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LIGHT OBSCURATION PARTICLE COUNTER FUEL CONTAMINATION LIMITS

Joel Schmitigal

United States Army, 6501 East 11 Mile Road, Mail Stop 110, Warren, Michigan United States of America, joel.schmitigal@us.army.mil Joel.A.Schmitigal.Civ@mail.mil

ABSTRACT

The United States Army conducted a survey of over 1200 fuel samples with the objective of evaluating the proposed limits for use by automatic light obscuration particle counters for the monitoring of aviation fuel cleanliness. The laboratory data collected supports the proposed ISO code limit, based on ISO 4406, of 19/17/14/13 limits for the 4µm(c)/ 6µm (c)/ 14µm (c)/ 30µm (c) size channels. The proposed limits were derived from 1.0 mg/L concentration levels for ISO 12103-1 A1Ultrafine and ISO 12103-1 A2 Fine test dusts, and down to a 5 ppm free water contamination. Based on this work the Department of Defense Tri-Service Petroleum, Oil and Lubricants Technical Steering Committee has recommended these limits for inclusion into Table I of MIL-STD-3004.

INTRODUCTION

The U.S. Army maintains the mission of providing quality fuel to U.S. and Allied troops in tactical environments. Presently, requirements as outlined require a dedicated group of specifically trained fuels personnel to perform several tests per day per installation looking for traces of sediment and water in the fuel (1) (2) (3).

The Army utilizes several techniques to ensure that aviation fuels are clean and dry. Despite the best of intentions the current test methods utilized by the Army have several drawbacks including: timeliness of data due to the turn-around time needed to get the test results, operator subjectivity, lack of detailed analysis, and limitations in providing reliable data. For these reasons the Army has been actively working to develop new methods for monitoring fuel contamination (4) (5).

The Army utilizes ASTM D4176 – Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures), as a final check of fuel to ensure aviation fuel is clear and bright before flight operations.

Fuel filter effectiveness is evaluated by quality assurance testing though conducting periodic fuel sampling for gravimetric analysis. The Army currently utilizes two methods for measuring particulate contamination by gravimetric analysis: ASTM D2276 - Standard Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling, and ASTM D5452 - Standard Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration. Additionally free water content is determined by performing ASTM D3240 – Standard Test Method for Undissolved Water in Aviation Turbine Fuels, commonly termed AquaGlo testing.

Current standards, such as MIL-STD-3004, Department of Defense Standard Practice for Quality Assurance/Surveillance for Fuels, Lubricants, and Related Products and Field Manual No. 10-67-2, Department of the Army Manual for Petroleum Laboratory Testing and Operations, specifies limits for free water and particulate matter in aviation fuels. Specifically, free water contamination in jet fuel cannot exceed 10 parts per million (PPM) (1) and particulate matter contamination cannot exceed 2.0 mg/L for Intra-Governmental transfer receipts and 1.0 mg/L on issue to aircraft, or up to 10 mg/L for product used as a diesel product for ground use (1). At a minimum free water and particulate by color (as specified in the appendix of ASTM D2276) are checked daily, while filter effectiveness is checked every 30 days by gravimetric analysis (ASTM D2276).

One of the problems with the gravimetric methods is the poor repeatability and reproducibility of the methods, ASTM D2276 has a repeatability of 0.25 mg/L and reproducibility of 0.62 mg/L at the 1.0 mg/L contaminate level based on a 5 liter sample, where as the Army utilizes 1 liter samples increasing the associated error. While the published repeatability and reproducibility of ASTM D5452 only spans from 0 to 0.6 mg/L applying the provided formulas to the 1.0 mg/L contaminate level provides a repeatability of 0.42 mg/L and reproducibility of 0.73 mg/L, sample volume used to calculate these values in not provided in ASTM D5452, but again 5 liter samples were used to develop these formulas used for these calculations.

PROJECT BACKGROUND

The U.S. Army Tank Automotive Research Development and Engineering Center (TARDEC) has been actively perusing advanced technologies to monitor aviation fuel for particulate and water contamination. The application of light obscuration particle counters for this purpose has risen to the top of available technologies in terms of performance and availability.

The use of particle counting and automatic particle counters for monitoring contamination is frequently used in the hydraulics/hydraulic fluid industry. In 1999 ISO adopted ISO 11171 *Hydraulic fluid power — Calibration of automatic particle counters for liquids*, replacing ISO 4402, as an international standard for the calibration of liquid particle counters giving NIST traceability to particle size measurement, and providing an area equivalent diameter of particles measured. To simplify the reporting of particle counter data international standard ISO 4406:1999 *Hydraulic fluid power — Fluids — Method for coding the level of contamination by solid particles* by grouping the numbers of particles into broad classes or codes, generally an increase in one ISO code number is caused by a doubling of the contamination level. The

