University of Dayton Research Institute

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Chemical Analysis Investigation of Jet Fuel Contamination Event: Punta Gorda Airport, FL, May 9th, 2019

Technical Report

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Introduction

A chemical analysis study was performed to investigate the possibility of diesel exhaust fluid (DEF) contamination of jet fuel as a root cause for some emergency landings of business jets fueled at the Punta Gorda airport (KPGD) on May 9, 2019. Various fluid and hardware samples were sent by local authorities to, and received by, the University of Dayton Research Institute (UDRI) Fuel Science Group for chemical analysis. Received samples included fuel system icing inhibitor (FSII, also known under the trade name of Prist) from KPGD bulk storage, fluid sampled from the #6 refueling truck FSII pail at KPGD, and numerous fuel and hardware samples taken from affected aircraft fuel tanks/filter housings and other suspected locations at the time of the event. A summary of all samples received and chemical analysis results are listed in Table 1. This report outlines the analysis conducted on the received samples and an explanation of the results.

Experimental Description

Aqueous phase liquid samples were analyzed by Refractive Index measurement to determine the approximate concentration of water and FSII. The presence of urea and FSII in fuel water bottoms was determined using gas chromatography-mass spectrometry (GC-MS); extracted ion chromatography for the *m/z* 60 fragment in conjunction with library search routines and analysis of standards were performed to positively identify the urea and FSII in a liquid mixture. Solid particulate sample, taken from a filter/screen, was dissolved in methanol to identify the presence of a urea-alkane clathrate via GC-MS. Jet fuel samples were not able to be analyzed for urea since the urea (or more appropriately the urea-clathrate) was insoluble in the fuel and could not be suitably sampled. Only when water bottoms were present in the fuel could the presence of urea be tested. A nitrogen-specific GC detector was also used as an alternative technique to verify the presence of urea in aqueous samples, in addition to GC-MS detection.

Results

The results of the testing indicated that the “drum” sample of FSII (**Sample 3**) was pure, unadulterated FSII. However, the “Truck 6” samples of FSII (**Samples 1 and 2**) were heavily contaminated with both water and the chemical urea. The water and urea likely came from the addition of Diesel Exhaust Fluid (DEF); DEF is a mixture of urea (33 wt%) and water (67 wt%) used to reduce tailpipe pollutant NO_x emissions of diesel trucks. Urea is known to be soluble in both water and FSII, but when urea is added to hydrocarbon jet fuels, the urea forms a solid precipitate (known as a “clathrate”) with some of the fuel molecules (namely the normal alkanes). The clathrate is a solid, white to off-white precipitate, and is known to interfere with fuel filtering and fuel system operation. Even small amounts of urea can be detrimental to the safe operation of aircraft due to plugging of fuel filters, nozzles, and other critical fuel system components. Based on the chemical analysis of the samples submitted, the FSII pail of the KPGD refueling truck #6 appears to be the source of urea, which subsequently contaminated fuel uploads that were being treated with FSII.

Table 1. Summary of Samples Submitted for Investigation of Contamination

Sample Information					Previous Test Results (Dates)	Approximate FSII Concentration (vol%)		Approximate DEF Conc. (vol%) in Aqueous Phase (GC-NCD)	General Observations	Data Figure No.
No.	Identification	Type	Shipper POC	Date UDRI Rec'd		Aqueous Phase (Refractive index)	Fuel Phase (GC-MS)			
1	KPGD fuel truck #6 FSII pail-sample poured (00197-002.001)	FSII	KPGD FBO-Intertek-Humberto Hill-954-448-6051	5/15/2019	#1 Intertek-(05-10-19)-Water Content 24.729%	84	n/a	33	FSII, with water and urea	A-1
2	KPGD fuel truck #6 FSII pail-sample from injection system (00197-003.001)	FSII		5/15/2019	#1 Intertek-(05-11-19)-Water Content 41.61% #2-Intertek 2nd lab-(05-14-19)-18.121% Water Content	75	n/a	46	FSII, with water and urea	A-2
3	KPGD FSII 55 gallon drum used to service truck pails (00197-004.001)	FSII		5/15/2019	#1 Intertek- (05-11-19)-0.018% Water Content #2-Intertek 2nd lab-(05-14-19)-.008% Water Content	100	n/a	<0.1	mainly FSII, contains some BHT, **no urea present**	A-3
4	N744AT, (Red can, ~10 mL), liquid from A/F fuel filters	Jet A	Air Trek-shipped by KPGD FBO 314-226-5410	5/15/2019	none	42	n/a	2	Water, diegme and urea, no fuel present	A-4
5	N601AT RH Wing Inbd/Fwd drain sum (water & fuel)p	Jet A	Air Trek-driven to UDRI by Aviation Tech from Louisville-Greg Vanwinkle 502-361-7130	5/16/2019	none	59	0.100	42	(water bottom) water, FSII, urea	A-5
6	N601AT RH Wing Inbd/Fwd drain sump (fuel only)	Jet A		5/16/2019	none	‡	0.099	‡	no water bottom	
7	N601AT RH Wing Inbd/aft drain sump (water & fuel)	Jet A		5/16/2019	none	53	0.110	16	(water bottom) water, FSII, urea	A-6
8	KPGD Fuel Truck #6 (fuel only)	Jet A	KPGD FBO-Intertek-Humberto Hill-954-448-6051	5/17/2019	#1 Intertek-(05-10-09)-pass	‡	<0.001	‡	Jet fuel, no water bottom, not analyzed	

Table 1 (continued). Summary of Samples Submitted for Investigation of Contamination

