# The Quest for a Quantitative Jet Fuel Contamination Limit—Particle Counting “RESOLVED”

**Vic Hughes & Paula Zard**

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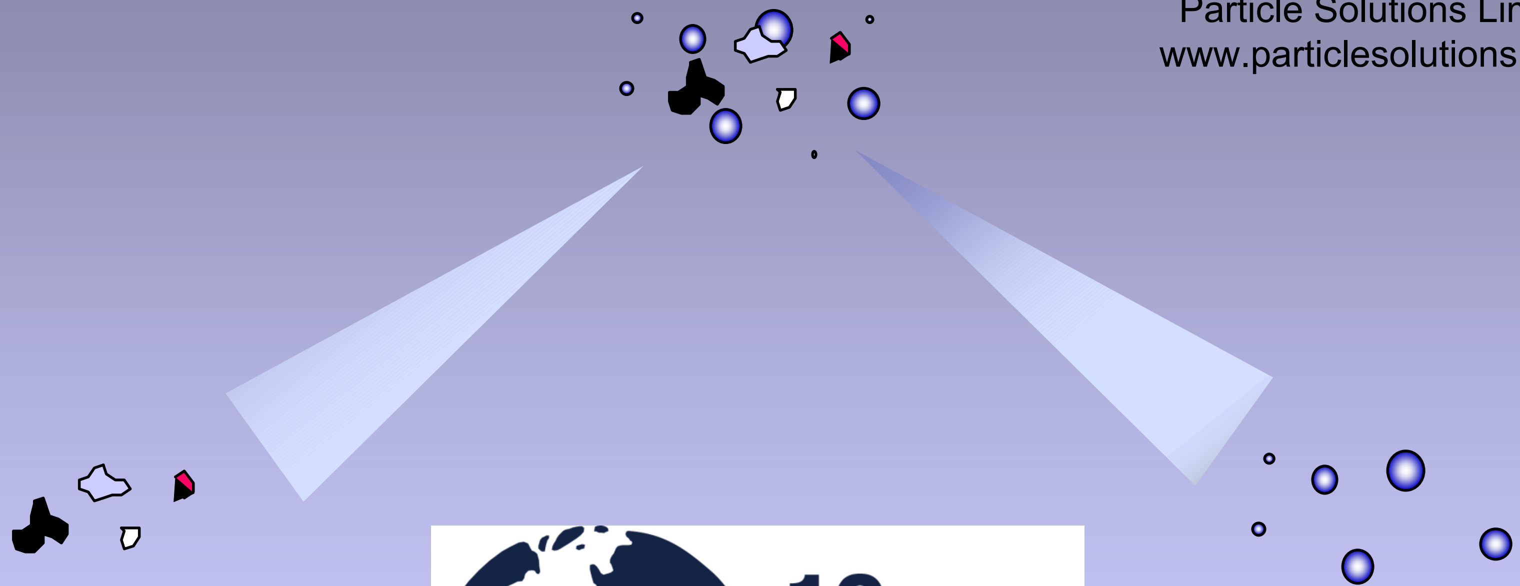
www.particlesolutions.co.uk



10th International Filtration Conference

# A Bit on History of Jet Fuel Cleanliness

**C&B**
**(Clear & Bright)**

Clear, bright and visually free from solid matter and undissolved water at ambient temperature

- gross contamination normally seen with this test
- easy to carry out, but easy to get wrong!
- Subjective!
- Can't detect <40μm

In 1998, LHR almost stocked-out of Jet fuel because of an appearance issue. Also isolated islands around the coast of the UK were close to having no fuel because of the "White bits" problem!! In the meantime, Avgas also had problems with "Black Bits".

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**Gravimetric**


- only measures total particulate matter
- no indication of size
- takes many hours to produce a result

Not only that, many operators in the industry are aware that the results obtained are often in error. Negative weights are common!!!

# A Bibliography of alternative methods

Hughes, **"Can a Quantitative Contaminants Specification be Realised for Jet Fuel?",** 5th IFC, **2002**.

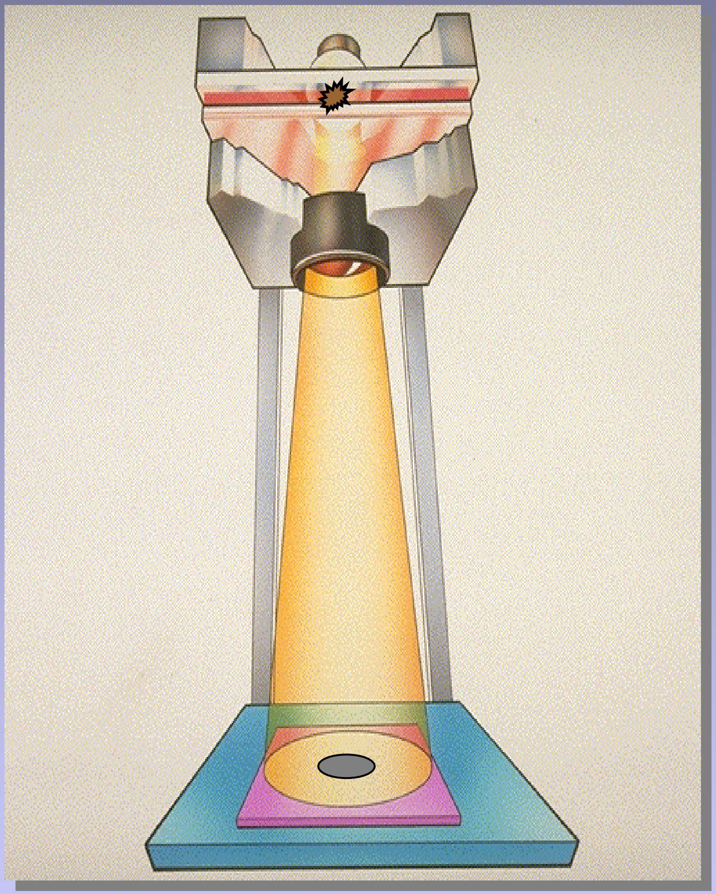
Kitson-Smith et al., **"Experience with the use of a particle counter in measuring fuel contaminants."** 9th International Conference on Stability, Handling and Use of Liquid Fuels, September 18-22, **2005.**

Hughes et al., "Can a Quantitative Contaminants Specification be Realised for Jet Fuel? - Part II", 6th IFC, 2004. **"Aviation Turbine Fuel Particle Counting."** 7th IFC, **2005**.

Kitson-Smith & Hughes, **"The use of electronic sensors in field measurements of aviation jet fuel cleanliness."** 10th International Conference on Stability, Handling and Use of Liquid Fuels, October 5-11, 2007.

# AVIATION JET FUEL APC

More objective,
Quantitative,
Timely,
Reliable,
Has the potential to pervade the industry yielding a common terminology from refiner to airline operator.



