ASTM New Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

April 15, 2024

Yong-Li McFarland, Chair

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EOFT and EOWTT Surveillance Panel Membership

20 members

Beth Schwab, Afton Chemical
Michael Kunselman, Center for Quality Assurance
Robert Stockwell, Chevron Oronite
Quanchang Li, ExxonMobil
Michael Deegan, Ford
Ron Shah, Infineum
Joe Franklin, Intertek
Karina Gil, Intertek
Michael Johnscher, ISP
Litchi Xie, Lubrizol Additive (Zhuhai) Co., Ltd.
Victoria Fein, Lubrizol
Jason Bowden, OH Technologies Inc
Greg Miiller, Savant Group
Maggie Smerdon, Savant Labs
Sean Alston, SGS North America
Jared Cavaliere, SwRI
Becky Grinfield, SwRI
Yong-Li McFarland*, SwRI
John Loop, TMC
Amy Ross, Valvoline

*Chair

EOFT D6795 and EOWT D6794 New Item

- ASTM D6795 is up for 5-year review in 2024, ASTM D6794 is up for review in 2025
- Looking for participants for suggested edits and improvements- Maggie, Rachelle, Victoria, John L volunteered
- Let Yong-Li know if can help by April 3
- Group will meet in May

New EOGT WK86363, ILS# 1854

- Ford request for a new Engine Oil Gelation Test (EOGT): request to add a new test filterability test to better screen oils for field issues
- Current status
  - Method: 2 drafts (large volume (600g) and small volume (200 g)) uploaded on ASTM Collaboration Area, email Jared with procedure questions
  - Oils: 11 potential reference oils offered; 17 oils received at TMC
  - Screening Tests, Pilot Study, and ILS: Screening Test completed, start additional Proposal testing
  - Timing: Pilot Study tests run by June, ILS tests to be run by October, and final method ballot in January or February 2025

Other Notes/updates – April 12

- Previously FTIR on filter was looked at – Ford to check if there's method on how IR was done (ATR, combined FTIR-EDS, can data be shared or quantified?- Rob to look into): See slides
- Concern about variation in results due to different extraction processes
- Is XRF/XRD possible?
- Was Ca sulfonate grease used in previous system? – No grease was used in manufacturing process
- dispersing procedure consistent?
- Filter media questions; filter confirmation; consider changing porosity
- particle count, particle size on EOT and SOT
- TMC to confirm on burette tip size
- request Type III water contaminant analysis from ISP and SwRI
- Angela, Rachelle, (ask Philipp to join) and YM to set up fishbone diagram: See slides from subgroup
- ask ISP to repeat differentiating run-Confirm if possible: ISP estimates tests could complete in August
- used about 50% of Oils F and K from TMC
- ISP to show video of how they run EOGT during April 15 meeting: please watch for any differences or items for clarification