Sample Information					Previous Test Results (Dates)	Approximate FSII Concentration (vol%)		Approximate DEF Conc. (vol%) in Aqueous Phase (GC-NCD)	General Observations	Data Figure No.
No.	Identification	Type	Shipper POC	Date UDRI Rec'd		Aqueous Phase (Refractive index)	Fuel Phase (GC-MS)			
9	Naples, FL truck #3 (1 gal can)	Jet A	Naples Airport Authority-David Hill-239-595-0266	5/20/2019	#1 Intertek-(05-17-19) pass	‡	n/a	‡	visible water present at bottom of 1 gal can sample but cannot isolate water drop	
10	Naples truck #4	Jet A		5/20/2019	none	‡	n/a	‡	visible water present at bottom of 1 gal can sample; **no urea present in this water**	A-7
11	Naples truck #7	Jet A		5/20/2019	none	‡	n/a	‡	no visible water	
12	Naples tank #2	Jet A		5/20/2019	#1 Intertek-(05-17-19) pass	‡	n/a	‡	no visible water	
13	N601AT filters (4) (large and small filters, left and right engine)	Hardware (filters)	Air Trek-driven to UDRI by Aviation Tech from Louisville-Greg Vanwinkle 502-361-7130	5/17/2019	none	‡	n/a	‡	inspected, precipitate observed on Right hand engine, large filter. Analysis indicated urea. No other filters from N601AT had precipitate	A-8
14	N601AT fuel screens (only 1 received, not 2)	Hardware (screens & solid precipitate)		5/17/2019	none	‡	n/a	‡	white precipitate, diluted in methanol, verified as urea clathrate	A-9, 10
15	N744AT filters (small, large, left, right)	Hardware (filters)	Air Trek-Wayne Carr 941-628-4290	5/20/2019	none	‡	n/a	‡	filters contain white precipitate, trace diegme, trace urea	
16	N823SF sump	Jet A	43602	5/20/2019	Textron Aviation-Tampa	‡	n/a	‡	not tested	
17	N823SF fuel cell after fuel was drained	Fuel residue	43602	5/20/2019	Textron Aviation-Tampa	41	n/a	n/a	contains water, diegme, **no urea present**	

‡No or insufficient water bottoms present in sample to test, e.g., fuel only sample.
n/a – not determined

Samples of the fuel from fuel filters and from on-board sump samples were taken from aircraft N744AT and N601AT between 5-14-19 and 5-21-19 (**Samples 4-7 and 13-15**). If water bottoms were present in these samples (such as **Samples 5 and 7**), the water was tested for FSII using refractive index measurements and tested for urea using GC-MS. The water bottoms in the tanks of both of these aircraft were contaminated with high levels of DEF. No fuel samples were tested for DEF because DEF creates an insoluble precipitate and the urea component is not substantially soluble in the fuel in the presence of an aqueous phase. The three fuel samples from the aircraft (**Samples 5-7**), along with the fuel sample from Truck 6 (**Sample 8**) were tested for FSII concentration by GC-MS.

A fuel screen from aircraft N601AT (**Sample 14**) was visually examined and found to contain a white precipitate. The white solid was diluted in methanol and tested by GC-MS, the results of which verified the presence of urea as a clathrate formed from reaction of urea (from DEF) with the fuel. Fuel from the right hand engine (both forward and aft) of aircraft N601AT contained water bottoms (**Samples 5 and 7**): these water bottoms contained urea (as mentioned above). In addition, white, solid precipitate could be seen on the larger right hand side fuel filter for N601AT (one of the four filters in **Sample 13**). The precipitate was identified as the urea clathrate. Materials from the other aircraft, N744AT, were also tested (large and small filters, left and right hand engines - **Sample 15**), and trace urea was found on all of these filter samples. A small amount of fluid from N744AT (**Sample 4**), was taken from a fuel filter and found to be mostly water, FSII and urea (not jet fuel as indicated on the sample list).

The four 1-gallon samples of fuel from Naples, FL (**Samples 9-12**) were received and examined. The bottoms of the cans were sampled for a possible water layer. There was visible water in two samples (**Samples 9 and 10**: Trucks 3 and 4), which were isolated and tested for urea. No urea was observed in these truck sample water bottoms.

Another set of samples were received from Textron Aviation –Tampa (**Samples 16 and 17**) pertaining to aircraft N823SF. One sample (**Sample 16**) contained fuel, and one sample (**Sample 17**) contained fuel with an obvious water bottom. The water bottom was sampled and tested for urea contamination. No urea contamination was observed.

The test results are summarized in Table 1. Gas chromatography with a nitrogen detector (Nitrogen Chemiluminescence Detector – NCD) was used as a second method to verify the presence of urea in the samples as shown. As mentioned previously, we have been unable to test fuel samples for urea; the urea clathrate that forms is not soluble in fuel and therefore precipitates (forms a solid). Whenever urea is measured, it is measured from a water bottom, or in deposits from a filter or screen, which can be easily dissolved in methanol. The appendix shows data obtained by GC-MS and GC-NCD testing on some of the samples examined.

Conclusions

In conclusion, the data clearly show that urea and water (the two components of DEF), were detected in many of the samples submitted from aircraft N601AT and N744AT. In addition, DEF components were detected in FSII samples for KPGD fuel truck #6, but not in the FSII from the original drum. DEF contamination was not observed in any of the Naples samples nor in any samples from N823SF. From these observations, it seems likely that DEF was added to a partially full FSII container/pail on refueling truck #6 at the Punta Gorda Airport. The FSII-DEF mixture remained a single phase liquid until the truck uploaded fuel with additive onto aircraft. Once mixed on the aircraft, the urea component of DEF combined with the jet fuel to create a solid, clumpy white deposit which collected on aircraft fuel filters, screens, and in fuel water bottoms.

Acknowledgements

The University of Dayton Research Institute would like to thank the Fuels and Energy Branch of the Air Force Research Laboratory at Wright-Patterson AFB, OH for allowing access to their chemical analysis and fuels research facilities for this work.