ISO11171-type instrument

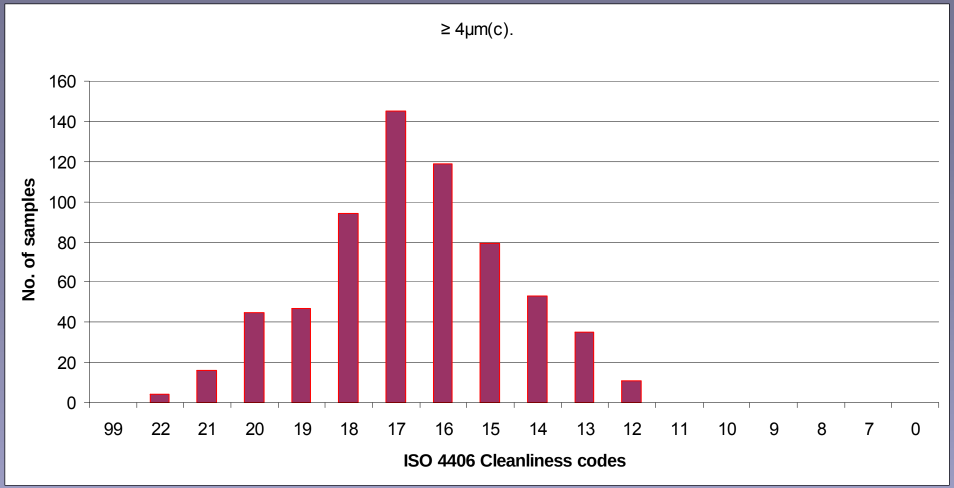
| Jet Fuel particle counting sizes, µm(c) | | | | | |
|---|---|---|---|---|---|
| ≥4 | ≥6 | ≥14 | ≥20 | ≥25 | ≥30 |

The Energy Institute now has three particle counting methods included in its Test Methods portfolio – IP564, 565 and 577.

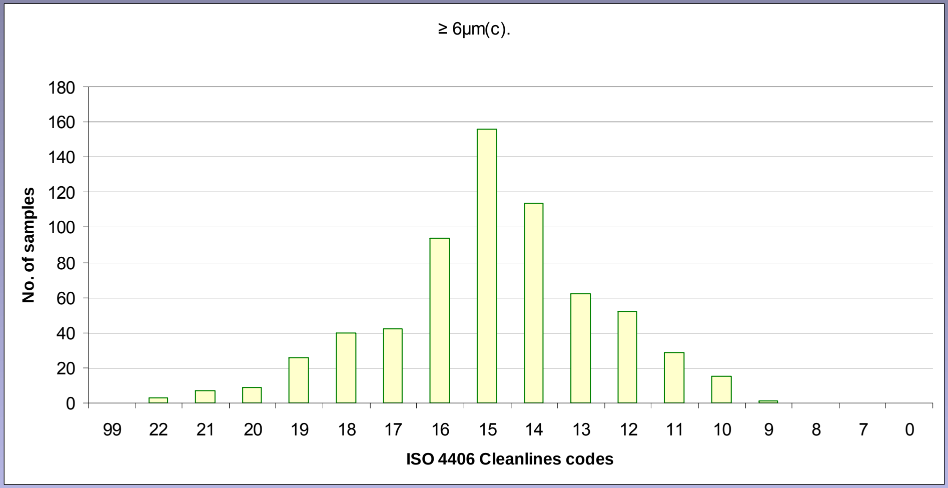
# ADOPTION IN AVIATION JET FUEL SPECIFICATIONS

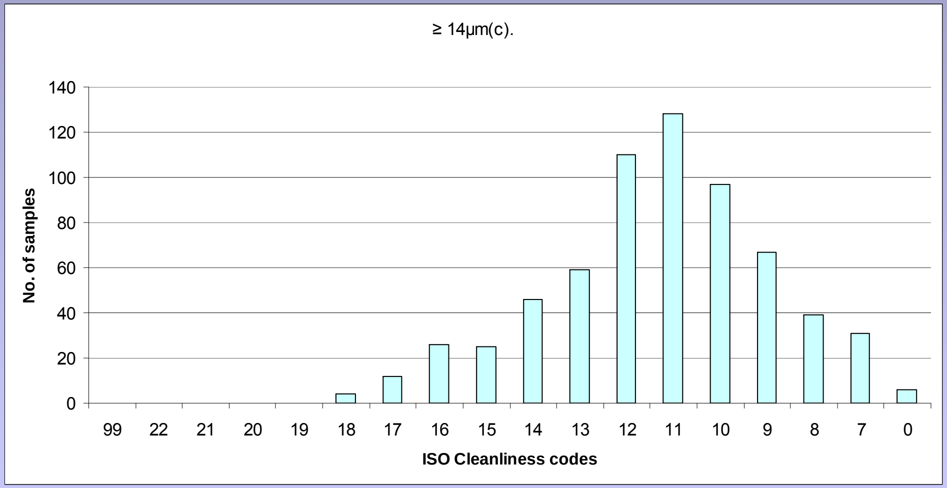
| Date | Action | Comments |
|---|---|---|
| 8th April 2008 | Def Stan 91-91 – Issue 6, Report counts | Note 4 read: “The implementation date for particle counting is 30th June 2009, but where possible, to help the data collection process, the results should be reported before that date. It is the Specification Authorities intention to replace Test 1.3 (gravimetric) with Test 1.4 (particle counting) at the earliest opportunity.” |
| July 2008 | Joint Inspection Group (JIG) Product Specifications, Issue 23 | |