Energy Institute (EI) has published guidance documents and test methods relating to fuel quality measurement using electronic sensors. In February 2012 the second edition of EI 1598 *Design, functional requirements and laboratory testing protocols for electronic sensors to monitor free water and/or particulate matter in aviation fuel* was published. In August 2012 EI published the first edition of EI 1570 *Handbook on electronic sensors for the detection of particulate and/or free water during aircraft refueling*. EI has also published three standard test methods for evaluating the particulate matter of fuels using light obscuration particle counters; IP 564 – *Determination of the level of cleanliness of aviation turbine fuel – Laboratory automatic particle counter method*; IP 565 – *Determination of the level of cleanliness of aviation turbine fuel – Portable automatic particle counter method*; IP 577 – *Determination of the level of cleanliness of aviation turbine fuel – Automatic particle counter method using light extinction.* ASTM International adopted ASTM D7619 *Standard Test Method for Sizing and Counting Particles in Light and Middle Distillate Fuels, by Automatic Particle Counter*, which utilizes the same instrumentation as IP 565.

DEF STAN 91-91 (UK), MIL-DTL-83133 (US), and MIL-DTL-5624 (US) all include a report only requirement for particle counting. The U.S. Army (5) (6) (7) (8), U.S. Navy (9) (10), U.S. Air Force, and DLA Energy (11) have conducted laboratory and field evaluations of particle counter technologies for fuel contamination monitoring. Testing has concluded that particle counters are unable to distinguish between free water and particulate contamination; however the technology has shown significant promise in monitoring fuel for total contamination, absent of the contaminate composition information.

Several interested parties, both commercial and military, have proposed limits based on light obscuration particle counting technologies based on ISO 4406:1999 detailed in Table 1 and references (12) (13) (5) (14) (15) (16) (17) (18). As a result of laboratory testing, the U.S. Army has proposed a working cleanliness limit (modified from ISO 4406) of 19/17/14/13 utilizing the 4µm (c)/ 6µm (c)/ 14µm (c)/ 30µm (c) size channels (5). The 30µm (c) size is included for the detection of free water in the fuel. The proposed ISO code limits of 19/17/14/13 are based on the 1.0 mg/L concentration levels for the A1 and A2 test dusts, and down to a 5 ppm free water presence.

	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*		
Diesel Fuel			
World Wide Fuel Charter 5th		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 11/8/7

Table 1. Proposed Particle Counter Limits
*addition of 30 micron channel proposed by U.S. Army for detection of free water.

APPROACH

The particle counter limit evaluation took place at TARDEC's Army Petroleum Laboratory (APL) in New Cumberland, Pennsylvania. APL provides quality surveillance of U.S. Government owned petroleum products worldwide and provides technical support to Army installations by providing laboratory testing services of their bulk fuels supplies including filter effectiveness testing to ensure that the product meets specifications and environmental requirements.

To evaluate the proposed light obscuration particle counter limit a particle counter specified by IP 564, and a particle counter specified in IP 565 and ASTM D7619 was provided to the APL to test field samples that come in from locations across the United States.

Each sample was particle count tested via IP 564 (first 59 samples) or IP 565/ASTM D7619 and ASTM D5452 to measure the particulate contamination content by gravimetric determination. Additionally free water content is determined by performing ASTM D3240 – Standard Test Method for Undissolved Water in Aviation Turbine Fuels was performed when particle counts exceeded the proposed 19/17/14/13 limits, but the gravimetric measurements were below 1.0 mg/L.

ANALYSIS

From September 2012 thru July 2015, 1614 samples were tested for foreign, particulate or free water, contamination. 1376 of the tested samples passed both the 1.0 mg/L gravimetric limit and the 19/17/14/13 particle count limit, with 238 samples failing either the particle count or gravimetric limit, or both test limits. 188 of the 238 samples tested failed the particle count limit and 157 of the 238 samples failed the gravimetric limit, 107 of the samples failed both the particle count and the gravimetric limit. 50 samples gave a false negative response to the proposed particle count limits by failing the gravimetric limit but passing the particle count limit.

81 samples gave a false positive response to the particle count limit by failing the particle count limit, but passing the gravimetric limit of 1.0 mg/L.

False Negatives

An analysis of the 50 false negatives which had a gravimetric measurement over 1.0 mg/L but a particle count under 19/17/14/13, showed that 36 of these samples may have a gravimetric contaminate load lower than the 1.0 mg/L limit based lack of precision of ASTM D5452. While the published repeatability of ASTM D5452, Equation 1, only spans from 0 to 0.6 mg/L applying the provided formulas to the 1.0 mg/L contaminate level provides a repeatability of 0.42 mg/L. Also the sample volume used to develop this formula was 5 liters, the testing at APL only utilizes 1 liter compounding to potential error.

$$r = 0.415x^{0.5}$$

Equation 1. ASTM D5452 repeatability

14 samples that have a high gravimetric measurement cannot be accounted for with particle counter readings. There is the potential that particles are present on the gravimetric filter pad that has a nominal pore size of 0.8µm that could be either smaller than 4µm (c) or greater than 200µm (c) in the case of the IP 564 instrumentation or 70µm (c) in the case of the IP 565 specified instrumentation. It has been requested that the laboratory photograph filter pads with a greater than 1.0 mg/L particle loading for visual analysis, as particles greater than 70µm or 200µm (c) would be visible to the naked eye and heavily contribute to the mass load of the tested filter. Examples of filters with large particles are shown in Figure 1. It has also been requested that failed gravimetric samples be performed in duplicate to determine if poor measurement repeatability of ASTM D5452 is leading to false negatives of the particle counter agreement.

