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# **Investigation of “Apple Jelly” Contaminant in Military Jet Fuel**



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## EXECUTIVE SUMMARY

### Project Objectives

The objective of this project was to characterize the aviation fuel contaminant known as apple jelly with respect to the compositional and process conditions required for its formation, and to determine possible methods, both compositional and process, whereby its formation can be reduced or prevented.

### Approach

The following tasks were involved in the investigation of this problem:

- Review past studies and pertinent literature for relevant information.
- Collect and analyze samples from military installations around the world.
- Conduct site visits to gather information regarding fuel-handling procedures and fuel storage/dispensing equipment.
- Based on all collected information, synthesize apple jelly in the laboratory.

### Results

The project team conducted 31 separate site visits to collect information and samples. Samples were also collected from sites other than those visited. SwRI received 139 samples of apple jelly, fuel, and other types of samples related to apple jelly contamination. Samples were analyzed for a number of compositional, physical, and chemical properties. The test results were analyzed to determine data relationships and information regarding the various properties of apple jelly. Based on these analyses, examples of both thin and thick apple jelly were synthesized in the laboratory. The synthetic apple jellies exhibited physical and chemical properties consistent with samples from the field.

### Conclusions and Recommendations

It is important to reiterate that we started this work with no constraints regarding what is and what is not apple jelly. We simply asked users in the field to submit samples of apple jelly whenever it was discovered. The samples we received varied in color, viscosity, and numerous other properties. We were told by some people that only thick apple jelly is really apple jelly, the rest is simply contaminated water bottoms. [We contend that all apple jelly is a form of contaminated water bottoms.] However, DESC instructed us to not limit our study because all the samples represented a potential problem for the user.

This work has demonstrated that apple jelly is a complex mixture. It begins with water and DiEGME. This mixture reacts with its environment, extracting and dissolving compounds from the materials with which it comes in contact.

Because apple jelly is such a complex and varied mixture, no one laboratory-synthesized apple jelly can represent all the possible compositions. Even a synthetic thin-apple jelly and a synthetic thick-apple jelly are not sufficient to represent all possible compositions.

In this work we started with apple jelly samples collected throughout the DoD/Air Force fuel-distribution system. The majority of our samples came from fuel systems delivering JP-8 to aircraft. There were differences in the fuels, although all the fuels were kerosene jet fuels. All the fuels contained corrosion inhibitor, FSII (fuel system icing inhibitor), and SDA (static dissipator additive) in varying amounts. The amount of water present in the various fuel systems differed greatly. All the fuel passed through filters, including particulate filters, filter/separators, and water-adsorbing filters. Other possible contaminants in the fuel systems included dirt, rust, paint, other fuels, elastomers, as well as myriad unknown contaminants.

Other than FSII, this work focused on only one JP-8 additive, SDA. We did so because there was evidence that SDA (or its components) accounts for some of the properties of the apple jelly samples we analyzed. That is not an indictment of SDA as a cause of apple jelly. Certain SDA components in the fuel simply become incorporated in the water/DiEGME as do other compounds in the fuel. In a system without SDA, some form of apple jelly could still form given the presence of water and DiEGME. This other apple jelly would have some properties similar to our samples but would also have some properties different from our samples. The other additives in the fuel may also make some contribution to the various properties of apple jelly. The extent of those contributions was not thoroughly investigated and would depend upon the other factors in the system. The same would be true if the water/DiEGME mixture formed in a diesel fuel system.

The work presented in this report explains the majority of the properties of the various apple jelly samples we received. We were able to demonstrate how thin and thick apple jelly, of the types we analyzed, could form. As mentioned above, all our samples came from somewhat similar fuel systems. Yet there are differences, and it is beyond the scope of this work to synthesize all possible combinations. As an example, our analyses showed sodium present in all the apple jelly samples. Possible sources include SDA, water-adsorbing filters, and also salt from the environment or even salt towers for drying fuel. We know that the majority of the sodium comes from the water-adsorbing filters. They are the only source sufficiently concentrated in sodium to provide the sodium levels observed in apple jelly, especially the thick apple jelly. The relative amounts contributed by any other source are impossible to ascertain. Still, some conclusions are possible. As an example, thin apple jelly tends to be lower in sodium. This probably means it has had less exposure to water-adsorbing filters. Hence, it also has little or no thickener so its viscosity is low.

The various properties of the apple jelly samples we analyzed are a result and a reflection of the various systems in which they were formed. It is a mistake to assume that there is only one type of apple jelly and that it is formed by only one means. However, the

mechanisms of formation put forth in this report do explain the formation of all apple jelly samples received during this investigation.

The only effective way to eliminate this problem contaminant (whether you call it apple jelly or contaminated water bottoms) is to eliminate either the DiEGME, or the water, or both. In fact, simply eliminating the water may not solve all problems since the DiEGME remains a potentially damaging compound if it is not properly mixed into the fuel.

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## TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
1 SCOPE OF WORK.....	1
2 OBJECTIVE.....	3
3 APPROACH .....	3
4 TEAM MEMBERS AND RESPONSIBILITIES .....	3
5 PREVIOUS STUDIES.....	3
6 DiEGME FUEL SYSTEM ICING INHIBITOR.....	8
6.1 FSII As A Biocide And/Or Biostat.....	9
7 SITE VISITS.....	9
7.1 Site Visit Summaries .....	11
7.1.1 DFSP Ludlow .....	12
7.1.2 Pease ANGB.....	15
7.1.3 Otis ANGB .....	15
7.1.4 Westover ARB.....	16
7.1.5 Bradley ANGB .....	17
7.1.6 Quonset Holland ANGB.....	18
7.1.7 NAS Brunswick.....	19
7.1.8 New Haven Jet Lines / DFSP New Haven .....	20
7.1.9 DPSP Portland .....	21
7.1.10 Bangor ANGB .....	23
7.1.11 DFSP Carteret.....	26
7.1.12 DFSP Jacksonville.....	27
7.1.13 McGuire AFB.....	28
7.1.14 DFSP Port Mahon.....	30
7.1.15 Dover AFB .....	31
7.1.16 DFSP Grand Forks.....	33
7.1.17 Grand Forks AFB .....	35
7.1.18 BP/AMOCO Mandan Refinery .....	37
7.1.19 Minot AFB.....	38
7.1.20 McConnell AFB .....	39
7.1.21 CONOCO Pipeline Company.....	41
7.1.22 Edwards AFB .....	42
7.1.23 Kinder Morgan Holding Facility .....	45
7.1.24 Beale AFB .....	46
7.1.25 Fairchild AFB.....	50
7.1.26 US Oil & Refining Company .....	52
7.1.27 McChord AFB .....	53
7.1.28 Hill AFB .....	55
7.1.29 NAS Corpus Christi.....	59
7.1.30 TEPPCO Bossier City & Barksdale AFB.....	60

## TABLE OF CONTENTS CONTINUED

<u>Section</u>	<u>Page</u>
7.1.31 Niagara Falls ARS .....	61
7.2 General Conclusions from the Site Visits.....	63
8 APPLE JELLY SAMPLE COLLECTION .....	66
9 LABORATORY ANALYSES.....	68
9.1 Compositional Analyses .....	68
9.1.1 Total Sulfur.....	69
9.1.2 Total Nitrogen.....	70
9.1.3 Total Sodium .....	70
9.1.4 Water and DiEGME .....	71
9.1.5 Chlorine .....	72
9.1.6 Ion Chromatography.....	73
9.2 Physical and Chemical Properties .....	76
9.2.1 Viscosity .....	76
9.2.2 Density.....	78
9.2.3 Total Acid Number .....	78
9.2.4 Total Base Number .....	78
9.2.5 Surface Tension .....	80
9.2.6 Conductivity .....	80
9.2.7 pH .....	82
9.3 Data Relationships .....	83
9.3.1 Total Sulfur.....	83
9.3.2 Total Nitrogen.....	86
9.3.3 Acid Number and Base Number.....	88
9.3.4 Total Sodium .....	90
9.3.5 Visual Color.....	92
9.3.6 Summary of Data Relationships .....	94
9.4 Thermogravimetric Analysis .....	94
9.5 Rheological Properties of Thick Apple Jelly.....	96
10 LABORATORY SYNTHESIS OF APPLE JELLY.....	100
10.1 Thin Apple Jelly .....	100
10.1.1 FT-IR Properties of Thin Apple Jelly .....	100
10.1.2 Minor Constituents of Thin Apple Jelly .....	102
10.1.3 Extraction Properties of SDA .....	104
10.1.4 Implications from Extraction Behavior of SDA .....	112
10.1.5 Effect of JP-8 Base Fuel .....	113
10.2 Thick Apple Jelly.....	123
10.2.1 FT-IR Properties of Thick Apple Jelly .....	123
10.2.2 Source of Sodium .....	127
10.2.3 FT-IR Properties of Water-adsorbing Filter Media .....	129

## TABLE OF CONTENTS CONTINUED

<u>Section</u>	<u>Page</u>
10.2.4 FT-IR Properties of Sodium Polyacrylate.....	134
10.2.5 Effect of SDA on Thick Apple Jelly.....	135
10.3 Effect of Water-adsorbing Filter Media on Thin Apple Jelly.....	141
11 SUMMARY AND RECOMMENDATIONS.....	142
11.1 Summary.....	142
11.2 Findings From The Field Investigations.....	145
11.3 Findings From the Laboratory Studies.....	147
11.4 Additional Findings.....	150
11.5 Recommendations.....	151
11.6 Possible Additional Work.....	152
12 ABBREVIATIONS AND ACRONYMS.....	153
13 REFERENCES.....	154
APPENDICES	
A. Sampling Instructions	
B. Listing of All Samples	
C. Photographs of First 69 Samples	
D. Results of Compositional Analyses	

## LIST OF TABLES

<u>Table</u>	<u>Page</u>
1. Chemical Synonyms for DiEGME.....	8
2. Partition Coefficients for DiEGME and EGME.....	9
3. List of Sites Visited.....	11
4. Initial List of Potential Sample Sites.....	66
5. Additional Apple Jelly Sample Sites.....	67
6. Total Chlorine Content by Inductively Coupled Plasma Spectrometry.....	72
7. Least-Squares, Linear Regression Correlation Coefficients ( $R^2$ ) of Selected Apple Jelly Properties.....	83
8. Extraction of SDA into DiEGME – Effect of Fuel.....	110
9. Composition of Thin Apple Jelly -- Effect of Fuel on 1:1 DiEGME/Water.....	114
10. Composition of Thin Apple Jelly -- Effect of DiEGME/Water Ratio.....	121
11. Composition of Thin Apple Jelly -- Effect of Fuel Composition.....	122
12. Brookfield Viscosities of Samples Shown in Figure 57.....	123
13. Sodium Content in Water Adsorption Filter.....	129
14. Analysis of Synthetic Thick Apple Jelly.....	136
15. Effect of Water-Adsorbent Filter Media on Thin Apple Jelly.....	141

## LIST OF FIGURES

<b><u>Figure</u></b>	<b><u>Page</u></b>
1. Supply Pattern for Selected Bases in the Northeastern United States .....	12
2. FAS Data From Beale AFB, 98L-0005 .....	49
3. FAS Data From Beale AFB, 98L-0024 .....	49
4. Clay Filters at Hill AFB.....	56
5. Additive Injection at Hill AFB .....	57
6. Total Sulfur Results .....	69
7. Total Nitrogen Results .....	70
8. Total Sodium Results.....	71
9. Water and DiEGME Concentrations .....	72
10. Ion Chromatography Results (All 16 Peaks).....	74
11. Ion Chromatography Results (SDA Peaks).....	75
12. Brookfield Viscosity Results .....	77
13. Sample Density Results .....	79
14. Total Acid Number Results .....	79
15. Total Base Number Results .....	80
16. Surface Tension Results .....	81
17. Conductivity of Apple Jelly Samples at Room Temperature .....	81
18. Conductivity of Water and DiEGME Mixtures at Room Temperature.....	82
19. pH of Selected Apple Jelly Samples.....	82
20. Total Sulfur vs. Total Sodium for Apple Jelly Samples.....	84
21. Comparison of Total Sulfur Levels of Apple Jelly and Fuel Layer Samples.....	85
22. Total Sulfur vs. Viscosity for Apple Jelly Samples.....	85
23. Total Nitrogen vs. Total Sodium for the Apple Jelly Samples.....	86
24. Comparison of Total Nitrogen Levels of Apple Jelly and Fuel Layer Samples.....	87
25. Total Nitrogen vs. Viscosity for Apple Jelly Samples.....	87
26. Viscosity vs. Total Acid Number for Apple Jelly Samples.....	88
27. Viscosity vs. Total Base Number for Apple Jelly Samples.....	88
28. Total Acid Number vs. Total Base Number for Apple Jelly Samples.....	88
29. Total Sodium vs. Viscosity for Apple Jelly Samples .....	90
30. Total Acid Number vs. Total Sodium for Apple Jelly Samples .....	91
31. Total Base Number vs. Total Sodium for Apple Jelly Samples .....	91
32. Visual Color Rating vs Total Sulfur for Apple Jelly Samples.....	93
33. Visual Color Rating vs. Total Nitrogen for Apple Jelly Samples .....	93
34. Visual Color Rating vs. Total Water for Apple Jelly Samples.....	94
35. TGA Scan of AP-002 -- Thin .....	95
36. TGA Scan of AP-032 -- Thick.....	95
37. TGA Residue vs. Viscosity for 11 Apple Jelly Samples .....	96
38. Viscosity vs. Shear Rate, AP-117 at 25°C.....	99
39. Viscosity vs. Shear Rate, AP-044 at 60°C.....	99
40. FT-IR Spectra of Water, DiEGME, and a 1:1 Blend of Each .....	100
41. FT-IR Spectra of Water/di-EGME and a Representative Thin Apple Jelly .....	101
42. Enlarged Region of Spectra Shown in Figure 41 .....	101
43. DiEGME Content Following Successive Extractions of SDA with DiEGME .....	105

## LIST OF FIGURES CONTINUED

<b><u>Figure</u></b>	<b><u>Page</u></b>
44. Total Acid and Base Numbers of DiEGME Following Successive Extractions of SDA .....	106
45. Total Nitrogen and Sulfur in DiEGME Following Successive Extractions of SDA .....	107
46. TGA Residue of DiEGME Following Successive Extractions of SDA .....	107
47. Total Water in DiEGME Following Successive Extractions of SDA .....	109
48. Total Sodium in DiEGME Following Successive Extractions of SDA .....	109
49. FT-IR Spectra of di-EGME/Water and JAW1-75-1 (1200 - 4000 $\text{cm}^{-1}$ ) .....	117
50. FT-IR Spectra of di-EGME/Water and JAW1-75-1 (2700 - 3058 $\text{cm}^{-1}$ ) .....	117
51. FT-IR Spectra of di-EGME/Water and JAW1-75-1 (1330 - 1420 $\text{cm}^{-1}$ ) .....	118
52. FT-IR Spectra of Thin Apple Jelly, di-EGME/Water, and JAW1-75-1 (1200 - 4000 $\text{cm}^{-1}$ ) .....	118
53. FT-IR Spectra of Thin Apple Jelly, di-EGME/Water, and JAW1-75-1 (2700 - 3215 $\text{cm}^{-1}$ ) .....	119
54. FT-IR Spectra of Thin Apple Jelly, di-EGME/Water, and JAW1-75-1 (1330 - 1420 $\text{cm}^{-1}$ ) .....	119
55. Effect of Heavy Fuel Components and SDA on 1:1 DiEGME/Water .....	122
56. FT-IR Spectra of Five Thick Apple Jelly Samples .....	123
57. FT-IR Spectra of Four Thick Apple Jelly Samples .....	124
58. FT-IR Spectra of Thin and Thick Apple Jelly Samples .....	125
59. FT-IR Spectra of Three Variably Viscous Apple Jelly Samples .....	126
60. Sodium Polyacrylate .....	128
61. FT-IR Spectra of Thick Apple Jelly and Dry Layer #2 .....	130
62. FT-IR Spectra of Wet and Dry Layer #2 .....	131
63. FT-IR Spectra of Original and Acidified Synthetic Apple Jelly JAW1-56-1 .....	133
64. FT-IR Spectrum of Sodium Polyacrylate .....	135
65. FT-IR Spectrum of Sodium Polyacrylate (1450 to 1760 $\text{cm}^{-1}$ ) .....	135
66. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly .....	138
67. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly (2800 to 3800 $\text{cm}^{-1}$ ) .....	139
68. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly (900 to 1800 $\text{cm}^{-1}$ ) .....	140



## 1 SCOPE OF WORK

Between 1980 and 1985, a representative of Imperial Oil made a presentation to Subcommittee J (aviation fuels) of ASTM Committee D2 concerning a contaminant found in the Alberta Products Pipeline (APPL). [1]\* The contaminant had a high viscosity and was eventually called “APPL jelly.” This is generally recognized as the first use of the term. Although no detailed analysis was conducted, the contaminant was believed to be associated with the ethylene glycol mono-methylether (EGME) icing inhibitor present in the fuel.

It is not clear whether the name eventually evolved into apple jelly or someone coined the name separately because of the appearance of the contaminant. However, since that time, the name has been applied to a range of contaminants found in aviation fuel delivery systems (primarily U.S. Air Force). At a November 2000 workshop on fuel system icing inhibitor [2] (FSII), the participants described apple jelly as “colored water bottoms from filter/separator sumps and tank low-point drains.”

In a recent Air Force report [3], apple jelly was described as follows:

“A moderately viscous material is appearing in military and commercial aviation fuel-distribution systems in the United States and other parts of the world. This anomaly is referred to as apple jelly due to its reddish brown color and other physical characteristics. As with any anomaly, occurrences that would go unnoticed in the past are reported with more frequency when the anomaly is given a name. This substance will disarm filter separators, causes premature change outs of filter elements and allows excess water to travel through the fuel-distribution system and possibly into an aircraft.”

Also, from that same Air Force report:

“... apple jelly has become a generic term in that not all samples of apple jelly look alike. The samples from the KC-135 and WPAFB are gelatinous. The

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\* Underscored numbers in brackets indicate references at the end of the document.

samples from Otis, Pease Filter Separator and Pease Composite visually resemble each other. The Norway and Grand Forks Storage Separator samples are visually similar, and the Grand Forks Curd sample does not visually resemble the other seven samples.”

The Air Force report [3] also refers to “documentation dating back to the 1950s addressing a material resembling apple jelly that was found in fuel controllers during flight tests of a new anti-icing additive.”

According to a Navy presentation [4] at the above-mentioned workshop, “a material commonly referred to as apple jelly has been a problem for many years and continues to be a problem.”

The above information helps demonstrate that the term apple jelly has been applied to many different contaminants. The differences include (but are not limited to) color, viscosity, and chemical composition. To some, apple jelly is thick and stringy, but the color is insignificant. To others, the viscosity does not matter, but it must be darkly colored. All seem to agree that apple jelly contains water and FSII and that the FSII can be either EGME or DiEGME (diethylene glycol monomethyl ether).

For the purposes of the study presented herein, Southwest Research Institute<sup>TM</sup> (SwRI) was instructed by the Defense Energy Support Center (DESC) to investigate apple jelly. No other restriction was placed on the scope because DESC wanted to address the apple jelly problem as currently encountered by personnel in the field. In our requests for samples from the field, no restrictions or descriptions were given to field personnel. We asked only that if personnel encountered apple jelly, they send a sample to us for analysis. It is important to remember this as you read the results of this work. SwRI was tasked to address the current apple jelly problem affecting the U.S. military. While we reviewed past work, it was not within the scope of this work to determine the nature of past apple jelly contaminants. Neither do we claim that our findings describe all contaminants that have ever been called apple jelly.



## **2 OBJECTIVE**

The objective of this project was to characterize the aviation fuel contaminant known as apple jelly with respect to the compositional and process conditions required for its formation, and to determine potential methods, both compositional and process, to reduce or prevent its formation.

## **3 APPROACH**

The following tasks were involved in the investigation of this problem:

- Review past studies and pertinent literature for relevant information.
- Collect and analyze samples from military installations around the world.
- Conduct site visits to gather information regarding fuel-handling procedures and fuel storage/dispensing equipment.
- Based on all collected information, synthesize apple jelly in the laboratory.

## **4 TEAM MEMBERS AND RESPONSIBILITIES**

SwRI assembled a team to conduct this investigation. The sub-contractors to SwRI were Consulting for Energy Efficiency and Environmental Excellence (C4e) and Calvin Martin of Martin & Associates. SwRI provided project leadership, laboratory analyses, and chemistry expertise. C4e provided expertise on Air Force fuel-handling procedures, relevant past experience, and contacts at Air Force installations. Mr. Martin provided expertise on DESC procedures and assisted with gathering information from DESC databases.

## **5 PREVIOUS STUDIES**

A study by the Naval Research Laboratory (NRL) [5] specifically addressed receipt filter/coalescers clogging during operations with either JP-8 or JP-5 that contain FSII additive. The clogging gel most often occurred when the filter/coalescer water

bottoms were not continually drained. The water bottoms had become saturated with FSII, making a solution that was close to 1:1 FSII and water. This changed the water to essentially a “very polar organic material,” which NRL found was capable of extracting polar constituents from the fuel with great efficiency. Thus, the filter/coalescer bottoms contained a very high concentration of polar fuel components.

The NRL study used ASTM D6426, “Standard Test Method for Determining Filterability of Distillate Fuel Oils” to test fuel samples and evaluate the possible formation of gel in the laboratory. DESC provided fuel samples from locations with recent problems. Representative samples of FSII were also obtained from different locations. Several properties of the FSII were investigated, including its solubility performance, purity, injection and mixing properties, and effect on filterability after injection.

The NRL report concluded that FSII alone does not cause the problem; however, its presence appears to be a necessary condition for the problem to occur. The frequency of reported problems was higher for JP-8 than for JP-5. NRL theorized that the formation of the brown gel is highly dependent on the content of small metal cations, such as sodium, in the fuel. These cations may react with other species in the water/DiEGME layer to form colloids.

In November 2000, the Air Force Research Laboratory (AFRL) published a report entitled, “Evaluation Report On Fuel Distribution Field Problem ‘Apple Jelly’ ” [3] and a paper entitled, “C-17 Fuel Sump Samples.” [6] The report discussed several potential causes of apple jelly and the analysis of eight apple jelly samples. Gas chromatographic analysis of six of the eight samples showed a range of DiEGME from 17 vol% to 60 vol%. The remainder of each sample was determined to be mostly water. The samples were also analyzed for elemental composition by inductively coupled plasma spectroscopy, total acid number (TAN), and pH. The highest TAN reported was 0.47. The results showed an unusually large concentration of sodium (as high as 1100 ppm), especially in the more viscous samples. Actual viscosity data were not given, however. Using X-ray

photoelectron spectroscopy (XPS) to analyze all eight samples, AFRL found that the sodium levels were not consistent with contamination from seawater or other salt sources.

After testing an unused water absorption cartridge using XPS, the middle layer of three layers of matting was found to contain “sodium agar... commonly used as the swelling agent in absorbent type cartridges.” [3] \* No conclusions were reached as to whether this was the source of the sodium; however, when a 60/40 DiEGME/water mixture was introduced, the matting swelled and seemed to decompose with the addition of DiEGME.

The AFRL report included the following recommendations:

- minimize water in the fuel-distribution system,
- add additional and better filtration to the fuel-distribution systems,
- determine the source of sodium found in the contaminated samples,
- add an antioxidant to DiEGME that is stored for extended periods of time or stored at elevated temperatures, and
- conduct research to determine a comprehensive phase diagram for FSII in fuel to determine the source and role of sodium in the formation of apple jelly.

In the C-17 paper, AFRL reported on an investigation of apple jelly in five C-17 aircraft that had vented fuel from one or both vent boxes either in flight or during ground fueling. All samples analyzed appeared to be the same apple jelly type material found in ground handling systems, and formation of apple jelly on the aircraft could not be ruled out.

The Canadian Association of Geophysical Contractors published a safety bulletin entitled “Jet Fuel (with Anti-Icing Additive) and Visible Water.” [7] According to the safety bulletin, a “large amount” of water passed through a fuel filter and eventually caused deceleration of a helicopter engine. The fuel (and water) in question had passed

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\* It is the author's understanding that sodium agar is no longer used in water absorbing filters. Current filters use polyacrylates as the water-adsorbing medium.

through a water-adsorbing filter element. Examination of the filter by the manufacturer did not indicate water passage, nor did the filter allow such passage in an actual water test. Further study of the problem resulted in the following findings (among others):

- a mixture of approximately 15% anti-icing additive in water will not be identified as water by some water sensors because of the density of the mixture
- a 1:1 mixture of anti-icing additive and water may allow as much as 10% water to pass through water-adsorbing filters
- standard, commercially available water-finding paste will not change color in a 1:1 mixture of anti-icing additive and water
- under certain conditions, the water-adsorbent medium in some older, water-adsorbing fuel filter elements can deteriorate; under these conditions, as the medium continues to adsorb water, it eventually reaches a point where it is extruded through the migration barrier and continues downstream

Hook and Danek [8] found that the glass transition temperature ( $T_g$ ) of a barrier coating in the wing tanks of F-111 aircraft was significantly depressed on exposure to water/DiEGME solutions. The drop in  $T_g$  was about 15°C with 10% DiEGME, about 24°C with 50% DiEGME, and about 72°C with pure DiEGME. One of the authors is also collecting reports of fuel tank failures resulting from exposure of sealants or fuel bladders to DiEGME, as a result of overdosing fuel with DiEGME or failure to properly mix the DiEGME with the fuel.

A report by the Australian Department of Defence [9] presented the results of several studies of various aspects of DiEGME and its role in aircraft degradation (most notably in the F-111). According to this report, the

“detrimental effects of DGME [DiGME] include (i) acceleration of corrosion of the D6AC steel used in critical areas of the F-111 aircraft including the wing pivot fittings and wing carry-thru-box, (ii) swelling of the aircraft’s sealants, (iii) softening of the epoxy barrier coating employed under the sealants, and (iv) delamination of paints in aircraft fuel tanks. The most serious long term effect of DGME [DiGME] on the F-111 aircraft has

been to increase F-111 aircraft fuel leaks, which are currently the aircraft's major operational limitation, to the extent that they are debilitating fleet operations.”

The following summarize some of what was known about apple jelly prior to this study:

- It is composed primarily of DiEGME and water, which account for about 98% of the mixture.
- The relative amounts of DiEGME and water vary. The typical ratio is about 60:40 (DiEGME:water).
- It contains sodium, and organic compounds.
- It is usually brown or reddish brown in color.
- It is variably viscous, from a viscosity approximately that of water to a very thick gel. As a comment to this information, it is noteworthy that a truly viscous gel would have a viscosity well beyond that of any blend of just water and DiEGME.
- Reports of apple jelly were most frequent during colder temperatures, but some reports were received during warm temperature periods.
- Mixtures of water and DiEGME are both corrosive and an aggressive solvent. The severity of corrosion and solvent attack depends on the concentrations of each component and the presence of other compounds (such as aromatic compounds) in the mixture.

From these facts the following can be stated:

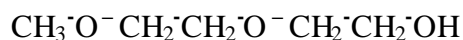
- Apple jelly is not composed solely of DiEGME and water, but contains other components.
- The rheology of apple jelly is not determined only by the DiEGME and water components, assuming the reports of gelatinous apple jellies were literally accurate.
- Not all apple jelly is the same; samples vary in both composition and rheology.
- Sodium appears to be present in apple jelly samples and some claims concerning a correlation between sodium content and viscosity have been made, although supporting data is almost non-existent. No clear position on the presence of other specific cations could be found.

## 6 DiEGME FUEL SYSTEM ICING INHIBITOR

The FSII currently used throughout the U.S. military is DiEGME. There are at least 20 synonyms [10] for DiEGME as presented in Table 1.

2-(2-methoxyethoxy)ethanol	methyl carbitol	poly-solv dm
diethylene glycol monomethyl ether	diethylene glycol methyl ether	diglycol monomethyl ether
ethylene diglycol monomethyl ether	beta-methoxy-beta'-hydroxydiethyl ether	2,2'-oxybisethanol monomethylether
Methoxydiglycol	dowanol dm	methyl dioxitol mecb
Methyl digol	3,6-dioxa-1-heptanol	ektasolve dm
Hicotol car	methoxyethoxyethanol	2-beta-methyl carbitol
Glycol ether dm	3,6-dioxaheptan-1-ol	

The molecular formula for DiEGME is:



For pure DiEGME, the flash point is 93.3°C (200°F), the boiling point is 193°C, and the molecular weight is 120.15. [11]

FSII is added to aviation fuels to lower the freezing point of free water present in the fuel system, thereby reducing the chance of ice crystal formation. FSII in the water can lower the freezing point to as low as -57°F. The typical treatment rate in fuel is 0.10 to 0.15 vol%. FSII functions by partitioning from the fuel into the water. The propensity of the FSII to partition into the water is known as the partition coefficient, which is measured as:

$$\text{Partition Coefficient} = \frac{V_w}{V_f}$$

Where:

$V_w$  = vol% FSII in water phase, at equilibrium

$V_f$  = vol% FSII in fuel phase, at equilibrium

The partition coefficients for EGME and DiEGME in a typical JP-4 and a typical JP-5 [12] are given in Table 2. These figures reveal that DiEGME has a much greater affinity for water than does EGME. It is also interesting to note that the partition coefficient for DiEGME is higher in JP-4 as compared to JP-5.

Table 2. Partition Coefficients for DiEGME and EGME		
Additive	Partition Coefficient	
	JP-5	JP-4
DiEGME	538	657
EGME	265	260

## 6.1 FSII As A Biocide And/Or Biostat

While FSII is added to aviation fuels primarily to inhibit ice formation, some control of microbial growth has historically been a widely accepted side effect of the presence of FSII in the water phase. Two terms must be defined as part of this discussion:

*Biocide* – the additive completely sterilizes the system to which it is added; there are no surviving organisms.

*Biostat* – when added to the system, the additive works to inhibit any additional growth but will not necessarily kill existing organisms.

The reports of FSII effectiveness as an anti-microbial agent are mixed. Krizovensky reported data that showed 10 to 17 vol% DiEGME in water provided both biocide and biostat properties. [10] Neihof reported that EGME (and to a similar degree DiEGME) [13] is not strictly a biocide. [14] Like Krizovensky, he maintained that the concentration of the additive in the water must reach about 15% to have any effectiveness. Neihof also postulated that the “anti-microbial action is likely to be due simply to making the osmotic concentration of solute in the water phase too high to permit growth.” Hill found that low concentrations of FSII may in fact stimulate growth. [15] Sonntag reported evidence of microbial growth in the presence of fuel with apple jelly. [16]

## 7 SITE VISITS

SwRI subcontracted C4e to conduct site visits as part of this investigation. The complete report of all site visits is available as a separate document. [17] A summary of the findings is presented herein.

C4e teamed with SwRI to review technical orders, conduct site visits, and collect field data to identify facility, equipment, and process factors that may contribute to the formation of apple jelly. Site visits were conducted at more than 30 affected bases and terminals in an attempt to collect information and samples. Prior to the field visits, the C4e teams reviewed available information concerning apple jelly-like contamination in turbine fuels. Additionally, the C4e investigators identified some changes in Air Force fuel and fuel-handling practices that have taken place in recent years. During their site visits, team members were asked to be mindful of these changes and how they might contribute to the formation of apple jelly. These changes include:

- The conversion from EGME to DiEGME.
- The conversion from JP-4 to JP-8 and JP-8+100.
- The Air Force adoption of the cone-down, center-sump, self-cleaning-design, bulk storage tank.
- Use of the API 1581 filter element in place of the DoD (MIL-F-8901) filter separator element.
- Installation of water absorption media cartridges on R-11 fueling vehicles to accommodate JP-8+100.
- Reduced inventory levels and settling time.

During the site visits, the C4e teams identified the following as additional items for investigation during site visits:

- Practices to minimize the presence of water in fuel and fuel systems.
- The type and condition of bottom sediment and water (BS&W), product-recovery equipment, and the process.
- Additive injection locations and procedures.
- The impact of temperature change on the formation of apple jelly.

Table 3 contains a list of all the sites visited. The majority of these sites were visited because they reported finding apple jelly within the previous 24 months (1999-2000). Teams were dispatched to identify the specific location (truck sump, hydrant filter



separator, etc.) and work backward through the logistics support system to document variables (materials, designs, equipment, maintenance, and operations) that may contribute to the formation of apple jelly. However, some of the sites were visited because they had not found apple jelly in their systems. At these sites the visit teams were instructed to look for procedures/processes that might explain why no apple jelly had been found. Although apple jelly has been reported at a few overseas locations, site visits were restricted to the Continental United States.