# THE FIRST DEF STAN 91-91 JET FUEL DATA SETS




5 Sources
14 refineries
2008-2009
http://www.dstan.mod.uk/afc%20presentations.html



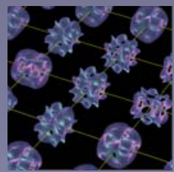



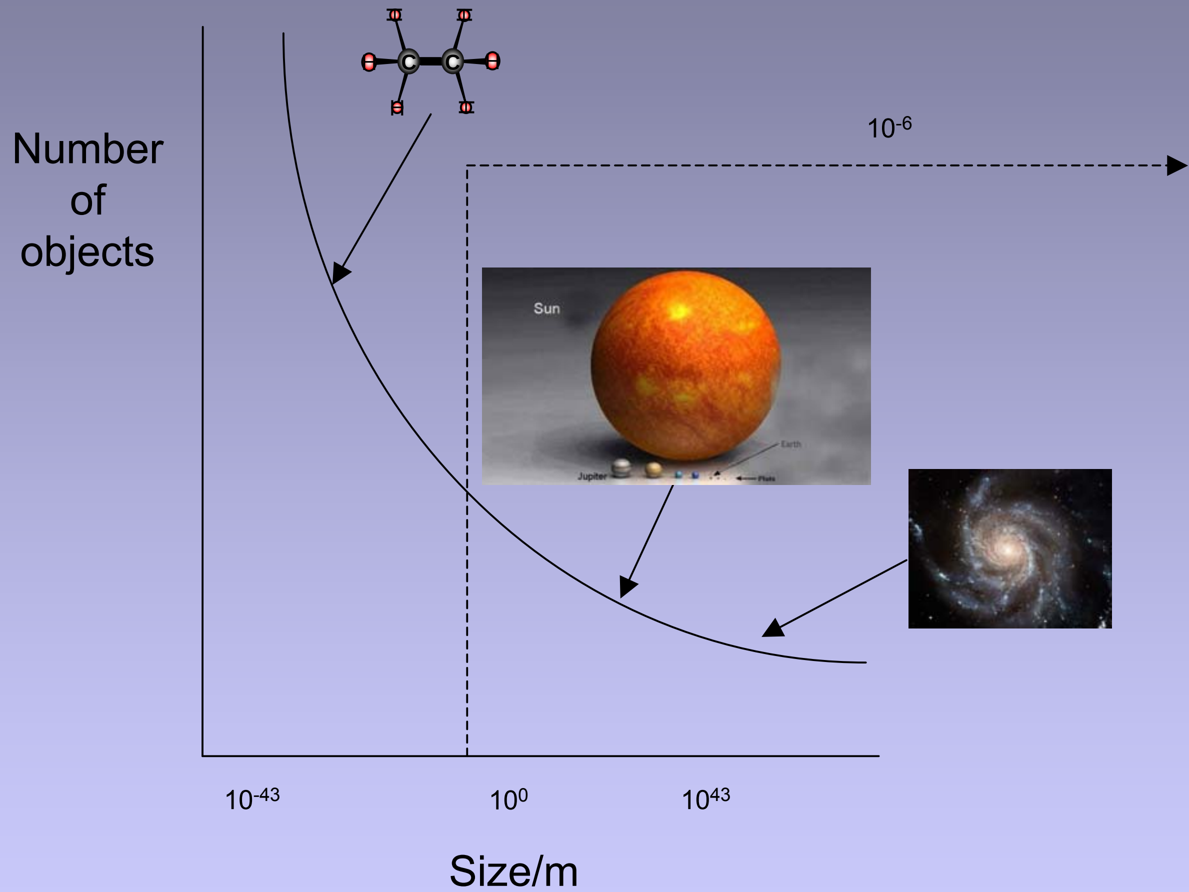

Approximately 20% of fuels appear to be dirtier than our previously proposed cleanliness limit of 18/16/14 – All of them meet the 1.0mg/l gravimetric limit!!

So what's happening…………..?????

# EINSTEIN WAS A WONDERFUL MAN!!

$$n = -k \ln d$$






Most natural particulate systems are a microcosm of this distribution …….



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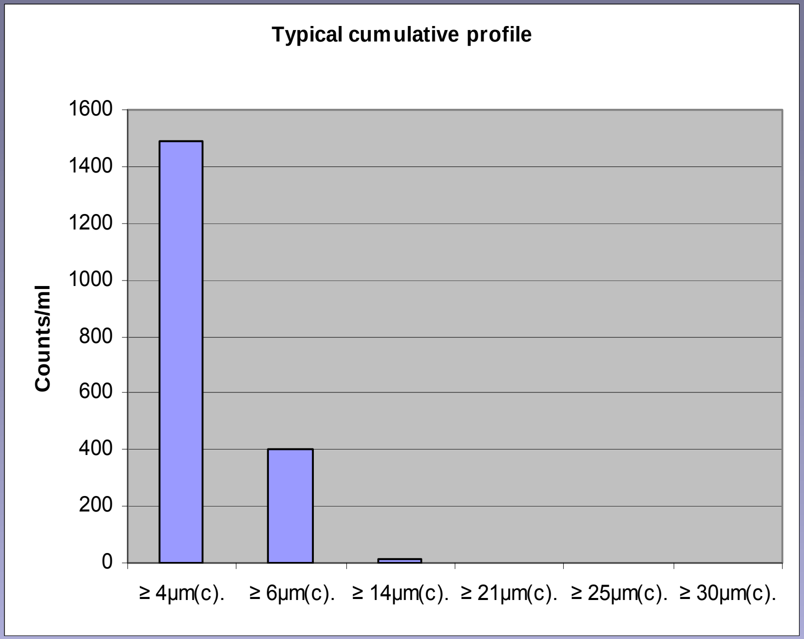
# Particle counting – Jet fuel data – looking good!

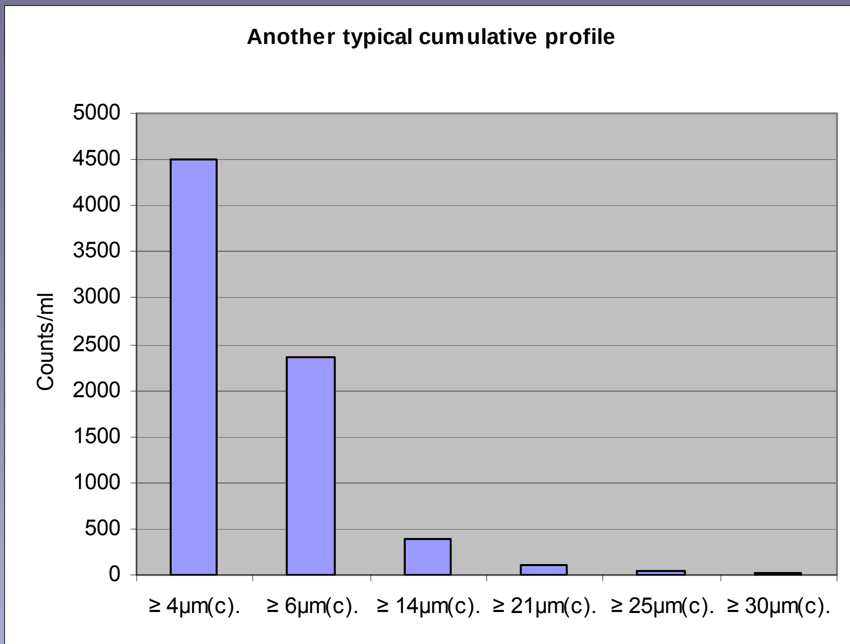


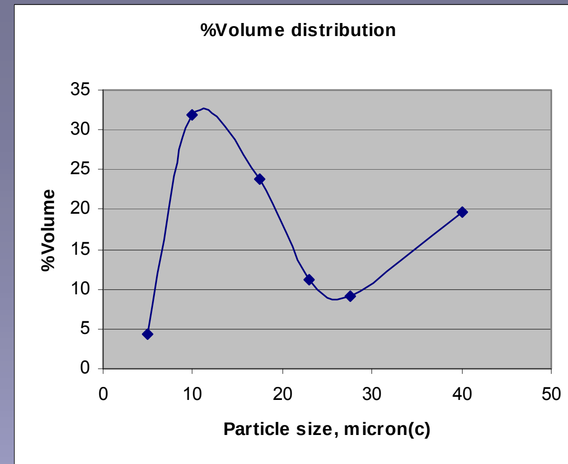

| Particle Count Data per ml | | | | | |
|---|---|---|---|---|---|
| ≥ 4µm(c) | ≥ 6µm(c) | ≥ 14µm(c) | ≥ 21µm(c) | ≥ 25µm(c) | ≥ 30µm(c) |
| 1486 | 402 | 15 | 2 | 0 | 0 |

| Particle Count Data - ISO Codes | | | | | |
|---|---|---|---|---|---|
| ≥ 4µm(c) | ≥ 6µm(c) | ≥ 14µm(c) | ≥ 21µm(c) | ≥ 25µm(c) | ≥ 30µm(c) |
| 18 | 16 | 11 | 8 | 6 | 4 |

Many of the results from the Def Stan 91-91 data set looked like this.

