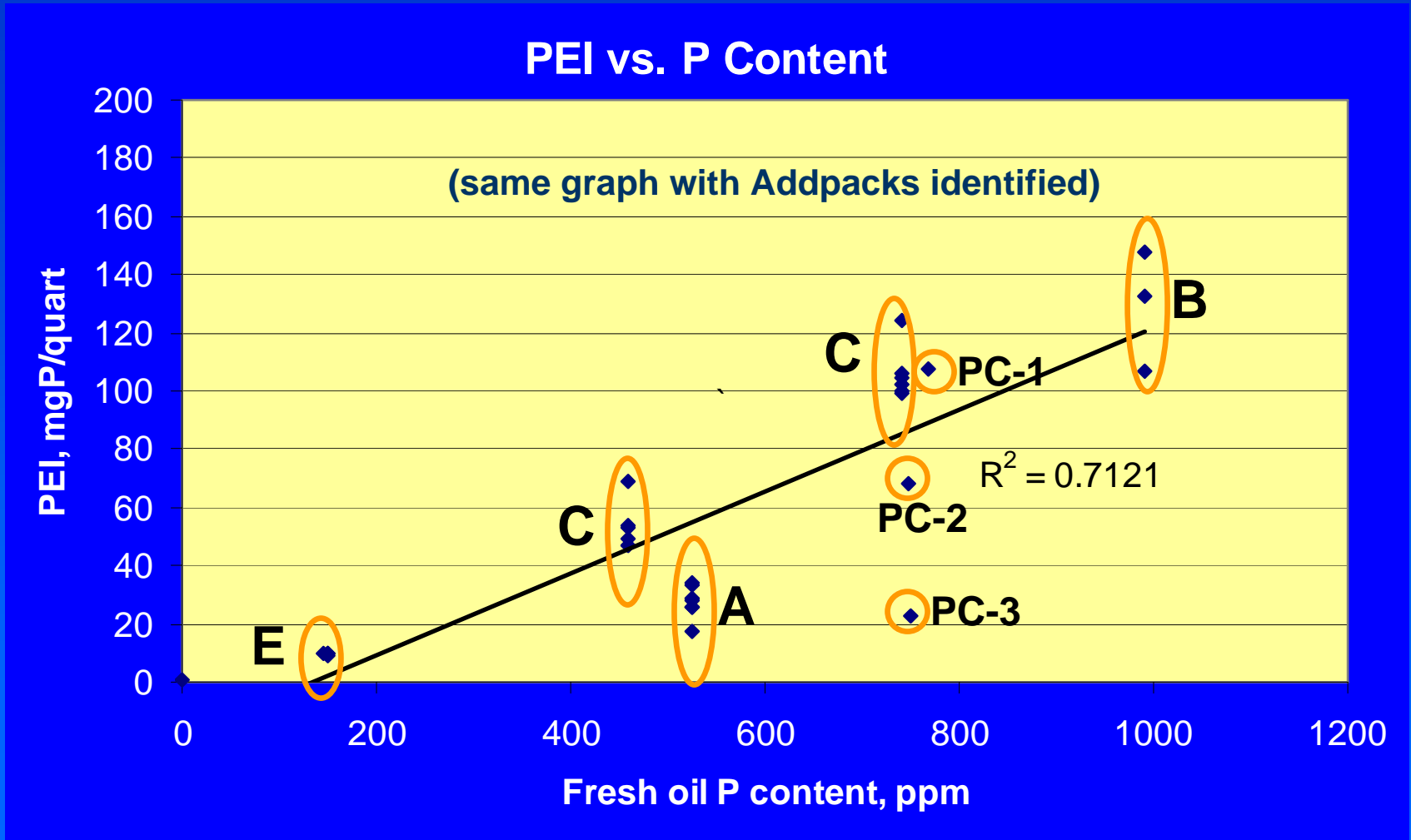
- There is no relationship between phosphorus retention and the phosphorus content of the fresh oil