<b>Table 3. List of Sites Visited</b>		
Barksdale AFB	Bangor ANGB	NAS Brunswick
Westover ARB	Pease ANGB	NAS Corpus Christi
McGuire AFB	Quonset Holland ANGB	BP/AMOCO Mandan Refinery
Hill AFB	Otis ANGB	Kinder Morgan Holding Facility
Grand Forks AFB	Bradley ANGB	TEPPCO Bossier City
McChord AFB	Niagara Falls AFRES & ANG	CONOCO Pipe Line Company
Edwards AFB	DFSP Carteret	US Oil & Refining Co
Beale AFB	DFSP Jacksonville	New Haven Jet Lines / DFSP New Haven
Minot AFB	DFSP Portland	
McConnell AFB	DSFP Grand Forks	
Dover AFB	DFSP Port Mahon	
Fairchild AFB	DFSP Ludlow	

## 7.1 Site Visit Summaries

Rainwater entry into on-base tanks is a common factor in apple jelly formation at many of the locations. This problem is compounded by ineffective product-recovery systems (sump drains) and the self-cleaning tank design that suctions bottom sediment and water into the tank issue line. Also, the use of filter separators that were not designed to handle fuel with additives, and the continued use of defective F/S vessels built to the old DoD standard, contribute to excessive water and apple jelly contamination. This is best illustrated by a discussion of processes and facilities at terminals and the supported bases with identified problems. Within the Northeastern United States, DFSP Ludlow supplies fuel to a number of installations that reported apple jelly problems. Conversely, other Northeastern DFSPs within the same logistics support system supply to sites with no reports of apple jelly. These DFSPs include Portland, ME, New Haven, CT, and Carteret, NJ. Figure 1 shows the fuel-distribution pattern for bases in the Northeast.

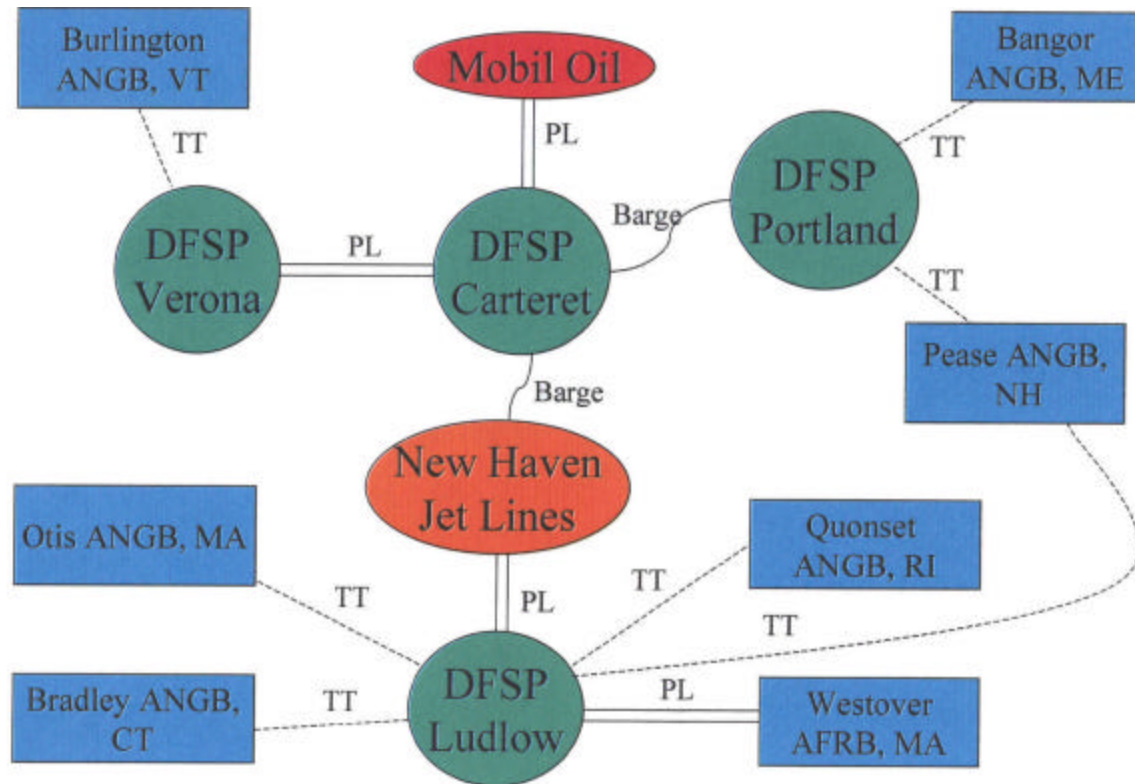


Figure 1. Supply Pattern for Selected Bases in the Northeastern United States

### 7.1.1 DFSP Ludlow

DFSP Ludlow receives fully additized JP-8 via pipeline shipments from DFSP New Haven, which receives its fuel via barge from DFSP Carteret. From DFSP New Haven, the fully additized JP-8 is moved through a commercial multi-product pipeline that also transports #2 fuel oil, gasoline, kerosene, and Jet A fuel. Pipeline time from New Haven is approximately 12 to 13 hours; the fuel moves at a rate of 2,000 bbls per hour. Pipeline drag reducer (PDR) is used in the pipeline that supplies Ludlow, but it is not used in JP-8 shipments. The use of PDR normally starts in December to meet the increased demand for heating oils. PDR is injected at New Haven but not within two hours before or after a JP-8 or other aviation fuel shipment. When asked about the controls to insure against the accidental injection of PDR, a pipeline company representative reported that there had been one such incident, but company personnel identified the error and cut the contaminated JP-8 product into a diesel tank.

DFSP Ludlow stores only JP-8, which is not filtered on receipt. DFSP Ludlow has two dedicated storage tanks, each with a 55,000-bbl capacity; however, the safe fill level is restricted to 41,000 bbls because of the small capacity of the tank dikes. The low product level results in increased surface area for water accumulations that leak past the floating-roof seals and into the JP-8 stored in the tanks. This storage system was originally built by the Air Force to support Westover ARB and was later sold to a commercial pipeline company. The steel, above-ground, floating-roof tanks have flat bottoms and a fixed discharge (suction line) that is approximately 1 foot above the tank bottom. The bottom of Tank #2 is epoxy coated, and Tank #1 is scheduled for cleaning, epoxy coating and installation of a cone roof. The terminal manager reported a 1-inch water bottom is always maintained in both tanks. Approximately 5,000 gallons of water bottoms are removed from the two tanks 3 to 5 times a year. The most water drained from the two tanks at any one time totaled 5,300 gallons. A vacuum truck is used to draw the water bottoms from the tank sumps. According to the terminal manager, the procedure involves draining the tank bottoms until the “black gunky stuff ends, and the product becomes milky.” The water bottoms are typically dark amber. The all-level sample prior to receipt on the day of the visit (February 22, 2001) was clear and bright; however, the post-receipt sample was cloudy.

Though static dissipator additive (SDA) and FSII are injected at DFSP Carteret, DFSP Ludlow must re-inject the additives because exposure of the JP-8 to excessive water leaches out the FSII. SDA is added as necessary to increase the conductivity level as the product is pumped into the tanks. SDA is diluted with JP-8 in a ratio of 1 to 9 and is stored in a 50-gallon tank prior to blending into the bulk fuel. Ludlow personnel attempt to achieve a fuel conductivity of 200 pico-Siemens per meter (pS/m) when adding the SDA. When bases request an increase in additive levels, the additional additive is often added directly into the delivery trucks through one of the domes on the top of the tank. FSII is injected in the issue line upstream from the pump by use of a Hammond’s turbine injector. Ludlow personnel attempt to achieve an FSII level of 0.12 vol%. The FSII is stored in a steel tank that is not equipped with a drier. At the time of the visit, Ludlow personnel were in the process of refilling the FSII tank, which was last filled during April

of 2000. Drums of FSII are stored outside, standing upright rather than horizontally. The tops of the drums were covered with water from recent rains. Additional FSII is rarely injected at DFSP Ludlow during the cold winter months, because of the minimal exposure to rainwater intrusion during periods of freezing temperatures.

An American Bowser F/S vessel is installed in the issue line; however, this vessel has been modified to use micronic filters only. Velcon 2-micron filter elements (F0629PLF1) are used (12 elements per bank of filters) on the issue line for trucks and the pipeline. The coalescer elements from this vessel may have been eliminated to reduce the cost of frequent replacement given the amount of free-water/FSII mixtures in the tanks. The truck fill rate is 900 gpm for a single truck and 1,000 gpm for two trucks. They pump JP-8 to Westover ANGB by pipeline at a rate of 15,000 bbl/hr.

Team members climbed both bulk tanks. Snow and ice had accumulated on the top of both floating-roofs. Removal of the snow and ice is hampered by the safe fill capacity that is restricted to 75% of the tank capacity—the top tank shell is too high to throw the snow over. The floating roof on one tank appeared to list at approximately 5 degrees because of the uneven distribution of the snow and ice accumulation, which is an unsafe condition. The limited fill capacity and the tilt of the roof, which interferes with the floating-roof seal, facilitate rainwater entry into the tank. Because of the tilting position of the roof, team members did not climb down onto the roof to examine the seal. The terminal manager/operator (the only person assigned to the terminal) was on this roof attempting to shovel the snow/ice to the center of the roof to balance the weight.

DFSP Ludlow routinely supplies six installations that were included in the onsite apple jelly investigations. These are Pease ANGB, Otis ANGB, Westover ARB, Bradley ANGB, Quonset Holland ANGB, and NAS Brunswick. All of these locations, with the exception of Quonset Holland ANGB and NAS Brunswick, report periodically draining apple jelly-like material from their receipt F/S sumps and, less frequently, from issue F/S sumps.

### **7.1.2 Pease ANGB**

JP-8 is delivered to Pease AFB by tank trucks from DFSP Ludlow and DFSP Portland. Pease personnel reported that the JP-8 from DFSP Portland is always clear and bright, while JP-8 received from DFSP Ludlow is often cloudy. Some of the deliveries from DFSP Ludlow are so cloudy that one cannot see the bottom of the delivering tank truck. Pease personnel also reported that they “never” have a problem when receiving tank trucks from DFSP Portland. After receiving from DFSP Ludlow, dark contaminants are drained from receipt vessels, generally starting in late October and early November when fuel temperatures drop to 40°F or below. The contaminants become lighter in color as the winter progresses. Receipt filter vessels are inside a heated pump house, and the elements are usually changed at approximately 11-month intervals because of excessive differential pressure. Issue filters are changed every three years, and they report they have never drained apple jelly-like materials from the pump-house issue F/S vessels. All fixed facility F/S vessels are within a heated pump house. The receipt temperature during January ranges from a high of 33°F to a low of 28°F, with an average of 30°F. During August, the temperatures range from a high of 85° F to a low of 65° F, with an average of 65°F to 70°F. The JP-8 is stored in two fixed-roof tanks with floating pans. One has a 12,500-bbl capacity, and the other has a 10,500-bbl capacity. The tanks are a self-cleaning design with the bottom sloped to the center and a suction line over the sump. The water and sediment drain line is a 3-inch line that empties into a 4,000-gallon product recovery tank. Reclaimed product is pumped through the F/S back into the bulk tank. Pease AFB has three R-11 refuelers, one Oshkosh and two Kovatch. They also have three HSV-12 hydrant-servicing vehicles. Pease also has an aggressive sump-draining program and a newer-designed product-recovery system.

### **7.1.3 Otis ANGB**

Apple jelly is routinely encountered at Otis ANGB from October through April and is primarily found in the receipt F/S sumps. Otis personnel have been successful at containing apple jelly to receipt and pump house issue vessels since March of 1999.

During March of 1999, they had to change all of the elements in their refuelers and the receipt and issue vessels after being overwhelmed by apple jelly. They were routinely draining apple jelly from their receipt F/S vessels until January 2001, when they changed their receipt filtration mode due to a major tank-construction project. Product is delivered to Otis ANGB from DFSP Ludlow. Otis ANGB has two 600-gpm horizontal F/S units and two 600-gpm horizontal issue F/S vessels. The heated pump house, which houses the receipt and issue F/S vessels, was recently taken out of service due to major tank construction. An R-14 Air Transportable Hydrant Refueling System (ATHRS) F/S unit was placed in service outside the pump house for the fuel receipt and issue filtration. No further incidences of apple jelly have been experienced since placing the R-14 in service. Under normal operating conditions, a Hammond Injector Model 800 injects JP-8+100 into a 4-inch truck fill stand pipeline downstream from the pump house. During the tank construction project, injection of the +100 additive has been suspended. Otis ANGB had Facet M-100 elements on hand at the time of our visit, which they planned to install in their receipt and issue F/S vessels upon completion of the construction project. The Facet M-100 elements are API 1581, 4<sup>th</sup> Edition elements that were designed specifically for use with JP-8+100. The Otis bulk storage system consists of two 40-year old steel ASTs. Tank #15 has a capacity of 19,000 bbls. It has a geodesic dome that leaked badly until it was recently repaired. Tank #16 has a fixed roof and a capacity of 1,300 bbls.

The product recovery systems on the operating tanks do an inadequate job of removing water from the tanks. A major construction project is scheduled to replace the existing system. Otis ANGB has five Oshkosh and two Kovatch R-11s equipped with Facet water-absorption elements. The water-absorption elements in the refuelers seem to be holding up well since the fleet-wide change following the contamination incident in March 1999.

#### **7.1.4 Westover ARB**

Westover occasionally drained apple jelly-like material from the receipt filter, prior to the construction of the two 20,000-bbl bulk receipt tanks. The apple-jelly

contamination occurred between late fall and early December. Following the completion of the two bulk receipt tanks, Westover discontinued the use of receipt filters and has not experienced further apple jelly occurrences. The same filter separators previously used for the receipts are now used to filter the JP-8 transferred to operating storage. The new receipt tanks are generally allowed to settle at least 24 hours. The sumps are drained prior to transfer to the operating storage system, through piping directly connected to a new product-recovery system. Westover personnel reported finding apple jelly in the receipt filters prior to Desert Storm and afterwards, but not during the period of heavy product movement in support of Desert Storm activities. Since Desert Storm, they have reported finding apple jelly on four occasions.

Westover receives JP-8 via pipeline from DFSP Ludlow. The pipeline from Ludlow to Westover is an 8-inch line with a static line fill of 29,600 gallons. Product receipts range in temperature from 32°F to 35°F during January and from 60°F to 66°F during August. Westover has three R-11s (one Oshkosh and two Kovatch) four HSV-12s, and two MH2C (Type II) hose carts. The R-11 refueling trucks were equipped with standard coalescer elements at the time of the site investigation (February 2001), though water-absorption cartridges were on hand for installation during the next filter change.

The Westover product recovery system was one of the better systems observed during the onsite investigations. The two new bulk tanks have geodesic domes that were well engineered and built. Reclaimed JP-8 is pumped back into the bulk storage through a filter/separator.

#### **7.1.5 Bradley ANGB**

DFSP Ludlow supplies JP-8 by tank truck to Bradley ANGB. Bradley encountered apple jelly last winter in their pump house receipt and issue F/S vessels. The pump house is equipped with a heater normally operated with the thermostat set around 50°F. Heat is required in the pump house to prevent a water main and an emergency eyewash station from freezing. This fall, the heater failed and was not repaired until mid-

December. When the heater was out of service, a portable Herman Nelson H1 heater was used in the pump house for heat. With the heater out of service, there has been no reappearance of the apple jelly. There is some speculation that a reduction in the pump house temperature might have played a role in the disappearance of apple jelly.

JP-8 is shipped from the Ludlow DFSP in dedicated tank trucks with a transient time of 45 minutes. During the winter, product temperatures upon receipt range from 20°F to 30°F. Bradley has two 300-gpm vertical Quantek vessels with Velcon coalescers for receipts and two 600-gpm vertical Quantek vessels with Velcon coalescers for issues.

Bradley ANGB has two, steel, fixed-roof ASTs that were constructed in 1993. This relatively new system has some design features that are helpful in fuel handling, such as the product-recovery, water-drain system on the operating tanks. This system consists of one underground collection tank, with a 2,000-gallon capacity. The product recovery tank is connected to both bulk storage tanks and is used to receive water and cloudy fuel from the tank sumps via a 3-inch drain line. After separation, the fuel is pumped back through the receipt F/S to the operating tanks. The product recovery tank is gauged for water using a plumb bob, tape, and water finding paste. They have four Oshkosh R-11s with Facet absorption media elements installed during the fall of 2000. Again, the primary apple-jelly problems occurred during filtration of receipts from DFSP Ludlow.

#### ***7.1.6 Quonset Holland ANGB***

Quonset Holland ANGB receives JP-8 from DFSP Ludlow and periodically finds foreign matter in the receipt F/S sumps. The nature of the foreign matter is unknown. The average driving time for the delivery tank truck is 2¼ hours. FSII levels currently average 0.115% at the time of receipt, with a fuel-conductivity range from 184 pS/m to 244 pS/m and an average of 210 pS/m.

Quonset Holland ANGB has two vertical F/S vessels with coalescers for fuel receipts and a vertical vessel with coalescers for issues. There are two 2,500-bbl above-



ground steel tanks with fixed roofs and floating pans. The tanks are 100% epoxy coated. The two product-recovery systems are inadequate due to poor design. Operating personnel must drain water into buckets and carry it to a product container for settling. Product is recovered after water separation. Quonset Holland ANGB has three Oshkosh R11s with Facet absorption media elements. A Hammonds Model 800 injector is normally used to inject the JP-8+100 additive into a 3-inch pipeline downstream of the issue filters.

The Fuels Superintendent identified the material drained from the receipt and issue F/S vessels as normal BS&W and not apple jelly. Therefore, Quonset Holland ANGB has not reported apple-jelly findings. This is worth noting since DSFP Ludlow supplies JP-8 to Quonset Holland ANGB and was a possible source of apple jelly found in receipt and issue F/S vessels at a number of installation in the Northeast. Since the definition of apple jelly has always been open to interpretation, similar materials drained at other locations may have been reported as apple jelly at those locations.

#### **7.1.7 NAS Brunswick**

DFSP Ludlow delivers JP-8 via tank truck to NAS Brunswick. The fuel is received through two 600-gpm F/S vessels manufactured by M.E. Industries. The vessel model number is VFCS-C-6N38SB-3630T2N. Each of these vessels contains six coalescer elements, P/N CC-N38SB, and three separator screens, PN CS639T2N. After the fuel passes through one of the receipt F/S vessels, it passes through an M.E. Industries “Fuel Quality Monitor,” model # M2030V600 containing water-absorption cartridges. The receipt F/S vessels drain automatically to a product-recovery tank, so the operators do not see what is drained from the receipt filter sumps.

The fuel storage tanks have fixed roofs and cone bottoms that slope to center sumps. They have two each issue F/S vessels identical to the receipt vessels described above, with the exception that the sumps drain automatically. The issue F/S sumps are directly connected to a product-recovery system via a ¾-inch pipe. The only visual observation of materials drained from the sump is through a site glass installed in the

sump drain line. After the fuel passes through the receipt F/S vessel it goes through a “Fuel Quality Monitor” as described above.

NAS Brunswick personnel report that during the winter of 1998-1999 they did drain some apple jelly-like material from their F/S sumps. At that time, the FSII content of the fuel was running from 0.18% to 0.22%. After the FSII content was reduced to approximately 0.12%, they no longer noticed any of the thick dark liquid.

The NAS Brunswick fuel-handling process differs from that of the Air Force/ANG bases supported by DFSP Ludlow in two ways: First, NAS Brunswick uses a three-stage filtration process (coalescence, separation, and absorption) at each filtration step. The Air Force uses either coalescence/separation (in fixed facilities and hydrant servicing equipment) or a single absorption phase on R-11 refueling trucks. Second, the receipt filters automatically drain to a product-recovery system with no manual drain to allow the operator to examine materials drained from the sumps. Air Force facilities supplied by Ludlow have installed manual drains that permit close visual examination

#### ***7.1.8 New Haven Jet Lines / DFSP New Haven***

DFSP Ludlow receives its JP-8 via pipeline from DFSP New Haven. In turn, DFSP New Haven receives fully additized JP-8 from the Carteret DFSP by dedicated jet fuel barge once every three to four weeks. Transit time via barge is approximately 8 hours. The New Haven receipt tank is sampled following each receipt. An all-level sample is taken, in addition to the barge retain sample, for laboratory B-1 level testing. The fuel is not filtered at the barge off-load terminal, nor is the fuel filtered during receipt or issue from the tank.

The fuel is transferred through a dedicated line from the tank to a commercial multi-product pipeline for shipment to DFSP Ludlow. The multi-product pipeline from New Haven to Ludlow is approximately 90 miles long, and the average pumping rate is

3.3 miles per hour. The JP-8 is buffered in the multi-product pipeline with Jet A fuel. The JP-8 is sampled at the start, in the middle, and 1 hour before the transfer is complete.

The one 60,000-bbl Motiva above-ground steel tank with a geodesic dome is located in the New Haven commercial-terminal complex. The 60,000-bbl tank is equipped with two sumps and 1½-inch water drain valves. The discharge line is offset from the tank sumps by approximately 40 feet. The single, common receiving and discharge line is equipped with a “piccolo tube” diffuser and extends 30 feet from the tank wall toward the center and descends to within 21 to 25 inches from the bottom. All diffuser orifices are located on the top of the diffuser. (This minimizes turbulence in the tank and reduces the chance of BS&W contamination of the issued product.)

#### ***7.1.9 DFSP Portland***

Like DFSP Ludlow, DFSP Portland also supports Pease AFB. Since Pease AFB reported that they drain apple jelly from receipt F/S vessels when receiving deliveries from Ludlow, but not when receiving from Portland, comparison of the two DFSP provides an informative contrast. DFSP Portland receives JP-8 via spot charter barges where the fuel is transported in uncoated mild steel tanks. Most JP-8 has historically been received from the Sun Oil Marcus Hook Refinery, though some has come from DFSP Carteret (primarily refined at Mobil Oil Houston). JP-8 is received through a dedicated line from the pier into two above-ground, fixed-roof bulk tanks. One tank has a 55,000-bbl capacity and the other a 90,000-bbl capacity. Within the past three years, mild steel secondary-tank bottoms have been installed in the tanks. The new tank bottoms are 10 inches above the old bottom at the tank center and slope to 6 inches at the outer edge. The fill and discharge lines are separate. Tank #4 is equipped with a floating suction line that draws fuel 14 inches above the tank bottom at the lowest point. The other JP-8 tank, Tank #5, has a discharge suction line that extends approximately 8 to 10 feet into the tank and draws fuel at a point approximately 24 inches above the tank bottom. The entry into this suction line differs from the Air Force tank design in that the elbow curves toward the top of the tank rather than the bottom. The suction line is baffled at the end, widening the

vortex created by suction. This design minimizes turbulence and the potential of drawing bottom sediment and water into the suction line. The water sump is some forty feet away from the discharge line. Water is removed from the tanks twice each year through a 1½-inch drain line. While the tanks have two drain lines, only one drain line is used per tank because tank settling has resulted in a slight pitch of the tank floor. On average, the product turns over every 30 days. Significant cooling occurs during transfer of the product to the Portland DFSP. For example, this past August product was loaded at 95°F and had cooled on the Barge to 56°F just prior to discharge. The temperature of product in storage during August ranges from 60°F to 70°F. During January, the product temperature reached a low of 10°F, but averaged approximately 29°F with a high temperature of 33°F. When issued, the product is pumped through two separate vessels, one for micron filtration and the other for coalescence. The maximum flow rate through the vessels is 1,100 gpm. FSII content of the product is fairly consistent at 0.13 vol%, while the product conductivity ranges from 150 pS/m in the winter to 350 pS/m during the summer months. The Portland DFSP has a 6,000-gallon stainless steel FSII tank equipped with a dryer vent. Injection capability is provided via an injector manufactured by Gates City. The tank is empty and has not been used in recent years. They do have five drums of FSII on hand, which they have stored in a warehouse for three years.

Product is filtered through separate filter and coalescer vessels. Both are equipped with Velcon elements. The filter vessel contains 11 filter elements, and the coalescer vessel contains 22 coalescer elements. There have been three filter changes since the beginning of the contract period (once in August 1999, again on October 28, 2000, and most recently on December 7, 2000). The filter changes were made because of increased differential pressure across the elements.

The Portland DFSP ships solely by tank truck. A visual examination and laboratory analysis for FSII, conductivity, and API gravity are conducted each morning. Samples are retained for a 15-day period. Tank trucks loaded at Portland go to Pease (50 minutes away), Bangor (2.5 hours away), Brunswick (45 minutes away), and several Army National Guard units.

The major differences between the facilities at DFSP Portland and DFSP Ludlow are that Portland stores JP-8 in fixed-roof, rather than floating-roof tanks; and, that Portland provides coalescer removal of water, while Ludlow has modified its filter separator vessel to use only micron filters. Because Portland's fixed-roof tanks do not allow rainwater intrusion into the JP-8, they are not faced with the need to re-inject FSII and SDA.

#### **7.1.10 Bangor ANGB**

Bangor ANGB receives product primarily from DFSP Portland. While Bangor ANGB has found small amounts of apple jelly in hydrant system F/S vessels and flight-line servicing equipment, it has not experienced apple-jelly problems in its receipt filters since changing from DFSP Searsport (now closed) to DFSP Portland as their major supplier. During pipeline receipts from DFSP Searsport, and especially toward the end of the pipeline use during late 1997, dark-colored contaminants were found frequently. At that time, it was believed that the dark materials were the result of pigging the pipeline to prepare it for closure. Materials with an apple jelly-like appearance were first found in the receipt filter during a filter change driven by a rapid rise in the filter-differential pressure.

Since converting from pipeline receipts to tank truck receipts during 1997, Bangor ANGB found approximately 1 quart of apple jelly in one of the two hydrant system filter vessels in their Type III hydrant system. These two F/S are located within an enclosed pump house along with all the other F/S vessels. The temperature in the pump house is maintained at approximately 55 degrees during the winter (though operators occasionally turn up the thermostat). One unique feature about the vessel in which apple jelly was found (designated as H-2) is that it sits directly beneath the overhead heater for the building.

A small amount (a few tablespoons) of apple jelly-like material that "flowed like mercury" was found in the filter vessel of one of the four assigned R-11 refueling trucks

(Kovatch, Serial # 98L59), during change from coalescer elements to water-absorption-type elements. The R-11s were equipped with water-adsorbing elements during November 1999. The other three R-11s are manufactured by Oshkosh and have vertical filter vessels built to use the old standard DoD NSN 4330-00-983-0998 filter coalescer elements. All of the R-11s were equipped with water-absorption elements, manufactured by Facet, during November 1999.

Apple jelly was also found on several occasions in one of the three assigned HSV-12 hydrant-servicing vehicles (Serial #98L76). On each occasion, a small amount (a few tablespoons) of apple jelly was found while draining the filter vessel the morning following a day of unusually heavy use. At that time, the HSV-12s were parked in a heated barn over night. The HSV-12's are now parked outside, and no apple jelly has been found recently. Beta Systems manufactured the F/S on 98L76, during the 4<sup>th</sup> quarter of 1997, Model # 182FS2001, and Serial # 182F-049. This vessel uses three coalescers and two separator elements. They have not found any apple jelly-like material in either of the two older-model HSV-12's. These are equipped with Velcon vessels, Model # HV2856YTSR. They use seven coalescers (I-65688TB) and two separators (99-848V).

The Bangor ANGB receives JP-8 via tank trucks (not dedicated to JP-8) that are filled at DFSP Portland. The trucks travel approximately 130 miles. Receipt temperatures during January average 34°F and have reached a low of 28°F. During August, fuel temperatures are consistently 75°F. The product is filtered upon receipt via two vertical API standard F/S vessels manufactured by M.E. Industries; the vessels are epoxy-coated steel. The F/S vessels are drained following each receipt. The filter vessels are drained through a ¾-inch line equipped with a circular sight gauge. The Bangor ANG personnel have modified the sump drain to include a manual drain. No fuel receipt quality problems have been experienced since changing from pipeline to truck receipts during 1997. The pipeline receipts originated from Searsport DFSP and traveled 27 miles from the DFSP to the base. Bangor receives an average of six tank truck deliveries per day. They have the capability to offload three trucks simultaneously. A single truck can be offloaded within 35 minutes. The truck offloading system is equipped with two positive displacement

pumps. The offload procedure results in pumping a significant amount of air as the tank trucks approach empty and while evacuating the discharge lines. The fuel is received through the receipt F/S into two above-ground 10,000-bbl tanks. Bangor ANGB stores and issues approximately 800,000 gallons per month.

The two 10,000-bbl above-ground storage tanks (ASTs) were built in 1993/94 and placed into service during 1994. The tanks have fixed roofs, floating pans, and are fully epoxy coated. The tanks are the “self-cleaning” design with the tank bottoms sloped to the center and the suction line directly over the tank sump. Bottom water is drawn out of the sump through a  $\frac{3}{4}$ -inch drain line (inside the tank) with a 1-inch line outside the tank. The sump drain extends to within 1 inch of the bottom of the sumps. When draining the tank sumps, fuel is first drained into a bucket for a visual examination. If there is no BS&W seen in the bucket, the fuel is pumped through the receipt F/S and back to the tank for approximately two minutes and monitored through a pipeline sight gauge. The filter vessel sump drain lines empty into a 2,000-gallon underground product-recovery tank. Sight gauges on the hydrant and truck fill stand F/S sump lines are used to monitor the product, and manual drains were installed and used daily. The product-recovery system is equipped with a 2-inch return line that moves JP-8 from the product-recovery tank, through the receipt F/S, back to the bulk tank. A 50-gpm pump powers the return line.

The Bangor ANGB Type III system was installed during 1994 at about the same time the base converted from JP-4 to JP-8. All F/S vessels are located within a heated pump house. Two F/S vessels are used for hydrant issues, and two support the refueling truck fill stand. The Type III system is turned off at the end of the day and restarted the next duty day. The Type III system has a 10-inch pipeline loop.

FSII content is fairly consistent at 0.12 vol%, with a low of 0.10 vol% and a one-time high of 0.14 vol%. Conductivity ranges between 100 and 160 pS/m during the winter months and between 250 and 300 pS/m during the summer months. The few experiences with apple jelly at Bangor are associated with the decrease in ambient temperature with the onset of winter, which causes the dissolved water to precipitate from

the fuel. Also, Bangor ANGB is one of the few locations that has reported finding apple jelly in HSV-12 servicing vehicles. This is of concern since apple jelly-like materials may accumulate over time in low points within the hydrant system and be picked up by the fuel during surge operating conditions.

#### ***7.1.11 DFSP Carteret***

DoD products handled by DFSP Carteret include JP-8, JP-5, and F-76. Carteret was activated as a new DFSP following the closure of DFSP Stanton Island in 1999. As DFSP Stanton Island tanks were drawn down in preparation for closure, it was reported that significant amounts of apple jelly-like materials were found in the system. This may have contributed to the reports of apple-jelly problems in the Northeast during the late 1990s. JP-8 is shipped to Carteret via a commercial, multi-product pipeline, from breakout tanks in Linden, NJ. Four, fixed-roof, steel tanks constructed in the 1940s are dedicated to JP-8 storage. Since December 2000, unfiltered, neat JP-8 has been received into tank #100 (capacity 92,000 bbls) and additized in a tank-to-tank transfer to tanks # 125 and 126 (capacities 23,000 bbls each). Non-additized JP-8 is filtered as it enters tank #44 (capacity 74,000 bbls). Tank #44 is used primarily to fill barges and occasionally tank trucks. Receipt batches are limited to a maximum of 75,999 bbls. Three tanks are dedicated to storing JP-5: tanks #25-1 and 25-2, constructed in 1955, and tank #63, constructed in 1942. JP-5 is additized as it leaves the pipeline and enters Tank 63. Carteret also has tanker and barge capability and supplies fuel to DFSPs New Haven and Portland by barge and DFSP Verona by pipeline.

Tank # 100, the JP-8 primary receipt tank, has an epoxy-coated floor and epoxy coating 3 ½ feet up the sides. It also has an 8-inch water dam around the suction line that has a duckbill 6 inches from the bottom. The discharge (suction) and receipt lines are separated. The suction line is fixed. The tank was last cleaned in May 1999, when the epoxy coating was applied. Tank # 44 is also coated similar to Tank # 100 and has a discharge line 18-inches from the bottom of the tank, positioned in the center, with a diffuser on the end and slots on the side. The tank sump is approximately 35 feet away



from the discharge line. The 18-inch receipt line is also used to fill barges. An 8-inch line connects the tank to the tank truck loading rack. When Tank # 125 was last cleaned in March 1999, the coating was unacceptable and had to be sandblasted. The floor and walls are mild steel. The fixed discharge line is a duckbill approximately 3 feet from the side with a water dam 6 inches from the floor. The tank sump is approximately 4 feet from the discharge line. Tank # 126 is similar to Tank #125.