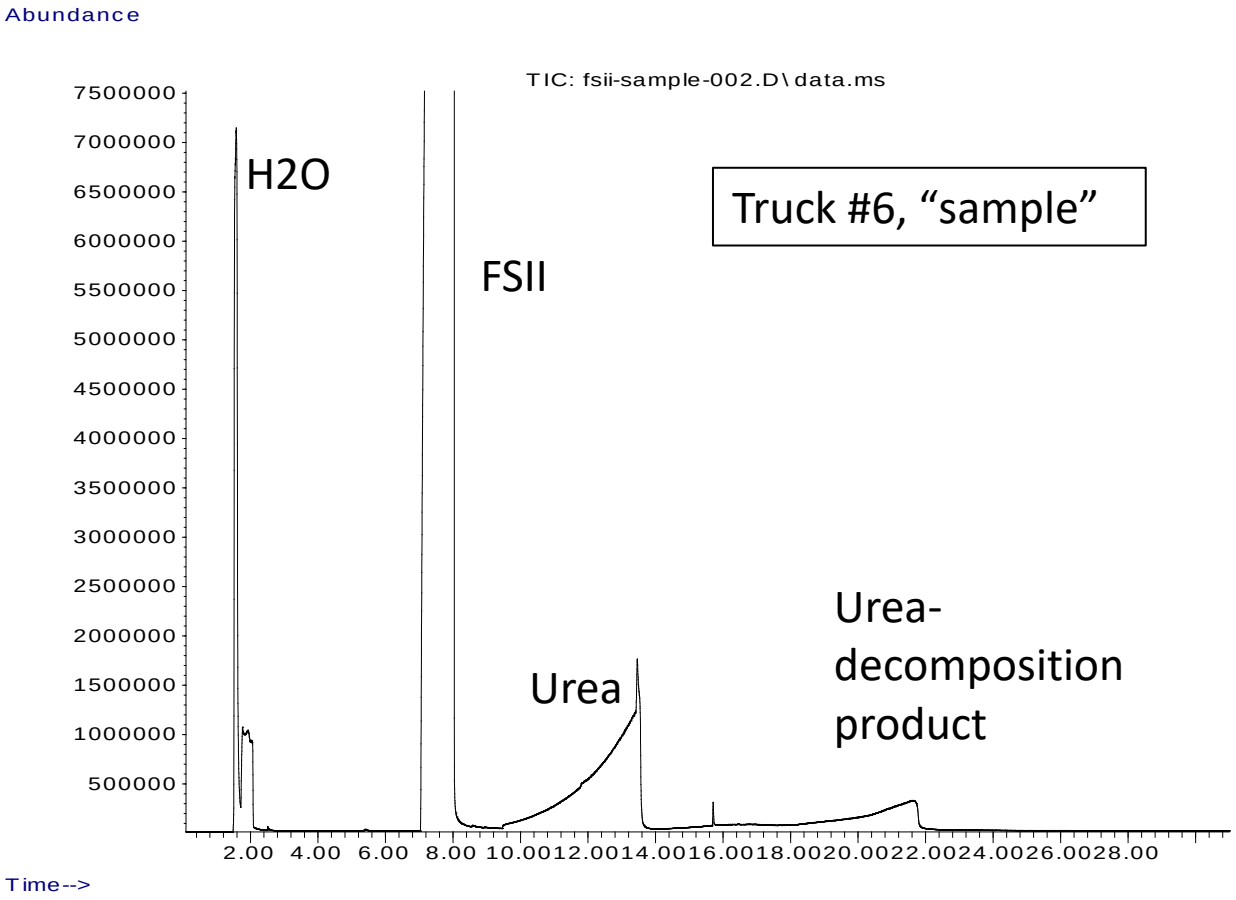

Figure A1. GC-MS of KPGD fuel truck #6 FSII pail, “sample poured”. This FSII sample is clearly contaminated with water and urea, the two components of DEF.

