U.S. ARMY TANK AUTOMOTIVE RESEARCH, DEVELOPMENT AND ENGINEERING CENTER

# A Users Perspective and Experience with Particle Counting in Liquid Fuels


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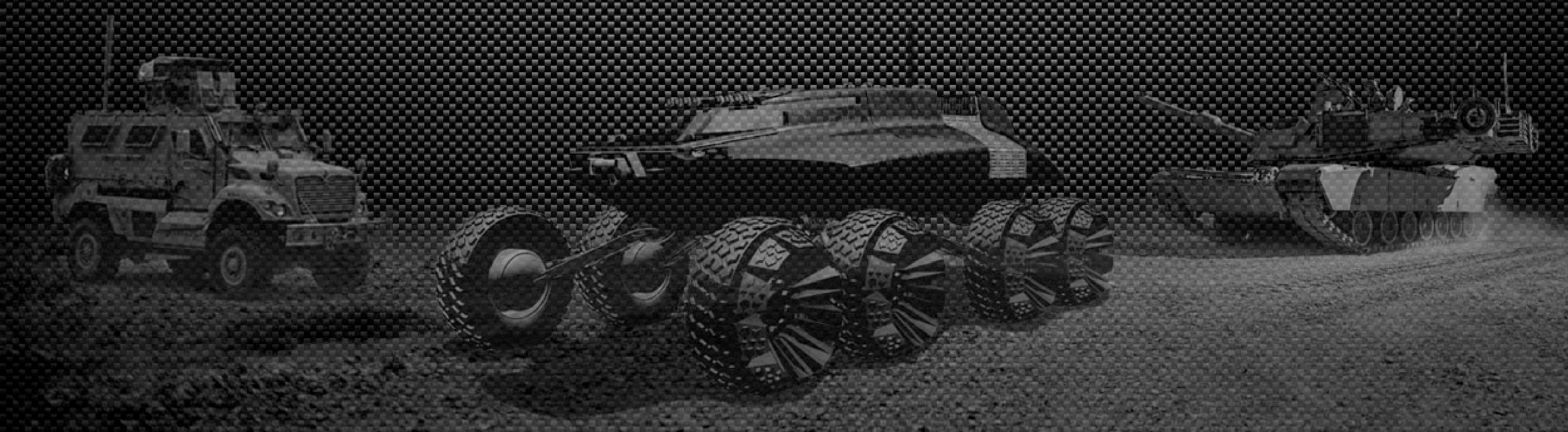
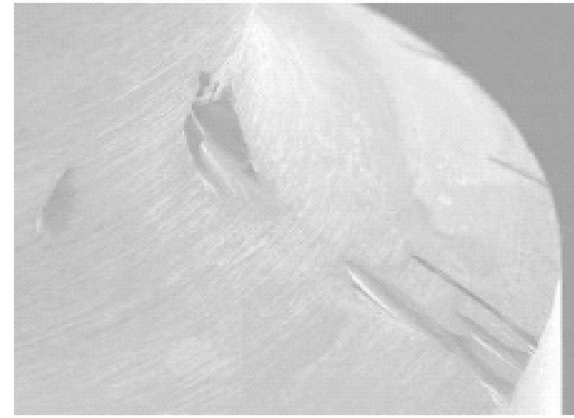
6 APR 2018

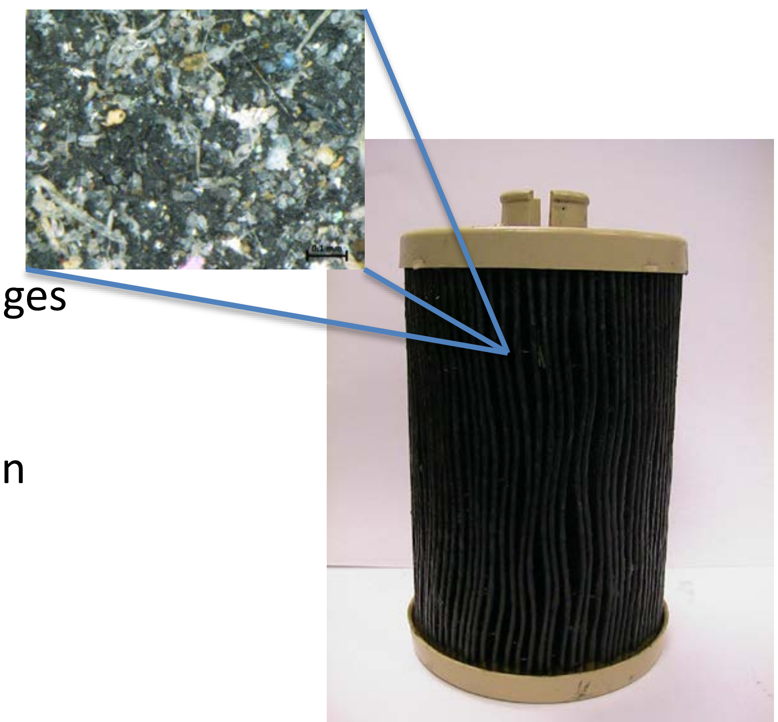




# Concern over particulate contamination



- Wear within the fuel system and fuel injection equipment due to particle erosion
  - Particle hardness is more important than particle density
- Filter plugging
  - Reduced filter life, or engine fuel starvation
    - Particle size and quantity are more important than contaminant mass



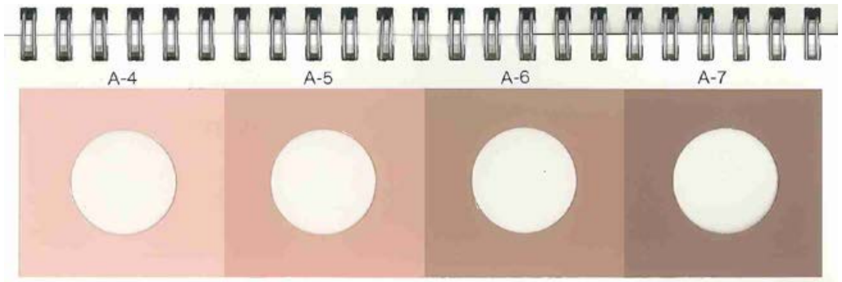
- Fuel system passage blockage
  - Ultrafine particulate can restrict fuel passages and movement between fine tolerance components
    - Particulate size is more important than contaminant mass

# Legacy Fuel Contamination Monitoring Methods

- ASTM D2276 – Particulate Contamination in Aviation Fuel by Line Sampling
  - gravimetric limit 1.0 mg/L (MIL-STD-3004, MIL-DTL-83133)
    - *Limit first appeared in MIL-T-5624G - 5 NOV 1965)*
- ASTM D5452 – Particulate Contamination in Aviation Fuels by Laboratory Filtration
  - gravimetric limit 1.0 mg/L  (MIL-STD-3004)
- ASTM D3240 – Undissolved Water in Aviation Turbine Fuels
  - 10 PPM (MIL-STD-3004, ATP 4-43)
- ASTM D4176 – Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)
  - Clear and Bright