Figure 1. Examples of filter monitors with large visible particulates

False Positives

An analysis of the 81 false positives which had a gravimetric measurement under 1.0 mg/L but a particle count over 19/17/14/13, showed that 36 of these samples may have a gravimetric contaminate load higher than the 1.0 mg/L limit based lack of precision of ASTM D5452. 2 of the 81 samples were Diesel No. 2 samples that where high in the 4µm (c) channel only. 78 samples where aviation fuels (JP-8 and F-24) that where high in the 6µm (c),14µm (c), and/or 30µm (c) channels indicating the potential for free water contamination. 6 of these samples were tested via ASTM D3240 and confirmed contain greater than 5.0 ppm free water, 9 samples contained 1-5 ppm, while 35 fuel samples were absent of free water, and 27 samples were not tested for free water. The utilization of glassware to transfer the fuel through the ASTM D3240 filter pad may have adversely impacted the free water measurements. The utilization of co-solvent as specified in Annex B of the test method may be a better method to determine water attribution to particle count measurements.

18/16/13 Limit Evaluation

Several organizations have proposed the use of 18/16/13 limits utilizing the 4µm (c)/ 6µm (c)/ 14µm (c) size channels as detailed in Table 1. The 1614 samples analyzed were evaluated against these proposed limits. 1105 of the tested samples passed both the 1.0 mg/L gravimetric limit and the 18/16/13 particle count limit, as opposed to the 1376 samples that passed both the 1.0 mg/L and 19/17/14/13 limits. Utilizing the 18/16/13 limits reduces the number of false negatives, failing the gravimetric limit but passing the particle count limit, from 50 down to 27 for a 46% reduction. The 18/16/13 limits increased the number of false positives from 81 up to 352 for a 352% increase.

Comparing the 18/16/13 particle count limit to 0.5 mg/L gravimetric limit specified in ATA 103 (19) to the data collected under this effort shows 727 of the samples collected failing either the particle count or the gravimetric limit, 314 of which failed both methods. 245 false negatives were recorded where the sample failed the gravimetric but passed the particle count and 168 false positives where the particle count was higher than the proposed limit but the gravimetric load was lower than the 0.5 mg/L limit. The false negative readings may again be affected by the repeatability of ASTM D5452 which is 0.293mg/L at the 0.5mg/L limit which calls into question up to 205 or the 245 false negatives, 103 of the 168 false positives may also be affected by this. Additionally the presence of free water at levels as low as 5 ppm, 15ppm specified in ATA 103, has shown give particle counts exceeding the 19/17/14/13 limits proposed by the U.S. DOD, and may be contributing to the number of false positives identified.

CONCLUSIONS AND RECOMENDATIONS

The laboratory data collected supports the U.S. Department of Defense ISO code limits of 19/17/14/13, developed from 1.0 mg/L concentration levels for the A1 and A2 test dusts, and down to a 5 ppm free water presence.

To identify the cause of false negatives filter monitor pads with a greater than 1.0 mg/L particle loading should be evaluated via visual analysis, as particles greater than 70µm or 200µm (c) would be visible to the naked eye and heavily contribute to the mass load of the tested filter.

Filter monitors that have sediment loading greater than 1.0mg/L should also be filtered in duplicate to determine if poor measurement repeatability of ASTM D5452 is leading to false negatives of the particle counter agreement.

The Department of Defense is moving forward with the 19/17/14/13 limits for the 4µm (c)/ 6µm (c)/ 14µm (c)/ 30µm (c) size channels for inclusion into Table I of MIL-STD-3004D change 1 as an acceptable method for particulate matter with the stipulation (requirement) to perform follow on testing for particulate matter via ASTM D5452 and water via ASTM D3240 for product exceeding the limits.

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LIST OF SYMBOLS ABBREVIATIONS AND ACRONYMS

µm	Micrometer
APL	Army Petroleum Agency
ASTM	ASTM International
ATA	Air Transport Association
AUST	Australia
EI	Energy Institute
IP	Institute of Petroleum
ISO	International Organization for Standardization
L	Liter
mg/L	Milligrams per Liter
MIL	Military
mL	Milliliter
ppm	Parts Per Million
STD	Standard
TARDEC	Tank Automotive Research Development and Engineering Center
U.S.	United States