FSII is stored in an 11,000-gallon tank with a nitrogen blanket. Since December 2000, the same Hammonds inject both FSII and SDA 2000D-40-1M injectors after the fuel leaves Tank # 100. FSII is injected to a final concentration of 0.11 to 0.13 vol%. Sufficient SDA is injected to reach a conductivity of 200-250 pS/m. The FSII concentration was traced from DFSP Carteret to New Haven with no significant loss of FSII evident.

There have been no reports of apple jelly since the terminal was activated; however, the appearance of a lime green tint in fuel receipts, especially JP-5, has been frequent since the department of transportation mandated the use of green-dyed water to pressure test the pipeline in December. Water is removed when needed as indicated by gauging. A vacuum truck removes BS&W. An immediate sample and all-level sample are taken and tested on all inbound and outbound shipments. A B-1 sample is taken on receipts after the fuel in the receipt tank has settled for a day.

#### ***7.1.12 DFSP Jacksonville***

Jacksonville receives fuel (JP-8) primarily from the Sun Refinery in Marcus Hook, PA. On occasion, product may also be received from DFSP Yorktown and DFSP Baltimore. JP-8 is primarily received by dedicated barge, the Bay Trader, on contract to the government, although spot-chartered barges may be used periodically. Travel time by barge is about 4.5 hours from Marcus Hook, 30 hours from Yorktown and about 8 hours from Baltimore. JP-8 is fully additized when received at DFSP Jacksonville. The terminal has the capability of adjusting additive levels as barges are discharged. Product

is received dry, and there are no water problems. Tanks are drained as required based on water-finding paste, but water is found only very infrequently.

The terminal has a total of 12 tanks but only eight are in use for JP-8. All are above-ground steel tanks equipped with fixed roofs and internal floating pans. Four tanks have bottoms, which slope to the side, and four bottoms slope to the center. Tanks are cleaned every 5 years. Product in tanks #1, #2, and #5 is rotated at least every six months. Product in tanks #4, #9 and #10 is rotated every 1 to 1.5 months; and, product in tanks #11, #12 (used to load barges) is rotated weekly.

Product is shipped to McGuire AFB through a dedicated pipeline and loaded in tank trucks (non-dedicated) for shipment to various other locations. Product is filtered when shipped by pipeline and trucks. Shipping time to McGuire AFB varies from 8 to 12 hours depending on the batch size. Filter changes average about every 15 million gallons.

DFSP Jacksonville personnel reported that they encountered a very small amount of apple jelly in the pipeline F/S to McGuire about 3 or 4 years ago.

### **7.1.13 McGuire AFB**

McGuire AFB was selected as a site to visit because it had no history of apple-jelly contamination. The plan was to contrast the facilities and fuel-handling procedures with locations that have experienced apple-jelly contamination. However, just prior to our visit, McGuire AFB fuels personnel discovered a jelly-like substance in the sump of Kovatch R-11 # 97L0165. The substance drained from the sump had a foamy appearance, as though a lot of air was mixed with the product. It had a pinkish-brown tint and had the appearance of a chocolate/strawberry milkshake. The appearance of the sample did not change significantly while sitting in the laboratory or even after it had been shipped to SwRI. It is suspected that this contaminant appeared because this particular R-11 had been recently used to flush four 100-foot sections of hose while preparing a Forward Area Refuel Point (FARP) cart for deployment. The hoses, which had been in storage for three

years at McGuire AFB, had been pressurized with water to 150 psi. They were drained and flushed by connecting one end to the single-point nozzle and the other to the bottom loader of the R-11. Five hundred gallons of fuel were circulated through each hose section; then the content of the hoses was evacuated into the R-11. They allowed 97L0165 to sit over night to allow the water to settle and discovered the contaminant while draining the sump the following morning. The R-11 was equipped with Facet water absorption elements, installed during July 2000. The hoses were not available for inspection because they had been deployed with the FARP cart. Very little information was available about the manufacture of or materials used in the hose. The ambient temperatures during the 2-day period that encompassed the flushing of the hose and the discovery of the contaminant ranged from 23°F to 28°F with wind chill factors of 1°F to 16°F.

A second incident occurred on February 27, 2001, when a F/S vessel shutdown during a transfer between hydrant systems. Some dark materials were drained from the filter sump. The filter elements had been in service for 35 months and an apparent water slug shutdown the F/S. Apple jelly was not found during an internal inspection of the F/S.

McGuire AFB receives fuel via a dedicated 6-inch, 11-mile pipeline from DFSP Jacksonville. McGuire is capable of receiving 648,000 gallons per day. A new tank truck off-loading facility will increase receipt capacity to 1.3 million gallons per day when completed. A pipeline modification, scheduled for completion in October 2001, will route both pipeline and tank truck receipts directly to the BRAC hydrant system, bypassing bulk storage. C-Station, used primarily for storage and as a fill stand, has six 25,000-gallon tanks and two fill stands.

McGuire has three 450,000-gallon floating-roof tanks with geodesic dome roofs. However, these tanks have been out of service for maintenance and repair since prior to their conversion from JP-4 to JP-8. Construction of a 2-million-gallon storage tank is scheduled for fiscal year 2002/2003.

The primary storage is the two Type III Hydrant Systems. The KC-10 hydrant system (BRAC) has two 1-million-gallon tanks with five 600-gpm pumps and delivers up to 2,400 gpm. It also has two fill stands that can sustain six R-11s per hour. The C-141 Hydrant System (DLA) is located within bulk storage and has two 850,000 gallon tanks with four 600-gpm pumps that deliver 2,400 gpm. They also have a Type II system with six 50,000-gallon tanks, three laterals with five outlets on each with a flow rate of 600 gpm. If one of the systems became inoperable, the system could be configured so that one hydrant system can service all spots with a reduced capacity. McGuire AFB has 12 R-11s with water-absorption elements installed in June-July 2000, six HSV-12s, and five MH2-C Hose Carts.

#### ***7.1.14 DFSP Port Mahon***

Port Mahon encountered apple jelly during December 2000. On two occasions they drained cloudy fuel from the F/S sump and got a dark material that was suspected to be apple jelly. This experience coincided with a drop in temperature and occurred at the same time that Dover AFB experienced apple jelly problems in their receipt F/S sumps.

A contractor operates DFSP Port Mahon for the primary support of Dover AFB. Occasionally, tank trucks are loaded for destinations directed by DESC to relieve temporary shortages or resupply delays. There are eight fixed-roof storage tanks for JP-8: four 30,000-bbl tanks, three 96,000-bbl tanks, and one 25,000-bbl tank. Tank #5 is a floating-roof tank but has been modified with a geodesic dome cover. The suction and discharge into tank #5 is typical of the other tanks. The single 8-inch suction line goes 30 feet into the tank, turns down 90 degrees, and comes within 3.5 inches of the tank floor. The tanks are cleaned on a 3-year cycle, and tank #5 was last cleaned in 1999. This 96,000-bbl tank was constructed in Aug 1964.

Product is received at Port Mahon by barge from Sun Oil, Marcus Hook, PA (75%) and Baltimore DFSP Terminal (25%). The U. S. Coast Guard limits barge size to 13,000 bbls because of the shallow bay. Eight to ten barges per month are received at Port

Mahon. Product received normally has all the additives necessary for JP-8, though additional SDA can be injected at Port Mahon if needed.

There are two 1,200-gpm vertical F/S vessels installed on the outbound lines. The vessels are Model # VFCS-C-12N38-5636TN2. Both vessels are API 1581, Group II, Class B. It was noted during the visit that the F/S drains were blocked with pipe plugs below the drain valves, and the vessel air eliminator lines were capped. The Terminal Manager stated that they were blocked to avoid a spill and the associated environmental impact. This modification arrangement inhibits checking the F/S sumps for water and relies on the internal float mechanism to stop the flow of fuel if the vessel sump becomes full. The internal float shutdown has a history of failure, allowing water to be transferred downstream of the F/S vessel.

DFSP Port Mahon supports Dover AFB. The only apple-jelly experience at both locations occurred during a 10-day period in December 2000. The fact that eight of the nine JP-8 storage tanks have fixed-roofs may explain why excessive free-water/FSII mixtures (apple jelly) have rarely been found at Port Mahon or Dover AFB. The one modified floating-roof tank with the geodesic dome cover, and a suction nozzle directed toward the tank floor that sits only 3½ inches above the floor might explain the apple jelly experience during December 2000; however, we were unable to confirm whether or not this tank was on issue. Some rainwater may have entered this tank and caused the formation of apple jelly. However, the drop in ambient temperature, and subsequent dissolution of dissolved water, that coincided with the discovery of the apple jelly are more likely the source of free water. Given the design and position of the intake nozzles, the free-water/FSII mixture would have been picked up by the discharge line and pushed to the F/S vessels.

#### **7.1.15 Dover AFB**

Dover AFB experienced apple jelly only during a 10-day period in December 2000. During this time, apple jelly was frequently drained from the sumps of receipt F/S

#1 & #2, which are installed in a heated pump house. When the problem occurred, the ambient temperature had dropped from 40°F to 20°F. It was also noted that slugs of water were coming through the F/S, and they were experiencing high AEL water readings. The Bulk Storage Supervisor reported that the F/S pressures decreased during the temperature drop from a high of 11-psi to a low of 5-psi differential pressure. After the temperature went back up, the differential pressure also went back up.

Product at Dover is received from the Port Mahon DFSP, which is approximately five miles away. Fuel is transferred from Port Mahon to Dover through two, 6-inch, dedicated pipelines. The normal transfer rate is 35,000 gallons per hour (gph) and during an upsurge, a boost pump is utilized that will increase the flow to 51,500 gph. The fuel is filtered at Port Mahon outbound to Dover. Line displacement time to Dover from Port Mahon is three hours. Pipeline shipments from Port Mahon are not filtered upon receipt and transfer from the bulk tanks. Dover AFB issues an average of 300,000 gallons of fuel per day. The FSII content of the fuel received at Dover is normally 0.13 vol% to 0.14 vol% on receipt and is normally 0.12 vol% to 0.13 vol% upon issue.

Dover AFB has three above-ground steel tanks used to store JP-8. Two of the bulk storage tanks are equipped with floating-roofs with geodesic covers. The third tank is an above-ground steel tank with fixed-roof and floating pan. This 55,000-bbl tank was built in 1971. It is equipped with a product recovery system with hand pump and ¾ inch line for water removal.

Dover AFB has three Beta, two Tri-State and one Page AVJET Hydrant servicers. The frequency of F/S element changes on this equipment averages three years, and the changes are based on the maximum use life. No apple jelly has been encountered in these hydrant servicers. There are nine R-11 refuelers equipped with absorption media elements. The Facet elements were installed in July 2000, and no problems have been encountered. Fuels Management at Dover has implemented an aggressive water-draining program. Operators drain the sumps of the refuelers and hydrant servicers at checkpoint and at the second shift change daily. Personnel report that they are not seeing anything abnormal.

There are two-Type III constant pressure hydrant systems installed at Dover. The F/S have API 1581, Group II, Class B, and 3<sup>rd</sup> edition elements. The first filtration occurs during transfer to the two, Type III, constant pressure, hydrant systems. Each hydrant system has two receipt and re-circulation F/S vessels and five issue F/S vessels. Each hydrant system is equipped with two above-ground steel fixed-roof 10,000-bbl operating tanks. The suction discharge lines in all four tanks are positioned within the tanks directly above the sumps. Again, the product recovery systems are equipped with ¾ inch lines. The four hydrant operating tanks are 100 percent epoxy coated and are equipped with three-inch diameter stripper lines. During surge operations, as much as 600,000 gallons of fuel may be issued per day, primarily by hydrant systems.

#### ***7.1.16 DFSP Grand Forks***

DFSP Grand Forks receives JP-8 from Conoco's Ponca City, OK Refinery via a multi-product (jet fuel, diesel fuel, and gasoline) pipeline. Pipeline Drag Reducer (PDR) is used in some of the pipeline product movements, but not JP-8. Because of the line fill in this pipeline, it takes approximately three weeks for a shipment to reach DFSP Grand Forks. DFSP Grand Forks receives a "heart cut" of the pipeline shipment, based on API gravity readings. The product at the front and end of the batch is not separated as product but is reportedly blended with "other" products. Grand Forks DFSP has four storage tanks, all built in 1959. Two of the tanks are 55,000-bbl steel tanks; two are 80,000-bbl tanks. The floors of the tanks are epoxy coated and the coating extends three-feet up the tank walls. The tanks have floating-roofs with geodesic dome covers. The roof drains have been removed. The tanks slope to the center and are equipped with 2-inch bottom sediment and water drain lines. The tanks are drained weekly and also after each receipt. Tank receipt and discharge lines are separate. The discharge (suction) lines are close to the bottom sumps. The distances range from 17 inches to 43 inches.

During January, product receipt temperatures range from 33°F to 35°F, while the temperature of JP-8 in storage ranges from -2°F to +20°F. During August, receipt

temperatures range from 65°F to 69°F, while storage temperatures range from 65° to 70°F. The JP-8 is not filtered during receipt at DFSP Grand Forks and it is not filtered during transfer to Grand Forks AFB.

Additives, SDA and FSII are injected using Hammond injectors (six model 6T-4L for the truck fill stand and a model 1400 for the pipeline). In each case, the same injection unit injects both additives. A 6" pipeline, which operates at 500 gpm, supplies the fill stands. The transfer pipeline to Grand Forks AFB is an 8-inch line with an average pumping rate of 650 gpm and a 210-psi average operating pressure (300 psi max). SDA is received in 55 gal drums and is blended with JP-8 at a ratio of 20 parts JP-8 to 1 part of SDA in a 27.5 gal tank. The JP-8 retain samples (fully additized) are used to dilute the SDA. FSII is received by bulk from Ashland Distribution Co. and is stored in a 6,353-gallon tank. The tank is equipped with a desiccant dryer, and the FSII turns over every six months. The DFSP personnel work hard to maintain a 0.11 vol% FSII injection ratio and keep meticulous records. FSII test results show a range from 0.10 vol% to 0.12 vol%. They report that the Hammonds injectors require constant monitoring. Fuels management personnel at Grand Forks AFB report that the quantity of FSII in prior years averaged 0.14 vol%. They also stated that following the reduction of FSII to 0.12 vol% and greater attention to removing water bottoms they have seen less apple jelly.

Tank truck deliveries are also made from DFSP Grand Forks to military activities in Minnesota (6 to 8 hours transit time), North Dakota (up to 4 hours transit time), Wisconsin (8 hours) and South Dakota (up to 15 hours driving time, necessitating an over night stop). Trucks are not dedicated but each truck is inspected and the manifolds drained prior to loading.

Due to the work of the DESC Apple Jelly Tiger Team and William Pulley, the QSR for this area, a tremendous amount of data were collected concerning JP-8 and operations at DFSP Grand Forks. Fuel quality data and temperature information were extracted from this data and are presented in graphic form in the full report from C4e. [17]



### **7.1.17 Grand Forks AFB**

Grand Forks AFB has been plagued with apple jelly for the past three years. It was worse three years ago but they continue to struggle with periodic occurrences. The very first occurrence was in receipt F/S #1 and #2 at their bulk storage facility. These two receipt F/S vessels historically yield the largest amount of apple jelly. Previously obtained quantities range from 2 quarts to 15 gallons. The second largest amounts are usually found in F/S #1 though #8 within the Type III Hydrant System, amounts range from 1 quart to 2 gallons. The third largest amounts comes from Tank # 3 and Tank # 4, which are above-ground floating-roof tanks with geodesic dome covers. From 2 quarts to 2 gallons are periodically drained from the sumps of these two tanks. The smallest amount comes from R-11 refueling units with 2 milliliters usually found in the F/S sump. The first occurrence during the fall season of 2000 was on October 16, 2000. Grand Forks AFB occasionally finds apple jelly in the transfer F/S and apple jelly is frequently found in R-11s parked in the four heated stalls over night. Base personnel noted this phenomenon and conducted an experiment during December 2000 and January 2001 that involved moving the trucks inside the heated storage area for several days, then outside, then back inside. Apple jelly was drained from the sumps while inside, but no apple jelly was drained from trucks kept outside overnight. They also conducted a similar experiment with the transfer pump house by turning the heat off for a period of time, and then back on. When the heat was on, apple jelly was produced. When the heat was off, no apple jelly was drained from the sumps. Base personnel reported that they take advantage of this phenomenon to reduce the differential pressure on vessels approaching the 25-psi differential pressure change criteria of the elements. They report that the Air Force has provided a waiver to extend the use of water absorption elements from the 22-psi differential pressure change requirement to 25-psi. On vehicles approaching the 25-psi limit, they frequently park those R-11 refuelers inside to allow the water absorption media to drain. This lowers the differential pressure and “extends the life” of the elements. It also results in the accumulation of apple jelly in the filter sumps.

JP-8 is shipped via pipeline to Grand Forks AFB from the DFSP at Grand Forks. Product is filtered, as it is received, through a Facet CDCS-D-7K39SB-1-2S636FM element located in Facility 511. As fuel is issued, it passes through a transfer/issue station located in Facility 501 and is filtered by a Bowser 1838 F/S vessel.

Grand Forks AFB receives JP-8 into two steel, epoxy-coated ASTs. One tank has a 30,000-bbl capacity and the other is a 25,000-bbl tank. Both were constructed in 1958. The tanks have floating-roofs and geodesic dome covers. The geodesic domes do not have a complete seal against the sides of the tanks and during periods of heavy rain and high winds, water enters the tank by blowing up under the skirts on the geodesic domes. They have an active project to extend the skirts of the geodesic domes to preclude water entry into the tanks. The tanks slope to the center, and the discharge (suction) lines are located approximately 15 inches from the bottom of the sumps. Product in these tanks is turned over every two to three days. During January, product storage temperatures typically range between 11°F to 22°F. The base reports that inventory levels have dropped by 45% over last two-three years. On average, only 50% of the existing storage capacity is currently in use.

The base has seven Oshkosh R-11 refuelers, and one Kovatch R-11. All are equipped with water-adsorbing elements manufactured by Facet (NSN 4330-010-439-2314, and PN 3 FGI 6335B). Grand Forks AFB also has four HSV-12s with Facet CA56-35B-51 coalescing elements. Because the HSV-12s tend to leak when taken from a warm environment to a cold environment, the HSV-12s are primarily (except for maintenance) parked outside rather than in the heated facility. No apple jelly has been found in the HSV-12s. Grand Forks has a Type II and a Type III System.

Analysis of receipt samples (using the AEL) shows that the product averages 3 ppm of undissolved water. FSII levels run between 0.10 vol% and 0.11 vol%. Base personnel report that the occurrence of apple jelly was greater three years ago and at that time FSII levels averaged 0.14 vol%.

### **7.1.18 BP/AMOCO Mandan Refinery**

The BP/AMOCO Mandan Refinery produces diesel fuel, heating fuel, gasoline, commercial jet A and JP-8. The crude oil is North Dakota sweet crude, Williston Basin. This crude is sometimes green in color and has API gravity between 41 and 42. They have on occasion purchased from Canada but have not done so in the last few years. The refinery capacity is 60 thousand barrels per day. The JP-8 is straight run distilled from one tower with no cracking or blending, however the JP-8 does pass through a salt dryer. There is a change from summer to winter grade diesels and #2 oil, but there is no change to the JP-8 process.

The refinery has three dedicated storage tanks. Tanks #757 and #717, constructed in 1954, are 43 MBBL steel, ASTs with floating-roofs and are used for truck rack loading. Tank #726 is a 96,000-bbl steel AST tank with a floating-roof and is used primarily for pipeline shipments. The roof is equipped with a flex-line roof drain. The sump wells are 18-inches deep and tanks are drained prior to issue or when rain or water intrusion is suspected. The receipt line extends straight 18 inches into the tanks with no loops or turns. It is offset from the suction line by 20 feet. The floors are epoxy-coated, the coating extends three feet up the sides. A three-inch diameter sight glass is used to visually observe the removal of water from the tank sumps. The laboratory supervisor reported that all water removed is clear with no discoloration. Storage tanks are drained after quality acceptance and before issue to customers. No water gauging is performed and quantity determination is by meter. The JP-8 contract requires storage tanks to be cleaned every 5 years.

Tank #726 is connected directly to the multi-product pipeline that feeds the BP/AMOCO terminal located in Morehead, MN. The refinery makes 20,000-bbl batches for pipeline shipment once per month because of the small capacity of the Morehead terminal. Side sample taps are affixed to all storage tanks, however since tank #726 is never full, all samples are obtained from a goose neck located 12 inches from the 3-inch water drain line. JP-8 is filtered from the refinery through a clay filter and two paper filters

prior to entering storage tanks. All JP-8 shipped by pipeline contains one additive, corrosion inhibitor/lubricity additive. The Morehead terminal injects FSII and SDA.

The refinery ships to Minot AFB twice per week by tank truck. FSII and SDA are injected at the tank truck loading rack. The FSII injector is manufactured by GATE-PAC, model, EI 2007-1. Setting is at 0.10 vol% to 0.15 vol% as per specification requirement. The FSII storage tank has a 10,000-gallon capacity; it is equipped with a dryer. FSII is received by tank truck from EquaStar, located in Bay Port, TX. The SDA injector is a GATE-PAC, Model, EI 0755-1, and the storage tank has a 500-gallon capacity. The tank trucks load at 600-gpm; and, the JP-8 passes through a F/S vessel as tank trucks load.

#### **7.1.19 Minot AFB**

Minot AFB initially experienced apple jelly during 1995, soon after the conversion from JP-4 to JP-8. Minot has experienced apple jelly on 16 separate occasions between December 1, 2000 and March 14, 2001. Apple jelly was found seven times each in issue F/S #2 and issue F/S #3, once in tank #4 product recovery, and once in a hose cart. Since October 2000, Minot has received JP-8 from the Mandan refinery by tank truck. The average travel time from the loading point to receipt is 3 hours. They report receiving significantly less water in the fuel from Mandan than was the case when they were supplied from DFSP Grand Forks. All JP-8 is filtered upon receipt through a Facet, SS636FF vessel. During January, product receipt temperatures range between 13°F and 28°F, while the temperatures of JP-8 at the time of issue range between 11°F and 34°F. During August, fuel receipt temperatures range from 65°F to 81°F, while storage temperature ranges from 67°F to 76°F. JP-8 received at Minot AFB contains all additives upon receipt. The base has two types of receipt F/S units: Facet, SS636FF/CC-K388B1, and Faudi, F.7-965 8806796. Personnel reported no significant difference between the Faudi coalescers and the NSN 4330-00-983-0998 coalescers ordered from stock. Issue/hydrant F/S vessels use Velcon separators, S0630VA, and NSN 4330-00-983-0998 coalescers manufactured by Velcon.

Minot Base Fuel Personnel are aware that combining FSII and water under certain conditions can cause a possible problem with apple jelly. They have a rigorous plan for water removal from all units and storage tanks. All systems and storage tanks are drained at the beginning of each shift. Apple jelly occurrences have lessened since the adoption of the new procedures; however, small amounts are found almost daily. All above-ground bulk-storage tanks are 100% epoxy-coated, modified floating-roof tanks with geodesic dome covers. Tank bottoms slope to the center where a ¾-inch line to the sump well is located to remove BS&W. The issue (suction) line is 12 inches from the bottom and located in the same area as the sump drain. During warm weather, the bulk tanks are drained for water daily. The drained water is put through the product recovery system. After settling, the product is pumped back to the tank with a hand pump. During cold weather, the hand pumps freeze and tanks are drained manually into jerry cans, which are emptied into bowsers. Minot AFB stores 50,000 bbls of JP-8 and 3,500 bbls of JP-4. JP-4 is available to assist engine starts during cold winter months. The JP-4 is stored in a single, fixed-roof tank. No apple jelly problems have been experienced with JP-4.

Minot AFB has seven Oshkosh R-11 refuelers and seven MH2C hose carts. They also have a Type II hydrant system with three independent pump houses. They expect to break ground for a new Type III hydrant system during fiscal year 2002.

When JP-8 was received in August 2000 from DFSP Grand Forks, the FSII levels were between 0.11 vol% and 0.14 vol%, averaging 0.12 vol%. From March 5, 2001 through March 21, 2001, the fuel was received from the BP/AMOCO Mandan refinery. The FSII levels for this fuel were between 0.12 vol% and 0.16 vol%; the average was 0.14 vol%.

#### ***7.1.20 McConnell AFB***

McConnell AFB has experienced apply jelly in a clear white form. The base personnel report that the substance is thick and gummy. This differs from other reports of apple jelly in that almost all other reported apple jelly is colored. This difference again

points out that the personnel in the field have widely varying ideas about what constitutes apple jelly.

Four KC135s have found apple jelly in 2 main tanks after reports of fuel gauge problems. All four aircraft had refueled at another location prior to reporting the problem to aircraft maintenance. The apple jelly found in the aircraft fuel cells was not as thick as the material drained from the fuel F/S mentioned above. During pre-flight inspection, KC-135 sumps are drained and the crews report never having observed apple jelly.

The Air National Guard uses the same JP-8 from the same system as McConnell AFB and has not experienced any apple jelly. The ANG has Oshkosh R-11 refuelers. Boeing engineers have reported a problem with apple jelly in their refuelers to the McConnell AFB Fuels Flight. Boeing loads their truck at the same location as the ANG at McConnell AFB.

McConnell has established a more aggressive policy to remove water from the fuel system as a means to reduce the occurrences of the apple jelly. JP-8 is received by dedicated pipeline from the CONOCO terminal approximately 1½ miles from the base. Tank trucks are received twice per year to exercise the truck receiving system. Pipeline receipts are usually scheduled for Wednesdays and are normally 614,000 gallons. After start-up, it takes about ten minutes to displace receipt lines. JP-8 is not filtered upon receipt, though the fuel is filtered as it leaves the CONOCO terminal. Tanks and F/S sumps are drained for water at each shift change, and tanks are drained each day while temperatures are below 55° F. The FSII concentration at time of receipt ranges between 0.105 vol% and 0.125 vol%. Fuels management personnel have requested that the FSII maximum level be reduced to 0.11 vol%.

All storage tanks are 100% epoxy coated. Tanks are above-ground steel with geodesic domes and floating-roofs. Two 35,000-bbl tanks were constructed in 1950, and two 10,000-bbl tanks in 1998. These tanks slope to a center sump, which has a ¾-inch water drain line. With one exception, suction lines extend to the center on a slight angle

parallel with the tank floor, approximately 18-inches from bottom. There are no elbows on the suction line and the inlets are directly over the sump. Tank #1, a 35,000-bbl tank, is the exception in that the suction line has an elbow that is angled away from the sump. McConnell has ten R-11 Kovach refuelers, a Type II and a Type III hydrant fueling systems and four Beta HSV-12s.

#### ***7.1.21 CONOCO Pipeline Company***

JP-8 is supplied to the CONOCO terminal by multi-product pipeline from Ponca City, OK. The product is sampled upon receipt and tanks are equipped with side taps for representative sampling. The contractor obtains all samples. All transmix is diverted into one of the CONOCO tanks to be blended with commercial product. JP-8 is stored in three dedicated, above-ground, steel tanks. Storage tanks are epoxy coated on the bottom and 1.5 feet up the sides. Tank bottoms slope to the edge and are equipped with water sump drains. Tank #1259 has a common receipt and issue line. It is a flat bottom tank equipped with four sump drains, one at the center and three around the edge. Tank #202 receipt line extends 5 feet into the tank and the suction line is 10 inches from the bottom with a ninety-degree elbow; the line is equipped with a vortex diffuser. Tank #1258 suction line extends 12 inches into the tank and is set on a pedestal 10 inches off the bottom. Sump wells are from 18 inches to 3 feet deep. Water draw-off lines are 2 inches in diameter. Storage tanks are cleaned at 5-year intervals in accordance with contract requirements. The terminal has three FSII tanks, one 4,000-gallon and two-2000 gallon tanks. The SDA tank has a 15 gallons capacity. The filter is equipped with 6-Velcon elements, FO-644 PLF. There are 7-Velcon coalescing elements, I-644 85 TB and three screens, SO 636 PV.

Water is removed after each receipt and all water is drained through the oil-water separator. Their procedure is to drain the sumps until “the brown color turns clear.” FSII and SDA are injected into a 6-inch receipt line during receipt, after the product passes through a 0.5-micron filter. After injection, the product travels through nine 90-degree elbows before entering the tank. Receipt pressure is 25 psi. The injector is a GATE-PAC

manufactured by Milton Roy. The FSII injector enters the JP-8 receipt line and is flush with the inside surface of the JP-8 receipt line. SDA is blended 9 to 1 and is also injected into the receipt line as described above. The FSII concentration has been lowered to 0.11 vol% at the request of McConnell AFB Fuels Management. The FSII concentration for fuel receipts ranges between 0.105 vol% and 0.125 vol%.

JP-8 is shipped to McConnell AFB by dedicated pipeline. It is fully additized prior to shipment and normally allowed to settle for 4 days. Pipeline shipments are filtered through the same 0.5-micron filter and coalescing elements as fuel receipts. Approximately 16 ounces of apple jelly-like materials are found in the separator unit each time the elements are changed. Element changes are based on differential pressure. Pipeline shipments to McConnell normally take 26 hours to complete.

#### ***7.1.22 Edwards AFB***

Edwards AFB first reported apple jelly on January 31, 2001. When sampling R-11 refueler 96L-144 using a single filter monitor, the Quality Control Specialist observed orange specks on the filter membrane. When observed under the microscope, the specks appeared to be apple jelly. [Note that this reported detection of apple jelly was downstream of the final filtration before the fuel enters the aircraft.] Subsequent use of the refueler resulted in collecting samples of apple jelly from the F/S sump. When the differential pressure in the F/S reached the maximum, the refueler was put in maintenance for element change. Two samples of the material from this refueler both appeared to be apple jelly to site-visit team members. In one sample the material adhered to the bottom of the container while the other sample was more fluid. The base has also observed a limited amount of apple jelly from Oshkosh R-11 refueler 91L-69. The two R-11 refuelers, 96L-144 and 91L-69, and the transfer F/S between bulk storage and the hydrant systems are the only locations where apple jelly has been found. One of the refuelers has a horizontal F/S and the other has a vertical F/S.



On March 13<sup>th</sup>, 2001, the differential pressure on refueler 96L-144 was recorded as 4 psi @ 400 gpm during re-circulation through the F/S. On March 19, 2001, during recirculation of 20,071 gallons of fuel, the differential pressure was recorded as 1 psi @ 400 gpm. After this re-circulation, about ¼ inch of apple jelly was collected in a quart jar from the F/S sump drain. The starting fuel temperature was 74°F, and upon completion of the transfer, the temperature of the fuel was 81°F. The same refueler was used on March 20, 2001 to transfer another 15,107 gallons through the F/S. The starting fuel temperature was 57°F, and the fuel temperature upon completing the transfer was 67°F. The differential pressure upon completion of the transfer was 2 psi @ 400 gpm, a slight increase from the pressure on March 19. The site visit team examined the elements removed from the transfer F/S between bulk storage and the hydrant systems and selected one that had an orange discoloration along the bottom portion of the element. This element was cut open and a sample of the orange material was removed and shipped to SwRI for analysis.

All products, except JP-8, are received by tank truck. JP-8 is received by pipeline from DFSP San Pedro by way of the Kinder Morgan Pipeline (KMP) to Carson (better known as Watson Station) breakout point. Product leaves Watson Station breakout point via KMP to Colton. Here it splits and goes to March AFB or Luke AFB or to CAL/NEV breakout tanks which are across the street at Colton. Product then leaves Colton in the CAL/NEV line for Las Vegas. As product travels to its primary destination, Las Vegas, small batches (5000 – 6000 bbls) are heart cut at Adelanto into tanks for subsequent shipment to Edwards AFB. The 6-inch pipeline from Adelanto to Edwards provides a receiving rate of 21,000 gph at Edwards. The average receipt quantity at Edwards is 100,000-120,000 gallons and requires 5 – 8 hours to complete. Pipeline displacement time from Adelanto to Edwards is 23.5 hours; therefore, it takes an average of 3 or 4 receipts to completely displace the line.

Adelanto has two 20,000-bbl floating-roof tanks and one 25,000-bbl tank both with geodesic domes. This location is completely automated. FSII and SDA are injected into the small batches (5,000 – 6000 bbls) of product for later shipment to Edwards after completion of B-1 testing.

Fuel issues vary considerably at Edwards depending on the aircraft testing in progress. At times de-fuels exceed issues. The average daily issues are normally about 75,000 gallons. De-fuels primarily involve home station aircraft.