# Legacy Fuel Contamination Monitoring Methods

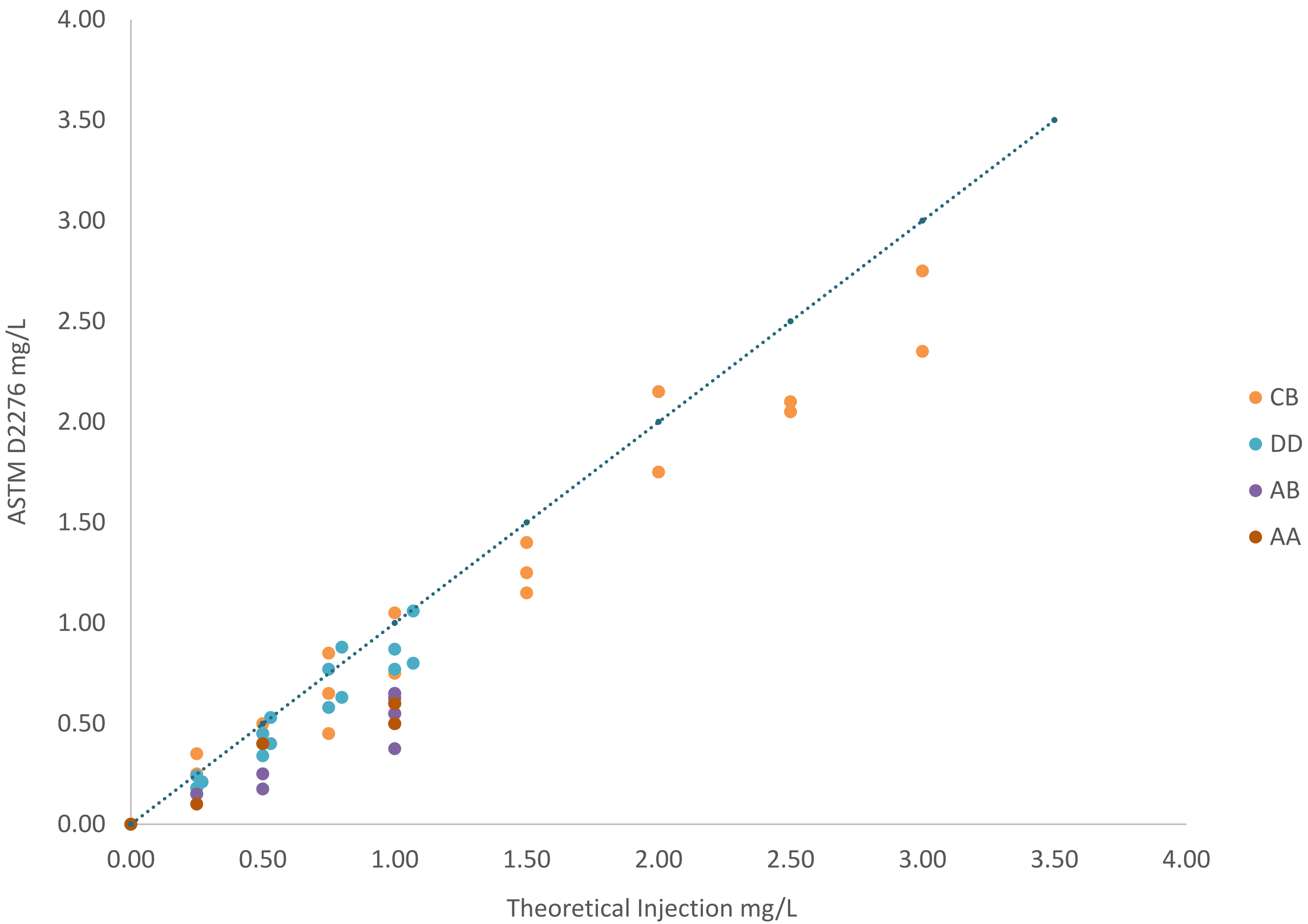
- Drawbacks:
  - Operator subjectivity (ASTM D2276 color comparison)




  - Lack of detail (ASTM D2276 gravimetric)

  - Large sample volumes (500mL – 5 Liters)
  - Potential contamination
  - Time consuming
  - Poor repeatability
  - Questionable accuracy

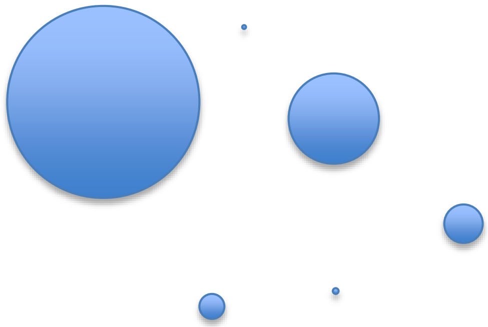
# EI 1581 Test Loop Injections vs ASTM D2276



# Particulate Hardness and size

| common name | formula | Mohs hardness | density $g/cm^3$ | ISO 12103-1 A1 [1] | 18 field samples median [2] |
|---|---|---|---|---|---|
| silica | $SiO_2$ | 7 | 2.65 | 69-77% | 36.60 |
| aluminum oxide | $Al_2O_3$ | 9 | 3.95 | 8-14% | 15.85 |
| Hematite (iron (III) oxide) | $Fe_2O_3$ | 5-6 | 5.30 | 4-7% | 3.4 |
| Magnetite | $Fe_3O_4$ | 5.5-6.5 | 5.15 | | |
| Calcium oxide | CaO | 3.5 | 3.34 | 2.5-5.5% | 7.45 |
| potassium chloride | KCl | 2 | 1.98 | 2-5% | |

| Ø µm | $\mu m^3$ |
|---|---|
| 0.8 | 0.27 |
| 1 | 0.52 |
| 4 | 33.51 |
| 6 | 113 |
| 14 | 1436 |
| 30 | 14137 |



[1] ISO 12103-1, A1 Ultrafine Test Dust - Powder Technology Inc.
[2] BFLRF No. 294 Characterization of CONUS and Saudi Arabian fine-grained soil samples

# Electronic Contamination Monitoring

- DoD has publish particle count limits in MIL-STD-3004 for aviation turbine fuel and in MIL-DTL-83133.

- MIL-DTL-5624 and DEF STAN 91-091 include a requirement to only report particle counting measurements.

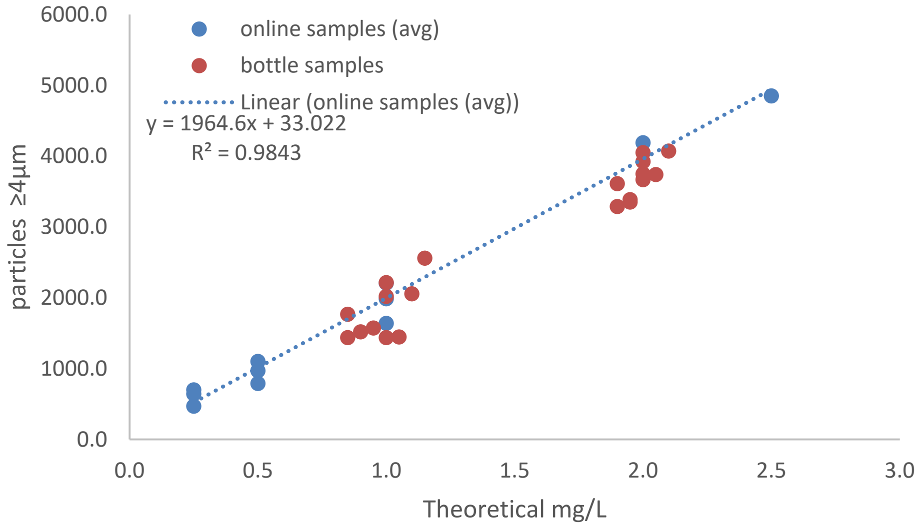
- IP 564/ASTM D8166 – Parker ACM20
- IP 565/ASTM D7619 – Stanhope-Seta AvCount Series
- IP 577 – Pamas S40