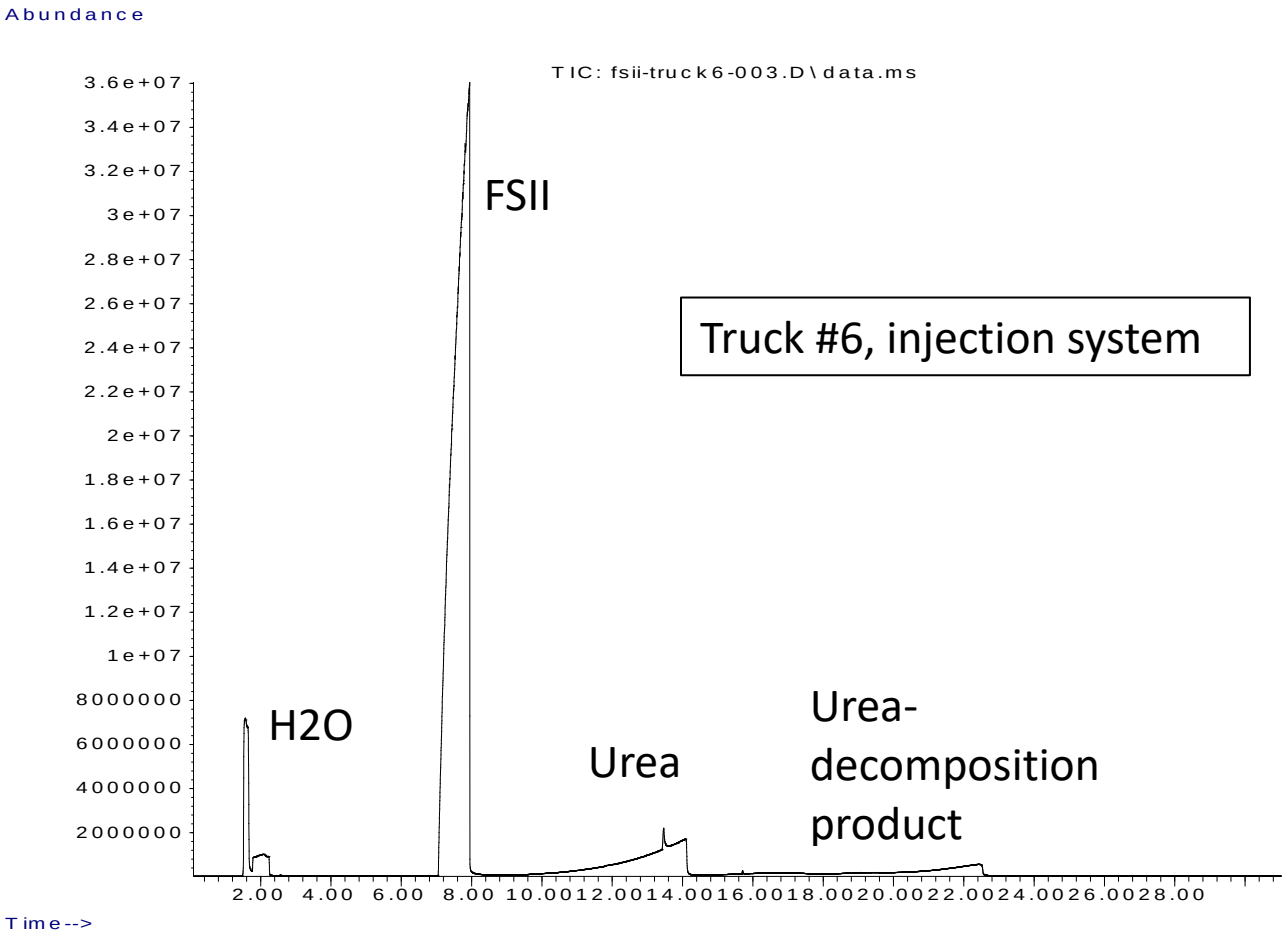

Figure A2. GC-MS of KPGD fuel truck #6 FSII pail, sample from "injection system". This sample is clearly contaminated with water and urea, the two components of DEF.

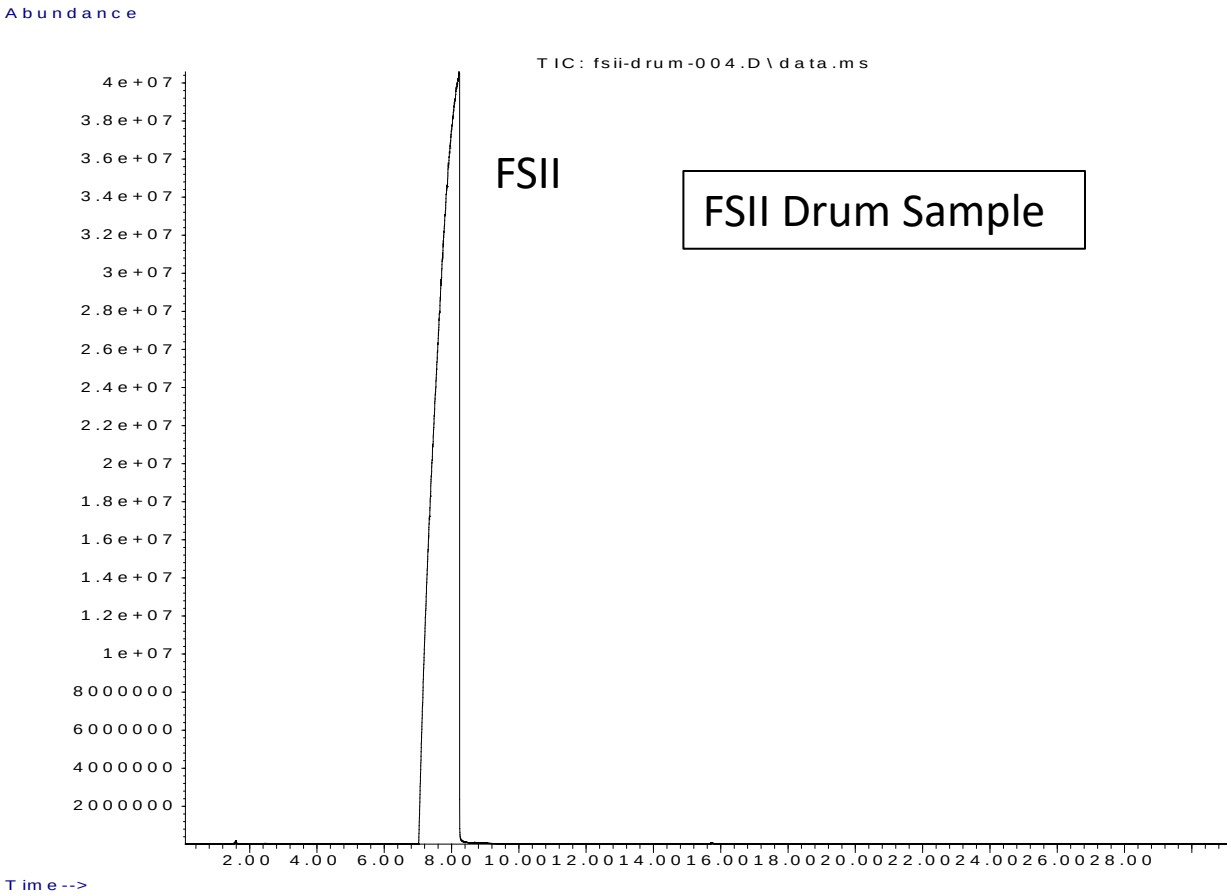

Figure A3. GC-MS of KPGD FSII 55 gallon "drum" used to service truck pail. This FSII sample contains NO water or urea, the two components of DEF.