# Particle counting – Jet fuel data – not so good?




| Particle Count Data per ml | | | | | |
|---|---|---|---|---|---|
| ≥ 4µm(c) | ≥ 6µm(c) | ≥ 14µm(c) | ≥ 21µm(c) | ≥ 25µm(c) | ≥ 30µm(c) |
| 4501 | 2357 | 379 | 103 | 46 | 19 |
| Particle Count Data - ISO Codes | | | | | |
| 19 | 18 | 16 | 14 | 13 | 11 |

Assuming this is all dirt with density 3000kg/m$^3$ the mass is 9.73mg/l – A BAD FAIL

BUT – If was all water with density 1000kg/m$^3$ the contaminant level could be 3.24 ppm water – A PASS

*Water droplets have a "non-Einsteinian" distribution – there is a minimum size below which a water droplet cannot exist! When combined with a particulate distribution the result is a complex distribution.*

# The Problem:

To separately determine particulate and water contaminants in fuel from light obscuration measurements.

Make water droplets smaller than detectable limit - 2 Options:

1. Solvate using a Cosolvent?

2. “Nano-encapsulation”

# Something to read?

*Cosolvency…..*

A.Q. Clark and J.S. Crichton, EP 1715323, Method for determining the concentration and/or size distribution of solid particles in middle distillate fuels, 2006.

S. I. Sinegubova, K. K. Il'in and D. G. Cherkasov. Mutual Solubility of Components and Critical Solution Points in the System Water-Isopropyl Alcohol-*n*-Dodecane in the Temperature Range 5–120°C, Russian Journal of Applied Chemistry, Vol. 78, No. 3, March, 2005

T.M. Letcher and P.M. Siswana, Liquid-liquid equilibria for mixtures of an alkanol + water + a methyl substituted benzene at 25°C, Fluid Phase Equilibria, 74, pp203-217, Elsevier Science, 1992.

*Micellar…..*

J.W. Mountain et al, US 6064480, Method of particle counting for water mixed lubricant, May 2000.

B.M. Verdegan and L. Thibodeau, Particle counting oil and water emulsions, Particulate Science and Technology, 7, pp23-34, Hemisphere Publishing Corp., 1989.

# Cosolvent approach – Choices:

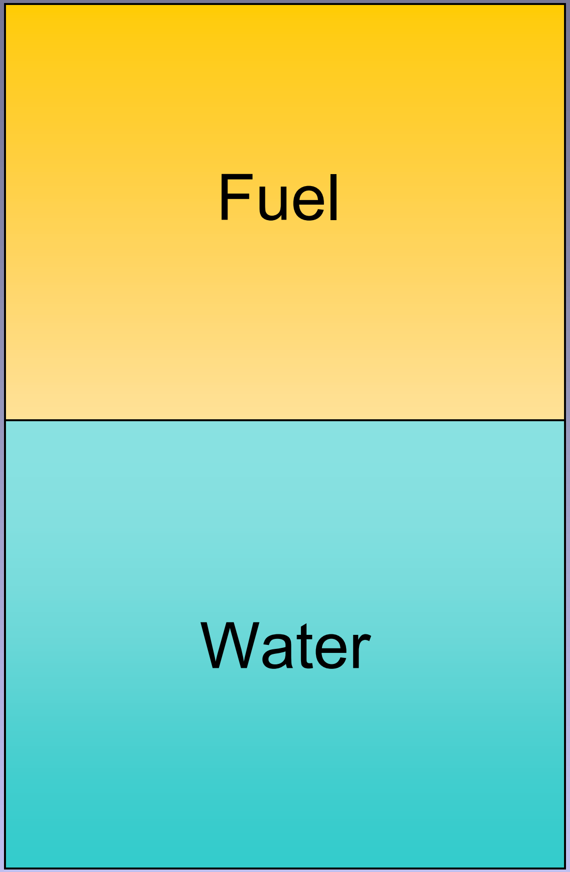
**High RMM Alcohol**

**Mono-EGME**

**Higher glycols, e.g Di-EGME and Tri-EGME**

**Low RMM Ketone**

**Low RMM Alcohol**




**S. I. Sinegubova, K. K. Il'in and D. G. Cherkasov**

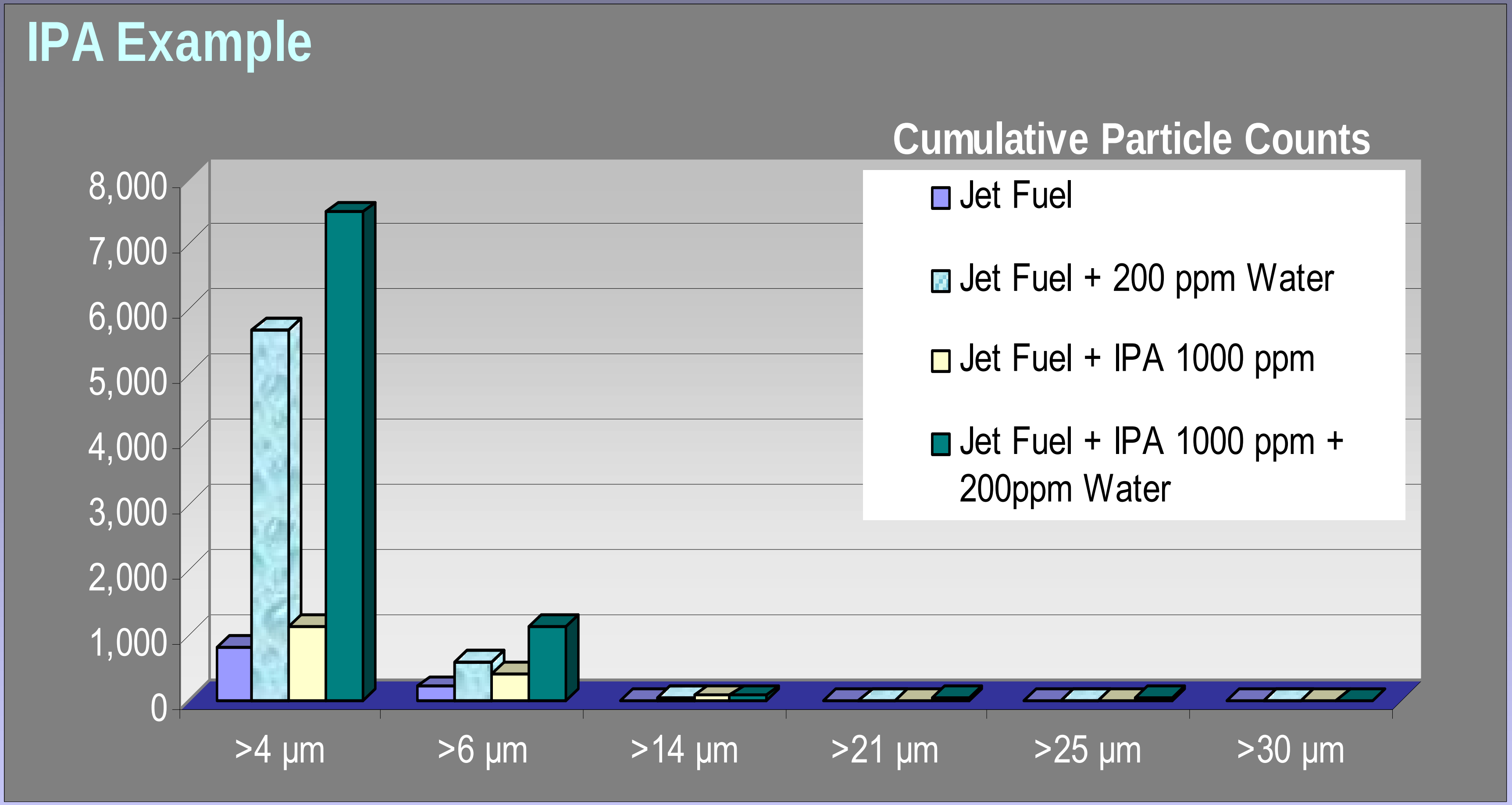
**Mutual Solubility of Components and Critical Solution Points in the System Water-Isopropyl Alcohol-*n*-Dodecane in the Temperature Range 5–120°C**
**Russian Journal of Applied Chemistry**
**Volume 78, Number 3 / March, 2005**
**In this ternary system, the miscibility point is determined by relative concentrations and temperature.**