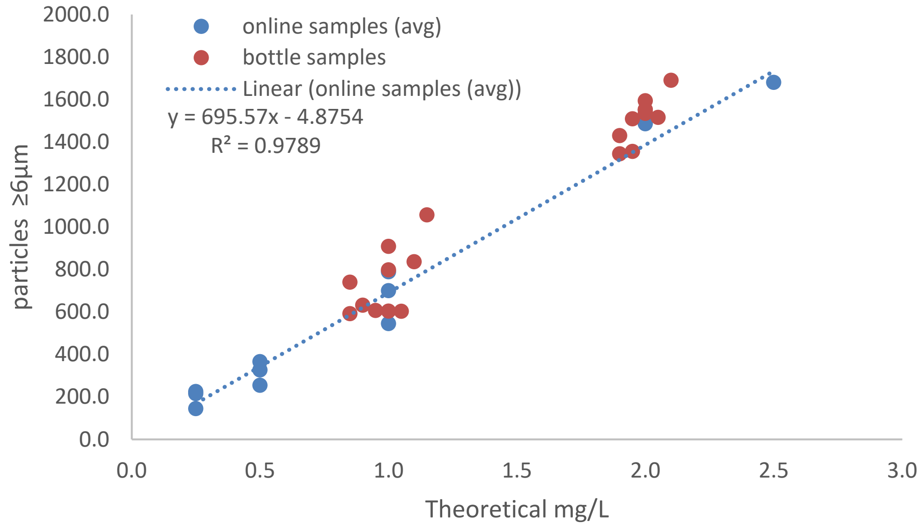

# Particle Counter Methodology

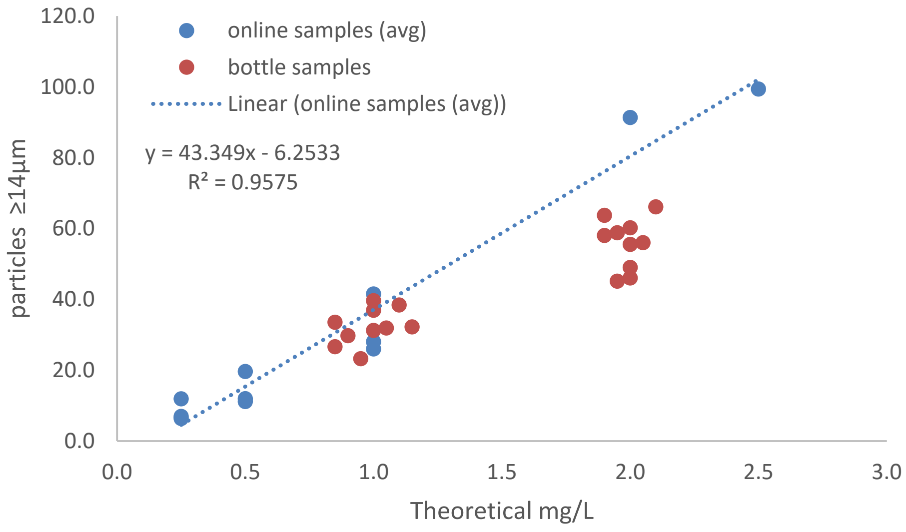
- Particle counts are taken utilizing calibration methodologies and standardized cleanliness code ratings
  - ISO 11171
  - ISO 4406
- Particle Count ISO 4406 code 19/17/14/13 limits of 4µm (c) /6µm (c)/14µm (c)/30µm (c) corresponding to 1.0 mg/L
- Limit can be applied directly to instruments reporting µm (b) if available

| Number of particles per millilitre | | Scale number |
|---|---|---|
| More than | Up to and including | |
| 2 500 000 | | >28 |
| 1 300 000 | 2 500 000 | 28 |
| 640 000 | 1 300 000 | 27 |
| 320 000 | 640 000 | 26 |
| 160 000 | 320 000 | 25 |
| 80 000 | 160 000 | 24 |
| 40 000 | 80 000 | 23 |
| 20 000 | 40 000 | 22 |
| 10 000 | 20 000 | 21 |
| 5 000 | 10 000 | 20 |
| 2 500 | 5 000 | 19 |
| 1 300 | 2 500 | 18 |
| 640 | 1 300 | 17 |
| 320 | 640 | 16 |
| 160 | 320 | 15 |
| 80 | 160 | 14 |
| 40 | 80 | 13 |
| 20 | 40 | 12 |
| 10 | 20 | 11 |
| 5 | 10 | 10 |
| 2,5 | 5 | 9 |
| 1,3 | 2,5 | 8 |
| 0,64 | 1,3 | 7 |
| 0,32 | 0,64 | 6 |
| 0,16 | 0,32 | 5 |
| 0,08 | 0,16 | 4 |
| 0,04 | 0,08 | 3 |
| 0,02 | 0,04 | 2 |
| 0,01 | 0,02 | 1 |
| 0,00 | 0,01 | 0 |

