

Examination of Avgas (N47515, Cessna 182)

Report Prepared for:

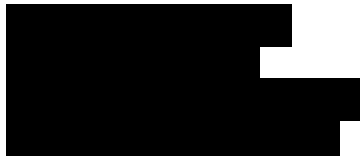
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July, 2015

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Executive Summary

- Report of the GC-MS results for three fuel samples from N47515, Cessna 182, received from NTSB.
- Report of the Karl Fischer coulombic titrimetric analysis of three samples from N47515, Cessna 182, received from NTSB.

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Distribution:

Since this report presents interim (pre-decisional) results of an ongoing project, the content is not for public dissemination. It is for the use of the sponsor only.

Introduction

The Unique Role of NIST

The Applied Chemicals and Materials Division at NIST, in essence, acts as the nation's reference laboratory for measurements, standards, data, and models for the thermophysical properties of chemical substances. The web site at <http://www.nist.gov/mml/acmd/> provides some information about the Division. Within this Division, the Experimental Properties of Fluids Group performs measurement work that is the required basis for all fluid characterizations and thermophysical model development. The web site at http://www.nist.gov/mml/acmd/fluids_properties/ provides information about this Group. One of the four main focus areas of the Group is the measurement and characterization of liquid and gaseous fuels.

Composition Analysis by Gas Chromatography with Mass Spectrometric Detection:

A good introduction to the metrology of both gas chromatography and mass spectrometry can be found in the recent editions of the CRC Handbook of Chemistry and Physics. No further introductory material will be presented here.[1]

Karl Fischer Coulombic Titrimetry:

The Karl Fischer (KF) titration is one of the most important techniques for the analysis of moisture. The method is very sensitive (1 ppm water, mass/mass) and reproducible to within 1-3 percent.[1] The classical Karl Fischer reagent consists of iodine, pyridine, and sulfur dioxide, with methanol or ethylene glycol monomethyl ether as the solvent. A more convenient approach to the Karl Fischer titration is the use of coulometrically generated iodine, usually obtained from dissolved potassium iodide in a pyridine-free amine solvent. A coulometric apparatus has two sets of electrodes. One set of electrodes, usually in the form of platinum screens separated by a glass frit, generates the iodine by anodic oxidation. The sensing electrodes consist of dual platinum probes.

Samples:

Three samples were presented for analysis in glass jars provided by NTSB. They are identified as follows:

1. NTSB N47515, Cessna 182 Left Wing Tank
2. NTSB N47515, Cessna 182 Sump Engine Tank
3. NTSB N47515, Cessna 182 Right Wing Tank

Samples 1 and 2 showed the typical blue coloration of 100LL. Sample 3 was of a much smaller volume, and had a slight yellow brown cast.

Results and Findings

Composition Analysis by Gas Chromatography – Mass Spectrometry Measurements

The samples were analyzed with gas chromatography (GC) with mass spectrometry (MS) detection.[2, 3] A 30 m capillary column of 5 % phenyl-95 %-dimethyl polysiloxane having a thickness of 0.25 μm and an internal diameter of 250 μm was used. The samples were injected manually into a split/splitless injector set with a 200 to 1 split ratio. The injector was operated at a temperature of 250 °C with a constant head pressure of 68.95 kPa (10 psig). High-purity helium was used as the carrier gas. The GC oven was ramped from 35 °C to 83 °C at 3 °C/min. The mass spectrometer scanned masses from 15 to 550 relative molecular mass (RMM) units. Two separate injections were performed for each sample. Spectral peaks were interpreted with guidance from the NIST/EPA/NIH Mass Spectral Database and the CRC Handbook of Basic Tables for Chemical Analysis.[2, 4] Peak heights were normalized to the tallest peak (the base peak), identified as 2,2,4-trimethyl pentane (RT = 1.5 minutes).

The results are presented as chromatograms in Figures 1-4, below. Figures 1-3 present the NTSB N47515 Cessna 182 Right Wing Tank, the NTSB N47515 Cessna 182 Right Wing Tank and the NTSB N47515 Cessna 182 Sump Engine Tank, respectively. Figure 4 presents all three chromatograms overlaid.