## Relative partitioning

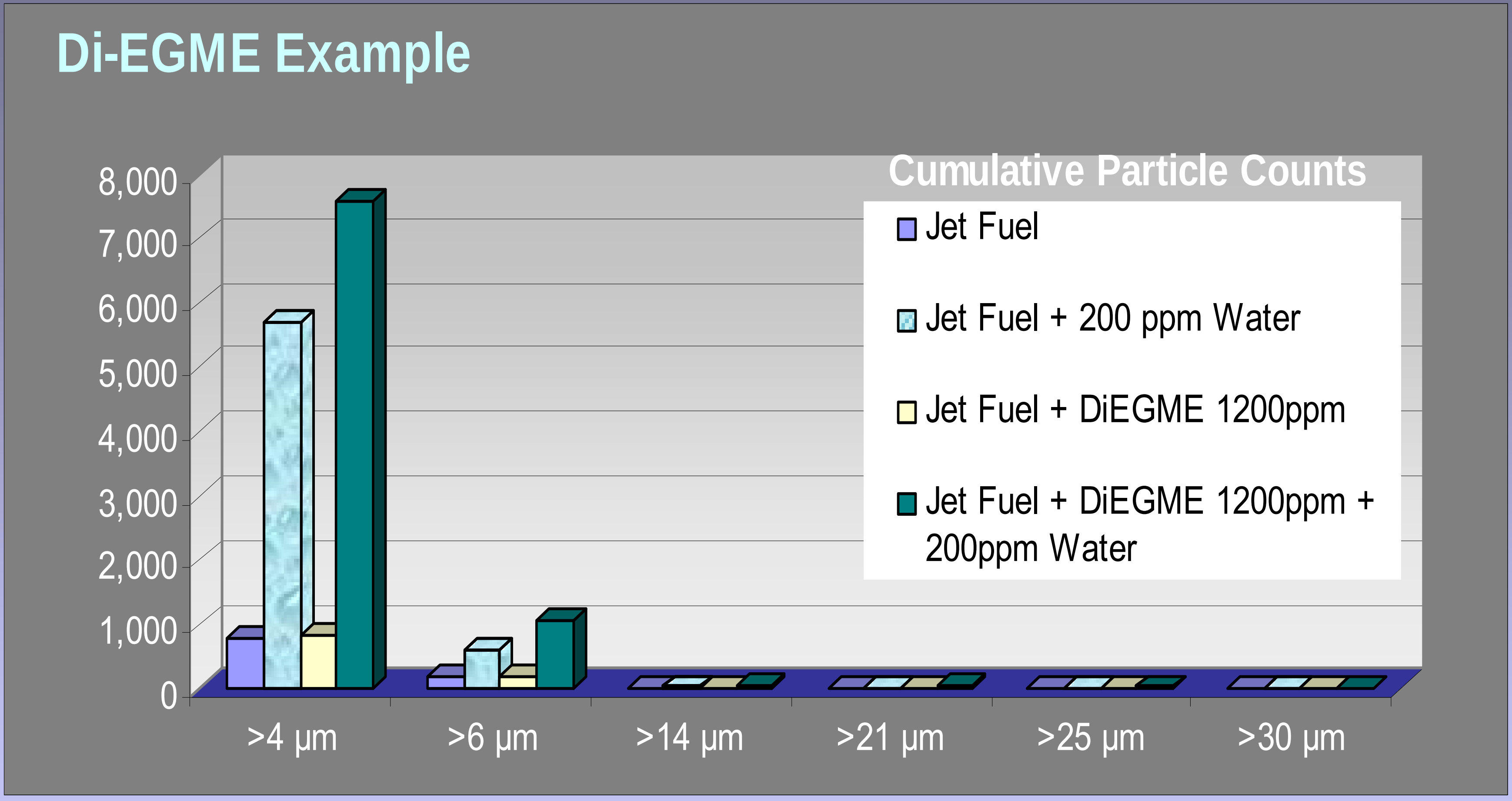
$$\log P_{\frac{oil}{water}} = \log\left(\frac{[cosolvent]_{oil}}{[cosolvent]_{water}}\right)$$

e.g. P (methanol) = -0.83 between water and octanol

# Cosolvent approach – Results: **IPA**



# Cosolvent approach – Results: **Di-EGME**




Di-EGME dissolves in the fuel but in the presence of free water, migrates to that phase resulting in increased counts (surfactancy issue?). Glycols have greater partitioning in the polar water phase than the apolar fuel phase.

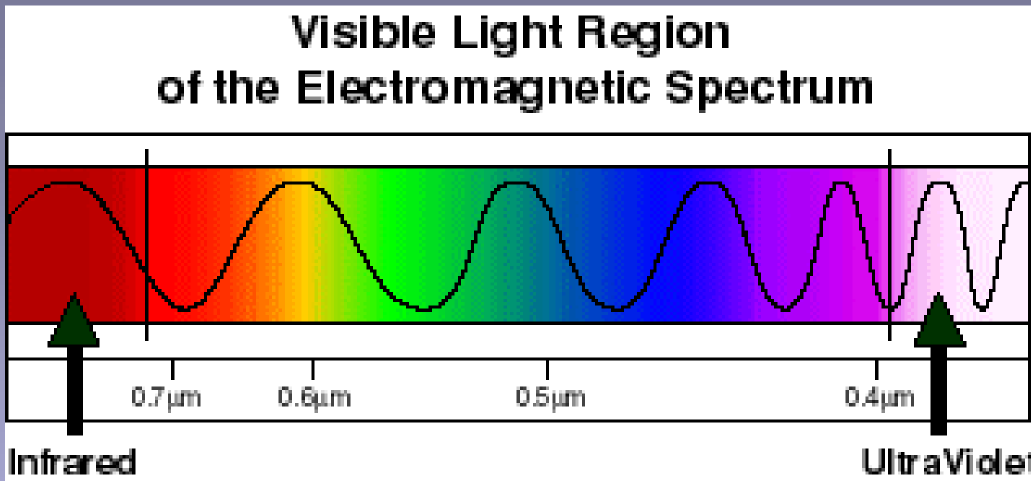
# Cosolvent approach – Summary:

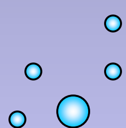
The cosolvent approach depends on partitioning coefficients remaining constant across range of applicable fuels.