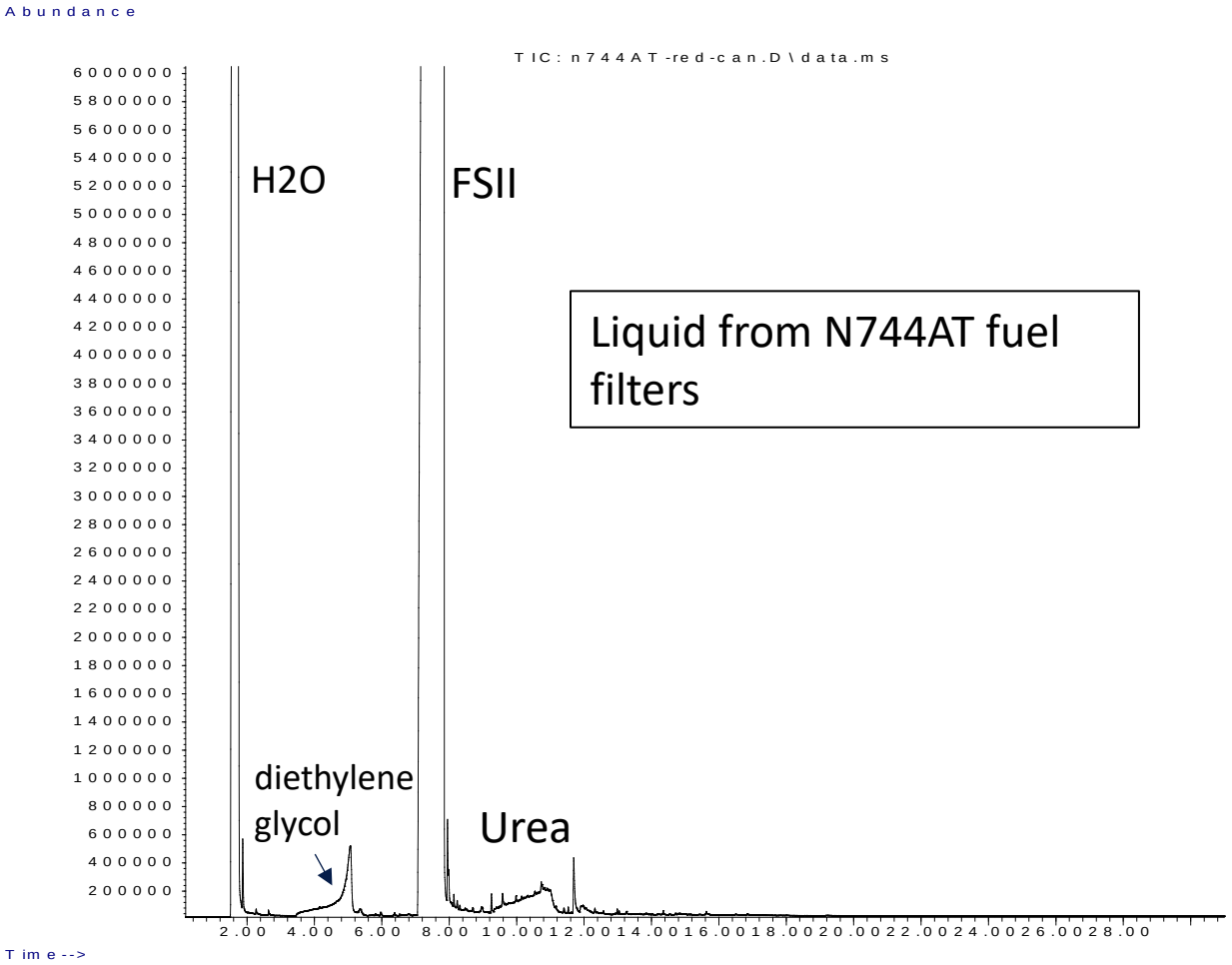

Figure A4. GC-MS of liquid taken from the N744AT fuel filters. This sample shows trace urea and diethylene glycol in a liquid sample that was mostly water and FSII.