Edwards has two above-ground JP-8 bulk tanks. Tank #19 has a capacity of 20,000-bbl and tank #28 is a 10,000-bbl tank. Both tanks are equipped with floating-roofs without covers. Seals on both tanks are in bad condition and tank #19 is out of round. After a recent heavy rain 2,000 gallons of water were removed from the two tanks and associated fill stand F/S sumps. Tank #19 has floating suction verified by drawings. Tank #28 was reported to also have floating suction, however drawings were not available to verify this. On tanks #19 and #28, the ineffective product recovery systems have been abandoned and removed. The tanks were retrofitted with 1½-inch valves and hoses that adapt to their 600-gallon bowser for a more effective system of removing water and emulsion from the tanks. Fuel is not filtered upon receipt, and the first filtration occurs as the product is transferred to hydrant operating storage. The transfer F/S uses American Bowser vessels with the old style DoD NSN 4330-00-983-0998 elements manufactured by Velcon, which are also used in the Type I and III hydrant system F/S vessels. The bulk storage area has the capability to load refuelers but is rarely used due to travel distance.

The three 8,000-bbl JP-7 tanks have suction lines directly above the center sump and are equipped with inadequate product recovery systems. It was noted that these tanks have sections of fiberglass-reinforced pipe and one flange has a small seep. FSII levels averaged 0.11 vol% in storage and at time of issue. When tested for fuel moisture content, one refueler had 5-ppm water with apple jelly.

Edwards AFB has eighteen R-11 refuelers (one Kovatch, seventeen Oshkosh) and five hydrant servicing vehicles (HSV-12s). Water absorption elements replaced the coalescer elements in the refueling trucks beginning in September 1999. Two trucks are dedicated for use with JP-8+100, though the quantity used is small. The +100 additive is injected at the truck fill stand.

Hydrant System 1 (facility 1724) has four 50,000-gallon horizontal cylinder tanks sloped to the sump end. Three are used for JP-8 and one for JP-4. This facility is used primarily to fill 95% of the refuelers at Edwards. There are 3 Bowser, 2 Gil, and 1 American Pipe and Steel F/S vessels with the old DoD-style elements manufactured by Velcon.

The Type III system has minimum use. It has two 12,000-bbl fixed-roof operating tanks. Product recovery systems equipped with hand pumps, ¾-inch lines and small (approximately 30-gallons) product recovery tanks. This system has minimum utilization because helicopters occupy the hydrant outlets, precluding access to the outlets by large aircraft. There are 2 Gil V-600 and 3 Gil V-1200 F/S vessels with the old style elements manufactured by Velcon.

### ***7.1.23 Kinder Morgan Holding Facility***

The breakout tank that supports Beale AFB is a 30,000-bbl holding tank. The tank is a flat bottom, floating-roof tank without cover. The tank has a floating suction line attached to the floating-roof. Product is received from the Rocklin breakout tank that is used for aviation and diesel fuels. The KMP pipeline from Rocklin to Erle Junction is 8 inches and from Erle Junction to Beale is 6 inches. Transit time is 9 hours with product moving at about 5 mph, providing a receiving rate of 1,350 bbls/hr into the KMP holding tank BE1. Product is filtered upon receipt through a Warner Lewis Model HP-1000 horizontal vessel rated at 1,000-gpm. This is a micronic filter for particulates and does nothing for water removal. Tank BS&W is drained into a 6,000-gallon wastewater tank. Wastewater is drained prior to each receipt and after heavy rains. The water drain line is equipped with a visa-flo gauge and draw off valve for water/fuel interface monitoring. The wastewater tank is emptied about twice each year.

SDA is aspirated in the concentrated form into the line and tank-heel at the beginning of each receipt, proportional to the expected quantity of the receipt. FSII is proportionally injected into the line using a Milroyal model MRI-69-113S17 injector as the product is received into the holding tank. This injector is equipped with a Rockwell Meter which measures the quantity of FSII injected and is then compared with the before

and after stick readings on the 6,000-gallon FSII tank. The FSII tank (BE-A1) has a pressure vacuum relief and breaths to the atmosphere. The FSII tank is replenished on average twice each year through DESC contract delivery by tank truck.

#### **7.1.24 Beale AFB**

In November 2000, Beale AFB found a gel-like substance when draining the F/S sump of Kovatch R-11 97L-358 on two occasions. On December 1, 2000, checkpoint personnel noticed globs of apple jelly coming from the F/S sump on R-11 refueler 98L-005. This was followed by periodically obtaining 50-ml trace quantities of apple jelly. Absorption media elements were installed in the refuelers during August 2000.

On December 13, 2000, traces of apple jelly contaminant were found in the F/S sump on MH-2C # 81W0104. Since there were no replacement elements available, the housing was cleaned and the hosecart was returned to service. On December 13, 2000, two other hosecarts (81W0109 and 81W0111) were opened and examined; no evidence of apple jelly was found.

On January 2, 2001, hosecart 81W0104 was re-inspected (filters removed) and 700 ml of apple jelly was found inside the vessel near the sump. The 700 ml sample from the hosecart along with apple jelly found in the sump of R-11 97L171, were forwarded to the Mukilteo Laboratory for analysis. Test results were received on January 17<sup>th</sup> for R-11 97L038 and hosecart 81W0104. The Mukilteo Laboratory reported that the samples contained FSII and water. No other constituents were identified. According to the laboratory reports, FSII made up 75.7% of the R-11 sample and only 38% of the hosecart sample. The R-11 sample was described as the “truest form of apple jelly” that they have seen at the Mukilteo Laboratory.

JP-8 for Beale is refined at ARCO, Ferndale, WA and tendered into DFSP Selby at Crocket, CA. From there it enters the Kinder/Morgan Pipeline (KMP), passing through their Concord, CA terminal and on to their Rocklin, CA terminal. From Concord it enters KMP's Chico, CA pipeline onto Erle Junction where it is cut over to Beale AFB. It then

goes into KMP's 30,000-bbl tank located next to the Beale tank farm. Once the product passes quality testing, tank lockouts are removed and Beale is then able to draw from the tank as needed. Additives (FSII and SDA) are injected into the line where it comes out of the ground, prior to passing through a 10 micron Velcon horizontal filter and into the KMP tank. The fuel also passes through a metering device just after leaving the filter chamber then on to the tank inlet that ends in a mixing nozzle. The tank has a floating-roof. The FSII tank and the injector pump are about 50 feet from the injection point.

Beale normally receives about 100,000 gallons of JP-8 twice a week. Issues are 25,000 to 30,000 gallons per day. Because inventory minimum levels were increased in December 2000, smaller and more frequent receipts are required. Settling times between receipts are less. Due to a history of water in the receipts, a haypack dehydrator was installed January 5, 2001, but the system is not operational. The contractor has not yet released it to the Air Force. This system makes no provisions for the proper containment and collection of water discharged from the vessel. Normally, when a dehydrator is installed, the separated water discharges into an oil/water separator. The visit team was told that the state of California EPA would not approve an oil/water separator.

The JP-8 fill stand at bulk storage has one Facet F/S model number VFCD-D-6N39SB-3S630FD. Six CA43-3SB coalescer elements and three SS330FD separator elements are required. There are two operational 15,000-bbl tanks (#10 and #11) both of which have geodesic domes and floating pans. Due to design of the geodesic domes, blowing rain penetrates the vents on the skirts of the domes. The vents are louvered, approximately four feet wide and surround the periphery of the tank. Both tanks have cone-down bottoms with center sumps and the discharge suction lines are positioned directly above the sumps. The tanks are equipped with product recovery systems, which discharge directly through 1½-inch lines into 30-gallon product recovery tanks. Collected water and fuel are drained into a 600-gallon bowser for separation and proper disposal. Tank water bottoms are inspected daily and drained as needed.

A third tank, #14, has been inoperative since 1996 and is programmed for extensive repair including a new bottom. This is a 10,000-barrel tank. Upon completion of this project, the availability of this tank should reduce the frequency of receipts and will accommodate longer settling time.

Beale AFB has four Kovatch R-11s and one Oshkosh R-11 in JP-8 service, as well as eight MH2C hose carts, which have NSN 4330-00-983-0998 filter elements. Five R-11 refuelers are used for JPTS support, three Oshkosh and two Kovatch.

Products stored at Beale AFB are JP-8 and JPTS. There are three above-ground steel JPTS tanks in the bulk storage area. These tanks have fixed dome roofs and there are no vents that allow rain to penetrate the tanks. These tanks are equipped with product recovery systems that facilitate removal of any water present in the tanks. Delivery of JPTS is by 20,000-gallon railcars. Prior to off-loading, the railcars are inspected for water and any water found is drained. Beale AFB personnel report they have not experienced any apple jelly problems in JPTS.

Data from the FAS were obtained and analyzed. [17] One interesting trend was noted in the data. Whenever there was even a slight increase free-water content it was followed by an increase in differential pressure in the water-adsorbing filters. A differential-pressure drop followed each of these pressure increases. While the pressure increase is expected, the drop in differential pressure that follows is not. This may indicate that material in or on the water-adsorbing elements is being washed downstream. Figures 2 and 3 present some of the FAS data that demonstrate this trend.

Beale AFB, 98L-0005

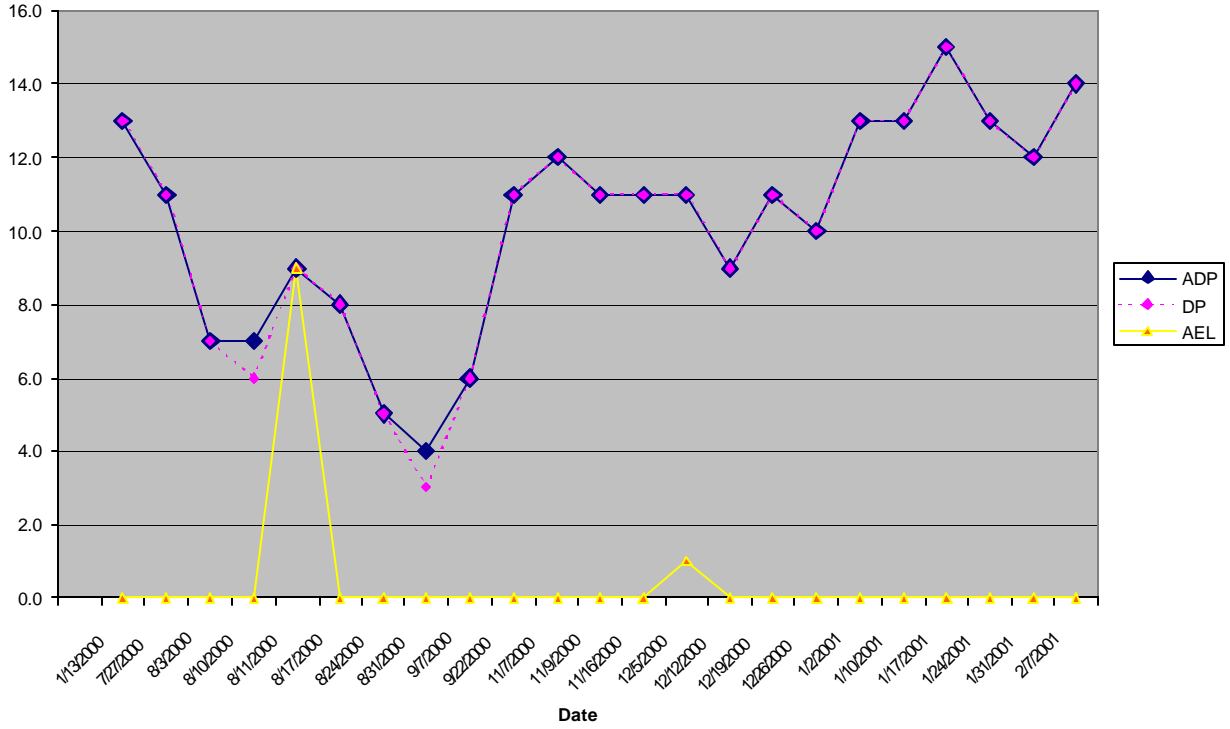


Figure 2. FAS Data From Beale AFB, 98L-0005  
Beale AFB, California 98L-0024

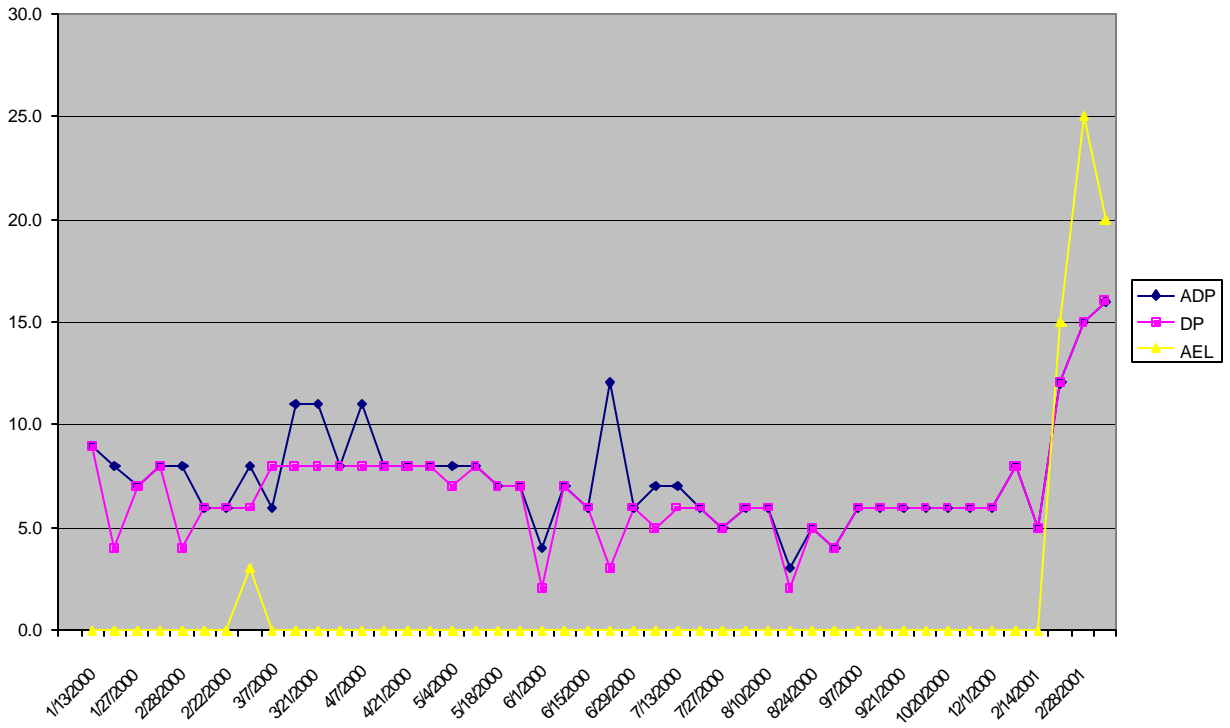


Figure 3. FAS Data From Beale AFB, 98L-0024

### **7.1.25 Fairchild AFB**

Fairchild AFB first encountered apple jelly on December 13, 2000, after differential pressures on the filter-separators of four Kovatch R-11 refuelers rapidly increased. In addition, apple jelly was drained from the cargo tank sump (upstream of the F/S) from one truck. The R-11 refuelers were equipped with Facet water absorption media elements during June 2000. The apple jelly was traced back to bulk tank #4, where they drained two or three quarters of apple jelly each day for several days. Following a transfer between Tank #4 and Tank #1 (20,000-bbl), apple jelly was also identified in Tank #1. Tanks #1 and #4 both have center-down cone bottoms with the suction lines located immediately above the bottom, sediment, and water sumps. Storage operators report that following heavy rainstorms, it is not unusual to drain 30 gallons of water from Tank #4. Little water is found following product receipts. An active tank and vehicle sump-draining program has significantly reduced the amount of apple jelly drained from bulk tanks and vehicles, though apple jelly continues to be found periodically.

JP-8 receipts range from 10,000 to 20,000 bbls. The JP-8 is moved by barge from DFSP Portland OR where it is loaded on barges that travel up the Columbia River for approximately 35 hours prior to arrival at DFSP Pasco. It takes about 12 hours to off-load a barge into Chevron's 30,000-bbl tank at Pasco, WA. After the tank has settled approximately a day and a half, the tank is sampled. Following certification, the product is scheduled into the Chevron pipeline for delivery (approximately 150 miles and 60 hours later) to the Chevron Detection/Injection point. At this point the fuel passes through two filter separators used simultaneously, in parallel. FSII and SDA are injected approximately 25 feet downstream of the F/S. There is no breakout tank at the Chevron Detection/Injection station, and the fuel moves directly into bulk storage at Fairchild AFB. The JP-8 moves through the Chevron Detection/Injection station at a rate that ranges from 16,800 to 31,500 gph through a six-inch pipeline. The above-ground FSII storage tank is not equipped with moisture control (such as a desiccant or nitrogen blanket) but has a standard vacuum pressure vent and breathes to the atmosphere. The Government owned deliveries of FSII (6,000 tank trucks) are sampled at the loading point, but FSII in the



Chevron tank is not sampled. FSII and SDA are injected with Gates City Injectors. The points of injection are approximately 10 inches apart. The Chevron facility is an automated station, though it is manned during receipts.

The bulk storage at Fairchild AFB consists of two fixed-roof 20,000-bbl tanks. One is equipped with a vapor recovery dome. In addition, there is a 5,000-bbl tank, and a 30,000-bbl floating-roof tank with a geodesic dome designated as Tank #4. The geodesic dome on Tank #4 is ineffective in keeping water from entering the tank. The floating-roof in Tank# 4 leaks due to an ineffective tank seal. FSII from the tank water bottoms was identified as damaging the epoxy around the tank perimeter. The product recovery system is ineffective.

Fairchild has 10 assigned R-11 refuelers, 6 Kovatch and 4 Oshkosh. Water absorption (JP-8+100) elements were installed in these refuelers during the period from June through December 2000. There are five hydrant servicers, 3 R-12Cs and 2 R-12s. The R-12C does not have de-fuel capability.

Fairchild AFB has a Type II and a Type III hydrant system. Hydrant System A is a Type II system with 10 each Bowser 842 vertical F/S vessels used to issue to a truck fill stand and KC-135 aircraft. This system is being replaced with a Type III system that is currently under construction. Hydrant System B is a Type III system with seven horizontal API Group II, Class B, 600 GPM vessels. The F/S vessels were manufactured by Facet (CFCS-D-7K395B-1-2 636FM). They use seven CA38-35B coalescer elements and two SS636FF separator elements. Two F/S vessels are used to filter transfers from bulk storage and five are used for hydrant issues. These F/S are flow rated at 600-gpm and are all the same model and manufacturer. The hydrant systems are normally used to refill JP-8 refuelers in lieu of traveling to the bulk storage area.

FSII readings on fuel receipts range between 0.09 vol% and 0.15 vol%.

### ***7.1.26 US Oil & Refining Company***

US Oil & Refining Company, which supports McChord AFB, refines 36,000 bbls of North Slope Alaska crude each day. They produce gasoline, kerosene, diesel, bunker fuels, and asphalt. The production yields about 10,000 bbls of vacuum gas oil a day. From this, they produce Jet A for Boeing and JP-8 for McChord. Jet fuel is manufactured continuously and they ship two to three times per week. The increased demand for JP-8 at McChord has pushed the refinery to the limit and they operate at maximum capacity. During 1996, US Oil bought the pipeline that historically supplies McChord AFB. They constructed a one-mile section of 6-inch diameter pipeline to connect the refinery to the old line. The pipeline previously pushed product from barge deliveries at the port to McChord. There was a history of water problems during this period, which seems to have been abated with the direct line from the refinery.

U.S. Oil produces a straight run jet fuel, and they use a “Merichem” caustic for removal acid and color bodies. The Merichem process does not require a water wash. Salt towers are used to extract moisture. Following caustic treatment, the fuel passes through two clay beds that are installed in series. An antioxidant is injected downstream of the clay filters, and the product is then held in one of the two 30,000-bbl tanks for Quality analysis and certification. Corrosion Inhibitor (Unicor J), FSII, and SDA are injected as the product is put into the dedicated pipeline for transfer to McChord. A Blend-Pak Plus Injector System made by Gates City Equipment, Atlanta, GA is used to inject the additives. Injected quantities are metered and closely monitored, and the system shuts down automatically when variances are unacceptable. No filter separator is provided for pipeline transfers, though F/S vessels are provided at the truck rack where Jet A is loaded for Boeing. Bottom water and sediment are drained from the tanks prior to each pipeline shipment. Only 60 feet of the 14.5-mile pipeline is above ground. Pipeline drag reducer (PDR) is not used in this dedicated JP-8 supply line, though it was not clear whether it is used in the movement of Alaskan crude.

### **7.1.27 McChord AFB**

The first encounter apple jelly at McChord AFB occurred during the fall of 1999 when apple jelly was noticed in the fill stand F/S vessel at bulk storage. There are three Warner Lewis horizontal filter vessels installed on the bulk storage fill stands. These are the old DoD standard vessel with Banner NSN 4330-00-983-0998 elements. The bulk storage supervisor, said when they noticed the substance, they didn't realize what it was and learned later that it was likely apple jelly. This experience motivated the bulk storage personnel to implement an aggressive tank bottom, water-draining program beginning the fall of 2000. They drain or perform a product recovery after every receipt and twice a week, minimum, on inactive tanks. This effort is likely the reason why no apple jelly has been seen in the F/Ss downstream of the bulk tanks since the fall of 1999. However, on February 13, 2001, the receipt F/S on the six-inch pipeline from the US Oil and Refinery was overwhelmed with a dark brown sticky substance. There are two 600-gpm API 1581 Group II Class B Facet horizontal F/S installed on the receipt line at the custody transfer point. The F/S differential pressure began increasing on each of the previous three receipts, which were 7,000 bbls each. The F/S vessels are in parallel and are rotated every other receipt. When the differential pressure reached the maximum allowable of 15 psi, the F/S vessels were shutdown, and upon removing the elements from the first vessel, personnel found the elements completely coated with the dark brown sticky material. The elements from the second vessel, which had also reached the maximum on differential pressure, were not completely covered, as was the case with the elements from the first vessel. An element from the first vessel having an abundance of the dark brown material was forwarded to the US Air Force Fuels Laboratory at Wright Patterson for an analysis. A second element was provided to the product supplier, the U.S. Oil & Refining Company. No finding had been reported at the time of our visit.

In its original liquid state, the heavy liquid penetrated the pleated paper filter and lodged in the outer coalescer sock portion of the element. It is assumed that a portion of the material washed through the element during the three 7,000 bbl transfers, as the differential pressure increased steadily.

Approximately 80% of the JP-8 product is received from US Oil and Refining via the 6-inch pipeline. This is a dedicated pipeline that extends from the refinery to McChord AFB, a distance of approximately 14.5 miles. The remaining 20% of product comes from the Manchester DFSP and is delivered by tank truck. Tank trucks are received from Manchester three days a week. The pipeline-receiving rate is 435 gpm and the hydrant transfer rate is 1,000 gpm. Two or three pipeline shipments are received each week. Product is received with an average temperature of 46°F in January and an average temperature of 69°F in August. The average FSII receipt content ranged from 0.10 vol% to 0.14 vol% with an average of 0.12 vol%.

McChord AFB has four above-ground steel tanks equipped with floating pans and geodesic domes. Tank #A-5, a 12,000-bbl tank, has a leaking geodesic dome. Because of frequent rainstorms and high winds in the Tacoma area, removing water from this tank is required often. The capacities of the four tanks are 5,000 bbls, 12,000 bbls, 17,000 bbls and 20,000 bbls, providing a total capacity of 54,000 bbls. Bulk storage personnel monitor these tanks closely for water.

This system has an automatic tank gauging system that measures water at the datum plate near the side of the tank. All four tanks were reported as having cone-down bottoms with a 7% slope. The suction lines are located directly above the center sumps. For the largest tank, it would require 9,000 gallons of water to reach the ATG system.

McChord AFB has ten R-11 refuelers (5 Kovatch and 5 Oshkosh) and four R-12 Hydrant Servicers assigned. Absorption media elements were installed June 2000-October 2000. Two Type III constant pressure hydrant systems are used primarily for support of wide body aircraft. Each system has two above-ground 10,000-bbl operating tanks. Tanks M-3 and M-4, completed two years ago, do not have the typical suction line over the sump. It was also noted, that the pump room in one system is without heat. The hydrant system supervisor reports that no heat in the pump room is better because of the reduction in thermal expansion when the systems are inactive.

### **7.1.28 Hill AFB**

The first “recent” finding of apple jelly at Hill AFB occurred during late December 2000, following a lull in flying activities resulting from “snow days” and the Christmas break. Small amounts of apple jelly were found in the filter housing of Kovatch R-11 refuelers (98L079 and 98L080) following a rapid increase in filter differential pressure. Water absorption media elements were in use and high differential pressures cause premature requirement to change elements in other trucks, including Oshkosh R-11, though no apple jelly was observed in the other refuelers. The weather during December cycled from very cold to warm with rain and then to very cold again. The JP-8 is refined at the Sinclair refinery in Wyoming at temperatures above 40°F degrees during the Winter and cools as it is transported by truck through the mountain passes to as low as 20°F upon arrival at Hill AFB.

A couple of years earlier when receiving by pipeline, personnel who had been at Hill AFB reported that they frequently saw apple jelly-like material; however, the fuel received by tank truck from the Sinclair Refinery in Wyoming is cleaner and drier.

JP-8 is received at Hill AFB without filtration, though a filtration system is currently being installed. Hill AFB normally receives eight to ten 7,000-gallon tank trucks that are pulling 3,000-gallon tank wagons. The product originated from the Sinclair Refinery in Rawlins, Wyoming, and as mentioned above, is subject to significant cooling during winter transport through the mountains between Wyoming and Utah.

Once during each six-month period, Hill AFB receives JP-8 by pipeline to exercise that receipt mode. The pipeline is a 6-inch diameter line that comes from the Chevron Refinery near Salt Lake City. No pipeline drag reducer (PDR) is used in this line. However, it was reported that the Pioneer Pipeline from Sinclair’s Rawlins Refinery, which does use PDR in diesel movements, connects to the Chevron line and provides approximately 100,000 bbls of Jet A to Delta Airlines at the SLC Airport each month. Additionally, fuel is occasionally pumped from SLC Refineries through the Chevron

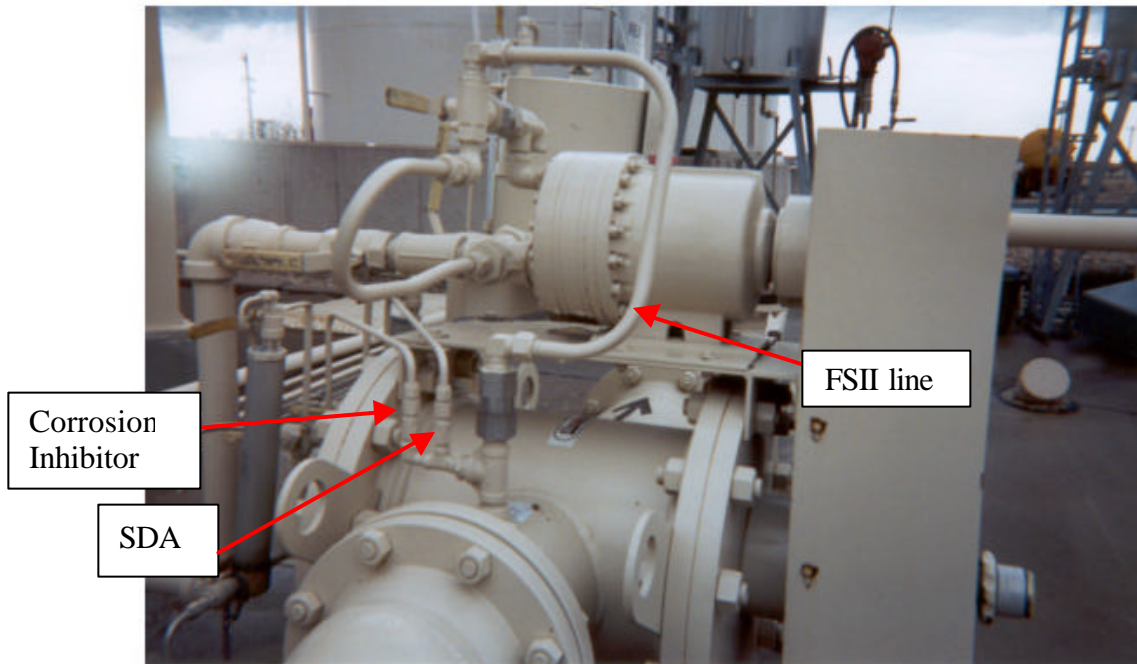
Pipeline to Pasco, Washington. Hill AFB is unique in that three additives are injected on base as the fuel is received: Corrosion Inhibitor, SDA, and FSII. Prior to injection, the fuel is filtered through one of two clay filters (manufactured by Eaton Metal Products). These on-base clay filters (see Figure 4) are set in parallel and only one of the two filters is equipped with a pressure differential gauge.

The three additives are injected using a Hammonds injector (model # D25XFCPHHHC / 1400-D25-1S-1S). The three additive streams are directed through a manifold into a common nipple prior to injection (see Figure 5), mixing the additives prior to injection. The three additives enter the pipeline upstream of the paddle wheel that powers the injector pistons.

Neither of the two FSII storage tanks is equipped with moisture control, such as a desiccant or nitrogen blanket. Fuels personnel knew neither the pumping rate or the inside diameter of the pipeline.



**Figure 4. Clay Filters at Hill AFB**



**Figure 5. Additive Injection at Hill AFB**

JP-8 is stored in four bulk tanks. The two largest (a 25,000-bbl tank and a 55,000-bbl tank) have fixed-roofs with floating pans. The two smaller tanks (an 18,000-bbl tank and a 12,000-bbl tank) have geodesic domes with floating-roofs. All four tanks have the self-cleaning design sloped to the center with the suction line directly over the sump, and fully epoxy coated. The BS&W drains or product recovery systems are ineffective and have been abandoned. Water is drained into a bowser through a long hose that goes over the tank dike. The sump of the tank designated as the issue tank is drained daily and the other tanks are drained weekly. Samples were taken from the bowser connected to the issue tank. The first one-quart sample, drained from the low point was a yellowish mixture of water and FSII, with no fuel interface. A drum thief was used to obtain a more representative sample. The fuel phase of the latter sample was unusually yellow, though the water phase seemed unremarkable. A thin layer of substance at the fuel water interface appeared typical of microbial growth. This sample was sent to the Air Force bio-environmental health laboratory for testing.

Two 40,000-gallon, horizontal tanks (Tanks # 39 and #40), which receive fuel from bulk storage without filtration, supply the truck fill stands. Storage operators usually drain from one pint to one quart of “orange colored water” from the tanks each morning. The fill stands are equipped with two vertical F/Ss (manufactured by Keene Corporation, model #330 V600 manufactured in 1974). These F/S vessels use the old DoD elements, FSN 4330-00-983-0998. Currently, this is the first filtration that JP-8 delivered by tank truck is subjected to before it enters the flight line refuelers. Most of the jet fuel issued by Hill AFB is JP-8+100. The +100 additive is injected downstream of the fill stand F/S vessels.

Hill AFB has fifteen assigned R-11 refuelers. Seven of these are 1989 model Oshkosh refuelers, three are 1991 model Oshkosh refuelers, and the remaining five are Kovatch 1998 vintage refuelers. According to the fuels management personnel, apple jelly has only been found in the 1998 Kovatch model R-11. However, the refueling vehicle maintenance manager stated that he finds apple jelly in the filter vessels of all the refuelers. Since his records showed that he had recently changed the filter elements in a Kovatch R-11, #98L083, he was asked if we could examine the filter element. The filter showed no evidence of apple jelly; however, the element was the filter coalescer type (63387TB) and not a water absorption type. This situation is exacerbated by the fact that refueler 98L083 is a designated JP-8+100 truck. Water absorption elements should be used for this application in accordance with T.O. 42B-1-1 because JP-8+100 disarms coalescer elements. The elements change information was reviewed for all fifteen vehicles and it appeared that one other R-11 (98L081) might have the old coalescer type elements based on the change dates. Later, it was determined that the elements, whichever type, had been changed earlier, but the automated report had not been updated. Water absorption elements were apparently installed in most of the R-11 refuelers during September and October of 1999. Since that time, the records reflect that two Oshkosh and three Kovatch R-11 experienced premature filter failure (each after 9 months or less installed time).



A review of product receipt records for 216 deliveries showed that FSII level on fuel receipts ranged from 0.0 vol% to 0.17 vol%. Greater than 90% of the shipments were between 0.10 vol% and 0.12 vol%.