# Phosphorus Volatility via TEOST

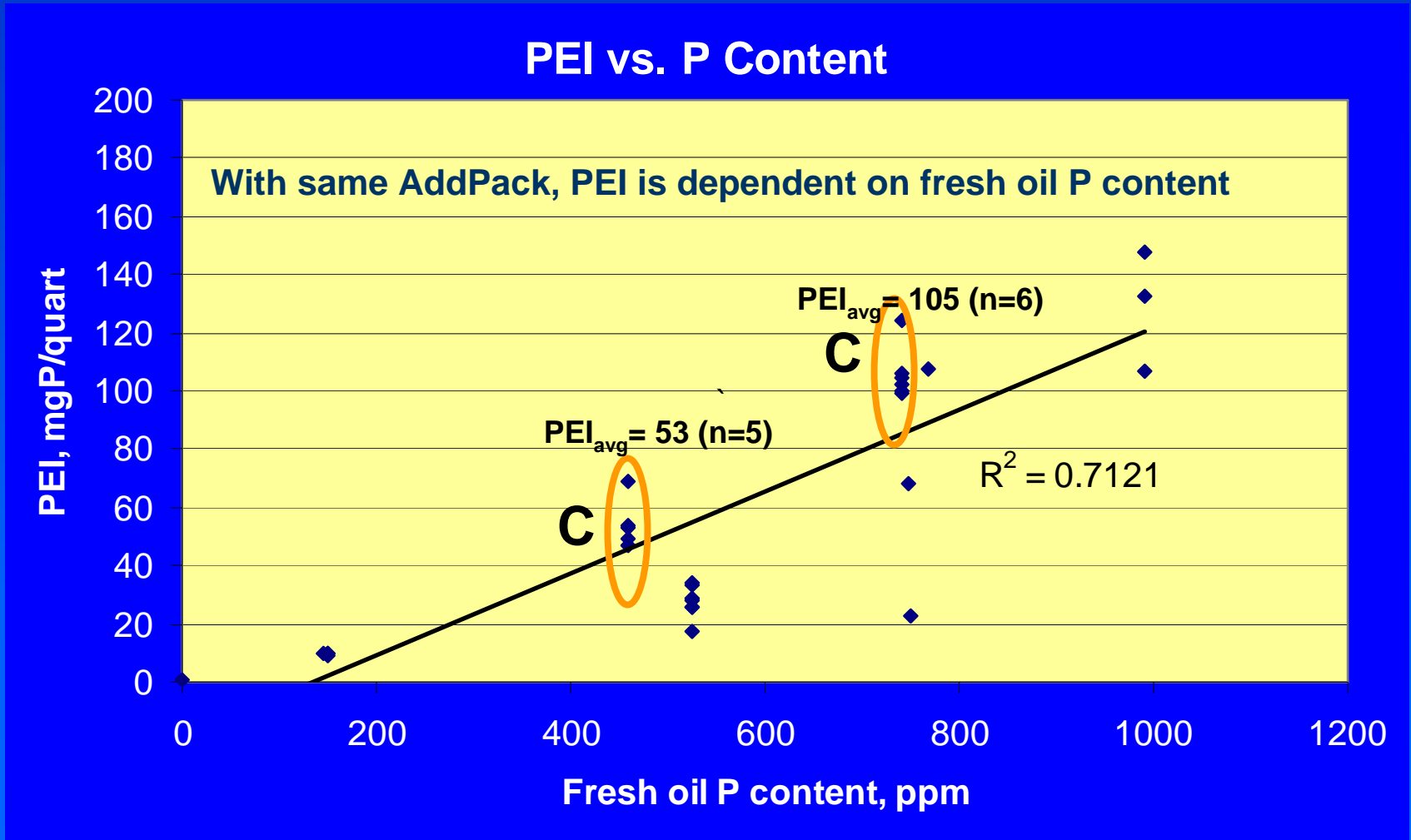


- There is a relationship between PEI and P content of the fresh oil
  - the higher the fresh oil content, the higher the PEI