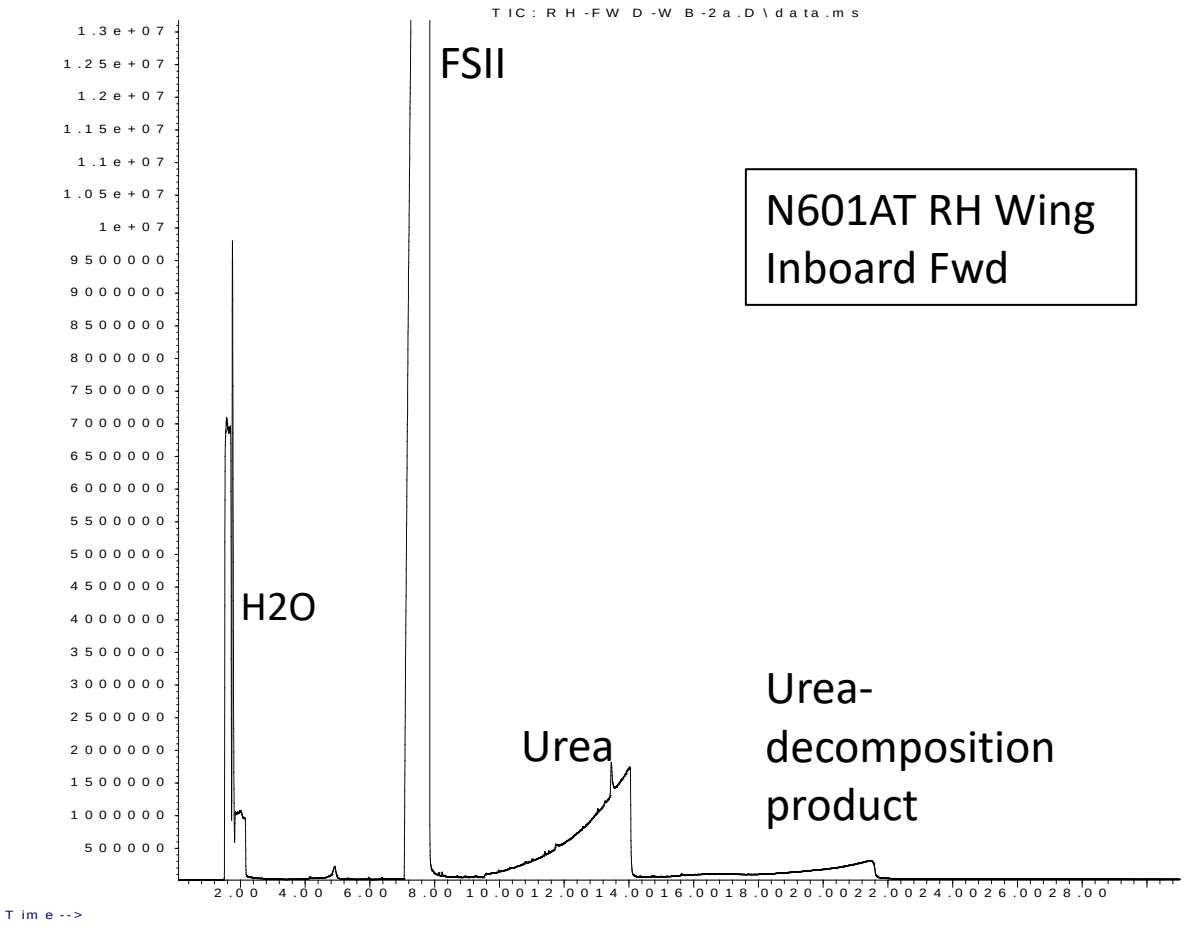

Figure A5. GC-MS of N601ATRH Wing, Inboard Forward Drain Sump (water bottom). This sample contains FSII, water, and urea in the water bottoms of a fuel sample taken from this drain sump.

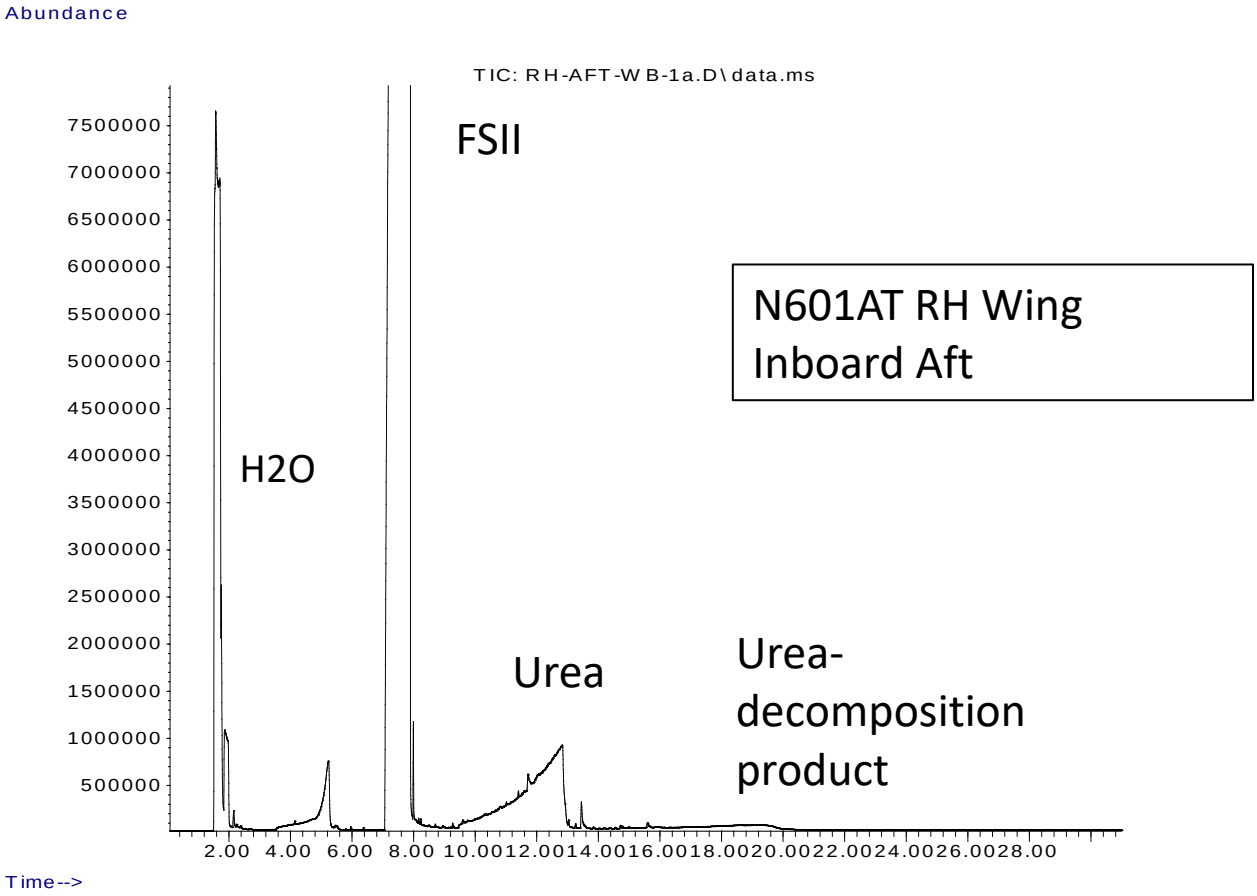

Figure A6. GC-MS of N601AT, Right Hand Wing, Inboard Forward Drain Sump (water bottom). This sample contains FSII, water, and urea in the water bottoms of a fuel sample taken from this drain sump.