Less than 4% of 814 samples reviewed showed any water at all in the AEL testing of flight line refueling equipment. Five of the 814 samples failed, having from 15 ppm to 20 ppm of free water. Approximately 15,000 to 20,000 gallons of JP-10 are downgraded to JP-8 each year.

#### ***7.1.29 NAS Corpus Christi***

Though a small operation with two 6,000-bbl tanks, NAS Corpus Christi has an aggressive fuel filtration program. The fuel is filtered upon receipt into Tank #1. After settling and draining any bottom water and sediment, the fuel is transferred from Tank One through a filter separator and then through a water absorption monitor into Tank #2. As the fuel is issued from Tank #2 to the fill stand it is filtered once again through separate filter separators and water absorption monitors. The two fixed-roof storage tanks have inverted cone bottoms. Fuel is drained manually into a bucket to check for water and then placed in the fuel reclamation tank. Doss Aviation provides along side fuel servicing using a fleet of new Isometrics built Refueling trucks. These are equipped with Beta Systems filter vessels that use the I-63887TB coalescers and the SO-6336S separators, and provide the fourth filtration prior to the fuel entering aircraft.

NAS Corpus Christi has no history of apple jelly. While it may be argued that this is because it is located in a warm climate (Texas Gulf Coast), temperatures do drop into the 30°F range on some occasions. There are two other key differences common to most Navy turbine fuel storage facilities that may be a factor in precluding apple jelly formation at NAS Corpus Christi. First, the product is stored in fixed-roof tanks that are not subject to rainwater intrusion. Second, the fuel goes through multiple, three-stage filtrations (coalescer, separation, and absorption).

### **7.1.30 TEPPCO Bossier City & Barksdale AFB**

The tanks at the Texas Eastern Pipeline Products Company (TEPPCO) terminal at Bossier City are not equipped with a means to drain bottom sediment and water (BS&W) from the tank bottoms prior to transferring the product to Barksdale AFB. Rainwater entry into JP-8 storage tanks at Barksdale AFB, and particularly Tank #4 is a significant problem.

The design of the Bossier City tanks is such that each time product is received any BS&W in the center tank sump is mixed with the incoming product. The lines used to receive JP-8 into these tanks enter from the bottom of the two tanks into a well that is approximately 16 inches deep. The receipt line rises approximately one inch above the tank floor and is capped by a diffuser resulting in the mixing each time new product is received. On the tank drawings, the receipt lines for the two tanks are identified as issue lines. New issues line have been installed and, while no drawing or photographs of the line inside the tanks were available, TEPPCO personnel report that the issue line draws fuel from approximately eight inches above the tank bottom. FSII and SDA are injected downstream of the bulk tanks as the JP-8 is transferred to Barksdale AFB via a 4-inch pipeline that stretches 3.98 miles to the base.

The SDA injection point is two feet upstream of the pump and the FSII injection point is four feet downstream of the pump. Government supplied FSII is stored in a bulk tank with a pressure relief system. The tank is not equipped with a desiccant and the FSII in the tank is not sampled for water content. We asked that the QSR draw a sample to be tested for water. The test results indicated that the moisture content of the FSII in this tank was within the specification level. Approximately three feet downstream of the point of FSII injection, the fuel enters a "Coriolas" meter manufactured by Smith Meter Company. The Coriolas has a seven-foot long venturi (approximately 2 inches in diameter), which increases the pressure on the upstream side and dramatically reduces the discharge pressure into the four-inch transfer line to Barksdale. The pump pressure was noted to be 150 psi and the pressure downstream of the venturi was 50 psi. The four-inch

line stretches 3.98 miles to Barksdale AFB where receiving pressure is approximately 4 psi. It is highly unlikely that this arrangement would result in adequate blending of the FSII into the fuel.

Product samples drawn upstream of the receipt filters at Barksdale AFB during our investigation were cloudy; however, downstream of the filters the product was clear and bright. The base personnel report a history of problems with wide fluctuations in the quantity of FSII and SDA in the fuel. However, laboratory records show that since August 2000, the quantities of FSII in the shipments have been consistently between 0.15 vol% and 0.17 vol% and the fuel conductivity readings range from 250 to 300 CUs.

The most significant amount of water in the Barksdale AFB fuel system enters the fuel at Tank #4, a 40,000-bbl tank that has experienced a rim fire, is out-of-round, and leaks badly during periods of heavy rains accompanied by moderate wind. As much as 500 gallons of water have been drained from Tank #4 after periods of heavy rain. The water problem is complicated by ineffective product recovery/water removal systems on several tanks. Another factor contributing to the water problem at Barksdale AFB is that inventory capacity significantly exceeds the average inventory. During the site investigations, a number of old floating-roof tanks modified with geodesic dome roofs were found to allow water entry into the tanks through the dome vents during periods of rain accompanied by moderate to heavy wind. When the product levels in the tanks are low the large area of exposed interior tank wall surface facilitates the collection of moisture and allows it to drain from the tank walls past the floating-roof or floating pan seal and into the tanks.

### ***7.1.31 Niagara Falls ARS***

The 914<sup>th</sup> Air Wing receives fuel via tank truck from the United Refinery located in Warren, Pa. An average of four tank trucks is received each day Monday through Friday. The trucking company dedicates these tank trucks exclusively to JP-8. The normal transient time to Niagara Falls is 2.5 hours. Product receipt temperatures have not been

recorded. During January 2001, the average storage-tank temperatures range from a high of 33°F to a low of 28°F with an average of 28°F.

Two 300-gpm Velcon horizontal receipt filter separators are installed in the pump house. Elements are changed at approximately 18-months based upon differential pressures. Also, there are two 600-gallon issue filter separators used to fill refuelers and transfer product to the NY ANG which support KC-135's with an installed type three constant pressure system. All of these vessels are equipped with Velcon coalescer elements (P/N I63885TB) and separators (SO-636V). Elements in these vessels are changed every 12 to 18 months based upon filter differential pressures.

The 914<sup>th</sup> AFRS has three above-ground steel tanks. Two of these have fixed roofs while the third tank is equipped with a geodesic dome. The two fixed-roof tanks slope to a center sump, while the tank with a geodesic dome slopes from the center to the outer edge of the tanks. The floor and walls of the two fixed roof tanks are 100% epoxy coated. In the geodesic dome tank, the bottom and five feet up the tank walls are epoxy coated. No specific information about tanks coatings is available. Water removal from the tanks is very difficult because the product recovery systems are equipped with ¾ inch lines. Apple jelly was found while draining the product recovery tank for the tank with the geodesic dome during our visit on 20 Feb 01. Product temperatures are from the automatic tank gauging (ATG) system and no historical temperature information is available. FSII levels were at 0.11% on February 8<sup>th</sup> and 0.10% on February 9<sup>th</sup>. Conductivity readings ranged from 316 to 490 CUs.

Apple jelly-like material was experienced earlier in the winter when draining the sumps on the receipt filter separators. Recently, apple jelly again appeared when draining issue filter separator 3. This was immediately after transferring product from Tank B.

The AFRS or ANG units have not experienced AJ problems in their R-11 refuelers (which are equipped with Facet water absorption media elements) or the one assigned Tri-State R-12 hydrant sevicer. Both units have a mix of Oshkosh and Kovatch R-11s. They

have found apple jelly only on their receipt and issue filter separators on the AFRS system.

Product is transferred to the ANG from the AFRS bulk storage system. The ANG has two AST operating tanks and a type-three hydrant system, which they use to support KC-135's. The ANG storage tanks have fixed roofs and center sumps with a 1.5-inch water drain line. The two tanks are completely epoxy-coated. JP-8 is received by the ANG through two 650-gpm vertical filter-separator vessels manufactured by Facet. These vessels are equipped with six Facet coalescers (P/N 6N39SB) and three separator elements (P/N 3-633F). The issue vessel, used to fill trucks, and the hydrant system filter-separators are horizontal vessels, also manufactured by Facet. These vessels are equipped with seven coalescers (7-K395 B-1) and two separators (2-S636FM).

## **7.2 General Conclusions from the Site Visits**

The following are general conclusions and comments derived from the site visits. Additional information and comments, based on the knowledge and experience of the C4e team members, but strictly germane to the investigation of apple jelly, are included in the C4e final report.

All of the bases that reported significant quantities of apple jelly had excessive water in the fuel they received or water intrusion (typically rain) into on-base tanks.

Design of fuel-handling facilities and equipment must be considered a factor. The use of fixed-roof storage tanks and discharge (suction) lines specifically designed to avoid the intake of water bottoms would reduce water intrusion and water-promulgation downstream. The Navy's use of a three-stage process for water removal (coalescence, separation, and absorption monitors), compared to the one or two-step process used by the Air Force, would also reduce water/FSII-related problems.

Reducing the amount of FSII injected into the fuel and moving the injection point closer to the aircraft could also reduce water/FSII-related problems. This option must be considered against other logistical concerns such as manpower requirements and infrastructure. A detailed study of this option is required before it could be implemented. Nonetheless, it is worthy of consideration.

The bases that were successful in reducing apple jelly incidents had previously introduced aggressive water removal programs. They had combined this with a reduction in the amount of FSII injected into their fuel.

At some Air Force bases, such as Barksdale, Fairchild, and Grand Forks, reduced fuel inventories had the effect of increasing available tank ullage and thereby increasing the amount of water in partially filled cone-roof tanks. There seemed to be a correlation between these factors and increased instances of apple jelly formation. At these locations, the cone-roof tanks are ineffective in preventing rainwater intrusion. This allows water accumulation on the interior tank walls that can leak past the floating pan or floating-roof seals. When product levels are low, the increased surface area above the floating-roof/pan collects a greater amount of water, which slips past the roof seal into the fuel. This problem is even worse at locations that continue to use floating-roof tanks, including DFSP Ludlow and Edwards AFB. Unfortunately, many of the geodesic dome and floating-roof tanks have been modified with Self-Cleaning tank bottoms, which exacerbates the problem by pumping the tank bottoms into filter housings and other equipment downstream. In this downstream equipment the water/FSII mixtures can react with other materials in the fuel and fuel system to cause additional problems.

The design of the Self-Cleaning fuel storage tanks maximizes the opportunity for fuel-water emulsification and entry into the fuel-distribution system. At the expense of fuel quality, this design intentionally sucks free-water/FSII mixtures and particles from bulk tank sumps and pumps it through filter separators not designed to handle additized fuel. The problem is worse at locations where floating-roof tanks or tanks with ineffective geodesic domes have been retrofitted with a Self-Cleaning tank bottom. Typically, these

tanks have ineffective product recovery/water removal systems. By comparison, little or no apple jelly problems have been reported in NATO or commercial aviation fuel tanks. The differences are the use of receipt filtration, fixed-roof tanks, and the effective water-bottom removal features of the NATO design. At commercial airports, aviation fuel storage tank designs purposefully avoid the intake of tank bottoms into fuel suction (issue) lines, while the opposite is often true at Air Force installations.

The size of the issue line on many newer hydrant systems at Air Force Bases creates a potential for free-water/FSII and other contamination to settle and accumulate in low points in the hydrant system loop. During peak operations this material can be picked up and forced further downstream in the fuel-distribution system. Increased low point draining frequency and seasonal draining--following dramatic temperature drops that cause water and FSII to drop out of fuel--would aid in reducing this problem. Additionally, some Type III hydrant systems are routinely shutdown at night and over weekends. This further promotes accumulation of water/FSII in low points.

The continued use of old DoD standard filter separator vessels, especially when combined with the self-cleaning tank design, jeopardizes fuel quality and provides a means for apple jelly to get into aircraft fuel systems. While marginally effective with JP-4, these filter vessels are ineffective with the denser JP-8, and allow the fuel to bypass the water separation stage. API/IP 1581 filter separators have replaced most of the old DoD standard design filter vessels, those that remain should be modified or replaced. Likewise, the DoD standard design coalescer elements under NSN 4330-00-983-0998 used in the old DoD standard vessels and some modified vessels have an off-the-shelf failure rate approaching 40%, regardless of manufacturer. Such vessels and filters continue in use in some fixed facilities, in Oshkosh R-11 refuelers, and in MH2-C hosecarts. Use of this coalescer element should be discontinued.

The thickest, most viscous apple jelly was always found in R-11s equipped with water absorption media filters. According to one manufacturer [18], water slugs with 20 vol%, or more, FSII can pass through these cartridges. The Navy practice of placing a F/S

element directly ahead of water-adsorbing media reduces the chance that a slug of water/FSII reaches the water-adsorbing element.

Interestingly, JP-8+100 bases, which were the first Air Force units to use the water absorption media elements, have had surprisingly few incidents of apple jelly in R-11 filter vessels. Moreover, apple jelly has not been reported in any of the aircraft that routinely use JP-8+100. The dispersion properties of the additive may help keep the entrained water/FSII in suspension and prevent it from agglomerating, settling out of the fuel or being adsorbed by the absorption cartridges. Also, the JP-8+100 bases tend to have stricter policies regarding the removal of water from the fuel system. Both of these factors probably combine to eliminate/reduce apple jelly formation at these bases.

Apple jelly has been found primarily in issue filters and flightline servicing equipment, following periods of heavy refueling activity.

## 8 APPLE JELLY SAMPLE COLLECTION

DESC provided SwRI with an initial list of 13 sites (See Table 4) to supply with apple-jelly sampling kits. The list was composed of sites with a prior history of apple-jelly occurrences and/or current problems. Each site was sent an apple-jelly sampling kit that included the following: one 500 mL (17 oz.) glass container; one 1-gallon epoxy-lined steel can; and three 125 mL (4 oz) glass containers. See Appendix A for a copy of the sampling instructions sent with the containers.

<b>Table 4. Initial List of Potential Sample Sites</b>		
Grand Forks AFB, ND	Otis ANG, MA	Tinker AFB, OK
McConnell AFB, KS	Barksdale, AFB, LA	Osan Air Base, Korea
Pease ANG, NH	Maine ANG, Bangor, ME	Misawa AB, Japan
Maxwell AFB, AL	Shaw AFB, SC	Kadena AB, Japan
Yokota AB, Japan		

As the project progressed, other locations notified SwRI of apple-jelly contamination. Each time SwRI was notified, a sampling kit was sent to the site for apple-jelly collection. The containers in these sampling kits varied, depending on the quantity of apple jelly



discovered at the site. Some sites had very small amounts of apple jelly to collect, which eliminated the need for the large 500-mL glass containers. Other sites did not require the fuel container because they had already changed out the fuel source upon apple-jelly contamination. There were other sources that received only vial-sized containers. Each situation was different and required consultation with the sites to determine the extent of apple-jelly contamination.

Many of the locations discovered apple-jelly contamination more than once. Each time apple jelly was observed, SwRI worked with personnel at the site to ensure that proper containers were sent for sample collection. Table 5 lists other locations that encountered apple-jelly contamination during the course of the project.

Niagara Falls ARS, NY	Ellsworth AFB, SD	McGuire AFB, NJ
Dover AFB, DE	Westover ARB, DE	Quonset State Airport, RI
Bradley ANGB, CT	Delaware ANG, DE	New York ANG, Scotia , NY
Eglin AFB, FL	Vienna, OH	Lajes Field, Europe
Edwards AFB, CA	Beale AFB, CA	Idaho ANG, Boise, ID
Elmendorf AFB, Alaska	McCord AFB, KS	Gulfport, MS
Fort Sam Houston, TX	Camp Bullis, TX	Fairchild AFB, WA
Travis AFB, Fairfield, CA	Charleston AFB, SC	
Williams International, Walled Lake, MI	Life Flight of Maine, Bangor, ME	

SwRI received 139 samples total, consisting of apple jelly, fuel and other types of samples related to apple-jelly contamination. Appendix B contains a listing of all the samples obtained under the apple jelly project. As samples arrived, they were immediately prepared for laboratory analysis. Preparation consisted of removing a portion of the apple jelly from the sample container and placing it in a 20-mL vial. The only exceptions were samples with extremely high viscosity. These were kept in their original containers because it was not possible to reliably transfer a sample to the small vial. For the first 69 samples, each vial was photographed next to a vial of clean water for comparison. These photographs are found in Appendix C. Examination of these photographs again demonstrates that there are significant visual differences in the apple jelly samples received.

## 9 LABORATORY ANALYSES

### 9.1 Compositional Analyses

As stated above, much was known about the chemical and physical properties of apple jelly at the beginning of this project, including the following:

- It is composed primarily of DiEGME and water, which account for about 98% of the mixture.
- The relative amounts of DiEGME and water vary. The typical ratio is about 60:40 (DiEGME:water) but can vary from that ratio.
- It contains other components such as sodium and organic compounds.
- It is usually brown or reddish brown in color.
- It is variably viscous, from a viscosity approximately that of water to a very thick gel, assuming prior reports were literally accurate.
- Mixtures of water and DiEGME are both corrosive and an aggressive solvent. The severity of corrosion and solvent attack depends on the concentrations of each component and the presence of other compounds (such as aromatic compounds) in the mixture.

We began this project with the understanding that the various properties of apple jelly are caused by one or more of the components other than water and DiEGME. That is because the properties of water and DiEGME alone do not account for all the properties of apple jelly. Water and DiEGME mixtures are known to be good solvents and are capable of dissolving both organic and inorganic compounds, depending on the concentrations of water and DiEGME. It was assumed that the other components of apple jelly are present in the mixture as the result of solvent attack by the water/DiEGME. It was also assumed that these other components are relatively common to the fuel and fuel system (i.e. fuel components, elastomers, coatings, fuel additives, common contaminants such as dirt, rust, salt, etc.). Studies by both the Air Force and the Navy had found some apparent correlation between apple jelly viscosity and sodium content of the apple jelly. Based on these understandings, we conducted numerous compositional analyses of the samples.

Initial compositional tests run included total sulfur, total nitrogen, TAN, total base number (TBN), water content, and DiEGME content. Some of these tests were chosen in part due to the fact that some prior studies had suggested their relevance. Also, our own experience in investigating petroleum product-related problems taught that such tests were an important first step in quickly defining the important issues. These results are presented in Appendix D and discussed in detail below.

### 9.1.1 Total Sulfur

Total sulfur was measured using ASTM method D 5453. The fuel and fuel layer samples were also analyzed for sulfur content for comparison to the apple jelly values. The results are plotted in Figure 6. The plot shows that there is no significant concentration of sulfur in the apple jelly samples as compared to the fuel and fuel layer samples. However, since there is zero sulfur in water and DiEGME, the sulfur level in apple jelly indicates that sulfur is coming from some source or sources. Potential sources include fuel and SDA, but not corrosion inhibitor. (Dimeracid corrosion inhibitors as typically used in JP-8 and JP-5 contain carbon, hydrogen, and oxygen, but essentially no nitrogen or sulfur.)

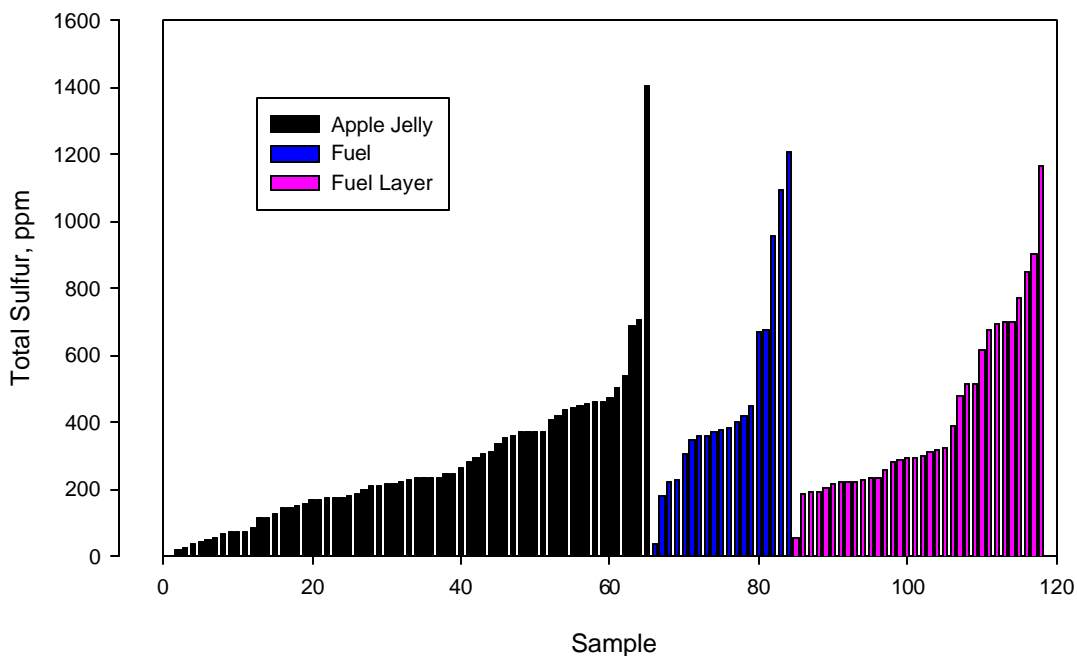


Figure 6. Total Sulfur Results

### 9.1.2 Total Nitrogen

The total nitrogen content of the apple jelly, fuel, and fuel layer samples was determined using ASTM Method D 4629. The total nitrogen results are plotted in Figure 7. In contrast to the total sulfur results, Figure 7 shows that nitrogen concentrates in the apple jelly samples to a far greater amount when compared to the fuel and fuel layer samples. The source(s) of the nitrogen were not available from this analysis. Potential sources include the fuel, fuel additives, and some elastomers.

### 9.1.3 Total Sodium

The total sodium content of the apple jelly, fuel, and fuel layer samples was determined using inductively coupled plasma (ICP) spectroscopy. The results are plotted in Figure 8. As with the total nitrogen results, sodium is present in the apple jelly samples to a significantly greater extent than in the fuel and fuel layer samples. Possible sources of sodium include SDA, salt from salt-dryers, dirt, and contaminated water. Some water-adsorbing filter elements also use sodium polyacrylates, which would be a large source of sodium. Sodium levels in all fuel and fuel layer samples were at or below the detection limit of the ICP procedure used, as expected.

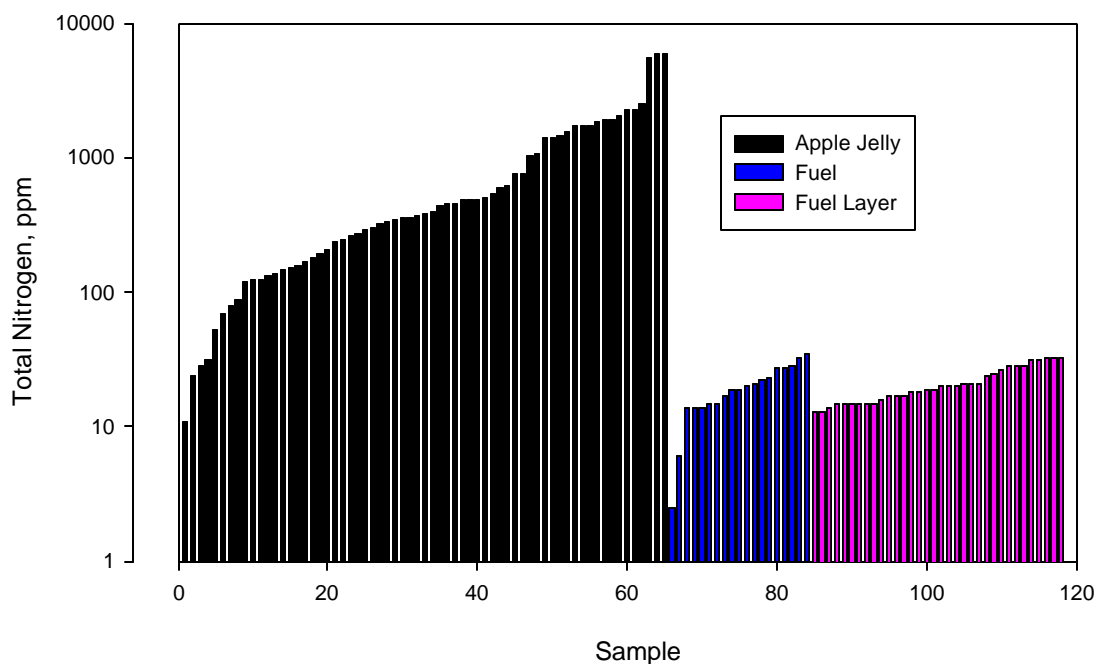
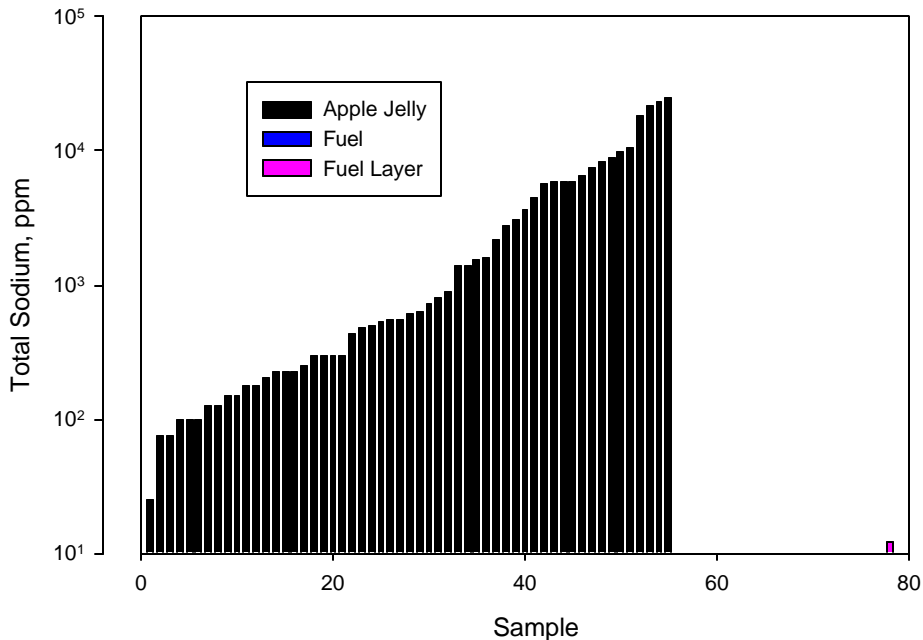


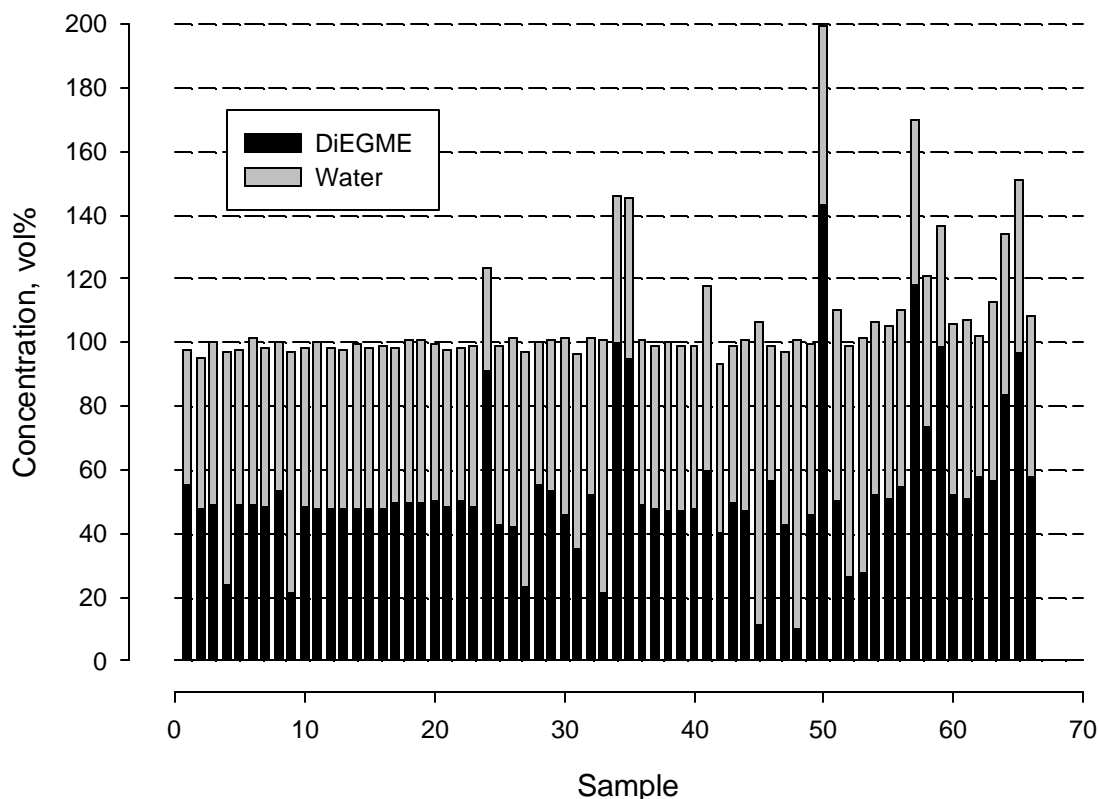
Figure 7. Total Nitrogen Results



**Figure 8. Total Sodium Results**

#### 9.1.4 Water and DiEGME

All the apple jelly samples were analyzed for water content and DiEGME content. Water was measured by Karl Fischer titration. DiEGME content was measured using ASTM D 5006 (water extraction followed by refractive index determination). The results for all the apple jelly samples are plotted in Figure 9. The water and DiEGME concentrations obtained for these samples are all beyond the published measurement range for the methods used. Also, dissolved species in the water/DiEGME mixtures can interfere with the measurement techniques, especially D 5006. As such, the water and DiEGME concentrations reported here should be considered approximate. However, the majority of the results are consistent with previous studies and with the assumption that apple jelly is composed of more than simply water and DiEGME. Note that the total vol% exceeds 100 in several of the samples. Only the thick samples (Brookfield viscosity > 10 cP as explained further in Section 9.2.1) exhibited this abnormality. Obviously, the results with greater than 100 vol% are incorrect. SwRI attempted to analyze the thick samples by gas chromatography without success. Material in the thick samples fouled the gas chromatography column and caused the analyses to fail. These results point out that materials present in the thick samples render such customary analytical techniques inaccurate or useless.



**Figure 9. Water and DiEGME Concentrations**

### 9.1.5 Chlorine

Chlorine analyses were conducted because the ion chromatography results (discussed in Section 9.1.6) suggested that chlorides were present in the apple jelly. One common additive (SDA) and four apple jelly samples were analyzed for chlorine content using ICP. SDA was analyzed because ion chromatography results also showed possible chlorine in the neat additive. The results are given in Table 6. These results show that the chlorine content does vary and may reach levels in excess of those found in the neat additive. Other sources of chlorine include salt-dryers and contaminated water.

<b>Table 6. Total Chlorine Content by Inductively Coupled Plasma Spectrometry</b>	
<b>Sample</b>	<b>Cl, ppm (wt)</b>
Stadis 450	240
AP-001	353
AP-006	64
AP-031	66
AP-044	159

### 9.1.6 *Ion Chromatography*

As mentioned previously, apple jelly is known to contain compounds other than water and DiEGME. Ion chromatography (IC) analyses were conducted on selected apple jelly samples in an attempt to gain additional information regarding the nature of other components of apple jelly. The ion chromatography instrumentation and procedure used was specific to anions. Cations were not detected. The IC results are displayed in Figure 10. SwRI was unable to positively identify many of the peaks (anions) in the IC analyses. This is because many of the ions detected in the apple jelly samples appeared to be complex and unlike the ions typically analyzed in our instrumentation. For this reason, we attempted to identify potential sources of some of the ions by comparing the apple jelly IC results with IC results from materials commonly found in JP-8. Our thought was that significant correlation between peaks from the apple jelly samples and peaks from known compounds would be evidence that the water/DiEGME had extracted components from those compounds. The purpose of the IC analyses was to provide evidence that components of apple jelly had actually been extracted from the fuel or compounds in the fuel. It was beyond the scope of the project to positively identify all the ions found in the apple jelly. The SDA was selected (rather than the corrosion inhibitor additive) for this comparative analysis for the following reasons:

- SDA is known to be a mixture of compounds with a purposefully-prominent ionic nature. This promises a potentially higher number of ions in the IC analysis thereby increasing the possibility of correlation with peaks in the apple jelly samples. This was verified by the presence of numerous strong anion peaks in neat SDA.
- Apple jelly was known to contain significant levels of sodium, nitrogen, and sulfur; all of which are present in SDA but not in the corrosion inhibitor. Also, corrosion inhibitors used in JP-8 are typically of the dimeracid variety, and therefore virtually non-ionic. Of course, these elements can also be present in fuel without SDA so the evidence, if any, could be inconclusive.
- The neat SDA has a dark red-brown color. The components of SDA that give it the dark color could also be responsible for some of the color in apple jelly if

extracted into the water/DiEGME. Again, both the fuel and corrosion inhibitor additive have dark colored components that could be responsible for some of the color in apple jelly.