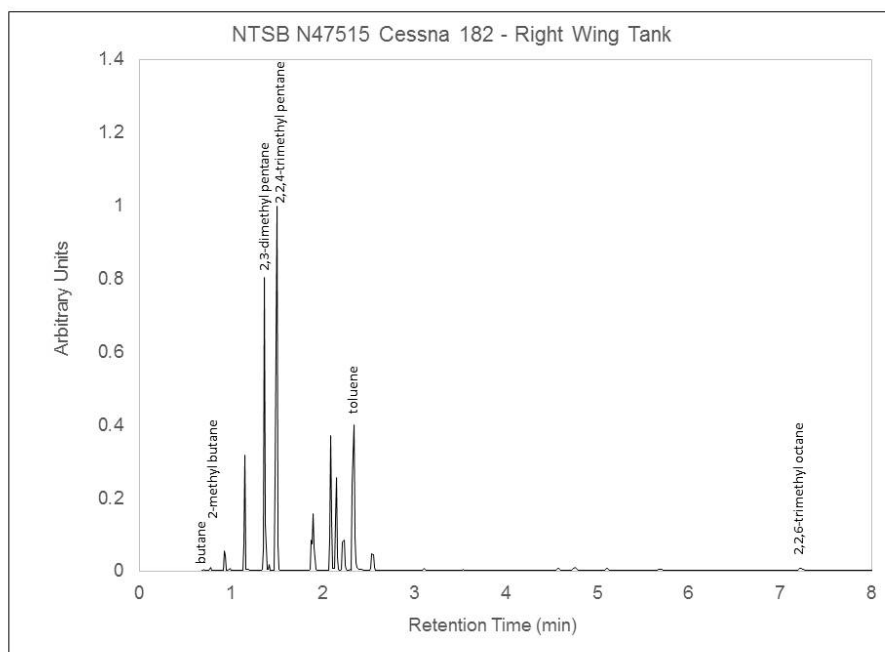


Figure 1: A chromatogram of the fluid identified as NTSB N47515 Cessna 182 Right Wing Tank. The method is provided in the text.

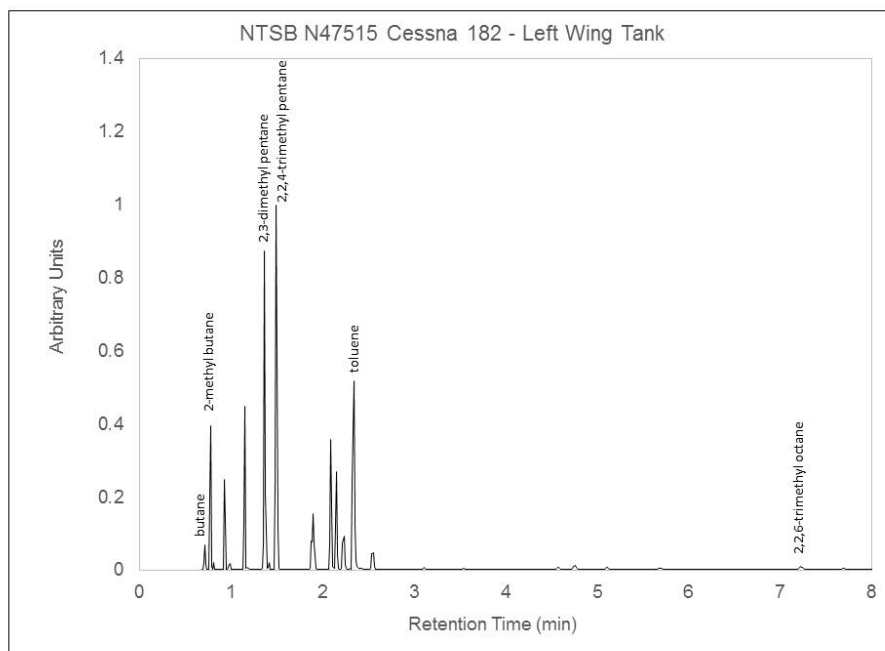


Figure 2: A chromatogram of the fluid identified as NTSB N47515 Cessna 182 Left Wing Tank. The method is provided in the text.