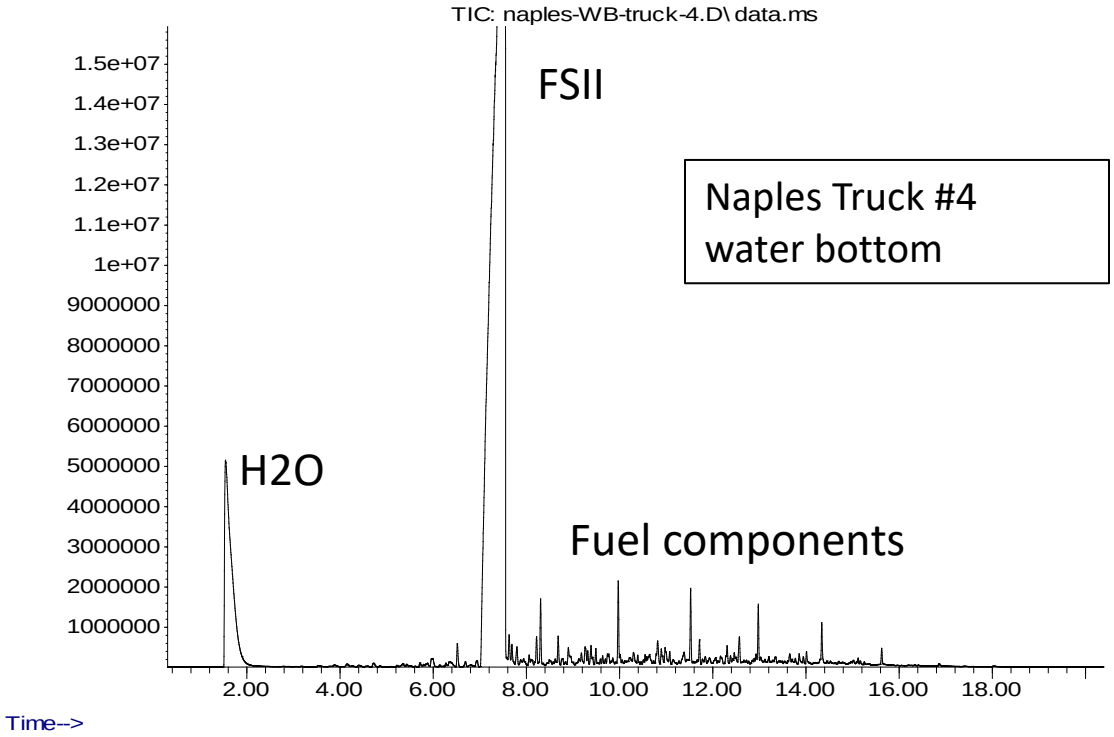

Figure A7. GC-MS of the water bottoms of Naples jet fuel sample, truck #4. This sample contained FSII, water, and some jet fuel components; no urea was detected.

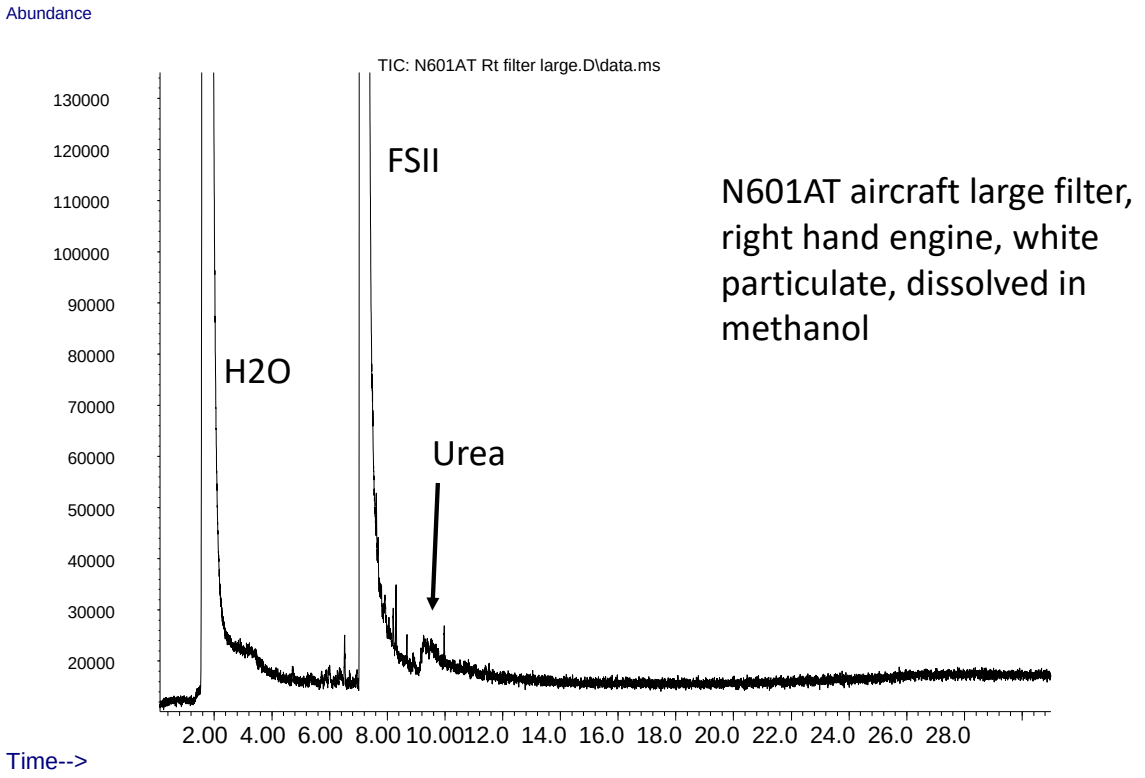

Figure A-8. GC-MS of particulate from the larger of the two right engine filters in aircraft N601AT. White solid precipitate, diluted in methanol (~5 mg solid in 1.0 mL methanol).