- Attempts to characterize neat corrosion inhibitor by ion chromatography failed. No significant anions could be detected, as expected.

IC analyses were conducted on 18 apple jelly samples and a sample of SDA. In the figures, the SDA results are in the first row of peaks (the light blue colored peaks). A total of 16 separate peaks were identified in the ion chromatograms although not all 16 were present in every sample. Figure 10 shows all 16 peaks and the corresponding peak area for each of the 19 samples. Notice also that the peak areas differ greatly from sample to sample.

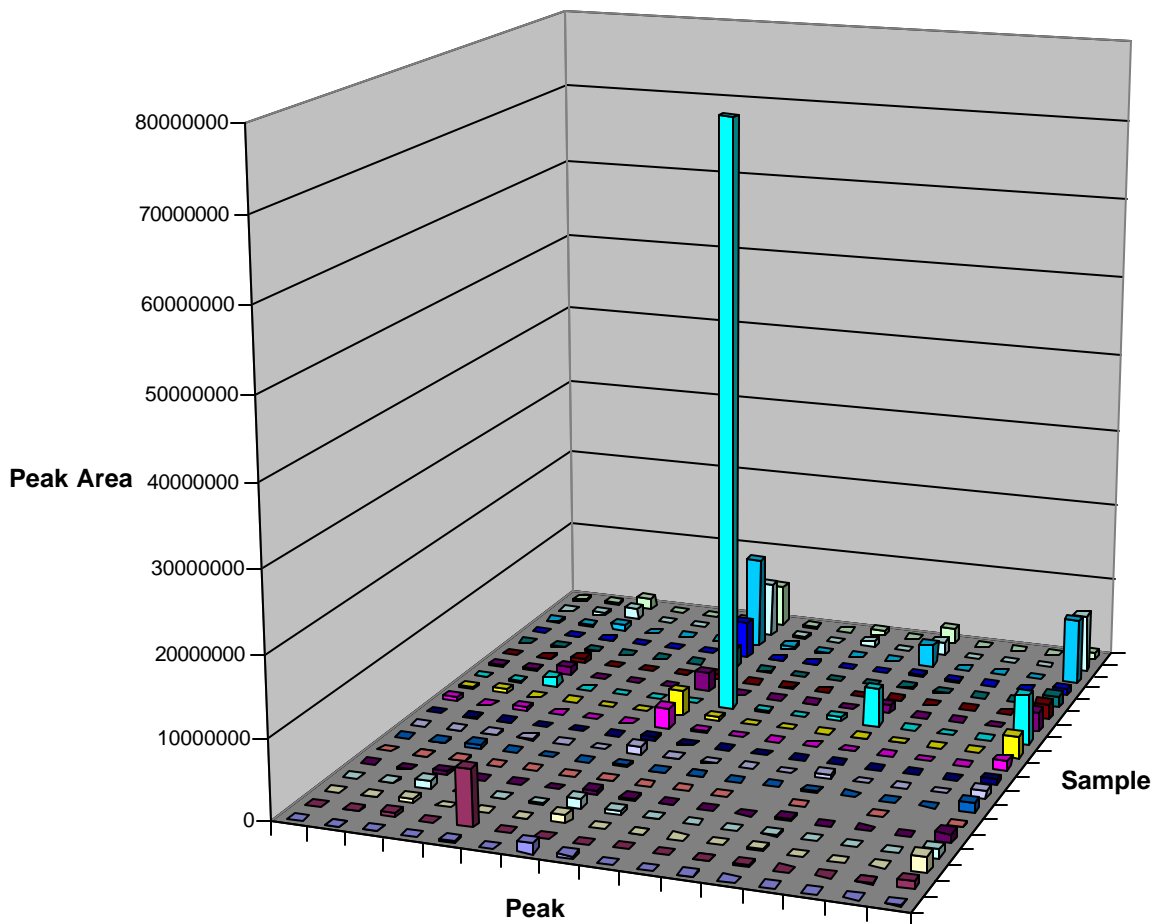


Figure 10. Ion Chromatography Results (All 16 Peaks)



As mentioned previously, SDA was analyzed to help confirm whether components of the additive were present (or possibly concentrating) in the apple jelly. To simplify this comparison, the results were plotted with only the peaks found in SDA. This plot is shown in Figure 11. Examination of Figure 11 confirms that several of the IC peaks found in the apple jelly samples could be attributed to SDA. This in turn demonstrates that water/DiEGME mixtures can extract material from fuel additives. No attempt was made to either identify individual components of the SDA or assign any particular component to a given IC peak. The tendency for SDA components to extract into apple jelly could be a significant factor in the high conductivity of most apple jelly samples compared to water/DiEGME blends (as discussed in Section 9.2.6). It is important to note that not all of the IC peaks correspond to SDA peaks. This confirms that, as expected, the water/DiEGME extracts other compounds during the formation of apple jelly.

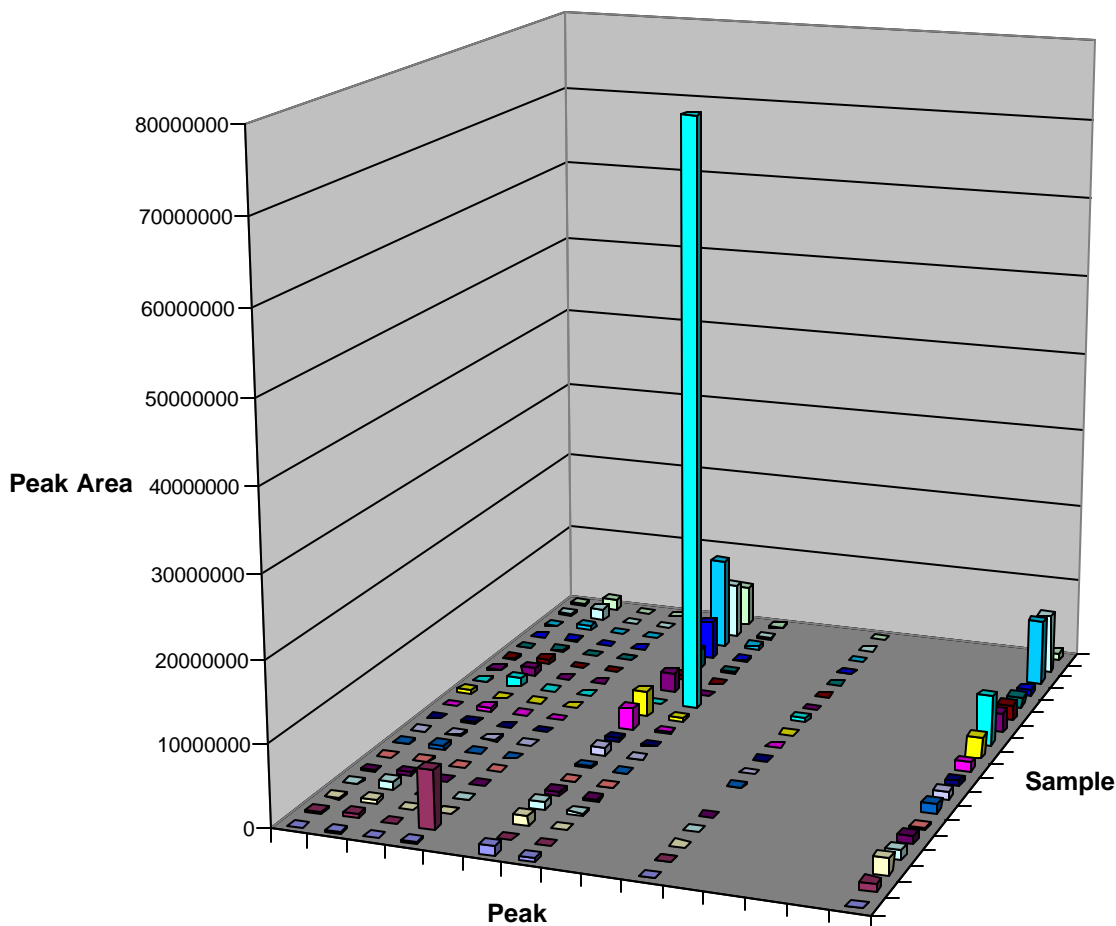


Figure 11. Ion Chromatography Results (SDA Peaks)

## 9.2 Physical and Chemical Properties

### 9.2.1 Viscosity

As discussed earlier in this report, it is well established that apple jelly is primarily composed of water and DiEGME. Before measuring the viscosity of apple jelly samples, the viscosity of water/DiEGME blends at 25°C of varying compositions were measured using a Brookfield rotational viscometer. The water in all the blends was de-ionized, and the DiEGME used throughout this entire report was reagent grade. Results (Figure 12a) indicate that the viscosity achieves a maximum value at about 75%(vol) DiEGME. This behavior indicates that a non-ideal solution is obtained, not surprising given the very significant hydrogen bonding that can be expected when water and DiEGME are mixed. Another observation consistent with this behavior was the noticeable heat of solution generated when the water and DiEGME blends were made. Notice however that even though significant intermolecular interactions via hydrogen bonding is occurring in blend of water and DiEGME, the maximum viscosity increase above an ideal solution relationship is only modest. This maximum viscosity increase is not, for instance, a factor of 100 or 1,000. Also, all solutions of water and DiEGME are Newtonian, as expected. The relevance of these observations will become more evident when the rheological properties of thick apple jelly samples are discussed later in this report.

Brookfield rotational viscosity was performed on 38 apple-jelly samples. Results range from 1.8 cP to greater than 10,000 cP. All results are plotted in Figure 12b. The line between thick and thin apple jelly was set at 10 cP because the range of viscosities for water/DiEGME blends is approximately 1 to 7.5 cP. Because viscosity values observed in apple jelly samples spanned over three orders of magnitude, a logarithmic display of viscosity data seemed appropriate. Therefore, the nearest power of 10, namely 10, was chosen as the discriminating line between thin and thickened apple jelly. Figure 12b shows that 27 samples were thin and 11 samples were thick. Also, all thin samples exhibited Newtonian behavior, and all thick samples exhibited non-Newtonian behavior. The non-Newtonian behavior of all thickened apple jelly is highly significant since it proves that the component responsible for increasing the water/DiEGME blend viscosity is not simply a highly viscous Newtonian fluid. This point is further discussed in Section 9.5.

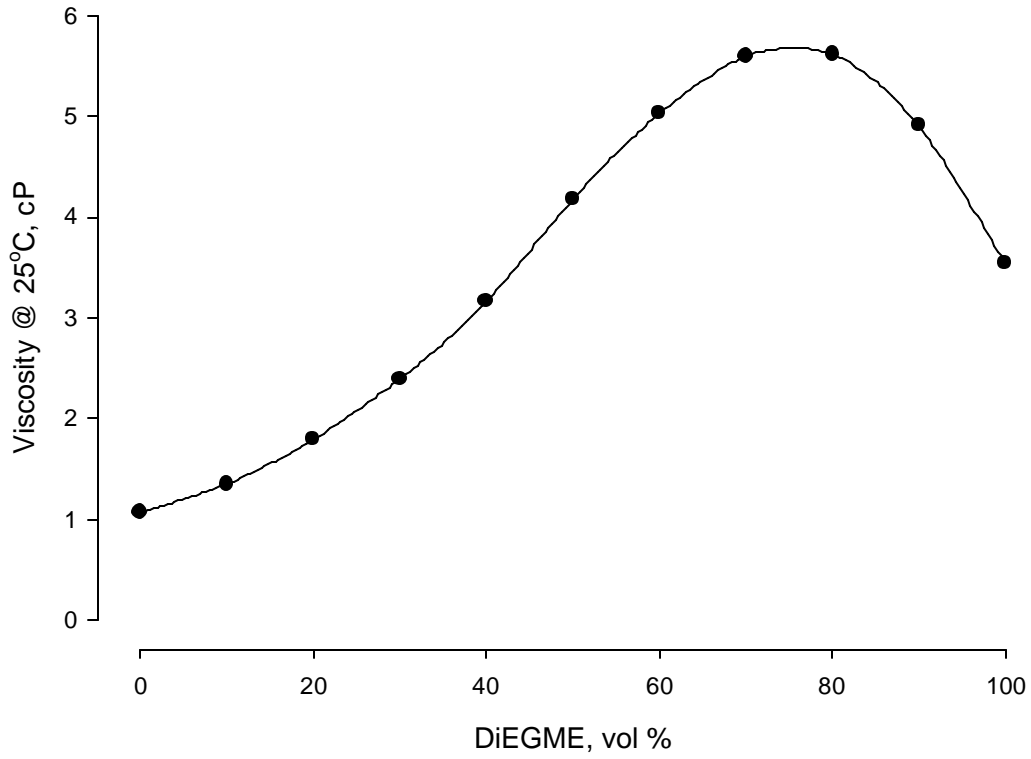


Figure 12a. Brookfield Viscosity Results

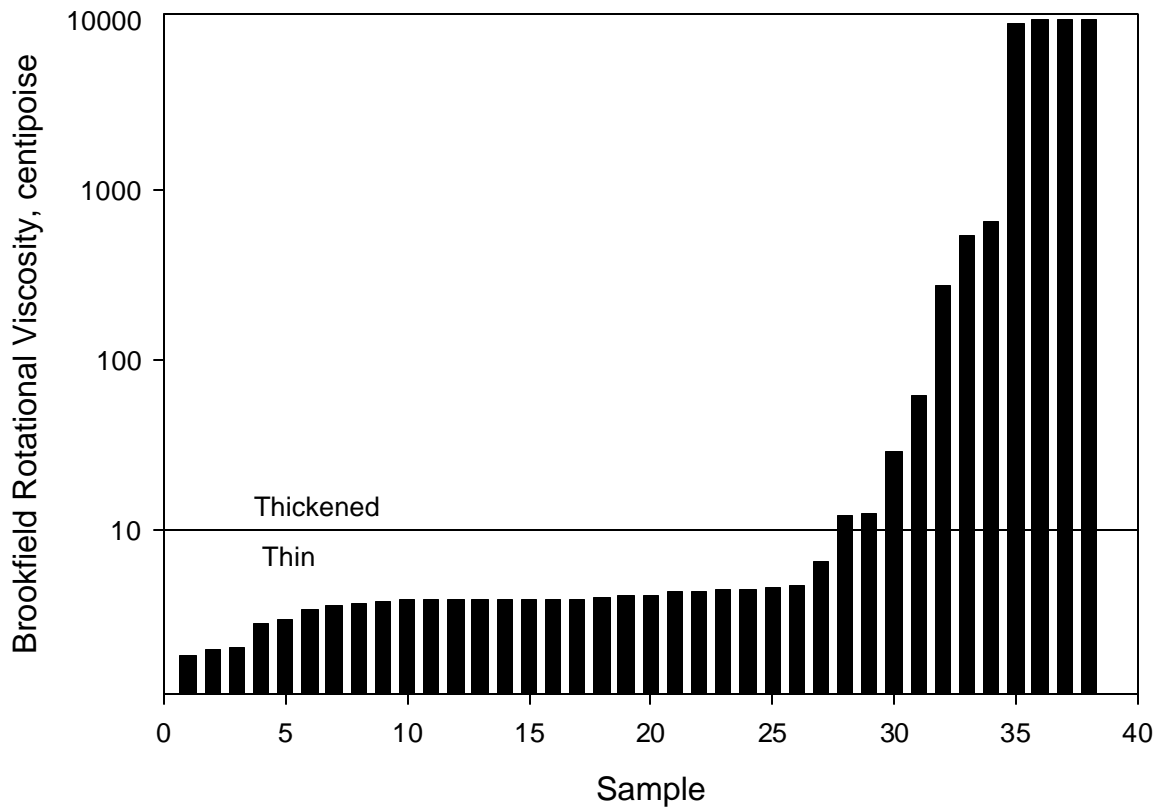


Figure 12b. Brookfield Viscosity Results

### **9.2.2 Density**

The density data are plotted in Figure 13. The densities of the fuel and fuel layer samples are consistent with aviation kerosene fuel. With only a few exceptions, the apple jelly samples have densities of approximately 1 g/mL; as expected with a composition of mostly water and DiEGME.

### **9.2.3 Total Acid Number**

Total acid number, TAN, was measured on each of the apple jelly samples using method D 664. Early in the project it was determined that many of the apple jelly samples would not completely dissolve in the titration solvent used in D 664. That solvent is composed of 495 parts isopropyl alcohol, 5 parts water, and 500 parts toluene. A suitable alternative solvent was found. The new solvent is composed of 33 parts toluene, 33 parts isopropyl alcohol, 33 parts dimethyl sulfoxide, and 1 part water. The TAN results for the apple jelly samples are plotted in Figure 14. The results range from 0.05 to 112 mg KOH/g. These results, especially for the thick apple jelly samples where TAN values were about 40 mg KOH/g or higher, were about 100 times higher than the highest TAN value reported by the Air Force. [3] For comparison purposes, the specification limit for TAN in JP-8 is a maximum of 0.015 mg KOH/g. It should be noted here that all apple jelly samples were completely miscible with water and with DiEGME. Also, no precipitate was ever observed with the addition of either water or DiEGME to any apple jelly sample. Finally, all thin apple jelly appeared to be stable solutions with no evidence of emulsions.

### **9.2.4 Total Base Number**

Total Base Number, TBN, was measured on the apple jelly samples using method D 2896. As with the TAN measurements, a modified titration solvent was required for the TBN measurements. The modified TBN solvent consisted of 1 part chlorobenzene, 1 part glacial acetic acid, and 1 part dimethyl sulfoxide. The TBN results are plotted in Figure 15. The results range from 0.26 to 97.63 mg KOH/g. Since no previous study could be found that included TBN evaluation, a comparison of our results with other results was not possible.

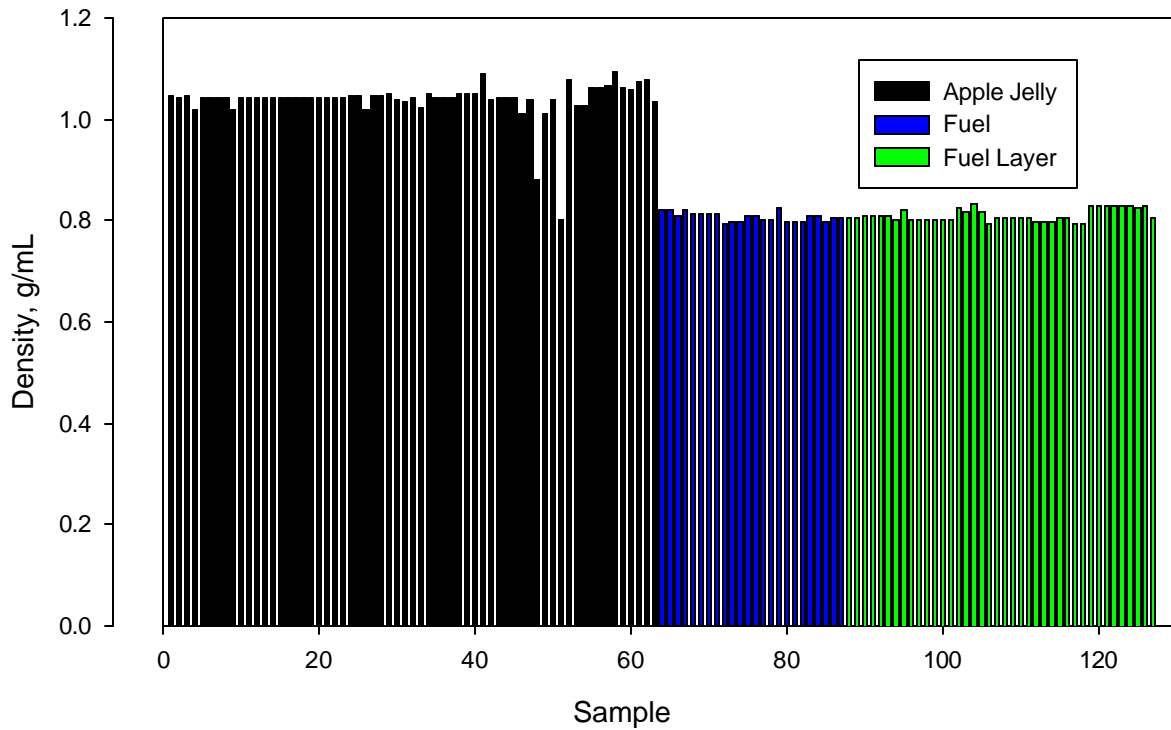


Figure 13. Sample Density Results

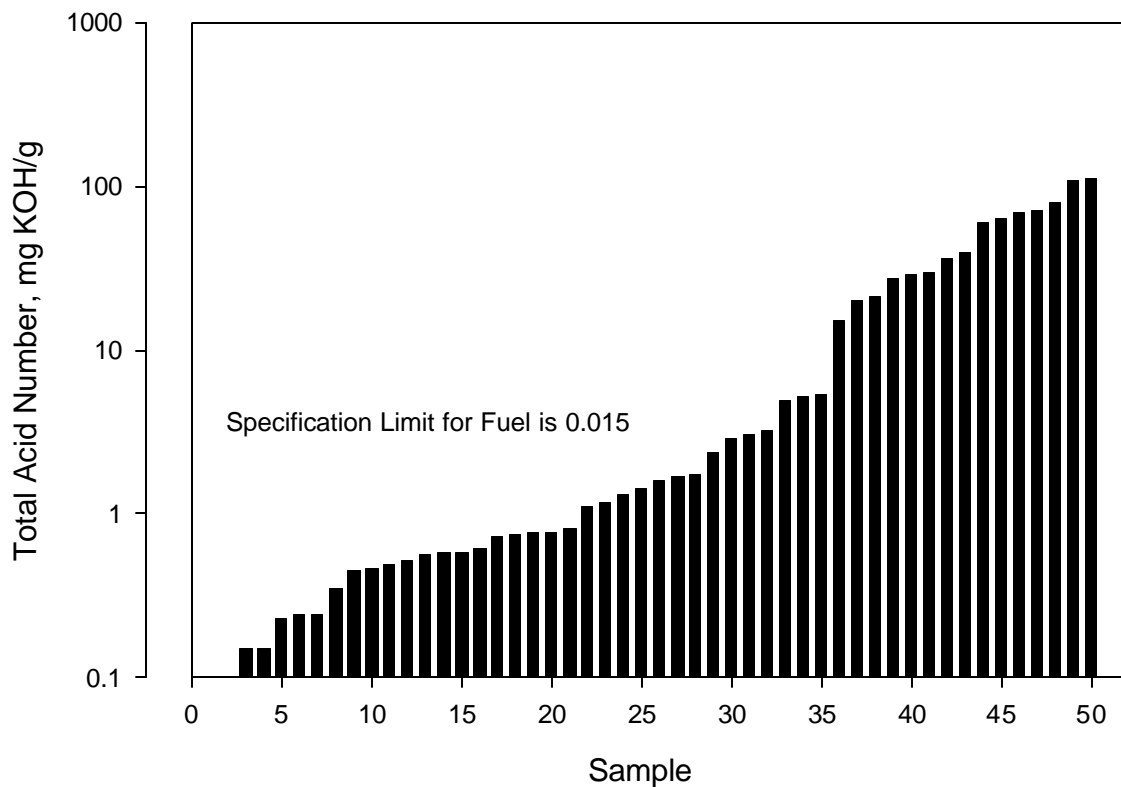
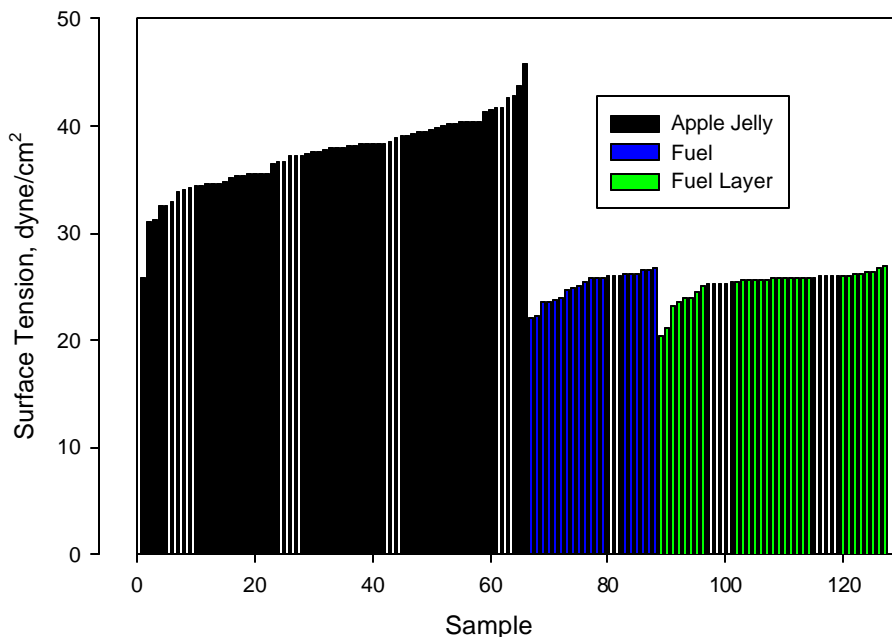


Figure 14. Total Acid Number Results



**Figure 15. Total Base Number Results**

### 9.2.5 Surface Tension

Surface tension was measured on the apple jelly, fuel, and fuel layer samples using a K12 processor tensiometer (manufactured by Kruss) with a Wilhelmy plate attachment. The results are plotted in Figure 16. The surface tension results for the fuel and fuel layer samples range between 20 and 30 dynes/cm<sup>2</sup>. This is typical for JP-8. The apple jelly results range from the high 20's to the high 40's. The much higher surface tension of apple jelly is consistent with the increased ionic character compared to fuel.

### 9.2.6 Conductivity

Figure 17 is a plot of the conductivity of the apple jelly samples. The results range from about  $1 \times 10^{10}$  pico Siemens per meter (pS/m) to over  $8 \times 10^{11}$  pS/m. These are extremely high conductivity values compared to JP-8, which typically has a conductivity of between 150 and 600 pS/m. Figure 18 is a plot of conductivity values for mixtures of pure water and DiEGME. Note that the maximum conductivity for these mixtures is  $2.2 \times 10^8$  pS/m. This comparison demonstrates that the conductivity of the apple jelly samples is due to more than simply water and DiEGME; there are other components of apple jelly that also contribute to the conductivity, and these contributions are typically very large. Note that the conductivity of the thickest apple jelly samples (viscosity > 1,000 cP at 25°C) was always at or near the top of the observed conductivity range.

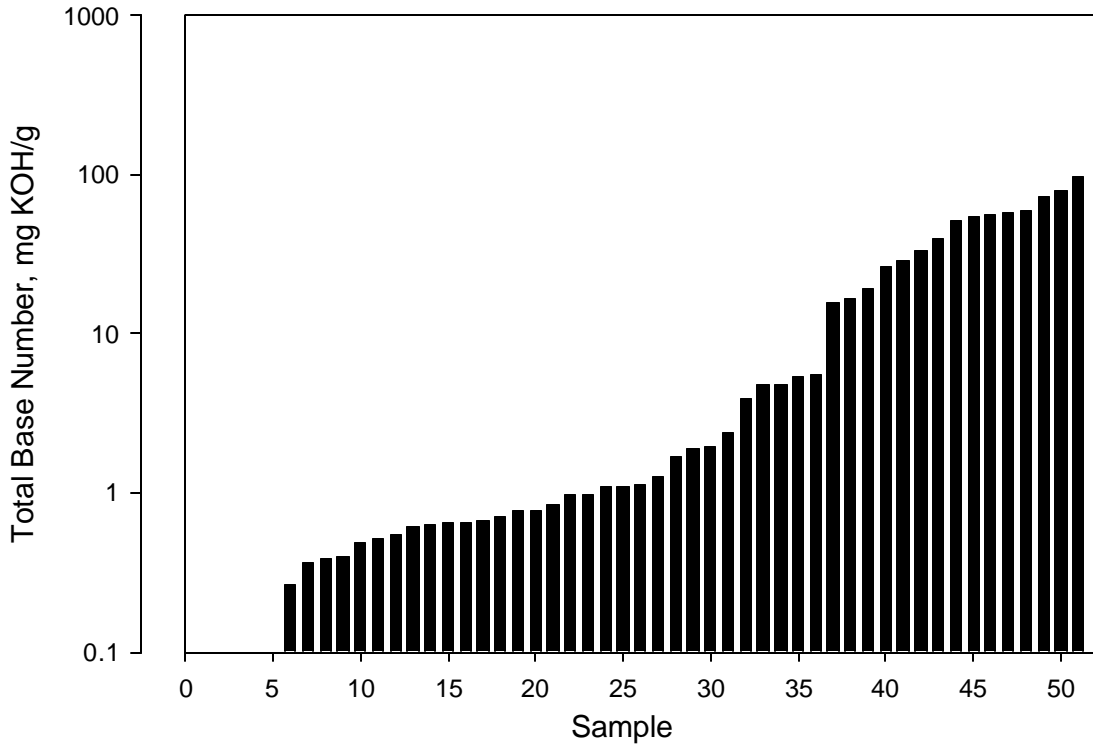


Figure 16. Surface Tension Results

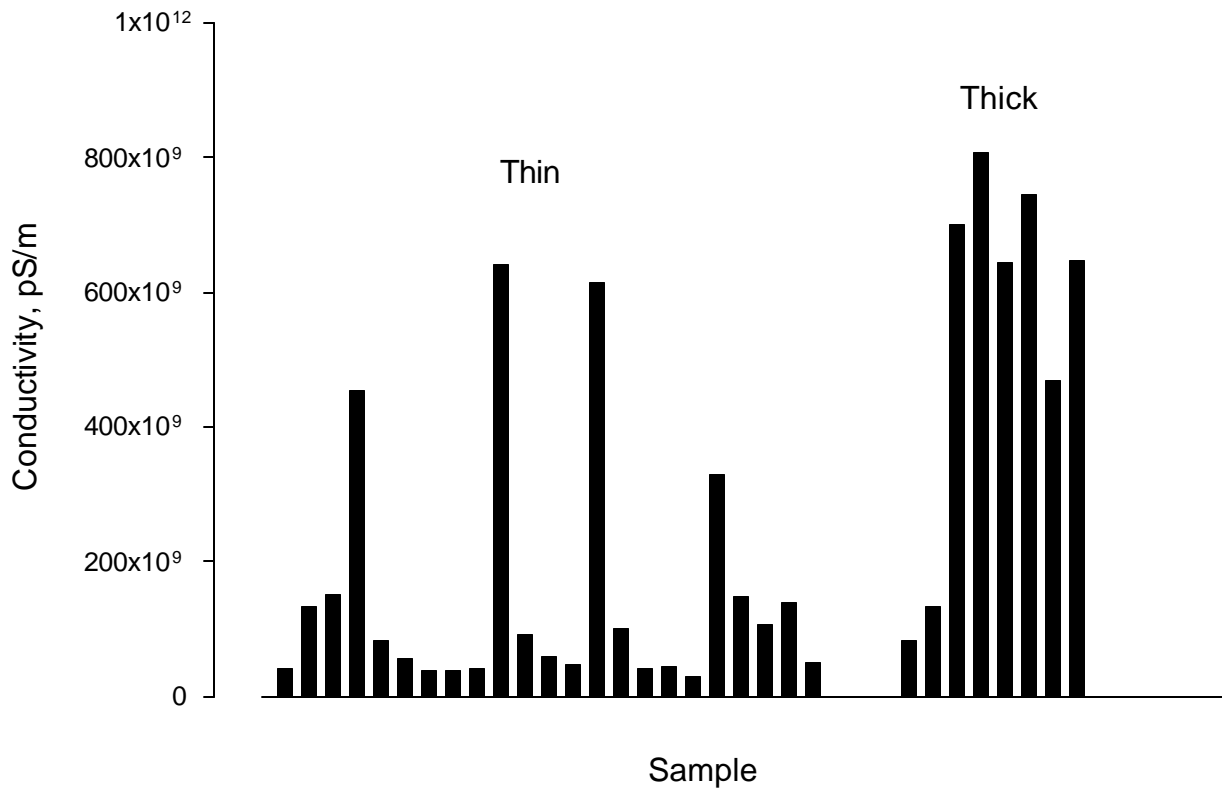
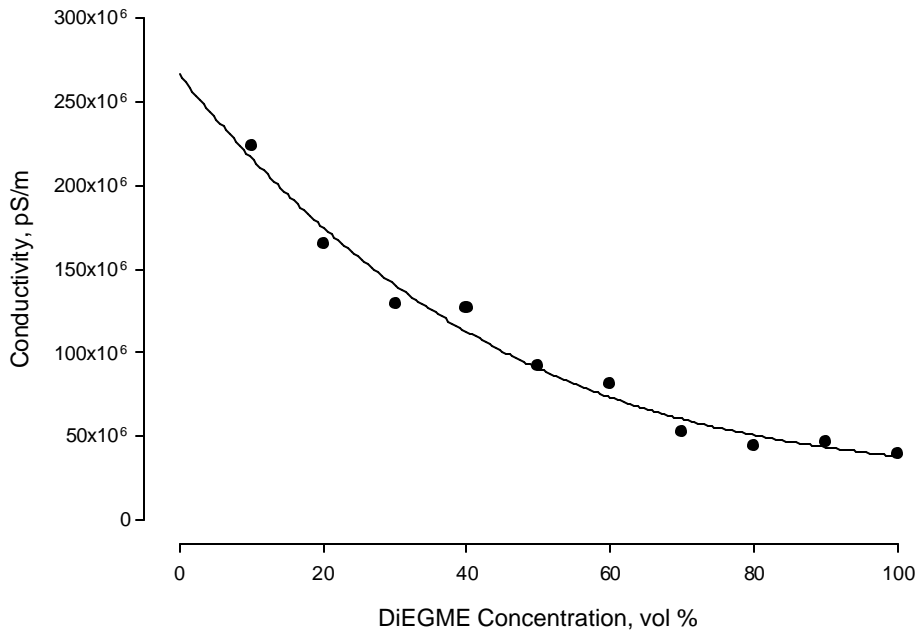


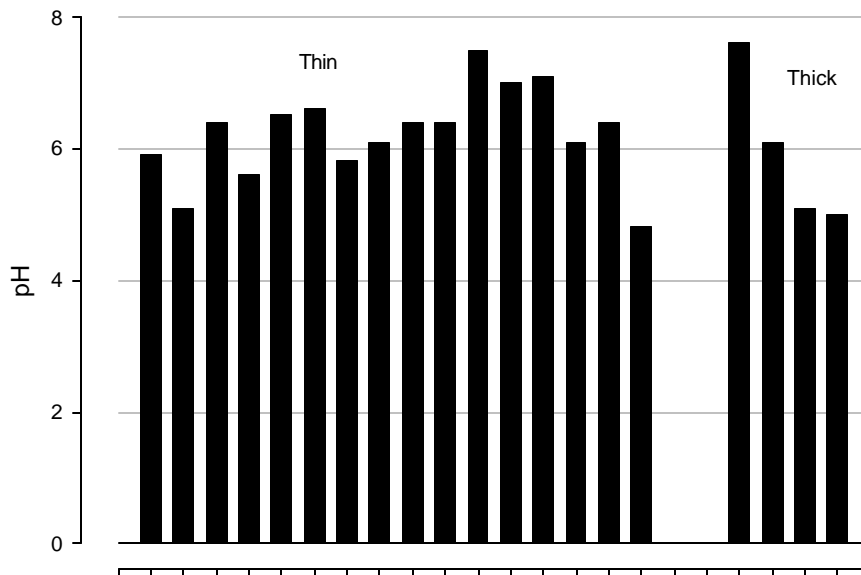
Figure 17. Conductivity of Apple Jelly Samples at Room Temperature



**Figure 18. Conductivity of Water and DiEGME Mixtures at Room Temperature**

### 9.2.7 pH

Figure 19 presents the measured pH values for several apple jelly samples. Only 4 thick apple jelly samples were measured because there was insufficient quantity of the others to obtain a reliable measurement. The results are in the range of 5 to 7.5 pH.