Calcium carbonate analysis

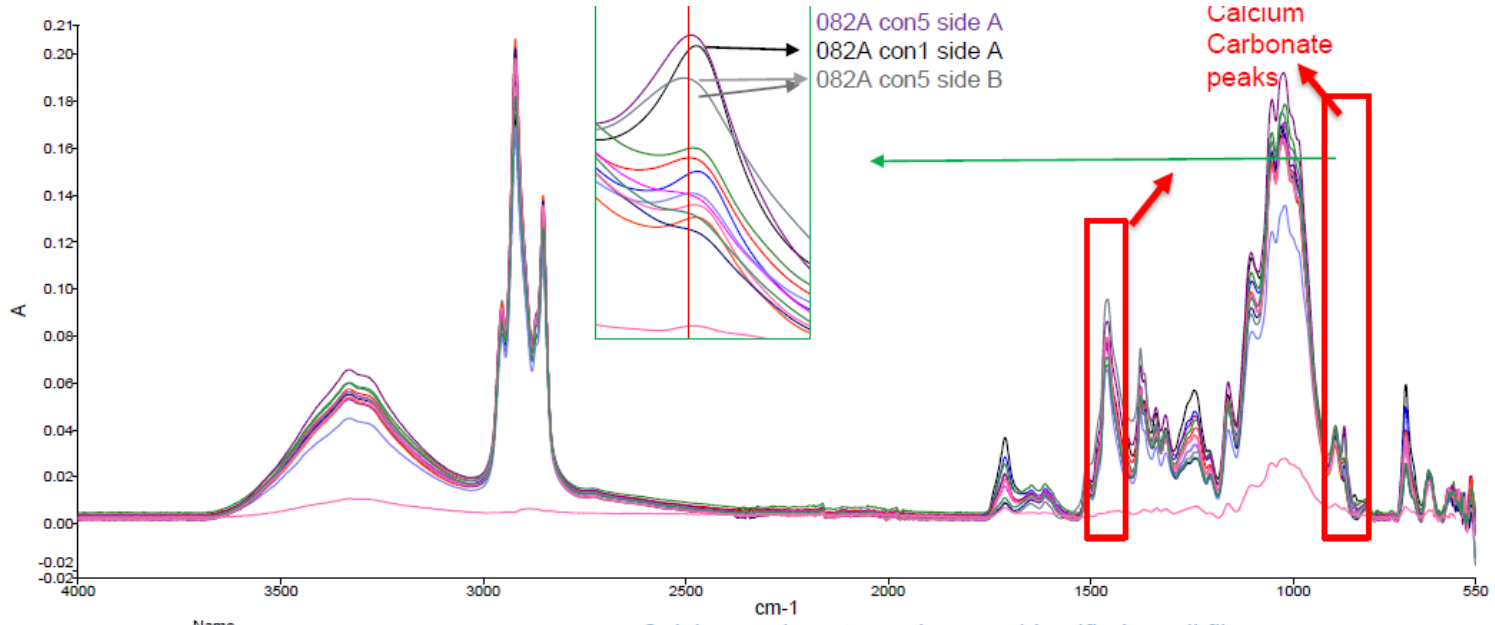
- The FTIR analysis suggests that calcium carbonate was present in all (12) spectra shown in Slide 1 (3 oil formulations x 2 test conditions x 2 sides per filter). The poor performing oil (082A) gave a higher carbonate peak than the other oils at roughly 800 cm-1, but it is not clear whether the difference in peak height is significant.

- The SEM/EDS analysis was conducted early in the study. It looked at post-test filters from the poor oil that were run with different test conditions. The filters were washed with pentane prior to analysis.

Post-test filter analysis – IR

082A = poor performing oil in the field
307A = good performing oil in the field

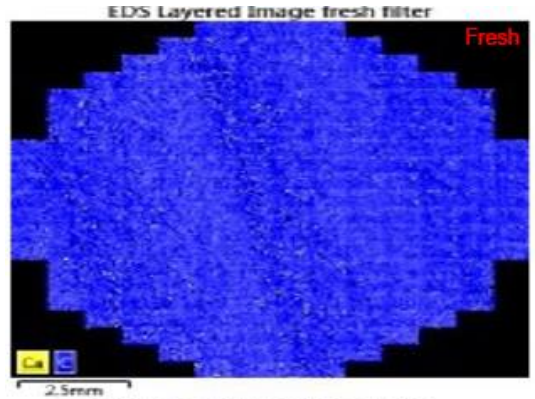
Identification of filters with calcium carbonate via FTIR ATR analysis

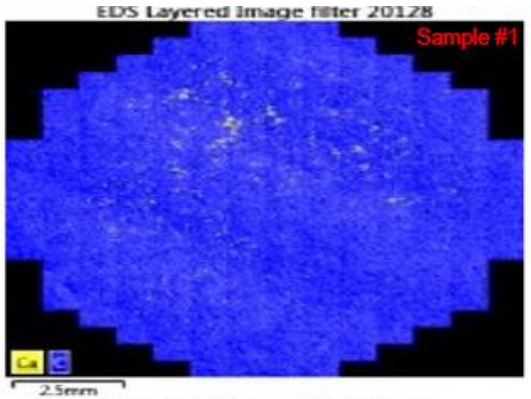


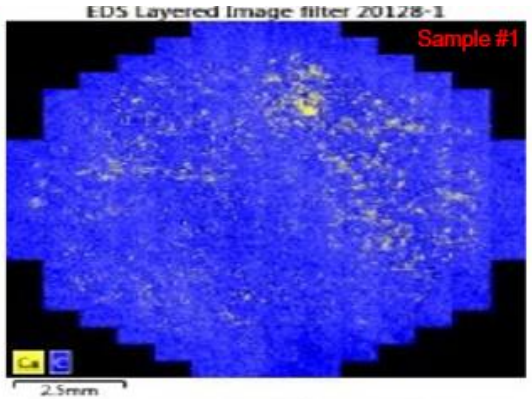
Name
IAR03968 082A condition 1 side A_1
IAR03968 082A condition 1 side B_1
IAR03968 307A condition 1 side A_1
IAR03968 307A condition 1 side B_1
IAR03968 323A condition 1 side A_1
IAR03986 323A condition 1 side B_1
IAR03986 082A condition 5 side A_1
IAR03986 082A condition 5 side B_1
IAR03986 307A condition 5 side A_1
IAR03986 307A condition 5 side B_1
IAR03986 323A condition 5 side A_1
IAR03986 323A condition 5 side B_1
IAR03886 fresh filter_1