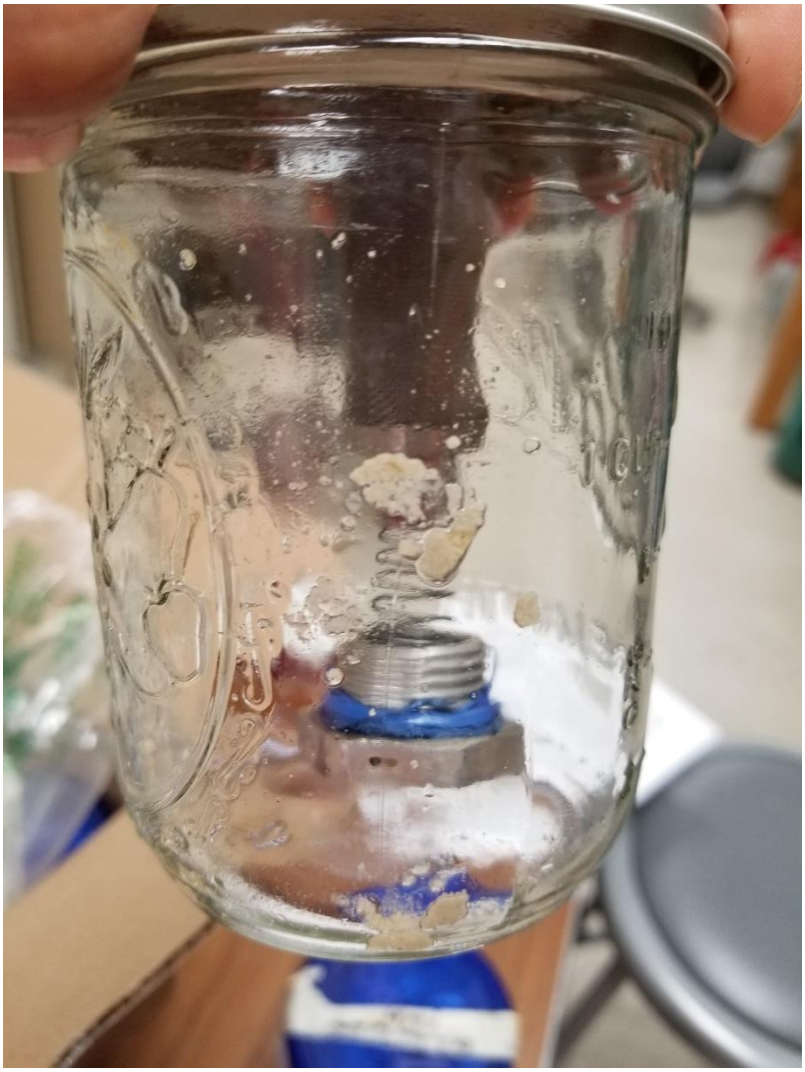

Figure A-9. Picture of the filter screen from the right hand engine of Aircraft N601AT. The filter screen is inside of the glass container, and the white solid on the inside of the glass container is the material sampled. This material was diluted in methanol and analyzed to verify the presence of urea.

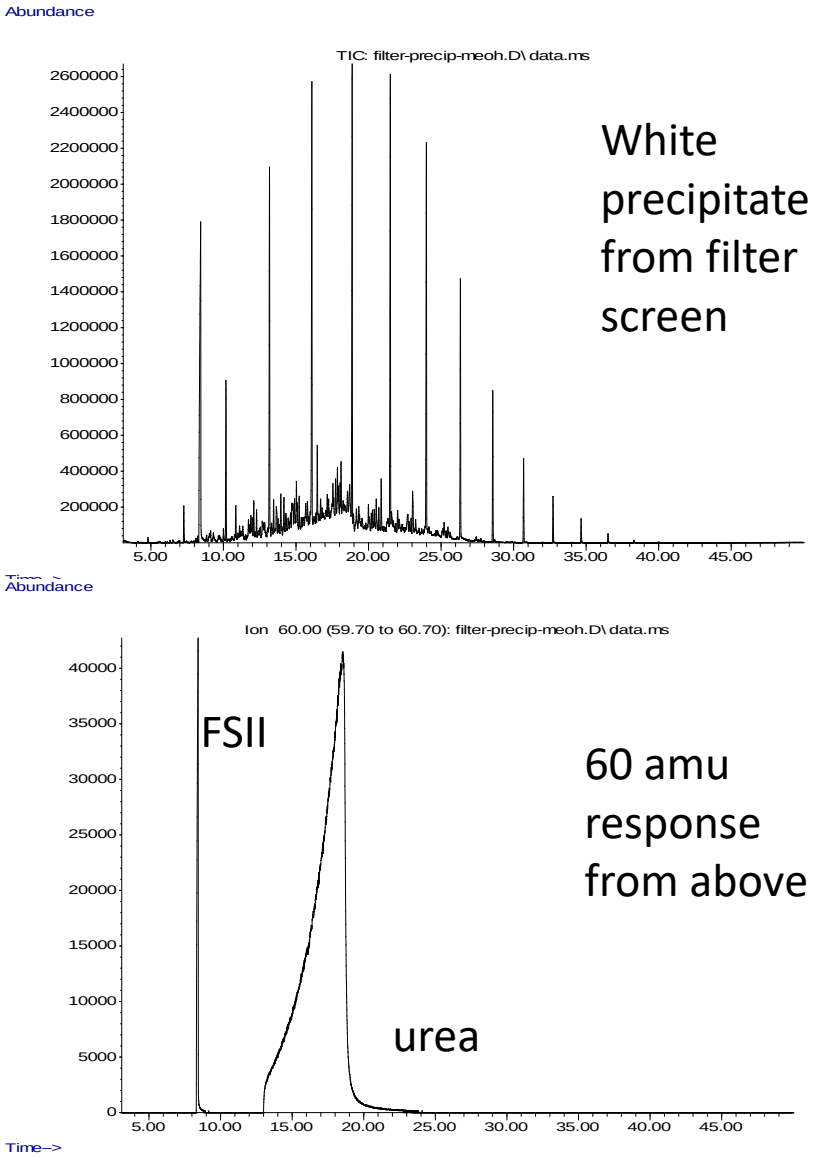

Figure A-10. GC-MS of the white solid taken from the filter screen of Aircraft N601AT. White solid precipitate, diluted in methanol (~10 mg solid in 1.0 mL methanol). Top shows complete signal; bottom shows signal for *m/z* 60 which is indicative of the FSII and urea response.