# ISO 12103-1 A3 medium test dust evaluation



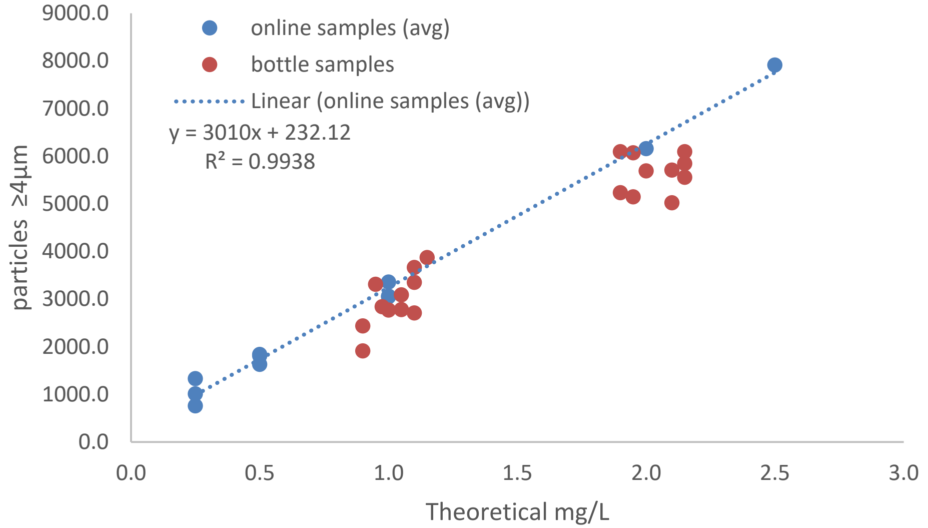



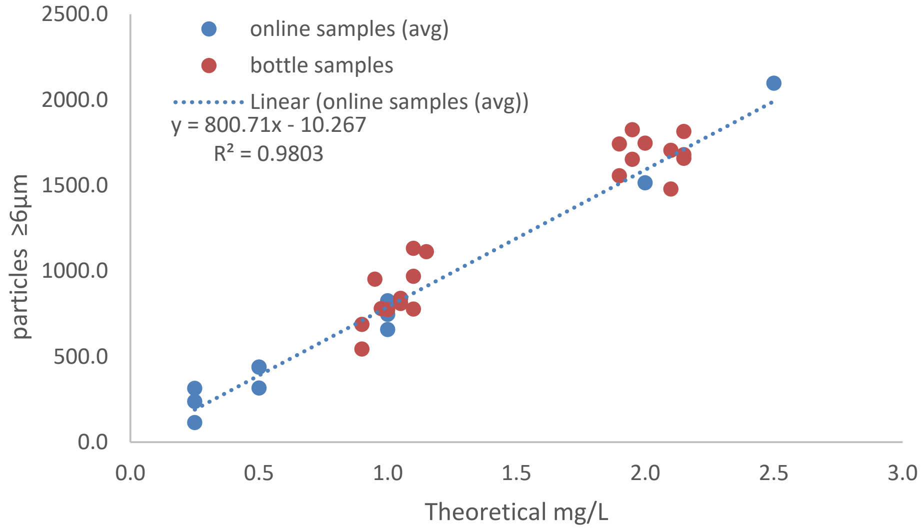



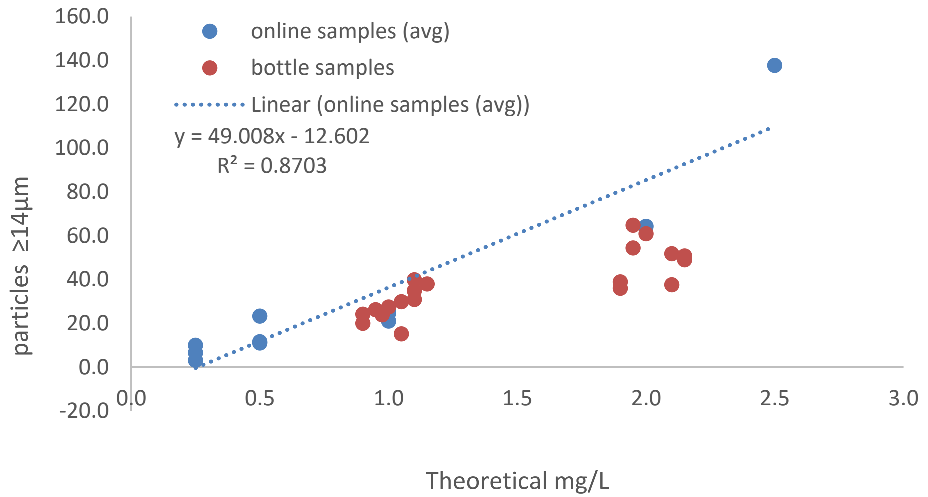

1.0 mg/L ~ 18/17/12

0.5 mg/L ~ 17/16/11

# ISO 12103-1 A2 fine test dust evaluation



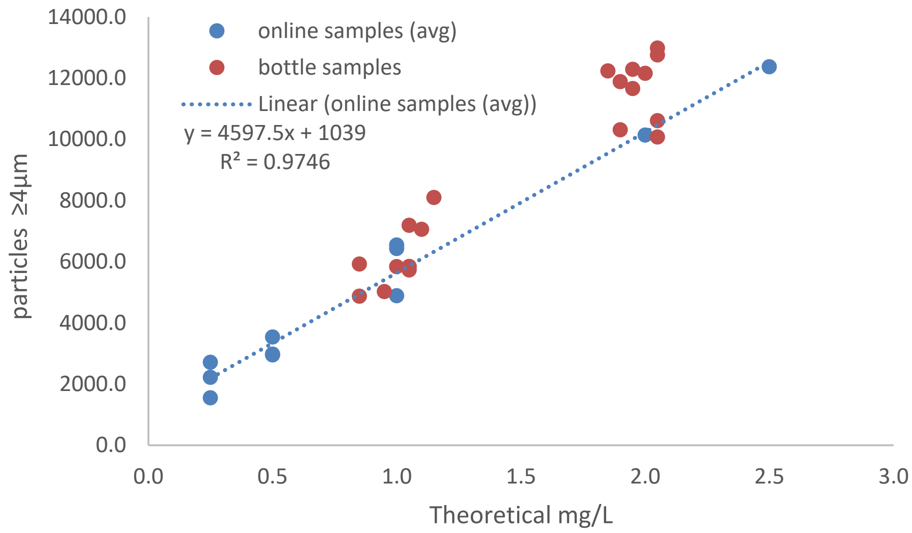



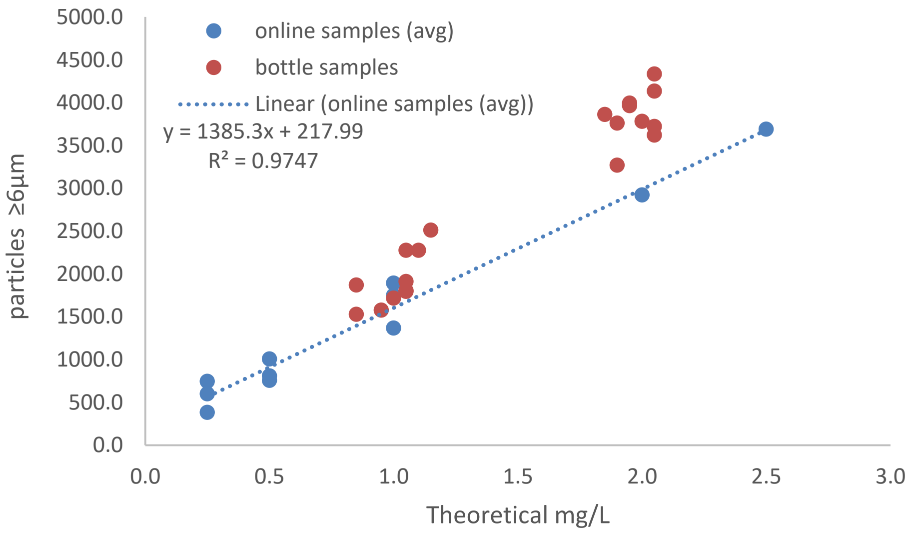