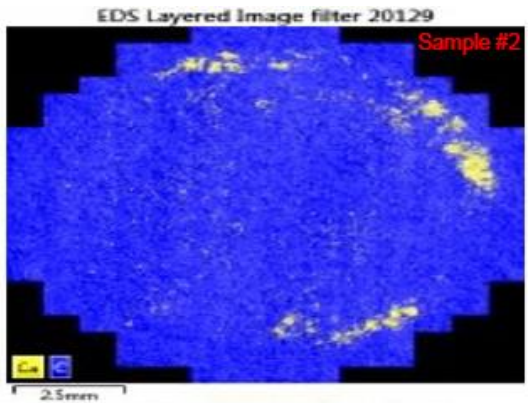
- Calcium carbonate peaks were identified on all filters.
- Filter 082A con5 side A & side B, and 082A con1 side A showed the highest Calcium carbonate peak intensities. The other filters showed very similar Calcium carbonate peak intensities.
- It is worth noting, this technique only analyse a small area of the samples. The result could be affected by which area of the sample was analysed.

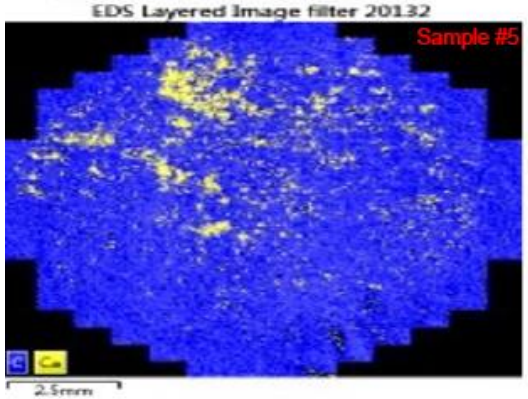
Post-test filter analysis – SEM EDS

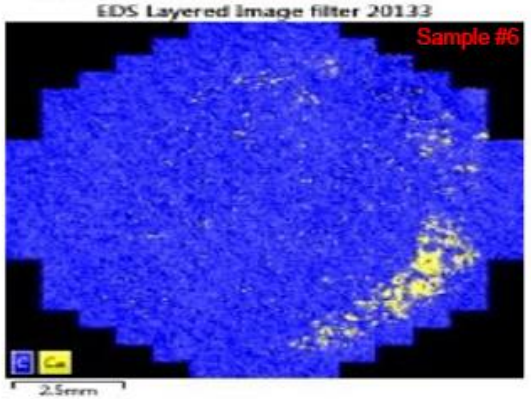


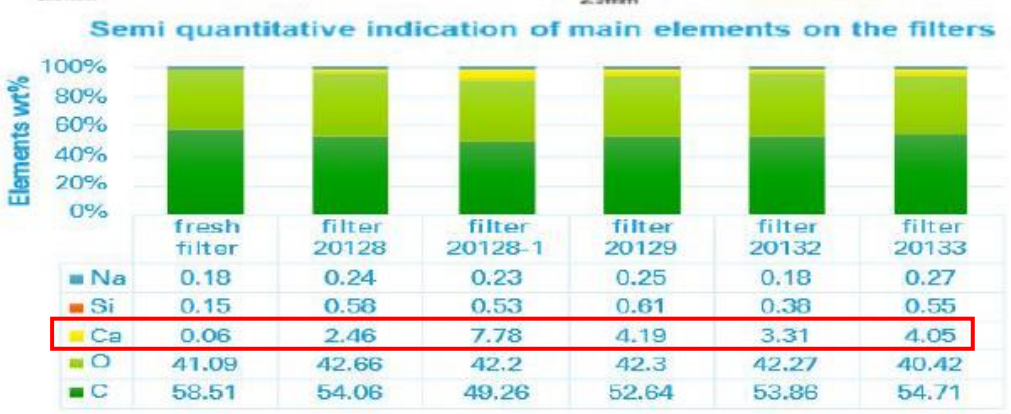








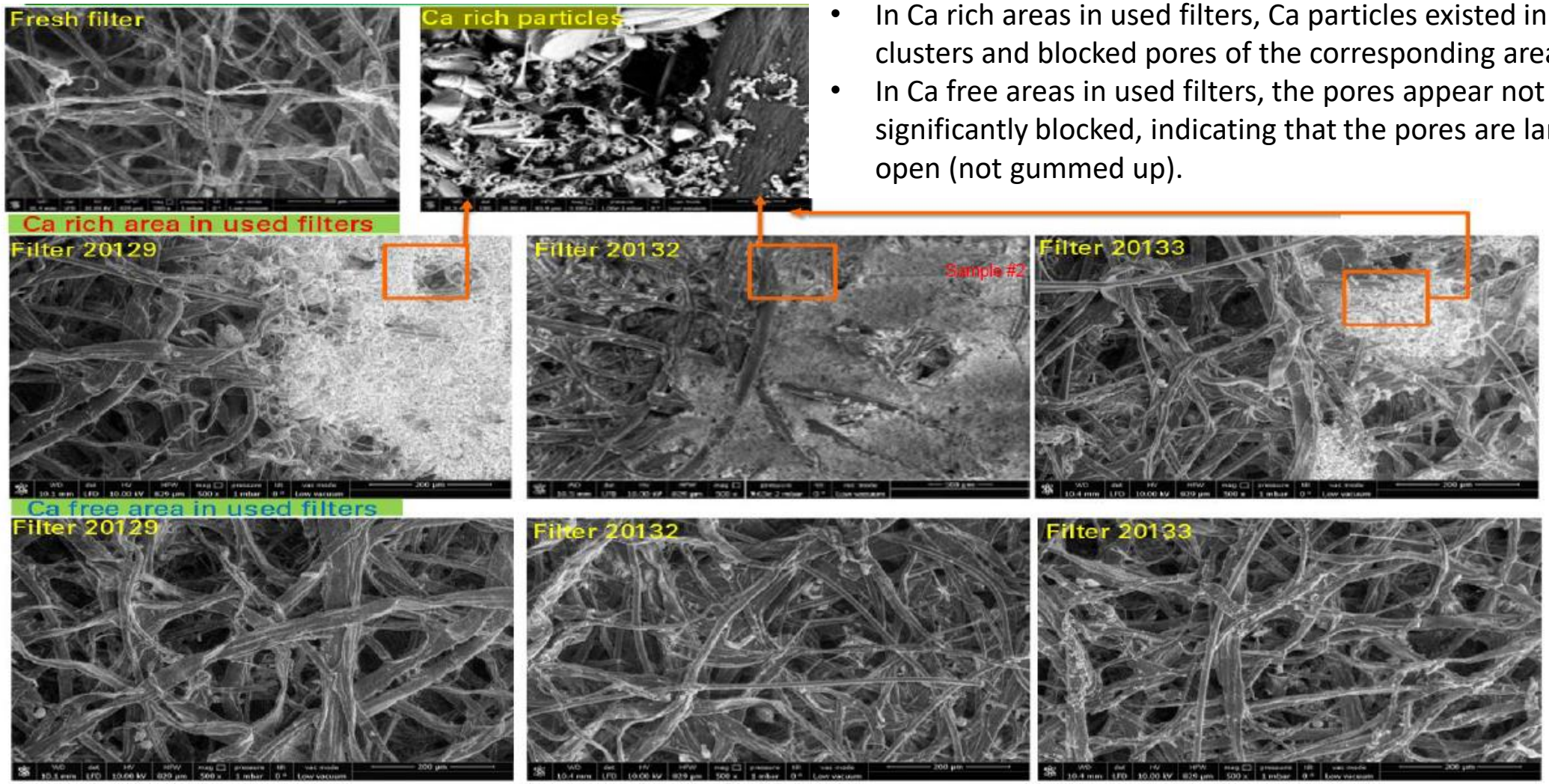




	fresh filter	filter 20128	filter 20128-1	filter 20129	filter 20132	filter 20133
Na	0.18	0.24	0.23	0.25	0.18	0.27
Si	0.15	0.58	0.53	0.61	0.38	0.55
Ca	0.06	2.46	7.78	4.19	3.31	4.05
O	41.09	42.66	42.2	42.3	42.27	40.42
C	58.51	54.06	49.26	52.64	53.86	54.71

- Compared to the fresh filter, more Calcium (Ca) was detected on the used filters
- Filter20128-1 showed higher Ca than the other used filters when filter 20128 showed lower Ca than the other filters. Further analysis will be carried out on all filters in 20128 batch.

Post-test filter analysis – SEM Images



- In Ca rich areas in used filters, Ca particles existed in clusters and blocked pores of the corresponding area.
- In Ca free areas in used filters, the pores appear not to be significantly blocked, indicating that the pores are largely open (not gummed up).