**Figure 19. pH of Selected Apple Jelly Samples**



### 9.3 Data Relationships

In some previous studies, cited above, it was suggested that there are statistical correlations between some properties of apple jelly. As an example, in one study, Beal and Hardy suggested a significant relationship between viscosity and sodium. [5] As a first level of investigation of correlations, we calculated the  $R^2$  correlation coefficients for combinations of sulfur, nitrogen, sodium, total acid number, total base number, and viscosity. The results are for a standard, least-squares linear regression. The results are given in Table 7. *[There were three apple jelly samples with viscosities outside the analytical range of our instrument ( $>10,000$  cP). These points were excluded from the statistical analysis but are included in the data plots for information.]* Notice that, in general, the poorest correlations are for sulfur content. Also notice that the highest correlations are for combinations of sodium, TAN, TBN, and viscosity. Plots of some of the data are given below along with discussion of the results.

Table 7. Least-Squares, Linear Regression Correlation Coefficients ( $R^2$ ) of Selected Apple Jelly Properties						
	Sulfur	Nitrogen	Sodium	TAN	TBN	Viscosity
Sulfur						
Nitrogen	0.0036					
Sodium	0.028	0.45				
TAN	0.026	0.46	0.92			
TBN	0.024	0.56	0.88	0.88		
Viscosity	$6.2 \times 10^{-5}$	0.37	0.87	0.86	0.95	

#### 9.3.1 Total Sulfur

Figure 20 is a plot of sulfur vs. sodium for the apple jelly samples. Examination of the plot shows extremely poor correlation of these data. The poor correlation between sulfur and sodium indicates that sodium salts of sulfur species, components of the SDA, are a statistically small part of the total sulfur of the apple jelly samples. Recall from Figure 6 that there was no significant concentration of sulfur in the apple jelly samples as compared to the fuel and fuel layer samples. This comparison is further demonstrated in Figure 21, which is a plot of the total sulfur level of apple jelly samples and corresponding

fuel layer samples. Notice that, with some exceptions, there is little significant difference in the sulfur concentrations of each. This indicates that, in general, there is not a preferential partitioning of sulfur species into the apple jelly. In fact, there is a slight preference of the sulfur species to remain in the fuel layer (19 of the 29 data pairs had higher fuel layer results). Figure 22 is a plot of viscosity vs. total sulfur for the apple jelly samples. There is virtually zero correlation of the data in this plot. This indicates that the sulfur containing species present in the apple jelly are not a statistically significant contributor to the thickening of the apple jelly. Similarly, the very poor correlation of sulfur with both TAN and TBN demonstrates that sulfur-containing species in the apple jelly are not statistically significant contributors to either of these properties.

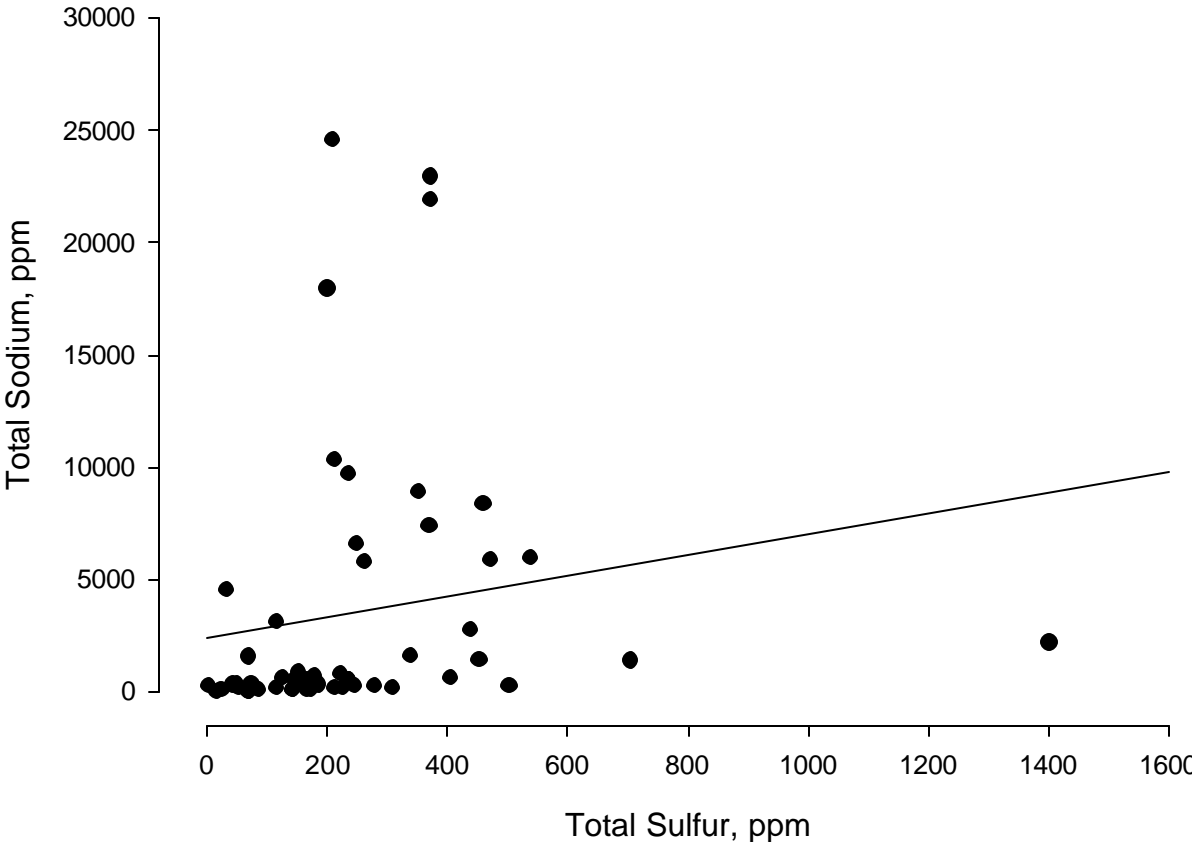


Figure 20. Total Sulfur vs. Total Sodium for Apple Jelly Samples ( $R^2 = 0.028$ )

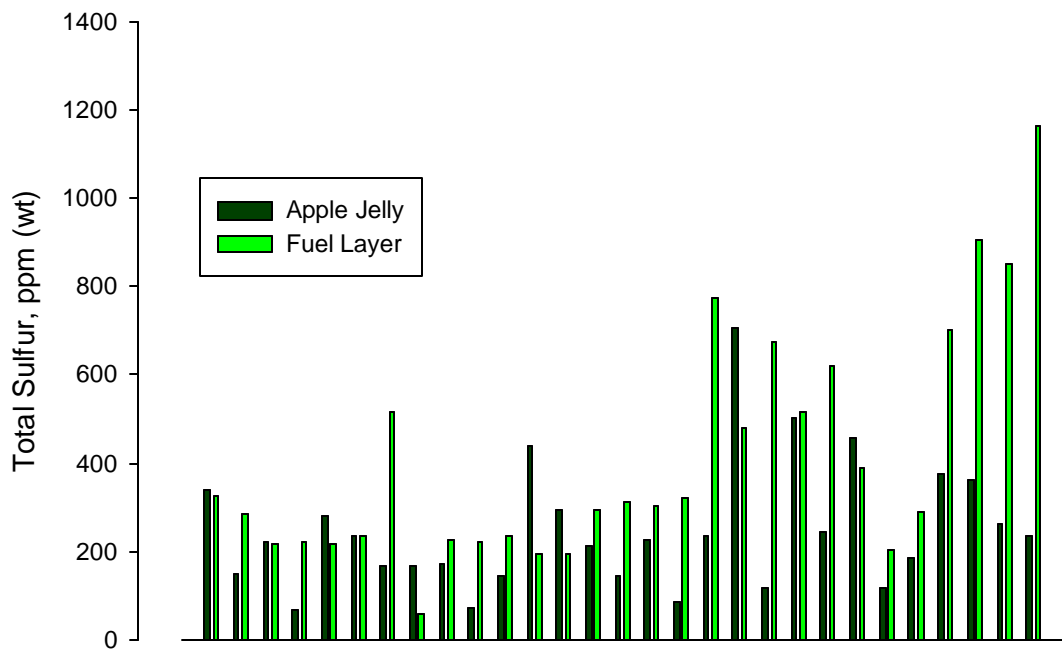


Figure 21. Comparison of Total Sulfur Levels of Apple Jelly and Fuel Layer Samples

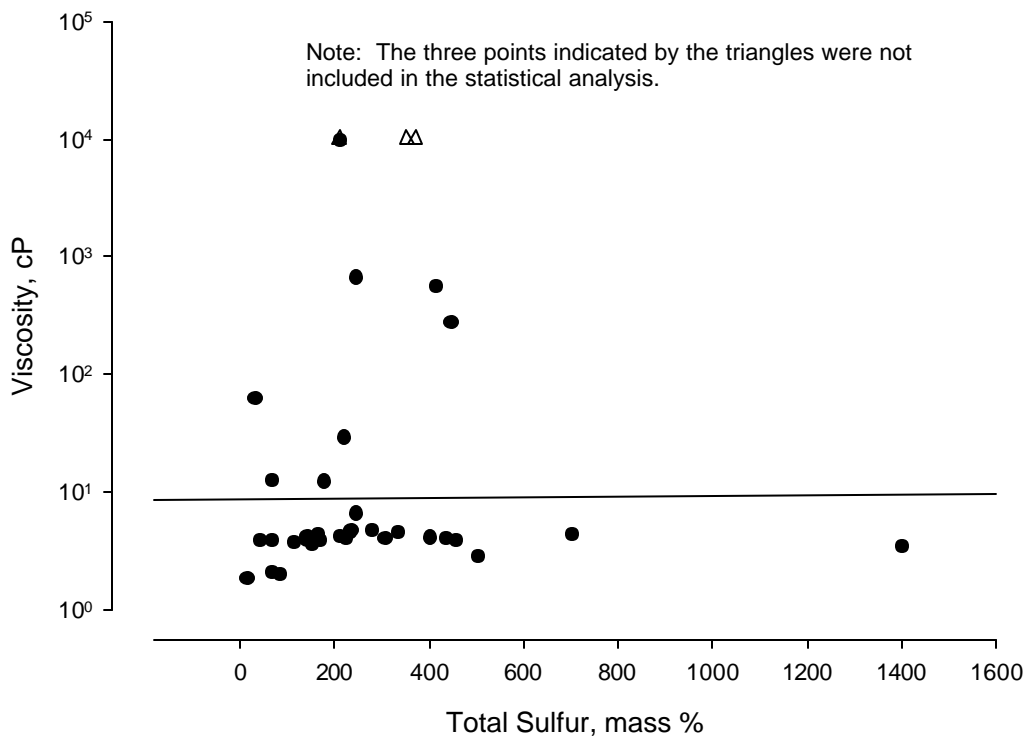


Figure 22. Total Sulfur vs. Viscosity for Apple Jelly Samples ( $R^2=6.2 \times 10^{-5}$ )

### 9.3.2 Total Nitrogen

Figure 23 is a plot of total nitrogen vs. total sodium for the apple jelly samples. The correlation coefficient is small ( $R^2 = 0.45$ ) but there is a general trend of increasing sodium content with increasing nitrogen content. If the source of sodium also contained nitrogen, the correlation coefficient would have to be much higher. However, an  $R^2$  of 0.45 implies an indirect relationship may exist between whatever is contributing to the sodium and the source of the nitrogen.

Recall from Figure 7 that there is a significant concentration of nitrogen species in the apple jelly samples as compared with the fuel and fuel layer samples. This comparison is demonstrated further in Figure 24. Note that for every sample pair, the apple jelly nitrogen levels are approximately 1 to 2 orders of magnitude greater than the corresponding fuel layer nitrogen. This indicates that, unlike sulfur, nitrogen-containing species tend to preferentially partition to the apple jelly as compared to the fuel.

Figure 25 is a plot of total nitrogen vs. apple jelly viscosity. Although the correlation is weak ( $R^2=0.37$ ), this plot shows a general trend of increasing viscosity with increasing nitrogen content. However, the low correlation proves that the species responsible for thickening the apple jelly does not, itself, contain nitrogen.

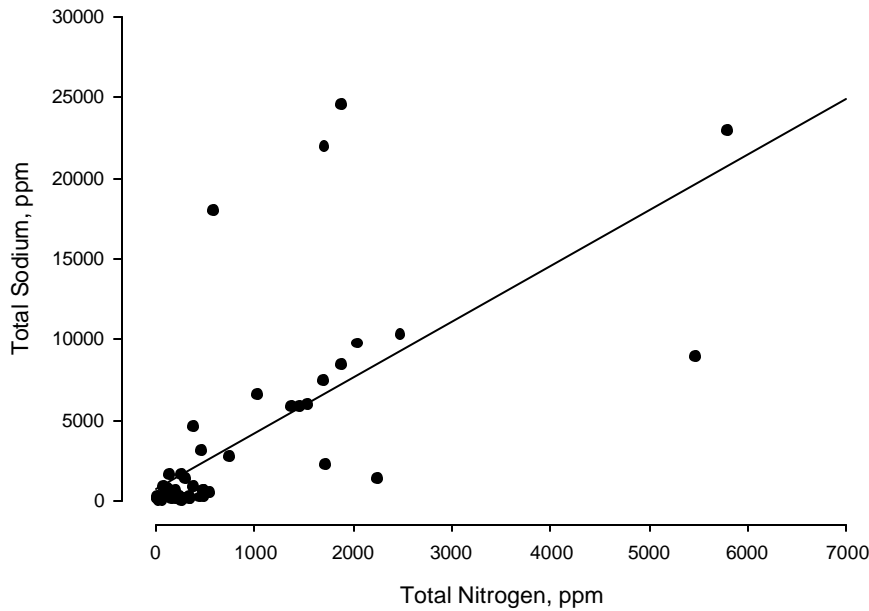


Figure 23. Total Nitrogen vs. Total Sodium for the Apple Jelly Samples ( $R^2=0.45$ )

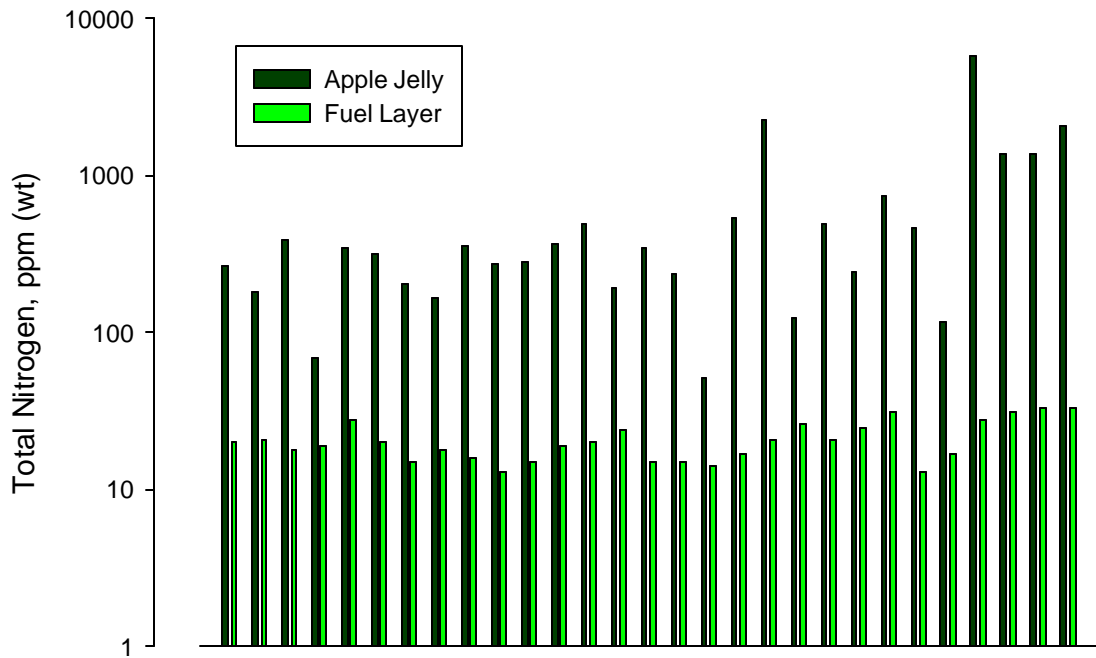


Figure 24. Comparison of Total Nitrogen Levels of Apple Jelly and Fuel Layer Samples

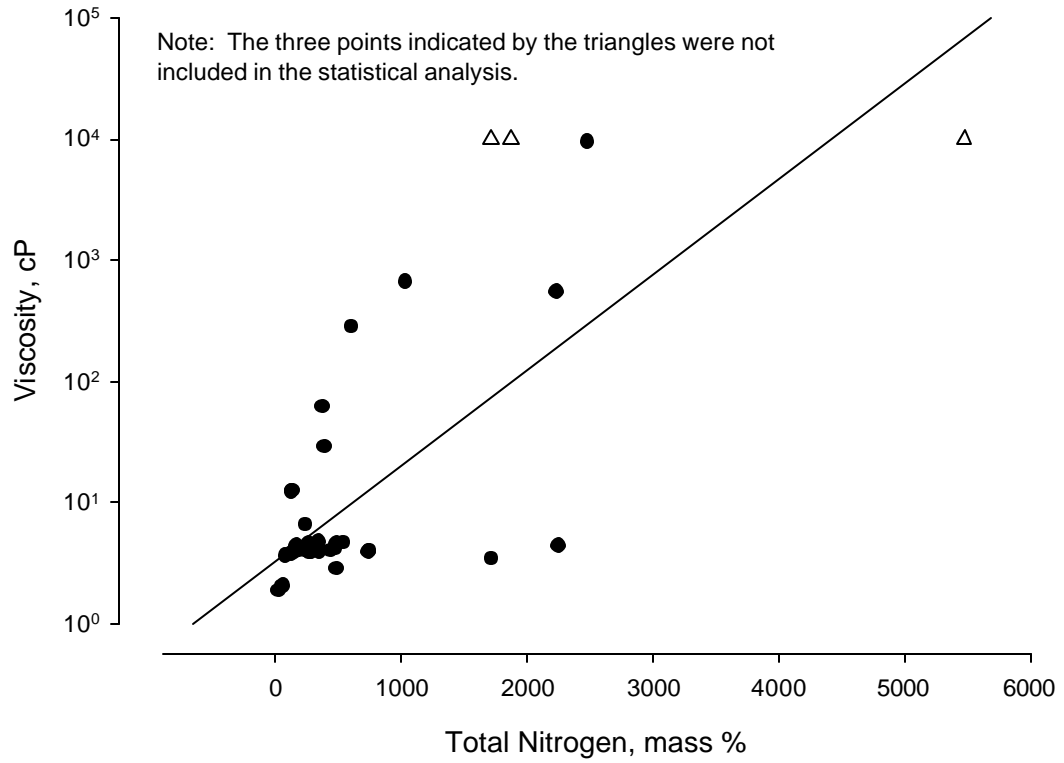


Figure 25. Total Nitrogen vs. Viscosity for Apple Jelly Samples ( $R^2=0.37$ )

### 9.3.3 Acid Number and Base Number

Figures 26 and 27 are plots of viscosity vs. TAN and TBN, respectively. Both plots show strong correlation, especially TBN ( $R^2=0.95$ ). Figure 28 is a plot of TAN vs. TBN. This plot shows that there is also strong correlation between TAN and TBN. The combination of both high acid numbers and high base numbers is the result of the presence of both weak acids and weak bases. The fact that both higher acid number and higher base number accompany higher viscosity indicates that both weak acids and bases are involved in the thickening of the apple jelly.

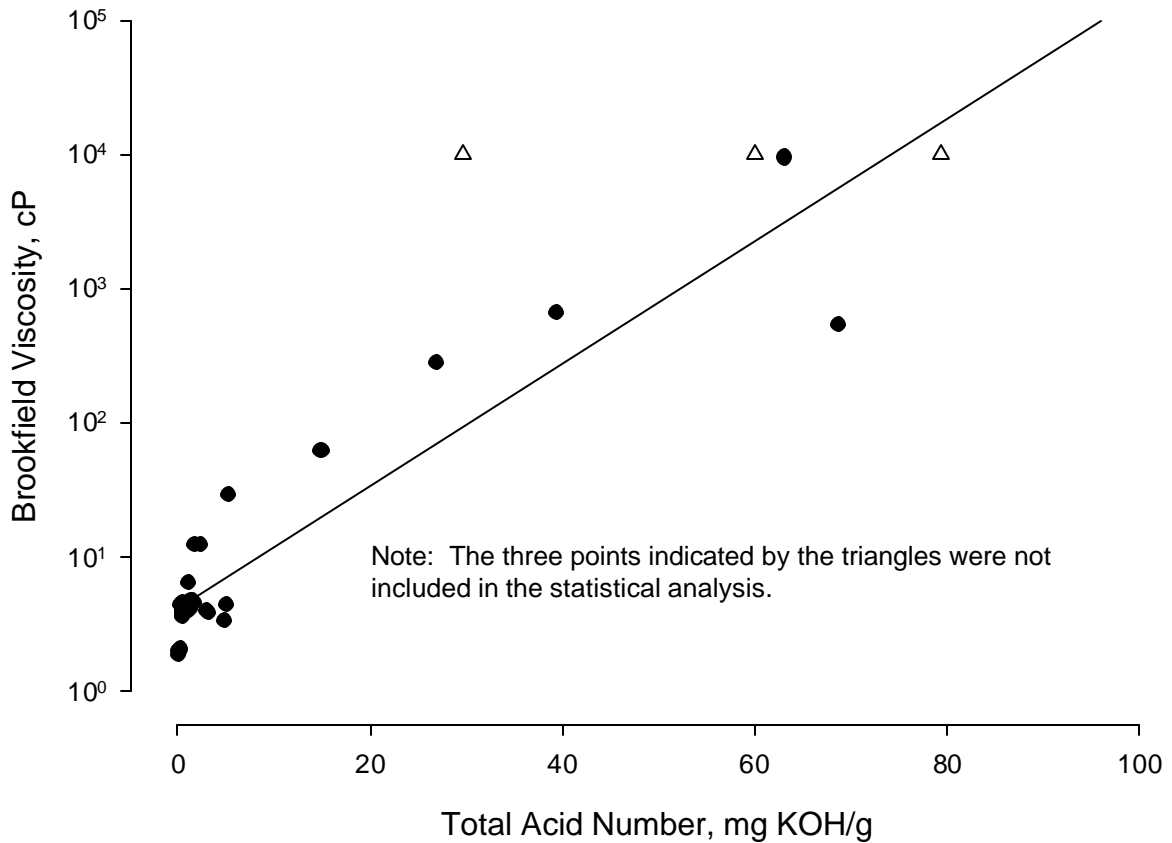


Figure 26. Viscosity vs. Total Acid Number for Apple Jelly Samples ( $R^2=0.86$ )

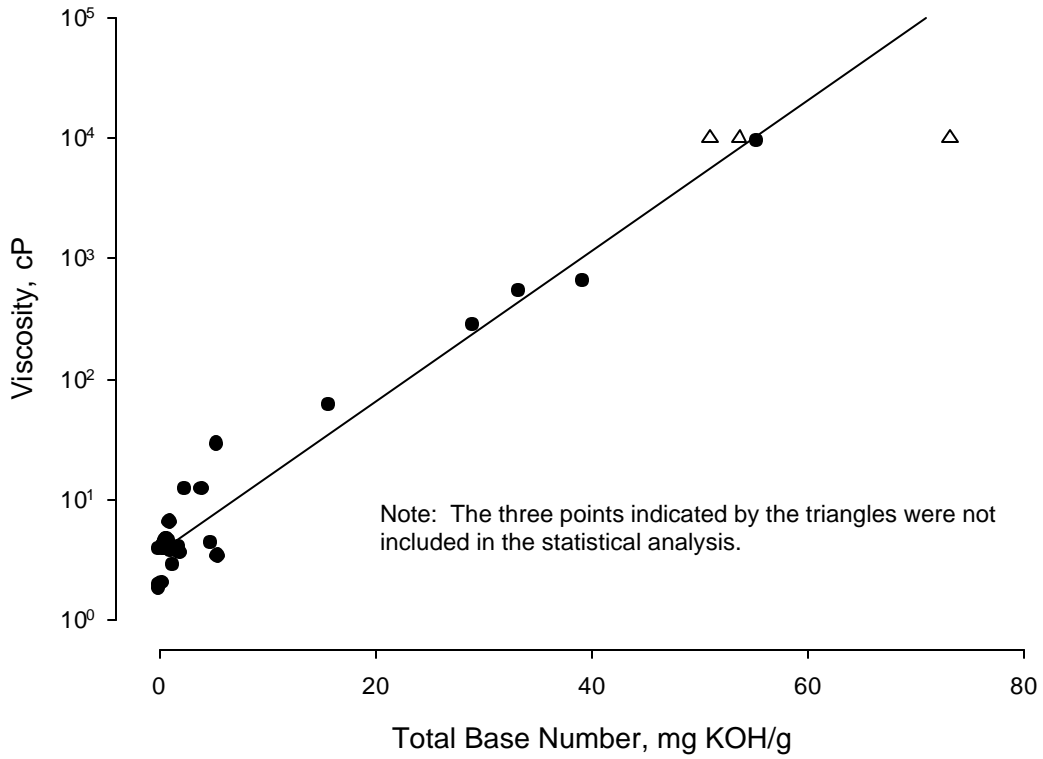


Figure 27. Viscosity vs. Total Base Number for Apple Jelly Samples ( $R^2=0.95$ )

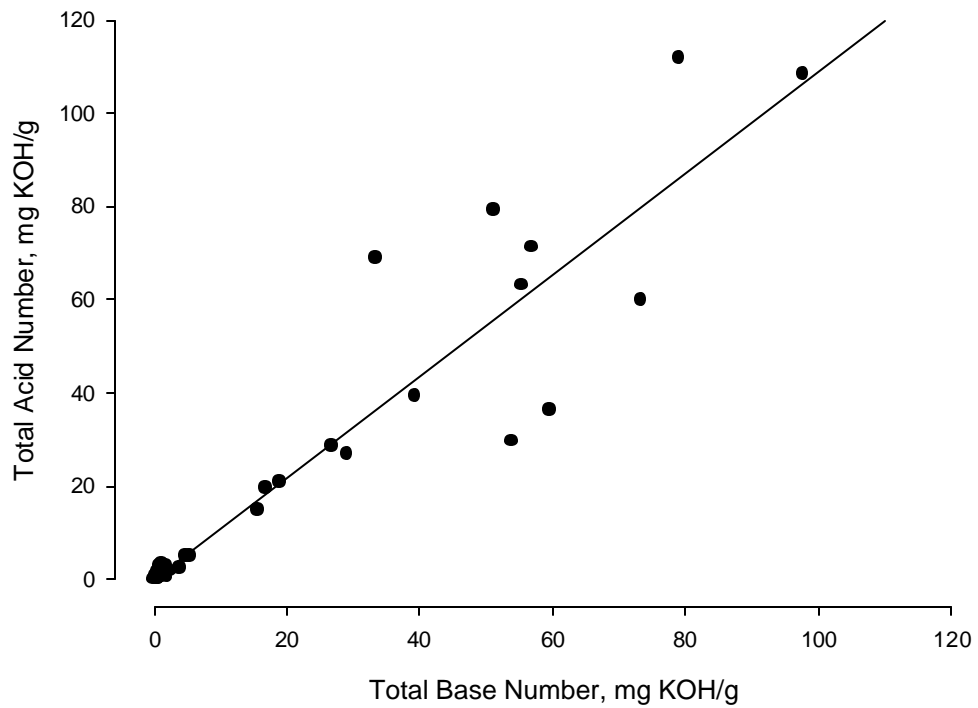


Figure 28. Total Acid Number vs. Total Base Number for Apple Jelly Samples ( $R^2=0.88$ )

### 9.3.4 Total Sodium

Figure 29 is a plot of viscosity vs. total sodium content. Here again, there is a strong correlation between the data. This correlation, in combination with the correlations from the previous section, implies that the thickener is composed of sodium ions and the conjugate base of a weak acid.

Figures 30 and 31 are plots of total sodium vs. TAN and TBN, respectively. These data also show strong correlation, indicating that a sodium-containing compound is strongly involved with both the acid number and the base number of the apple jelly samples.