The actual free water solubilising capacity will be finite for a given Temperature/Cosolvent/Fuel condition (will need to be determined).

An EI RR for particle counting methods 564 and 565 give mixed results in terms of statistical robustness for the use of IPA (the most promising cosolvent).

# Nano-emulsification approach – Resolver Principle:



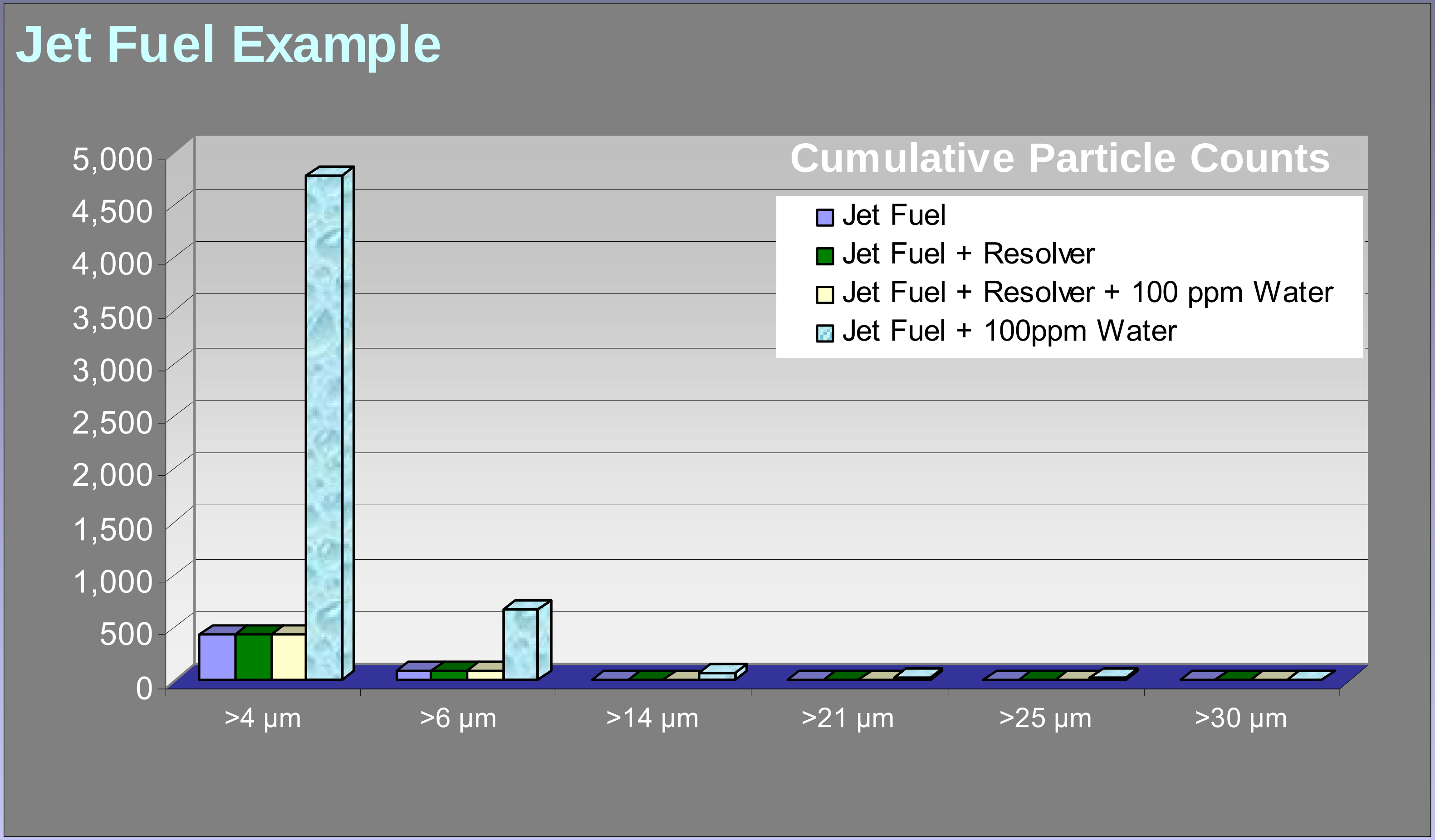



**Water droplets and particles <<wavelength of light will appear isotropic**

**Water droplets and particles >>wavelength of light will obscure the light. Droplets within an order of magnitude of the wavelength of light will scatter it.**

**Nanoemulsions typically comprise structures that are <0.1µm**

# Resolver in use:



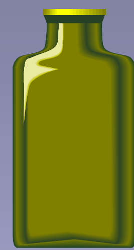

**Resolver:**

**A proprietary formulation of non-hazardous chemicals that have a high affinity for free water, will readily dissolve in a hydrocarbon liquid, will effectively "solubilize" any free water that may be present by creating an optically isotropic system.**

PROPOSED ANNEX TO IP PARTICLE COUNTING TEST METHODS:

## ANNEX *:  THE SEPARATE DETERMINATION OF PARTICULATE MATTER AND FREE WATER FROM PARTICLE COUNTING.

The following scheme describes the analytical process:



a.  Original sample with minimum sample size = twice the minimum size specified in *(relevant section of the particle counting method)*

b.  Using containers cleaned in accordance with *(relevant section of the particle counting method*), divide the original sample into 2 aliquots, each of a minimum volume defined by *(relevant section of the particle counting method).*  Label accordingly.

c.  Add the additive to one of the sub-samples.  The additive is supplied in 10cm3 vials.  The whole content of a vial should be added – if the sub-sample size is > 1 litre, then a further vial should be added for each 1 litre increase.