EOGT Root Cause Team

Fishbone Diagram

Angela Willis, 12 April 2024

Brief Overview - Fishbone Diagram for Root Cause

Multi-Step Process

1. Document problem statement
2. Brainstorm potential causes to problem by category
   - Six categories: Methods, Measurements, Equipment, Materials, People, and Environment
3. Determine activities needed to eliminate or confirm potential causes - includes prioritization of investigation
4. Eliminate potential causes using data-driven decisions
5. For causes that cannot be eliminated, identify sub-causes by asking "Why" questions
   - Determine activities needed to eliminate or confirm those sub-causes

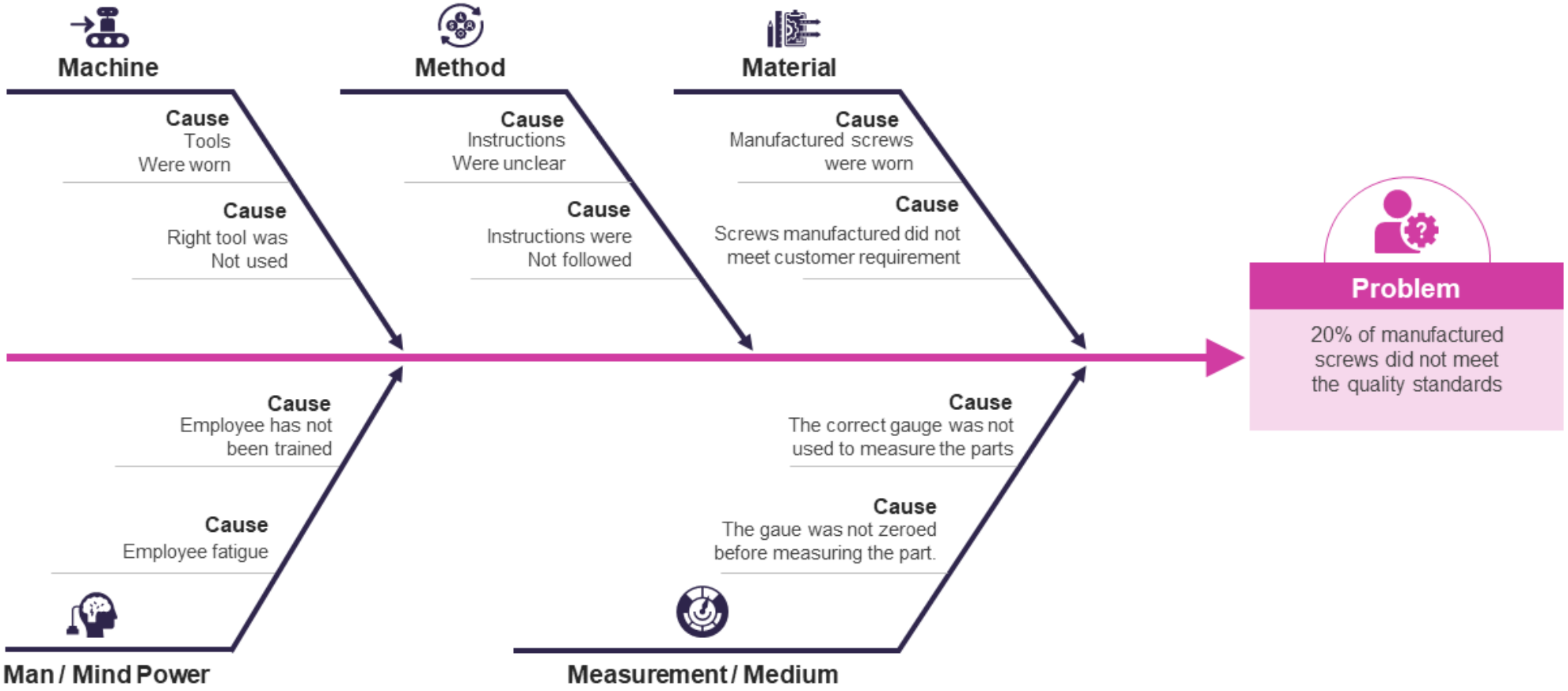
- Keep drilling down by asking "Why" questions and repeating the evaluation/elimination cycle until true root cause is identified.