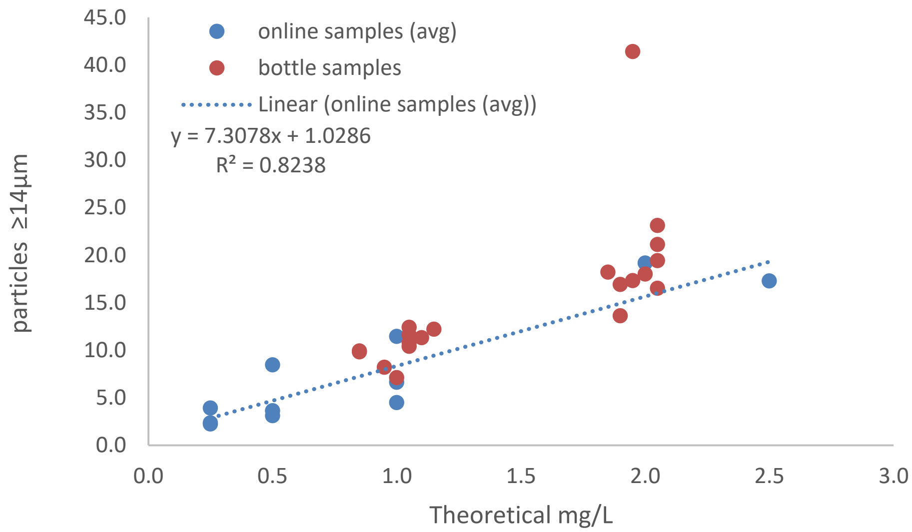

1.0 mg/L ~ 19/17/12

0.5 mg/L ~ 18/16/11

# ISO 12103-1 A1 ultrafine test dust evaluation



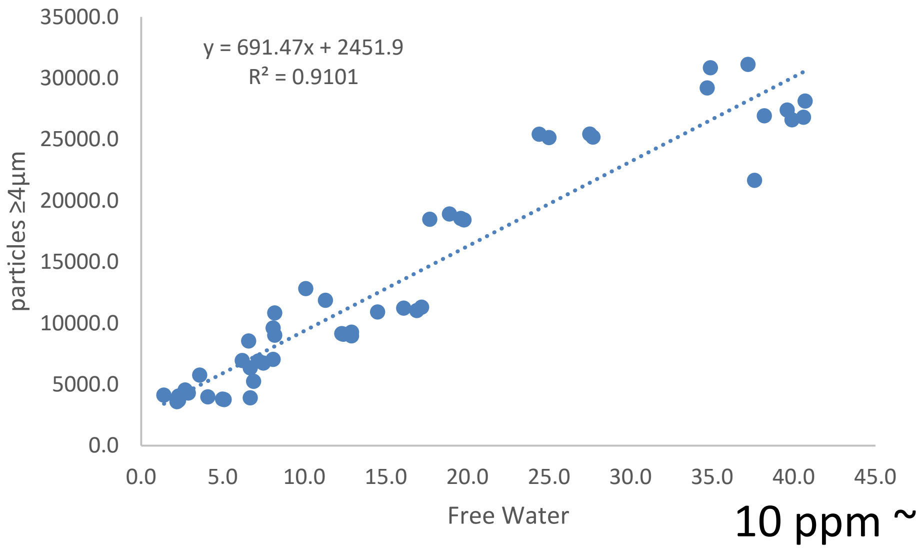



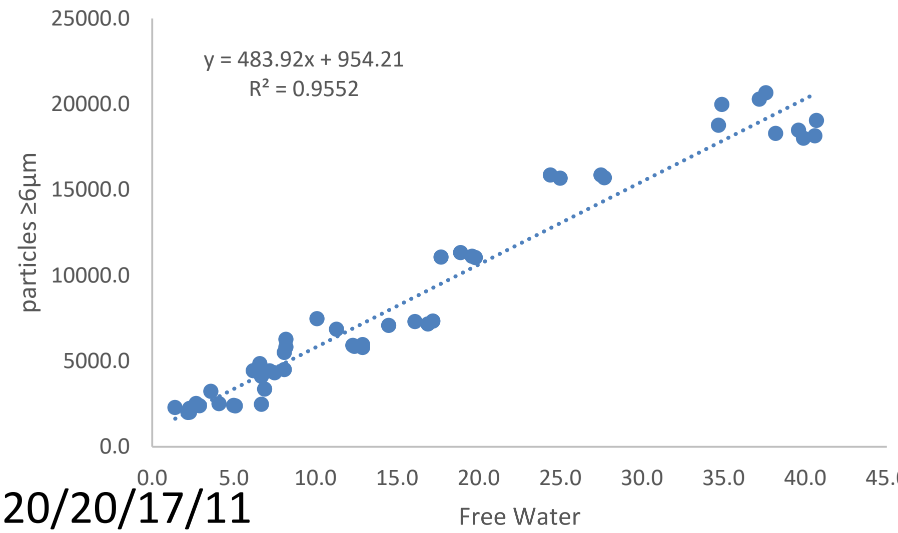




1.0 mg/L ~ 20/18/10

0.5 mg/L ~ 19/17/9

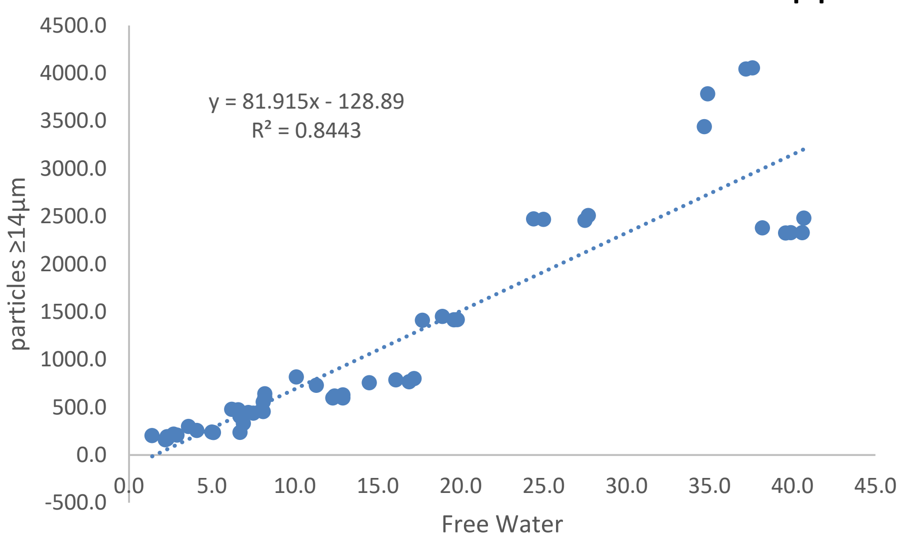
# Free water EI 1581 test loop full flow 100 gpm