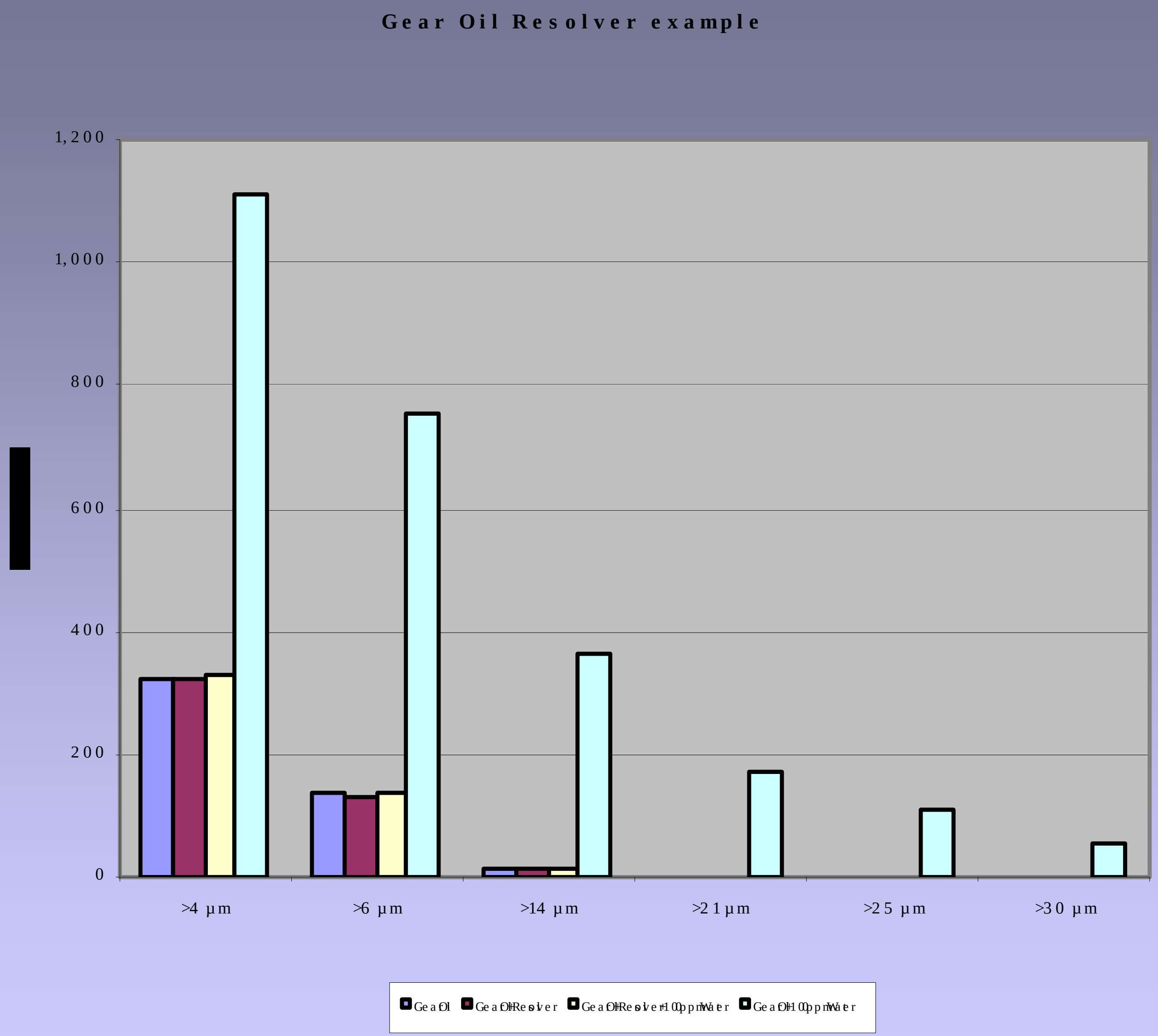
**Sample A**

**Sample B**

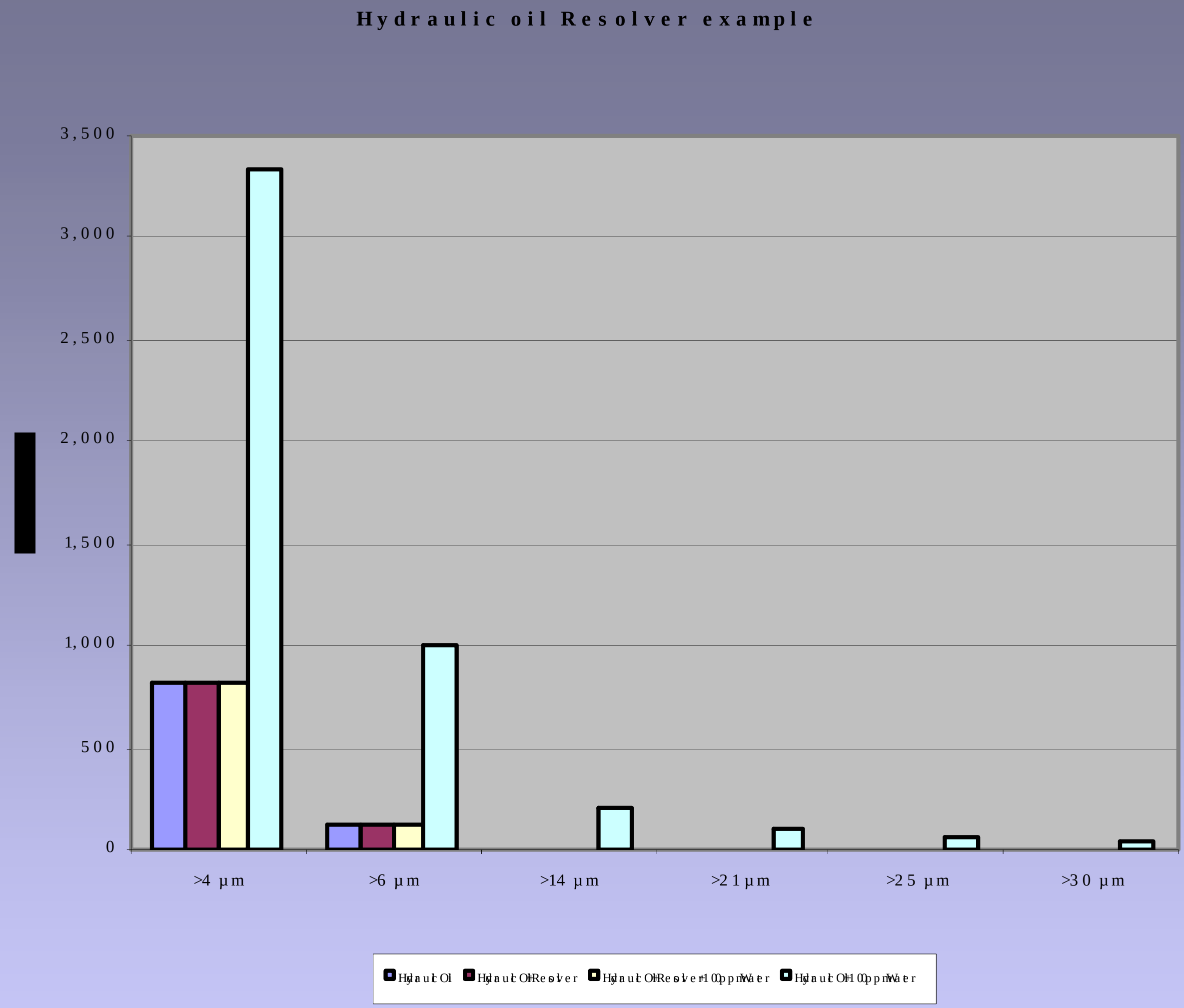
d.  Turn both samples end-over-end as described in *(insert relevant section)*

e.  Analyse both samples according to the *relevant section of the particle counting method)*