# Phosphorus Volatility via TEOST

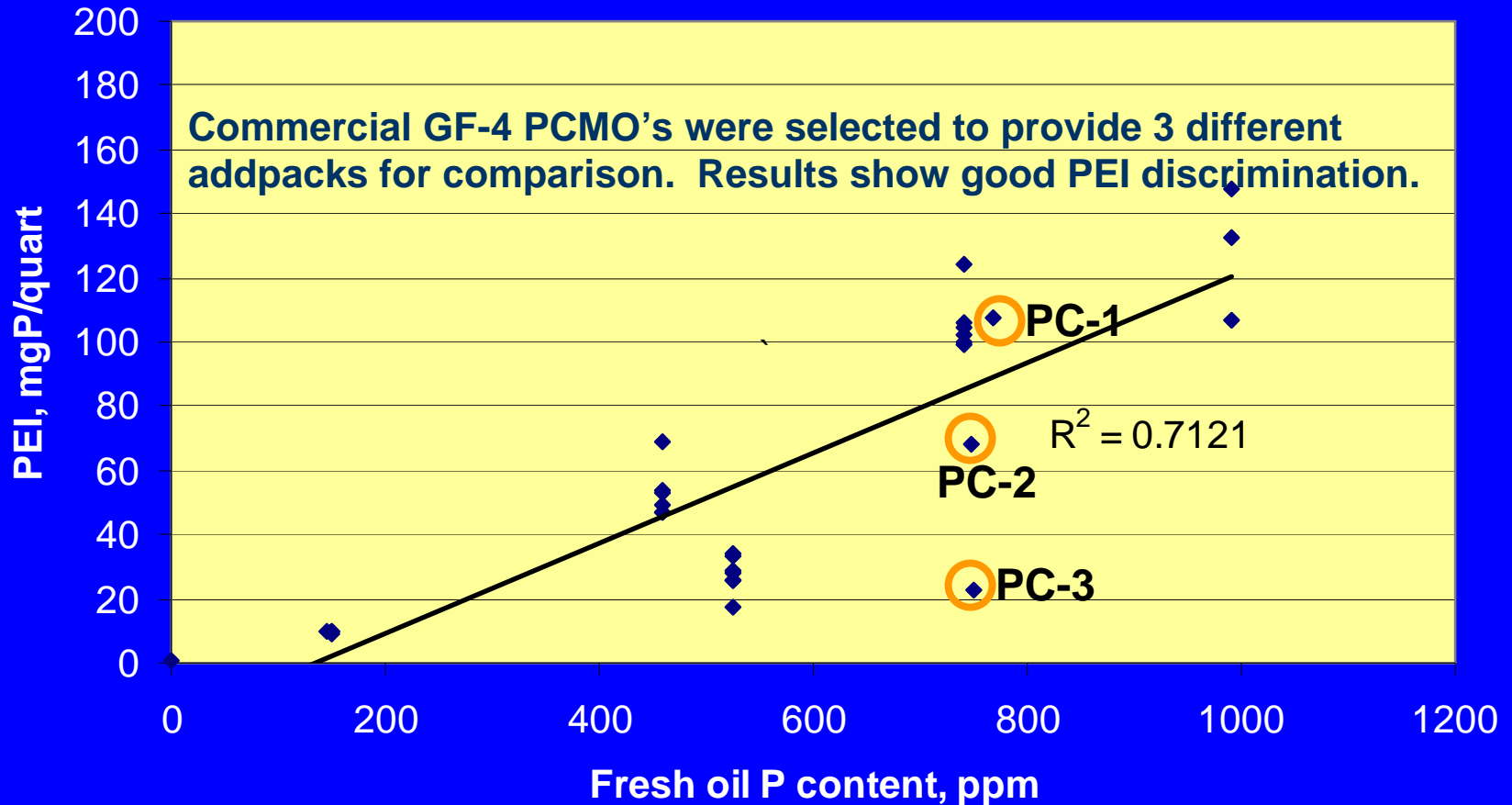


# Phosphorus Volatility via TEOST



# Phosphorus Volatility via TEOST

## PEI vs. P Content



# Phosphorus Volatility via TEOST

## Test Precision

- One sample with AddPack C was run in duplicate
- One sample with Addpack A was run in triplicate
- Tests run on different days with same operator

AddPk	Wv	Pv	Wf	Pf	%PR	PEI
A	2.59	138	8.42	~500	91.9%	36
A	2.53	122	8.41	~500	93.0%	31
A	3.65	93	8.43	~500	92.3%	34
C	4.12	256	8.43	~750	83.1%	106
C	3.76	269	8.41	~750	83.7%	102

$33.7 \pm 2.5$   
 $92.4\% \pm 0.6\%$

$104 \pm 2.8$   
 $83.4\% \pm 0.4\%$

# Phosphorus Volatility via TEOST

## Conclusions

- TEOST MHT-4PV (phosphorus volatility) appears to have acceptable discrimination and precision to be considered by ESCIT as candidate test method for measurement of phosphorus volatility
- ASTM D 7097 is an existing test method which does not require further method development
  - already part of ILSAC GF-4 & API SL/SM specifications
- Further evaluation of ESCIT oils is warranted

# Phosphorus Volatility via TEOST

## Acknowledgement

- Many thanks to Mike Russo in RTV Petroleum Applications Laboratory for his help in organizing the TEOST MHT-4 testing, and to Dee Gomez in RTV Analytical Laboratory for ICP testing