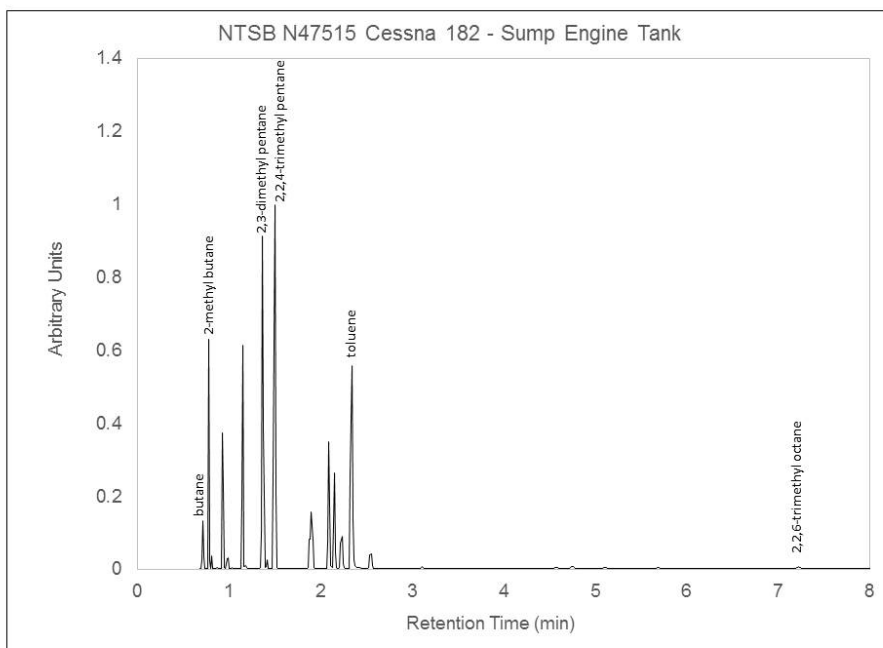


Figure 3: A chromatogram of the fluid identified as NTSB N47515 Cessna 182 Sump Engine Tank. The method is provided in the text.

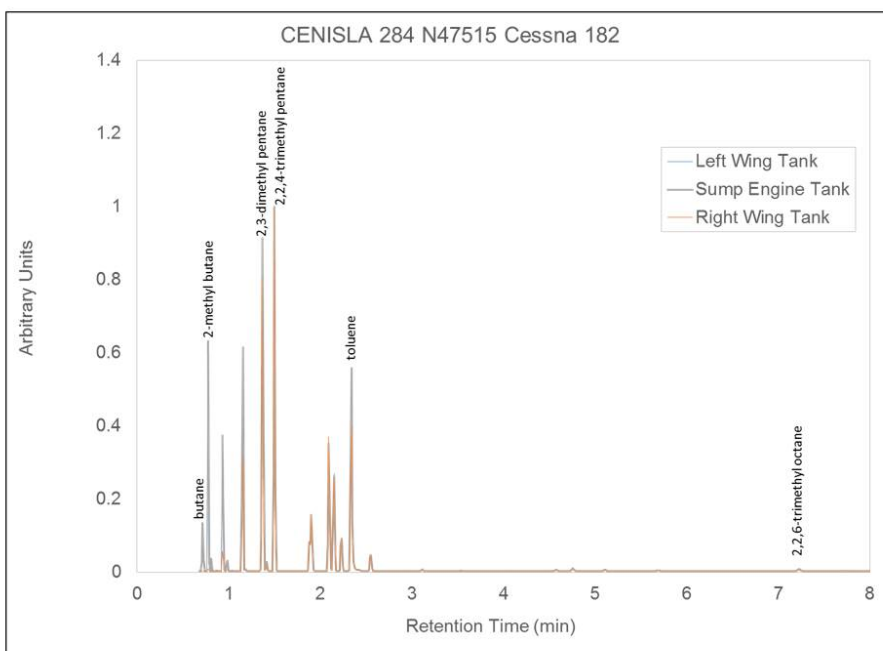


Figure 4: An overlay of the previous three chromatograms.

All three chromatograms are consistent with our prior analyses of samples of 100LL Avgas.[5, 6] In particular, a question arose regarding the sample labeled NTSB N47515 Cessna 182 Right

Wing Tank because it does not appear to have the usual blue coloration of 100LL. While this sample is also consistent with 100LL, it has a significantly lower light fraction (primarily composed of n-butane and 2-methylbutane) than the other two samples.

Moisture Analysis Measurements

Each sample was analyzed for water content by coulometric Karl Fischer (KF) titration. At least three replicate determinations were performed for each sample (unless otherwise noted). The general procedure for this type of measurement is given in ASTM Standard Test Method E 1064–00.[7]

Three replicate measurements of the sample labeled NTSB N47515 Left Wing Tank 100LL gave the following results: 1.5301 g of the sample was found to contain 117.6 µg of water (for a concentration of 76.9 ppm); 1.4493 g of the sample was found to contain 111.1 µg of water (for a concentration of 76.7 ppm); 1.2507 g of the sample was found to contain 90.8 µg of water (for a concentration of 72.6 ppm). Therefore, we found the water concentration in this sample to be 75 ppm with a standard deviation of 2 ppm.

Three replicate measurements of the sample labeled NTSB N47515 Engine Sump 100LL gave the following results: 1.2788 g of the sample was found to contain 75.3 µg of water (for a concentration of 58.9 ppm); 1.2740 g of the sample was found to contain 83.0 µg of water (for a concentration of 65.1 ppm); 1.2982 g of the sample was found to contain 80.1 µg of water (for a concentration of 61.7 ppm). Therefore, we found the water concentration in this sample to be 62 ppm with a standard deviation of 3 ppm.