# Gear Oil / Resolver example:

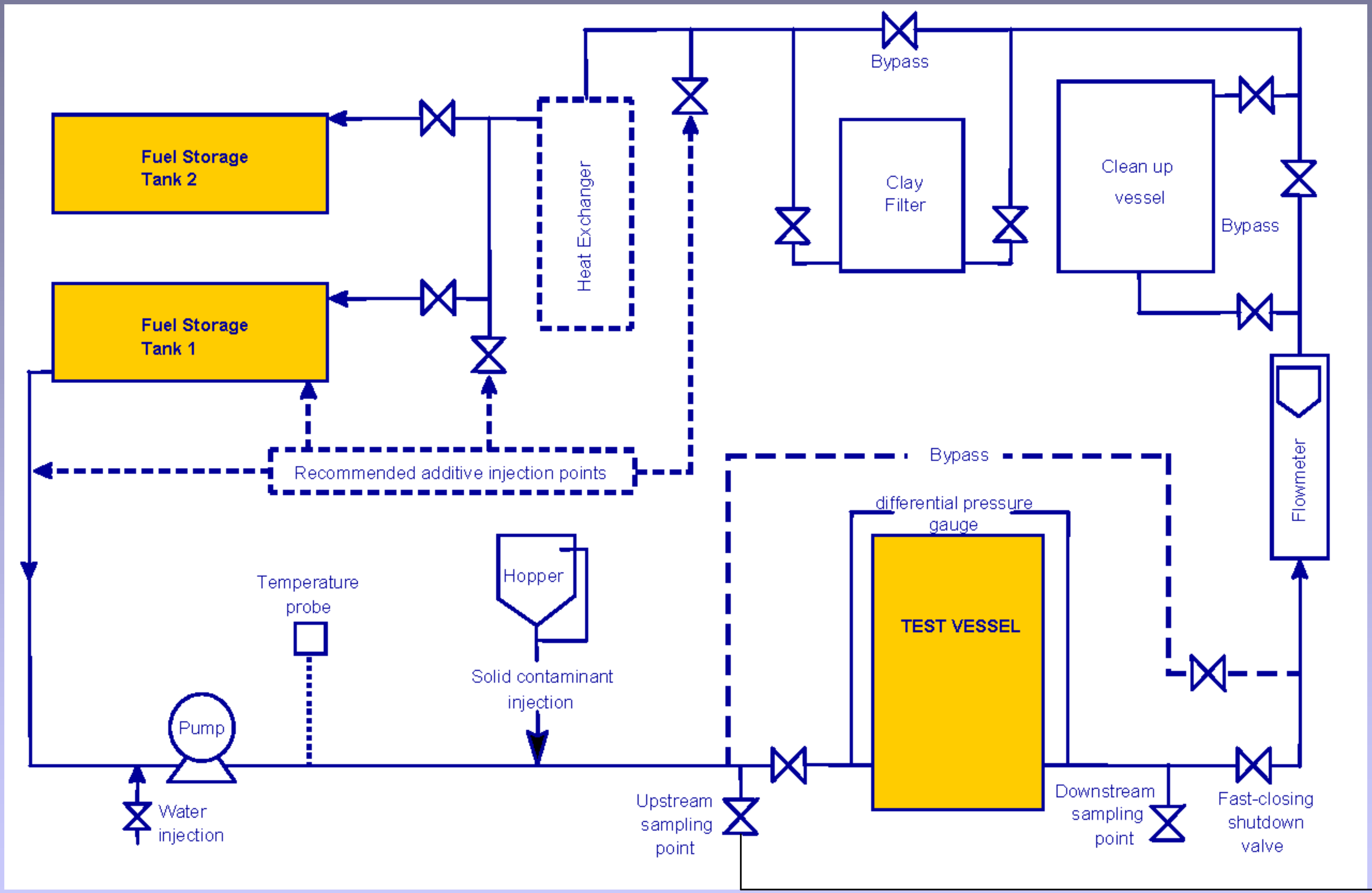


# Hydraulic Oil / Resolver example:



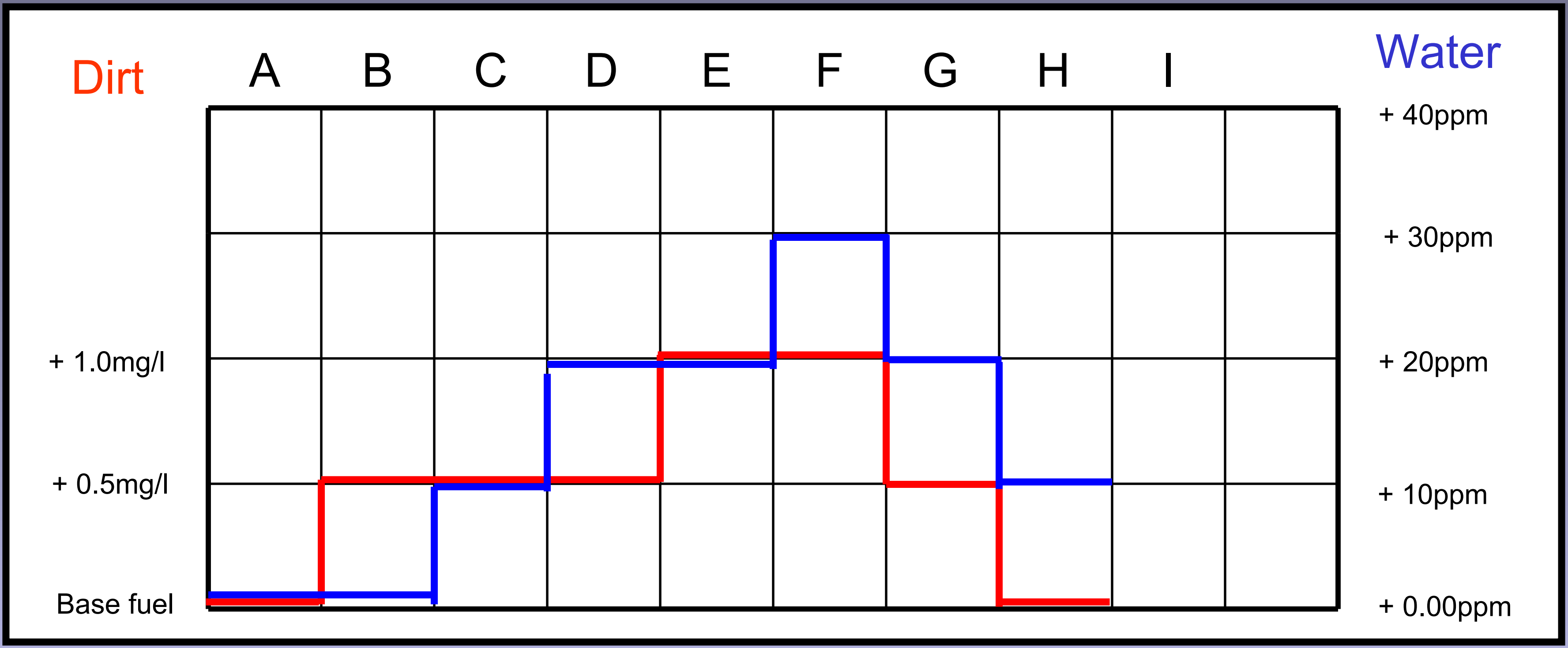
# Next Step – Ruggedness Trial:

**EI Filter Test Facility (schematic):**




Generate range of samples containing known levels of dirt and/or water.

# Test scheme:




For each condition A-I, a sample is taken by each of the operators and split into three. A1 is untreated, A2 is Resolver treated and A3 is cosolvent treated. When all test runs on a given condition have been completed in duplicate, the rig will be adjusted to the next condition. It takes only a few minutes to reach equilibrium. G is a repeat of D. F is the "spec" limit for dirt and water separately

Each operator will run his/her test method as described in IP 454, 455 or 577 resp. (Suggest 2 of each instrument)

# Summary

PSL has prepared a proposed testing scheme for EI -STB11 approval

EI-STB11 to engage contract rig facility

Complete test programme

Data analysis

Decision time.

Resolver