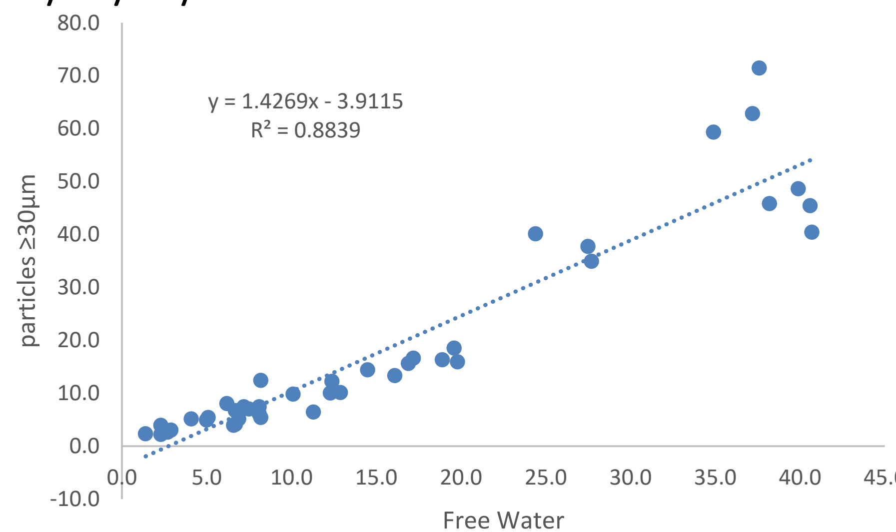


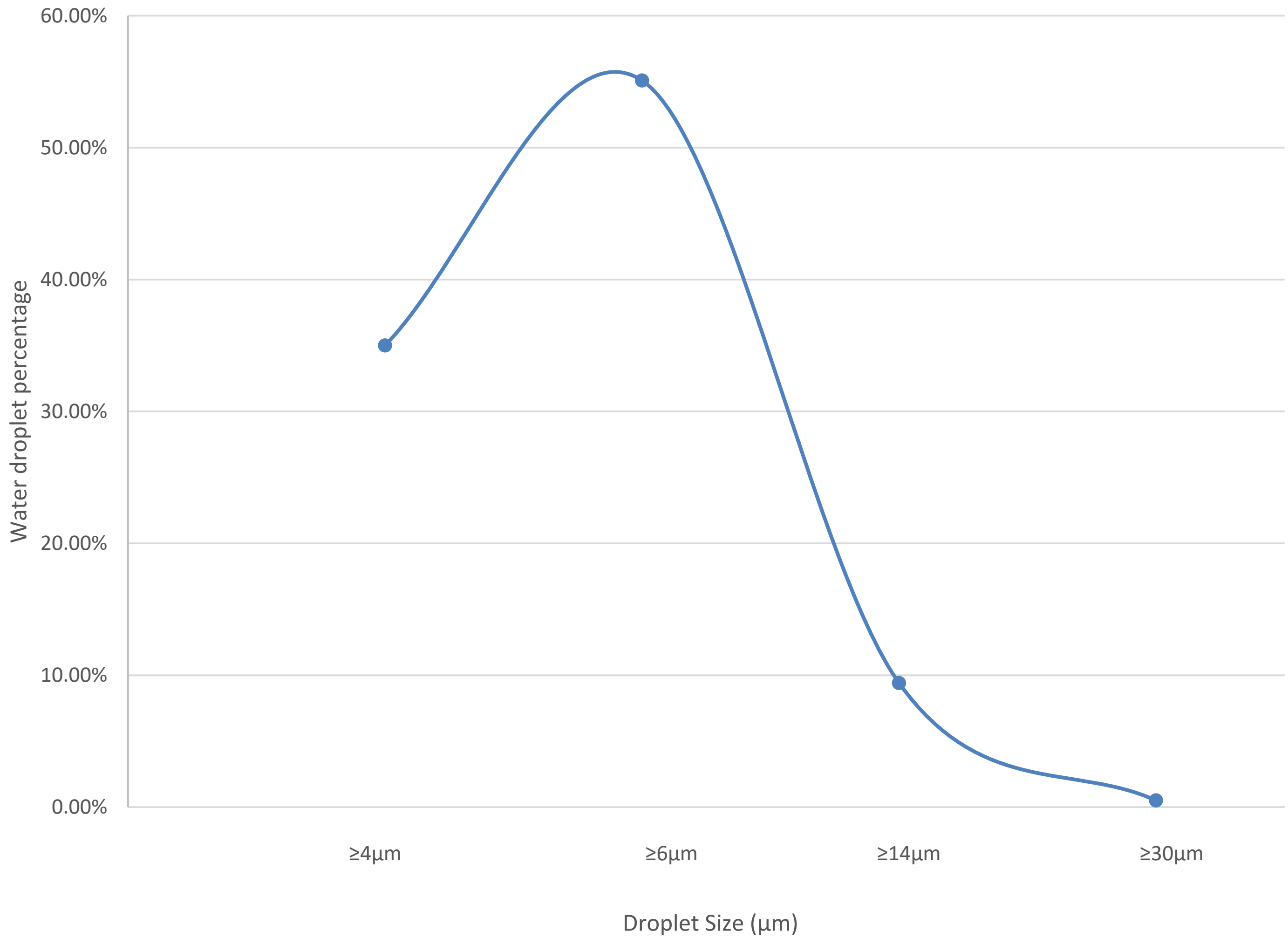

10 ppm ~ 20/20/17/11

5 ppm ~ 20/19/15/9



# Free water EI 1581 test loop full flow 100 gpm



# Effect of Temperature change on Free Water

- ASTM D3240 and IP 564 measurements where taken at two different EI 1581 testing facilities.
  - Fuel temperatures where then modified as the fuel was traveled from the pipeline to the instrument.
  - Data suggests that slight variations in free water content may be seen due to these variations in temperature via ASTM D3240 and the particle counting channels larger than 14 microns.

Site 1.

| Approximate Fuel Temperature | °F | 40 | 70 | 100 |
|---|---|---|---|---|
| ASTM D3240 | ppm | 6.5 | 4.5 | 2 |
| IP 564 | ≥4µm | 8013.5 | 2532.1 | 1102.6 |
| | ≥6µm | 2691.3 | 615.5 | 315 |
| | ≥14µm | 27.8 | 1.1 | 0.4 |
| | ≥21µm | 2.6 | 0.1 | 0.1 |
| | ≥25µm | 0.7 | 0.1 | 0.1 |
| | ≥30µm | 0.2 | 0.1 | 0 |

Site 2.

| Approximate Fuel Temperature | °F | 60 | 70 | 80 |
|---|---|---|---|---|
| ASTM D3240 | ppm | 0.8 | 0.4 | 0.2 |
| IP 564 | ≥4µm | 24501 | 20104 | 22922.1 |
| | ≥6µm | 7902.2 | 5345 | 7029.5 |
| | ≥14µm | 5.5 | 2.3 | 3.1 |
| | ≥21µm | 0.8 | 0.5 | 0.2 |
| | ≥25µm | 0.5 | 0.3 | 0.1 |
| | ≥30µm | 0.4 | 0.2 | 0 |

# Recommended Limits

| | Receipt | Vehicle Fuel Tank | Fuel Injector |
|---|---|---|---|
| **Aviation Fuel** | | | |
| DEF (AUST) 5695B | | 18/16/13 | |
| Parker | 18/16/13 | 14/10/7 | |
| Pamas / Parker / Particle Solutions | 19/17/12 | | |
| U.S. DOD | 19/17/14/13* | | |
| ATA 103 | | 19/17/14/13 | |
| USA into aircraft* | | 19/17/14/13 | |
| USAF into plane# | | 18/16/14 | |
| **Diesel Fuel** | | | |
| World Wide Fuel Charter 5th | | 18/16/13 | |
| Truck & Engine Manufacturers Association | | 18/16/13 | |
| DEF (AUST) 5695B | | 18/16/13 | |
| Caterpillar | | 18/16/13 | |
| Detroit Diesel | | 18/16/13 | |
| MTU | | 18/17/14 | |
| Bosch/Cummins | | 18/16/13 | |
| Donaldson | 22/21/18 | 14/13/11 | 12/9/6 |
| Pall | 17/15/12 | 15/14/11 | 12/9/6<br>11/8/7 |

* 4µm (c)/ 6µm (c)/ 14µm (c)/ 30µm (c)

# proposed

# Army Sampling effort 1.0 mg/L & 19/17/14/13

- September 2012 – February 2018

- ASTM D5452 Laboratory filtration (aviation)
- ASTM D6217 Laboratory filtration (diesel)

- IP 564 – Parker ACM20 (59 samples only)
- IP 565/ASTM D7619 – Stanhope-Seta AvCount

- 2550 samples analyzed

# Army Sampling effort 1.0 mg/L & 19/17/14/13

- 2550 samples analyzed
- 2177 samples passed both gravimetric and particle count

- 373 samples failed particle count or gravimetric
  - 295 out of 373 samples failed particle count
  - 247 out of 373 samples failed gravimetric
  - 169 out of 373 samples failed both particle count and gravimetric

- 78 False negatives (fails gravimetric, passes particle count)