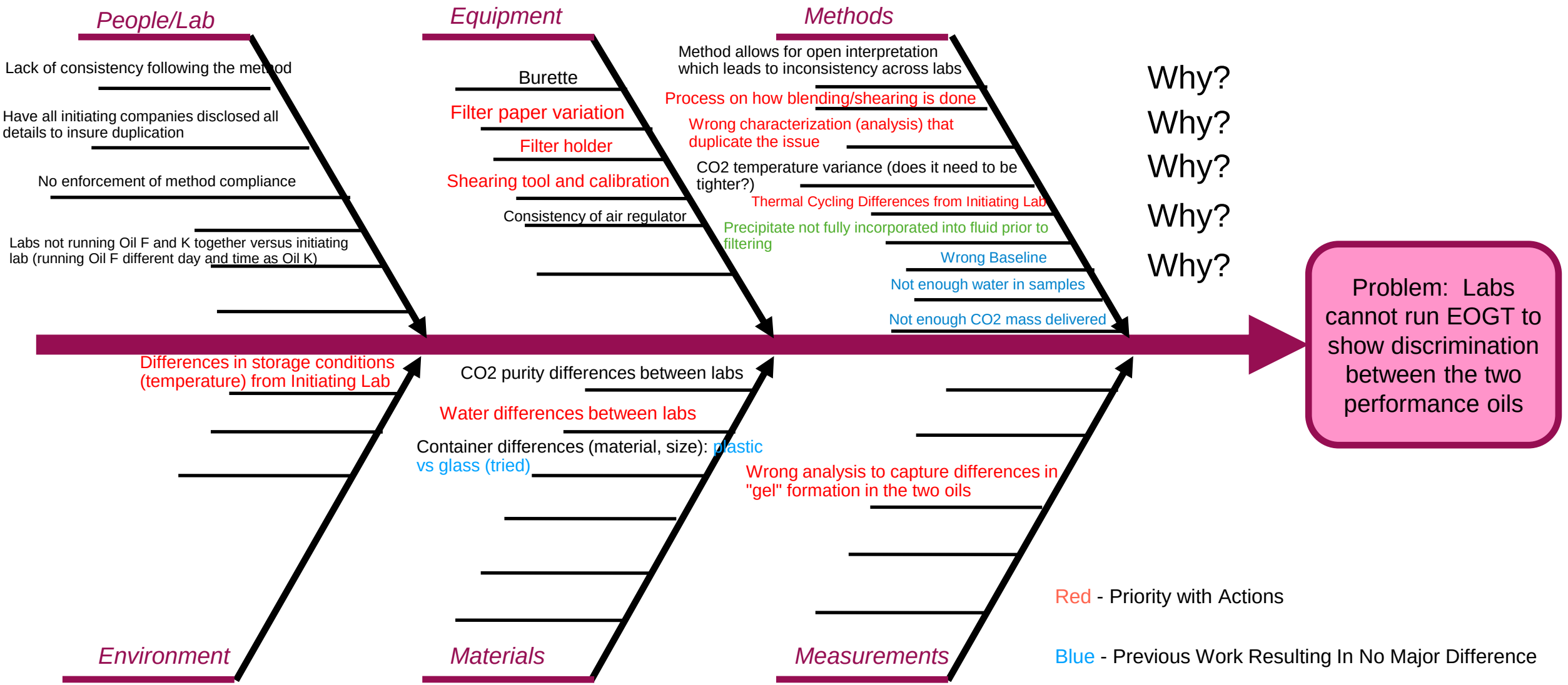
Fishbone Diagram is dynamic - serve as tool to prioritize activities and methodically identify the root cause

Example of Fishbone Diagram

Fishbone Diagram to Determine the Root Cause of Problem

Mentioned slide illustrates fishbone diagram that can be used by an organization to determine the root cause of a problem. It is also known as cause and effect diagram.