The sample labeled NTSB N47515 Right Fuel Tank 100LL was clear and pale brownish yellow. Due to a limited quantity of sample, only one titration was performed on this sample, and even that titration was performed with an order of magnitude less liquid than was typical for other fuel-rich samples. Therefore, the uncertainty statement at the beginning of this document does not apply, and the result is best viewed as only semi-quantitative. For this single titration, 0.2292 g of the sample was found to contain 38.5 µg of water (for a concentration of 168.0 ppm).

The moisture measurements are represented as histograms in Figure 5 below:

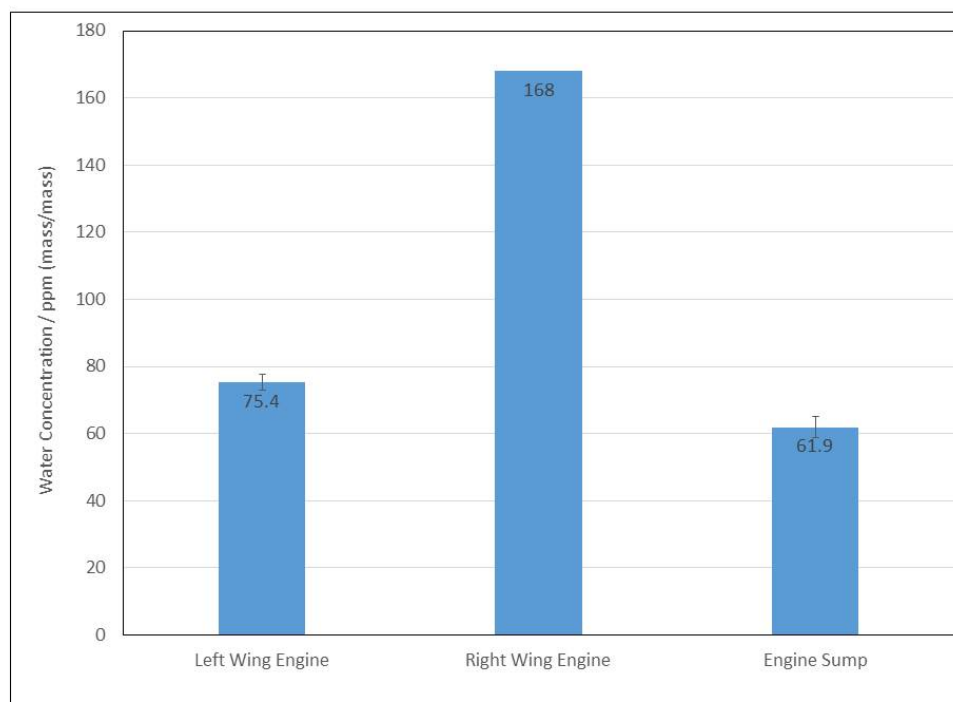


Figure 5: A histogram representation of the water content of the samples discussed above.

Conclusions:

The unusual appearance of the sample labeled NTSB N47515 Right Fuel Tank 100LL was inconsistent with the usual appearance of avgas100LL. Analysis by GC-MS indicates that the chemical composition of this fluid is consistent with 100LL, absent certain lighter components. It is possible that these constituents might have been lost by heating.

The water content is consistent with saturation but no separate phase of water was noted in any of the samples.

References:

- [1] Haynes, W.M., *CRC Handbook of Chemistry and Physics*, 96th ed. 2015, BocaRaton, FL: Taylor and Francis CRC Press.
- [2] Bruno, T.J., Svoronos, P.D.N., *CRC Handbook of Basic Tables for Chemical Analysis*, 3rd. ed. . 2011, Boca Raton: Taylor and Francis CRC Press.
- [3] Bruno, T.J., Svoronos, P.D.N., *CRC Handbook of Fundamental Spectroscopic Correlation Charts*. 2006, Boca Raton: Taylor and Francis CRC Press.
- [4] *NIST/EPA/NIH Mass Spectral Database, S. R. D.*, 2005. *SRD Program*, National Institute of Standards and Technology, Gaithersburg, MD. 2010.
- [5] Lovestead, T.M., Bruno, T.J., *Application of the advanced distillation curve method to aviation fuel avgas 100LL*. *Energy & Fuels*, 2009. **23**: p. 2176-2183.

- [6] Windom, B.C., Lovestead, T.M., Bruno, T.J., *Application of the advanced distillation curve method to the development of unleaded aviation gasoline*. Energy & Fuels, 2010. **24**: p. 3275 - 3284.
- [7] *ASTM Standard 1064-12 Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration, Book of Standards, West Conshohocken, PA: ASTM International*. 2012.