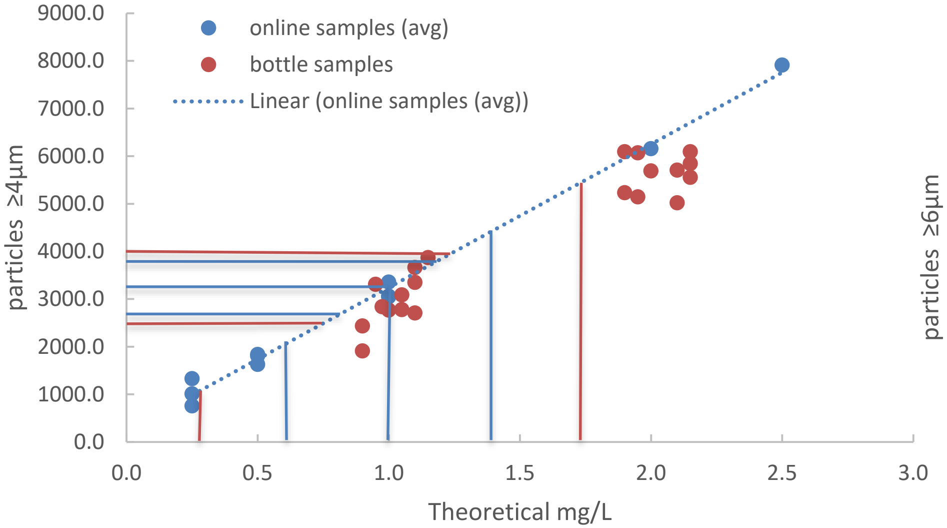
- 126 False positives (fails particle count, passes gravimetric)

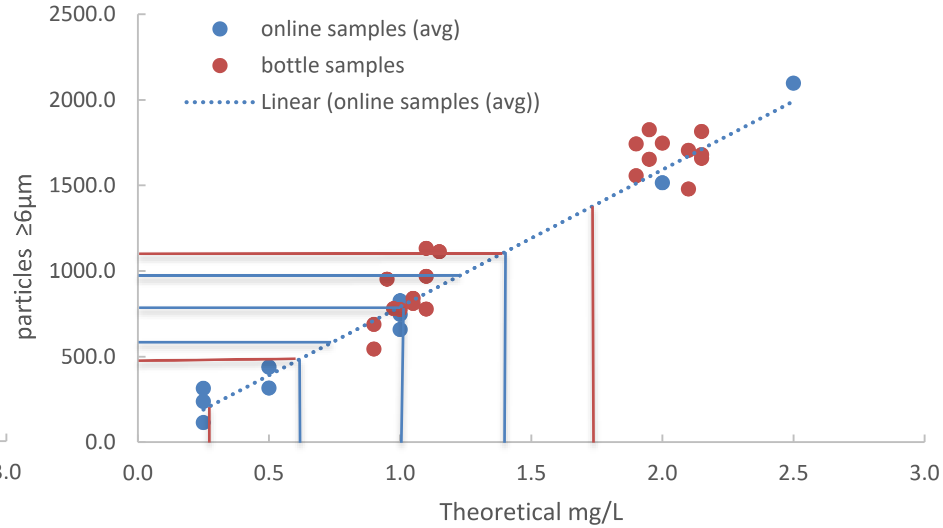
**92.0% agreement**

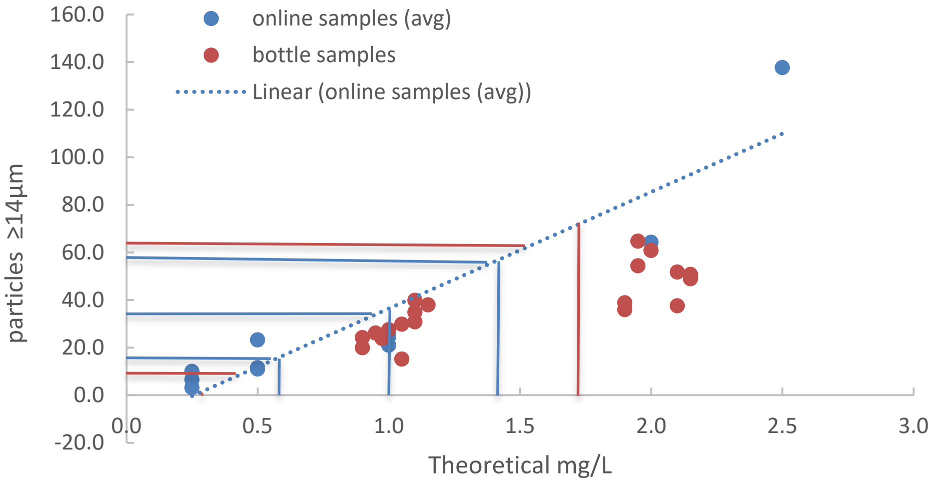
# Army Sampling effort 1.0 mg/L & 19/17/14/13

- 78 False negatives (fails gravimetric, passes particle count)
- ASTM D5452 repeatability (r) 0.0-0.6 mg/L
  - $r = 0.415x^{0.5}$
  - r at 1.0 mg/L = 0.415 mg/L
    - repeatability formula based on 5 liter sample
- 58 samples may be lower than 1.0 mg/L based on repeatability calculations.
- 20 samples have high gravimetric reading that is not accounted for by particle count data.
- Particles not seen by particle counter
  - Particle greater than 70µm (c) (Stanhope Seta) - 200µm (c) (Parker)
  - Particles less than 4µm (c)

# ISO 12103-1 A2 repeatability and reproducibility




ASTM D5452* extrapolated
IP 564
repeatability
Reproducibility

# Army Sampling effort 1.0 mg/L & 19/17/14/13

- 126 False positives (fails particle count, passes gravimetric)
  - 56 samples gravimetric data may be greater than 1.0 mg/L based on repeatability calculations.
  - 118 fuels high in 6µm (c),14µm (c), and/or 30µm (c) channels indicating free water contamination
  - Analyzed to determine if free water contributed to the high particle counts
    - 16 of 118 fuel samples confirmed >5 ppm free water contamination
    - 15 of 118 fuel samples with 1-5 ppm free water
    - 41 of 118 fuel samples confirmed to be absent of free water
    - 46 of 118 fuel samples untested

# Air Force Sampling effort 0.5 mg/L & 18/16/14

- 475 samples analyzed
- 435 samples passed both gravimetric and particle count

- 40 samples failed particle count or gravimetric
  - 27 out of 40 samples failed particle count
  - 18 out of 40 samples failed gravimetric
  - 5 out of 40 samples failed both particle count and gravimetric

- 13 False negatives (fails gravimetric, passes particle count)

- 22 False positives (fails particle count, passes gravimetric)

**92.6% agreement**

# USA 18/16/14 limits & 0.5 mg/L

- 2550 samples analyzed
- 1610 samples passed both gravimetric and particle count

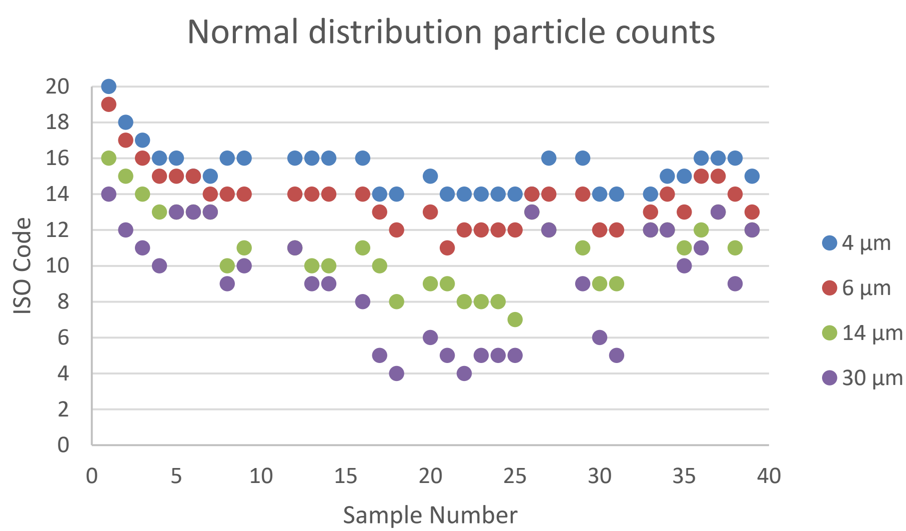
- 940 samples failed particle count or gravimetric
  - 595 out of 940 samples failed particle count
  - 696 out of 940 samples failed gravimetric
  - 351 out of 940 samples failed both particle count and gravimetric