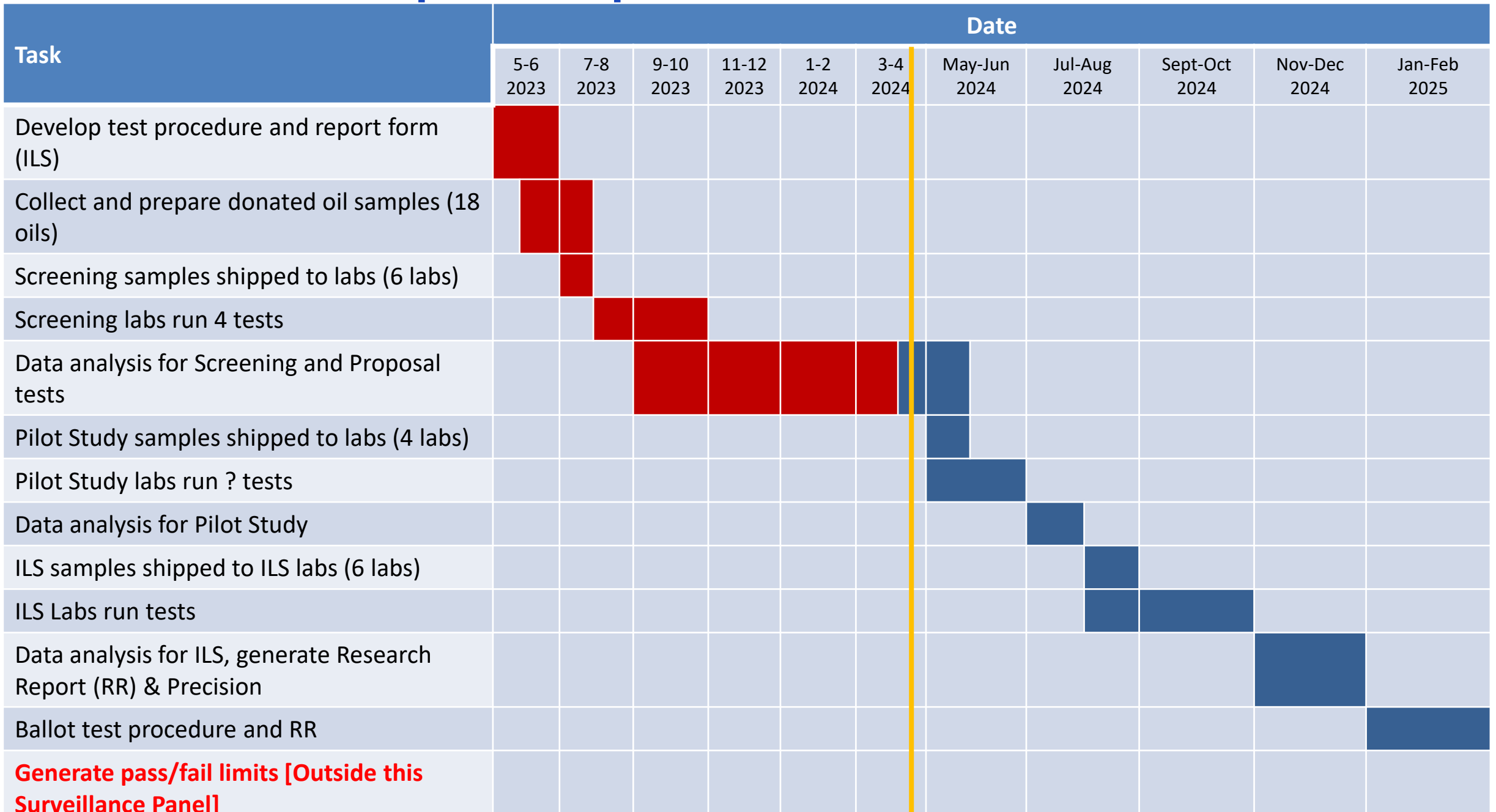

People/Lab
Lack of consistency following the method
Have all initiating companies disclosed all details to insure duplication
No enforcement of method compliance
Labs not running Oil F and K together versus initiating lab (running Oil F different day and time as Oil K)
Equipment
Burette
Filter paper variation
Filter holder
Shearing tool and calibration
Consistency of air regulator
Methods
Method allows for open interpretation which leads to inconsistency across labs
Process on how blending/shearing is done
Wrong characterization (analysis) that duplicate the issue
CO2 temperature variance (does it need to be tighter?)
Thermal Cycling Differences from Initiating Lab
Precipitate not fully incorporated into fluid prior to filtering
Wrong Baseline
Not enough water in samples
Not enough CO2 mass delivered
Why?
Why?
Why?
Why?
Why?
Problem: Labs cannot run EOGT to show discrimination between the two performance oils
Differences in storage conditions (temperature) from Initiating Lab
Environment
CO2 purity differences between labs
Water differences between labs
Container differences (material, size): plastic vs glass (tried)
Materials
Wrong analysis to capture differences in "gel" formation in the two oils
Measurements
Red - Priority with Actions
Blue - Previous Work Resulting In No Major Difference
Green - Previously Work Resulting in Difference

Previous Work By Main Team

- Increased CO2 mass into samples: *No differentiation*
  1. 2 tubes vs. 1 tube
  2. 4 hrs vs. 1 hr using 2 tubes
- Increased water concentration into samples - Up to 8%: *No differentiation*
- Included water into baseline sample - ran 0 hr (blended) filterability: *No differentiation*
- Precipitate not fully incorporated into samples prior to filterability: *Differentiation established*
  - A method was done by decanting off fluid and only filtering precipitate in samples
  - Resulted in differentiation between Oil F and K samples
  - *This creates the theory that homogenizing of EOT samples may be critical and merits more investigation.*

EOGT FISHBONE ACTION ITEMS LIST - Revised 12APRIL2024						
Category	**Cause**	**Sub-Cause**	**Action to Evaluate**	**Due Date**	**Status**	**Date Closed**
Method/Environment	Storage Time Temperature/Thermal Cycling	Cold temperature not being considered as a driving parameter	Savant performing "cold soak" on post test retains (repeat storage at 32F), bring to room temperature, then repeat filtration.	April 22	Started April 5	
Method	"Process of method execution" different between labs and initiating lab	Blending process using dispersing tool different between labs (not detailed in procedure)	Review ISP procedure execution video during Task Force Meeting (operators will be present).	April 15	Will share on April 15	
Environment	Differences in storage conditions from initiating lab	Storage temperature possibly differ over time (not in controlled environment)	At ISP, storage location was an office area. ISP will confirm if office area maintains 20-25C, or if temperature is lowered at during off-business hours (night time)	April 12	Heating system may go down to 16C, but unsure if office actually goes to that temp	4/12/24
Environment	Differences in storage conditions from initiating lab	Storage temperature possibly differ over time (not in controlled environment)	Request participating labs to monitor and report storage temperatures, as well as confirm they are in a controlled environment (set temperature, no fluctuations due to company energy savings)	April 22	YM to ask labs to provide response to TMC	
People/Lab	Labs not running Oil F and K together versus initiating lab. (Oil F run on different day from Oil K)		Angela to review data to check how many labs running 2 reference oils together and separately	Apr ?	Angela to share with this subgroup findings	
Equipment	Shearing tool and calibration	Differences in type of shearing tool used and how 18,000 rpm is calibrated may change results	ISP ask to share how rpm is calibrated	15-Apr	tool used hasn't been calibrated and uses setting 6 for 18,000 rpm; a new digital version has a connection to "calibrate"; ISP to check model number for new digital shearing tool;	
Materials	different water types used		Angela to share differences between Type I, II, III	Apr ?	Angela to share with this subgroup findings at next meeting	
Materials	filter paper variation	understand batch variability and if any differences in cutting filters	YM to ask OHT to provide 1) tolerances for pore size and beta efficiency from manf, 2) could share with main group their procedure for cutting filter paper and quality protecol to ensure integrity and compliance; 3) C of A possible to be shared	Apr ?	OHT to provide more details	
Equipment	Filter holder		YM ask labs to share part #, vendor and photo of holder to TMC	Apr ?		
Methods	homogenizing step		YM ask labs to check if homogenization step is accurately described in Data excel			

Draft Timeline – updated April 12



Action Items and Next Meeting

- Group to let Yongli know on direction for EOGT
- TMC to confirm burette tip opening with labs – waiting for 2 labs to report
- Root Cause subgroup will meet and report back to group on fishbone diagram.
- Savant to provide update on Cold Soak on post test retains
- Labs to monitor and report storage temperatures for where EOGT samples are stored and provide previous testing info to TMC
- Angela to review data on how many labs run 2 reference oils together vs separately
- ISP to provide model number for new digital shearing tool
- Angela to check water differences between Type I, II, and III
- OHT to provide info on filter tolerances for pore size and beta efficiency (if possible from OEM), share about process for 3rd party to cut filters and any quality protocol in process, and potential Certificate of Analysis for filters
- Labs to provide part number, vendor, and photo of filter holder to TMC; OHT also offered info on part
- Labs to check if homogenization step description is accurate and add details to Data Excel
- Eugene (Afton) to look into FTIR peak in ISP data around 2300 wavenumber to see what peak may be – To check for next meeting

- Next Meeting: Monday April 29 at 8:30AM CDT

Thank you for your support!

Participants		
Method Development (11)	Oil Donations (9)	Testing Labs (7)
Afton ExxonMobil Ford Infineum Intertek ISP Lubrizol Oronite Savant SwRI TMC	Afton Ford Infineum Lubrizol OH Technologies (donate filters only) Oronite Subaru TMC (collection, shipping only) Toyota	Afton (Screening, Pilot, and ILS) Intertek (Screening, Pilot, and ILS) ISP (Screening, Pilot, and ILS) Savant (Screening and ILS) SwRI (Screening, Pilot, and ILS) TMC (monitoring system only) Valvoline (Screening and ILS)