- 345 False negatives (fails gravimetric, passes particle count)

- 244 False positives (fails particle count, passes gravimetric)

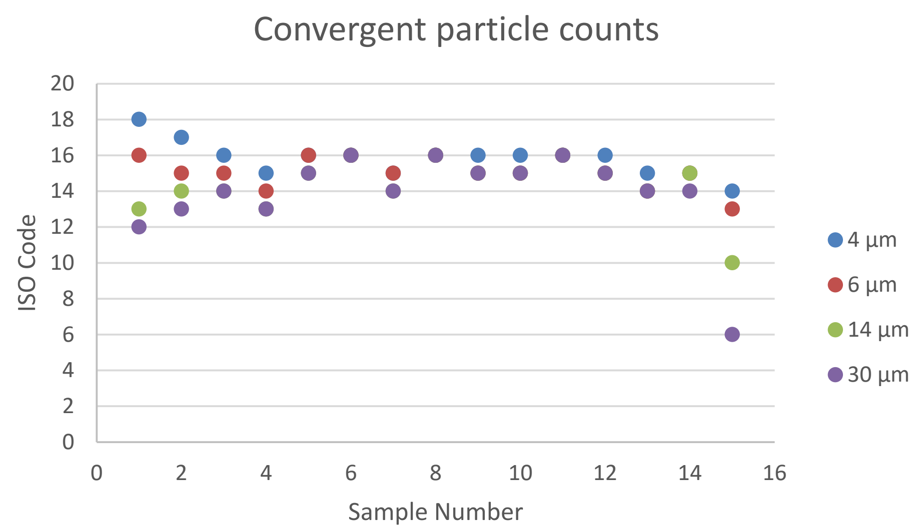
## 76.9% agreement

- Non Aviation Brigade Support Battalion




- Normal distribution of particle counts indicative of minimal sediment contamination

- Convergent particle counts where all channels read close together indications large water droplets present in the fuel.

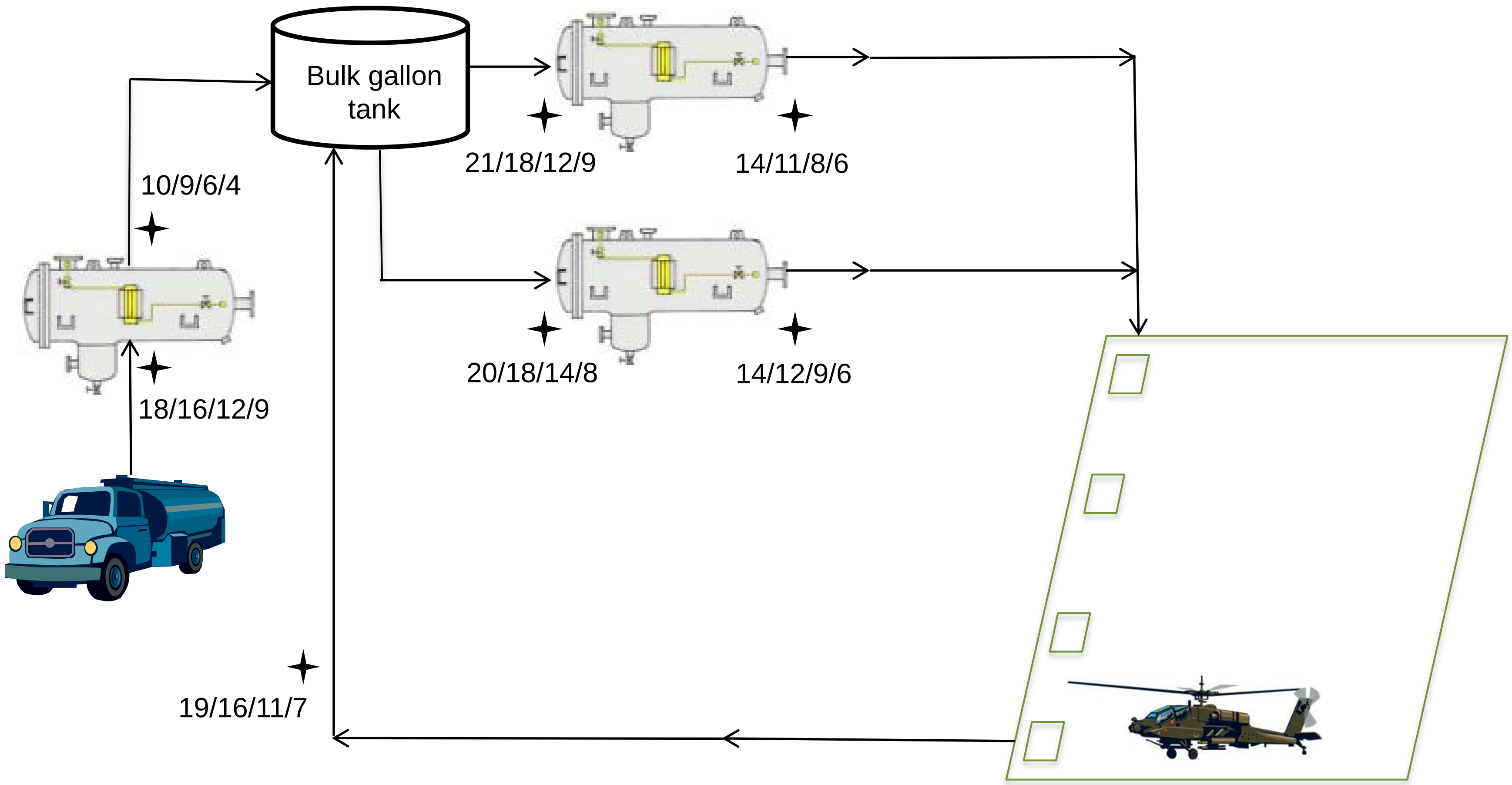


# Field success stories

- Non Aviation Brigade Support Battalion
- Water contamination confirmed! Led to microbial infestation outbreak.

# Field success stories

- Airfield Hydrant return line contamination
- Filters required replacement after 3 months of service
- Gravimetric measurements at fuel receipt and retail normal



# Discussion

- Low levels of free water can have a large impact in particle count readings, this may account for an unknown number of the false positive readings.
  - Recommend following the procedure to utilize chemical treatment to eliminate the interference of free water droplets from particle counts found in Annex B of IP 564, IP 565, IP 577; Appendix X2 of ASTM D7619 and Appendix X1 of ASTM D8166.

- The published particle count limit of 19/17/14/13 for the 4µm (c)/ 6µm (c)/ 14µm (c)/ 30µm (c) size channels in MIL-STD-3004 for aviation turbine fuel and MIL-DTL-83133 have shown to be in agreement 92% of the time.

# Discussion

- Intent of the DoD is to gain push of particle counting technology down to the skin of the aircraft.

- A particle count limit of 18/16/14 for into plane samples has a high correlation to 0.5 mg/L sediment.
  - 92.6% agreement for USAF samples ranging from 0.0 – 0.5 mg/L
  - 77.8% of the time for USA samples ranging from 0.0 – 1.0 mg/L.
    - 38% of these samples not in agreement are false positives samples
    - 62% false negatives

Questions?