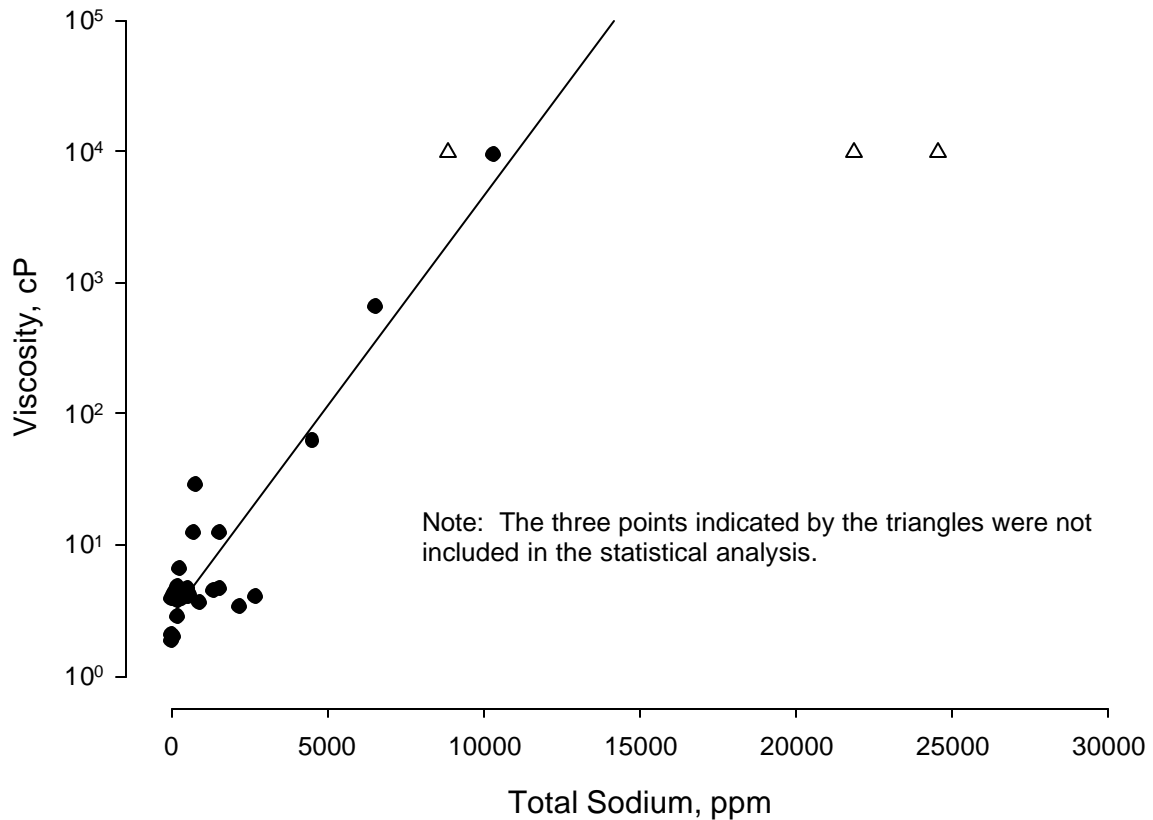
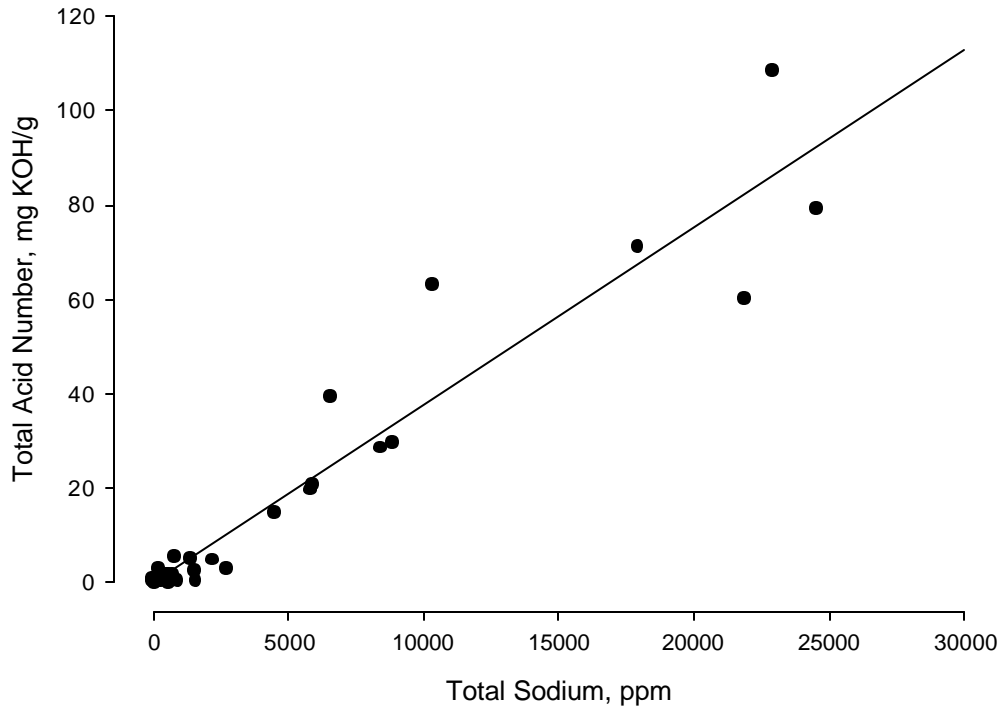
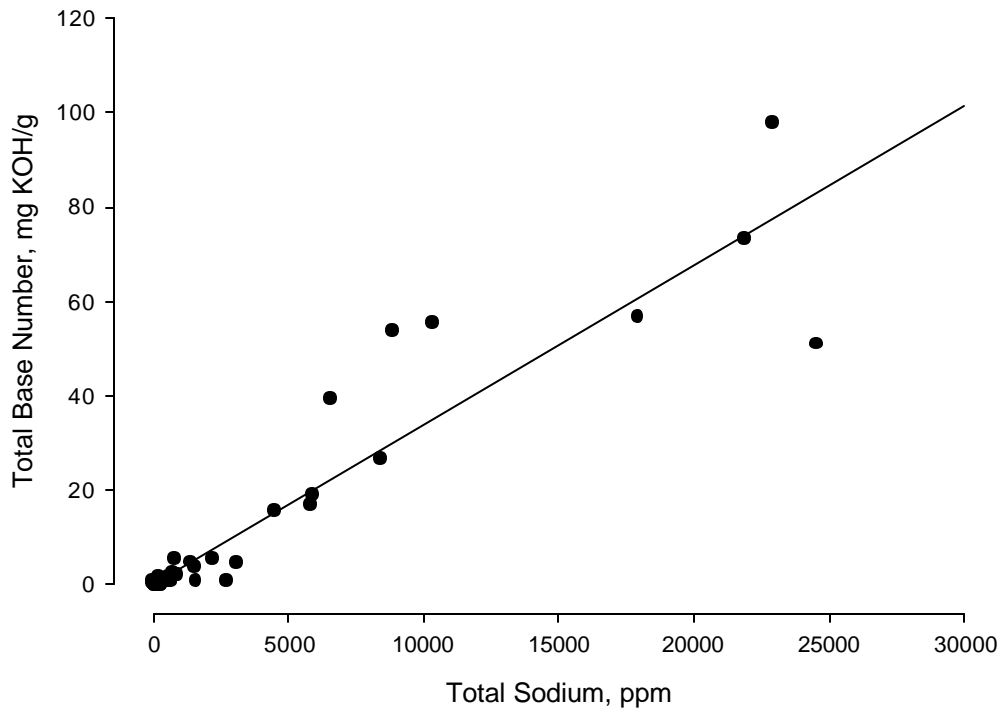


Figure 29. Total Sodium vs. Viscosity for Apple Jelly Samples ( $R^2=0.87$ )





**Figure 30. Total Acid Number vs. Total Sodium for Apple Jelly Samples ( $R^2=0.92$ )**



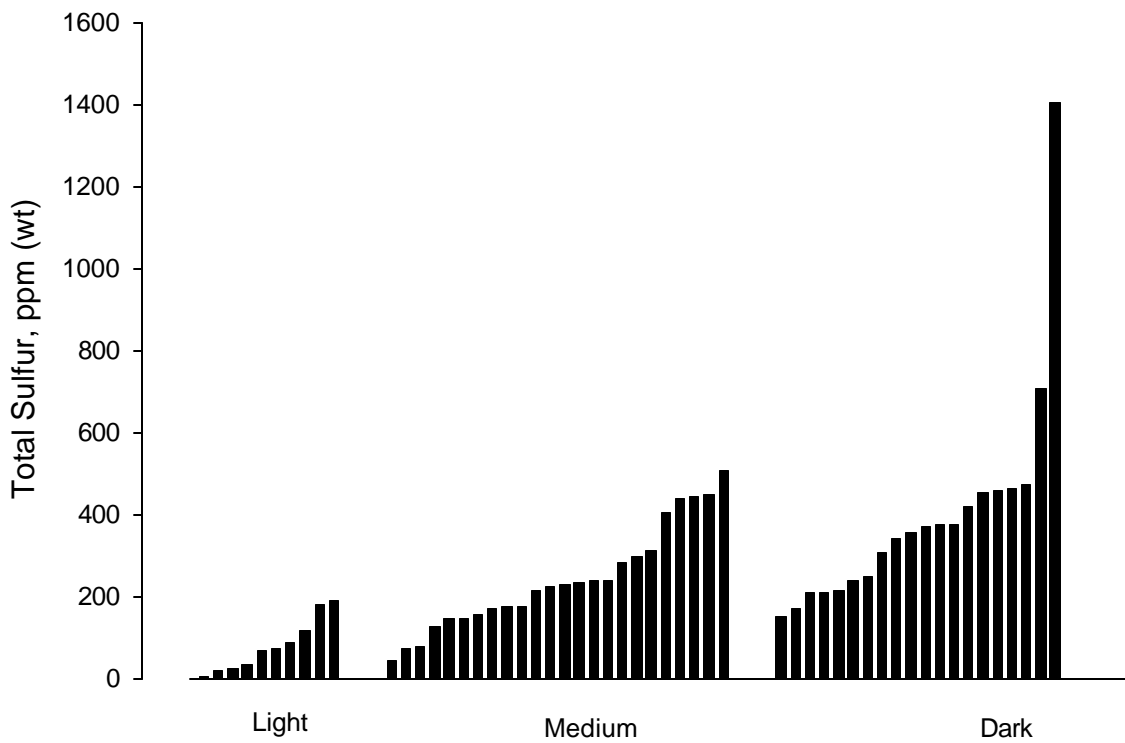
**Figure 31. Total Base Number vs. Total Sodium for Apple Jelly Samples ( $R^2=0.88$ )**

### 9.3.5 *Visual Color*

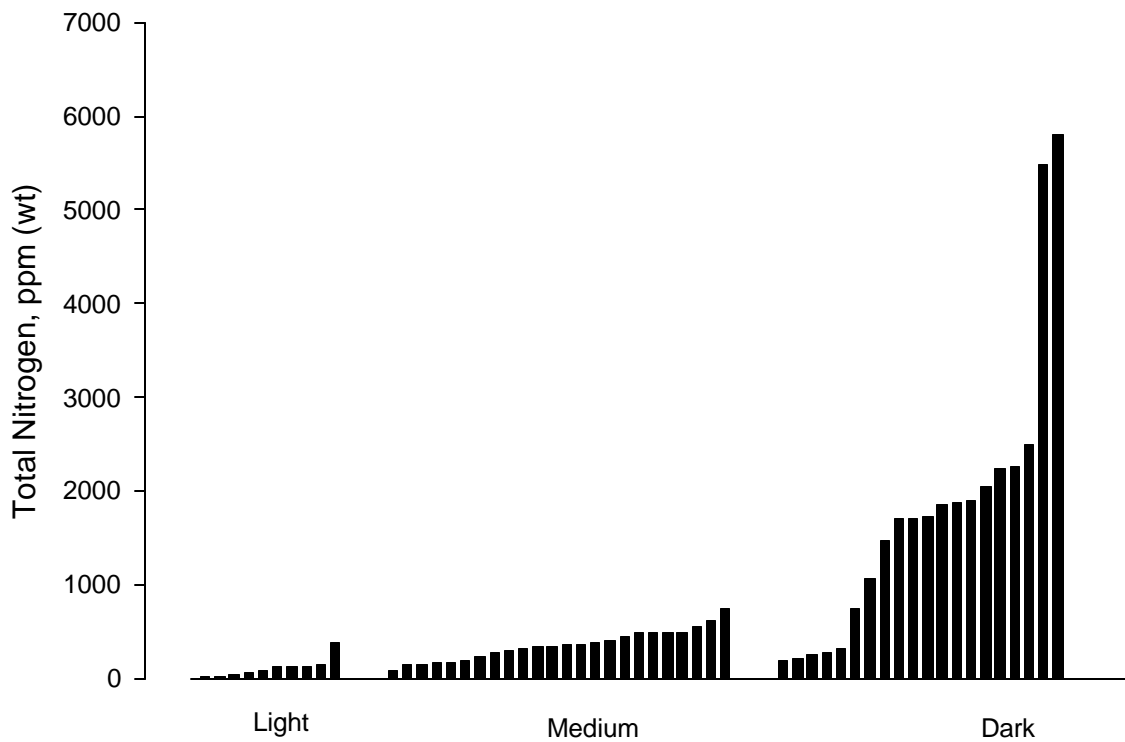
Since the apple jelly samples had visibly different colors, it was decided to investigate the correlation of color with sulfur content, nitrogen content and percent water. We had intended to obtain a spectrum for selected samples using an ultraviolet/visible spectrometer for the purpose of making the comparisons. Unfortunately, our spectrometer was inoperable so those spectra were not obtained. As a gross approximation, each apple jelly sample was visually ranked (by human eye) and assigned a ranking of light, medium, or dark. Samples were rated as follows: “light” when possessing a colorless to light amber appearance; “medium” when amber to orange; “dark” when dark orange to brown/black. These subjective rankings were then compared with the total sulfur content, total nitrogen content and percent water. The comparisons are presented in Figures 32, 33, and 34, respectively. Examination of the plots shows the following:

- 1) in general, total nitrogen was highest in the dark samples,
- 2) the relationship between sulfur content and color is similar to that for nitrogen although not as pronounced, and
- 3) the samples with the highest percentages of water (>60%) are all light in color.

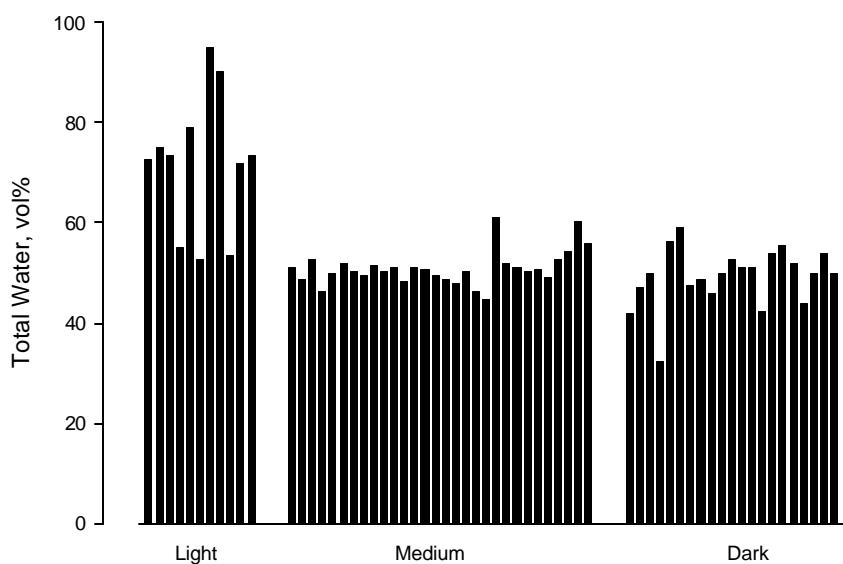
These observations imply that as water content increases in apple jelly samples, the species that cause dark color are present in decreasing concentration. This would only be true if those colored species were organic compounds. Apparently, when water is present beyond a certain threshold level, the highly colored species responsible for dark color are no longer soluble by an amount sufficient to impart dark color. This information, when combined with the earlier observations concerning nitrogen, implies that a primary source of dark color in apple jelly is organo-nitrogen compounds.



**Figure 32. Visual Color Rating vs. Total Sulfur for Apple Jelly Samples**



**Figure 33. Visual Color Rating vs. Total Nitrogen for Apple Jelly Samples**



**Figure 34. Visual Color Rating vs. Total Water for Apple Jelly Samples**

### 9.3.6 Summary of Data Relationships

The main points from this section are:

- Sulfur content had very poor correlation to the properties to which it was compared.
- Nitrogen content had better correlation than sulfur content. Nitrogen content correlated best with total base number ( $R^2=0.56$ ).
- Sodium content, total acid number, total base number, and viscosity all had very high correlation with each other.

## 9.4 Thermogravimetric Analysis

A white residue was obtained during the acid digestion of samples for the ICP metals analysis, but only for the thick apple jelly samples. This white precipitate was also found during other analyses such as acid number and base number. For the acid number and base number analyses, the precipitate formed when exposed to the titration solvent (as described earlier in this report). To investigate this further, thermogravimetric analysis, TGA, was performed on 11 apple jelly samples of various viscosities. Figures 35 and 36 are TGA scans of representative thin and thick samples, respectively. Notice in Figure 35 that there is very little residue left at temp above which the DiEGME is evaporated. In contrast, Figure 36 shows a residue of 5.7 %(wt) in the thick apple jelly sample. The absence or presence of a residue (depending on the viscosity) held true for all of the apple jelly samples, thin and thick, analyzed by TGA. This residue, found only in the thick apple jelly samples, implies the presence of a high molecular weight species.

Sample: AP-002 APPLE JELLY  
 Size: 60.9370 mg  
 Method: DECOMPOSITION  
 Comment: ISOTHERMAL METHOD

TGA

File: C:\...AP-002ISO061401.130  
 Operator: R\WARD  
 Run Date: 14-Jun-01 11:07

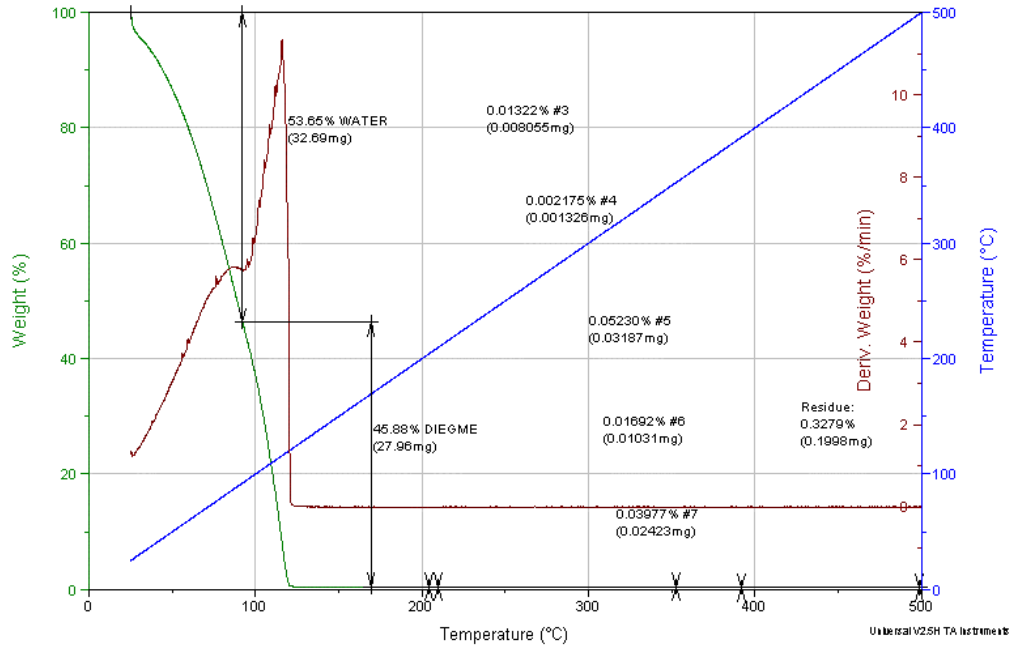


Figure 35. TGA Scan of AP-002 -- Thin

Sample: AP-032 APPLE JELLY  
 Size: 57.2600 mg  
 Method: DECOMPOSITION  
 Comment: ISOTHERMAL METHOD

TGA

File: C:\...AP-032ISO061801.135  
 Operator: R\WARD  
 Run Date: 18-Jun-01 08:49

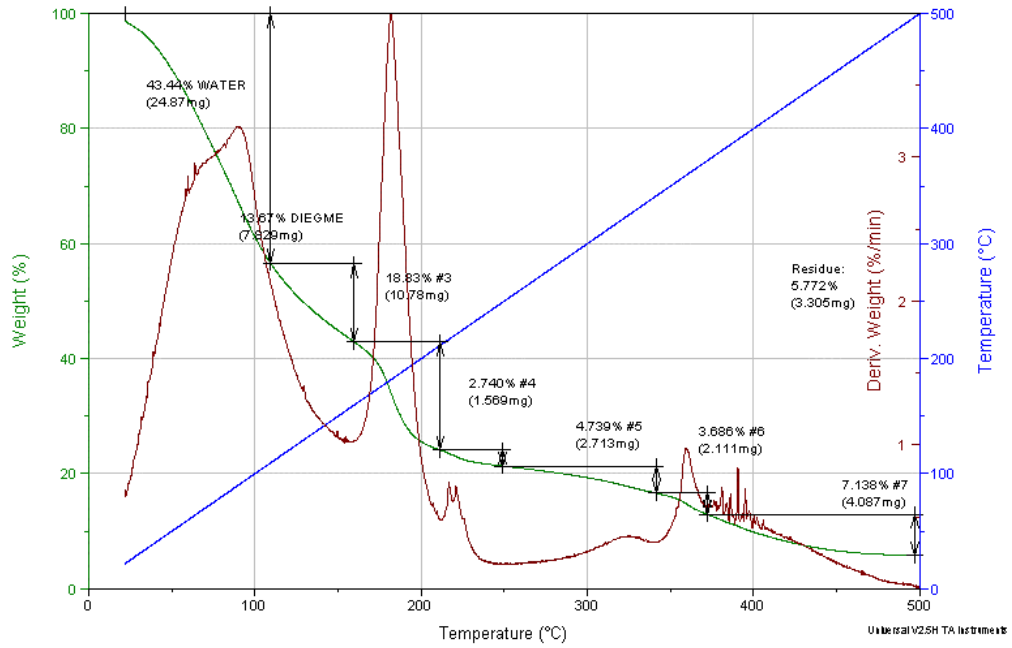
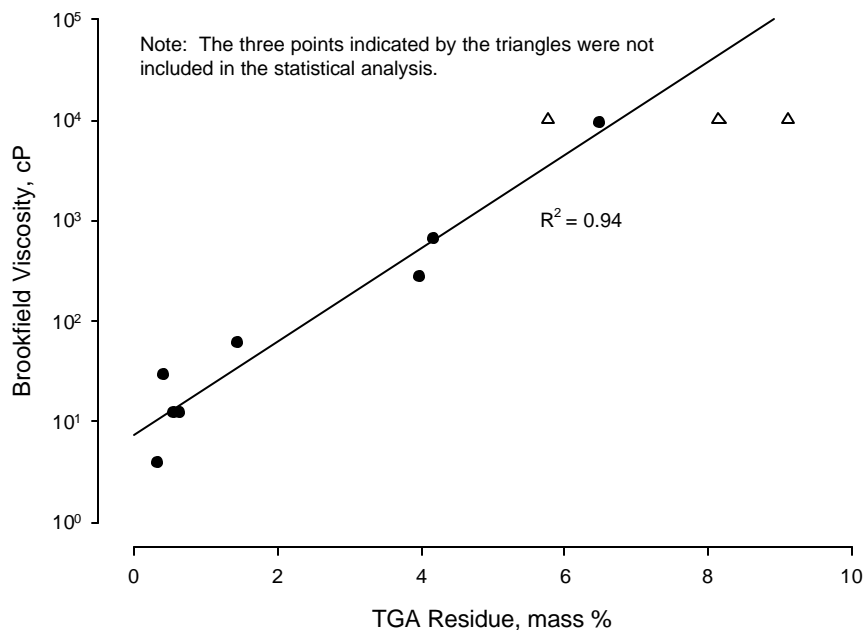


Figure 36. TGA Scan of AP-032 -- Thick

Figure 37 is a plot of the TGA residue vs. viscosity for the 11 samples analyzed. Notice there is a very strong trend of increasing viscosity with increasing TGA residue. This implies that the high molecular weight residue is a factor in the viscosity of the apple jelly. This is consistent with a thickener material in the thick apple jelly samples, as further discussed in the next section.



**Figure 37. TGA Residue vs. Viscosity for 11 Apple Jelly Samples**

## 9.5 Rheological Properties of Thick Apple Jelly

As discussed, the primary difference, between thin and thick apple jelly, is the significantly increased viscosity exhibited by the latter. Previously discussed data strongly suggest that the increased viscosity is due to the presence of a thickener. The data further suggest that the thickener:

- contains no nitrogen (poor correlation between viscosity and nitrogen),
- contains no sulfur (very poor correlation between viscosity and sulfur),
- contains sodium (high correlation between viscosity and sodium),
- titrates as basic in the TBN procedure (extremely high correlation between viscosity and TBN),
- and has a high molecular weight (extremely high correlation between viscosity and TGA residue).

Additional information concerning the rheological properties of thick apple jelly was obtained by examining the viscosity as a function of shear rate.

Figure 38 shows the Brookfield Viscosity at 25°C of thick apple jelly sample AP-117 as a function of shear rate, as indicated by rpm. [According to Brookfield, the precision of any measured viscosity is within 1%.] As seen, viscosity drops off significantly as shear rate increases from 0.6 rpm to 5 rpm. This is a type of non-Newtonian behavior known as pseudoplasticity. [19] Figure 39 shows the Brookfield Viscosity at 60°C for thick apple jelly sample AP-044 as a function of shear rate, both initially and after being sheared at 100 rpm for two hours. Both the initial viscosity and the viscosity after prolonged shearing at 100 rpm exhibit pseudoplasticity. Comparing the viscosities before and after prolonged shearing shows that permanent decreases in viscosity at any given shear rate have occurred. This is a form of time-dependent non-Newtonian behavior known as thixotropy. [19] Non-Newtonian behavior such as pseudoplasticity and thixotropy are very common in fluids containing a very high molecular weight thickening material. Therefore, this rheological behavior is consistent with the earlier data (such as the TGA residue versus viscosity data) that suggested the presence of such material in thick apple jelly.

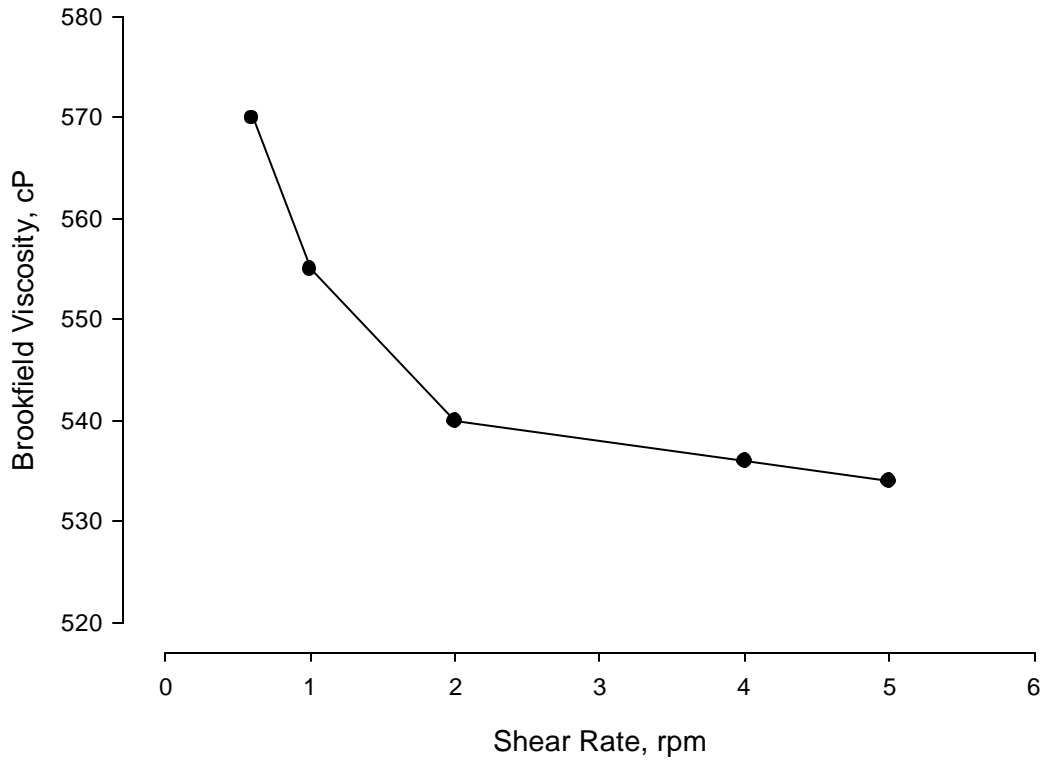
At this point, a brief discussion concerning some rudimentary elements of rheology may be useful. The terms thickener and thickened are used numerous times in this report. A common misconception that exists among those not experienced in the chemistry of true thickened materials is that to thicken something simply means to increase its viscosity. This is incorrect. To truly thicken a fluid and to simply increase its viscosity are two entirely different processes. For instance, if 95 ml of a 10 cP Newtonian fluid are mixed with 5 ml of a 10,000 cP Newtonian fluid, and if the two fluids are completely miscible and non-reactive with each other, and if the solution found is ideal or nearly so, then the final viscosity will be about 14 cP. This is because the logarithm of the final viscosities of such fluid mixtures is simply determined as the volume percent weighted sum of the logarithms of the individual component viscosities. The final fluid in this example mixture has not been thickened; it has only had its viscosity increased. The final fluid will typically be a single phase Newtonian fluid, just as its individual fluid components were

Newtonian. In contrast, fluids that are truly thickened, are typically two phase systems, even though they often appear to be only one phase on a macroscopic scale. Examples of true thickened fluids include tomato catsup, lubricating greases, and Jell-O-brand gelatin (an extreme case). All of these “fluids” are non-Newtonian, and all have one or more separate, discontinuous phases that thicken a continuous phase. The structure of the discontinuous phase (the thickener) will change as the entire fluid is mechanically stressed. This change in the thickener structure will in turn result in a change in the viscosity. Most thickened fluids decrease in viscosity as the fluid is stressed (pseudoplasticity). Often, thickened fluids will suffer permanent viscosity loss with sufficient shearing. Such behavior (thixotropy) corresponds to a permanent alteration of the microscopic structure of the thickener phase within the continuous phase. [19]

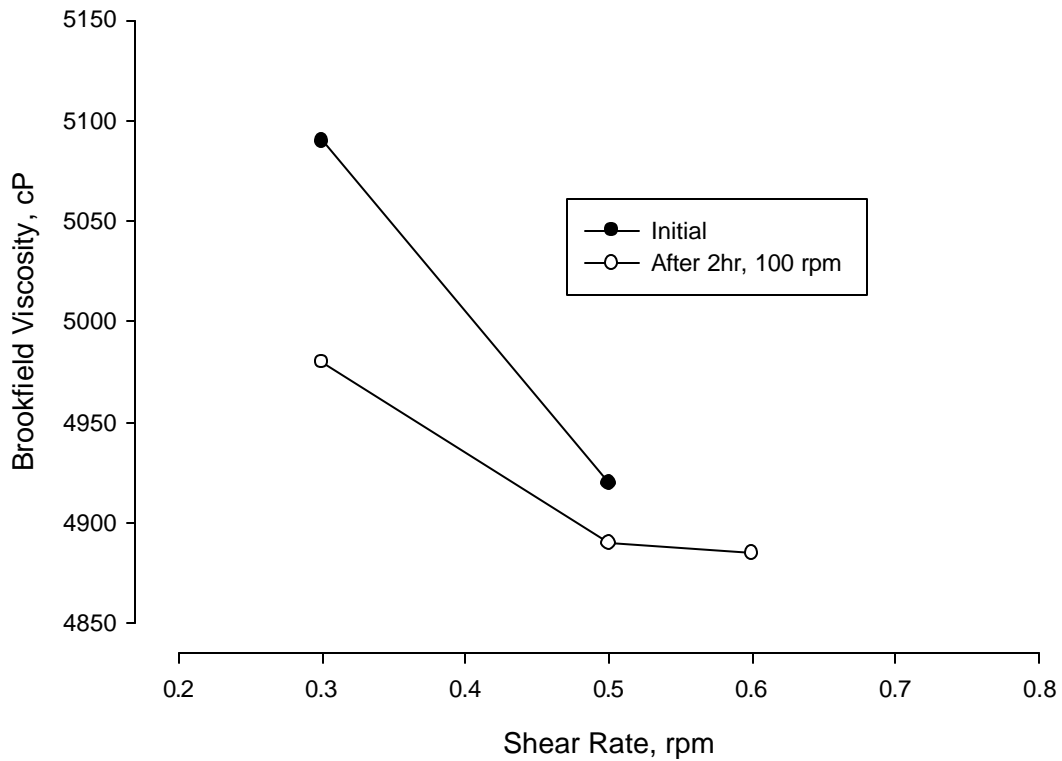
The fact that all thin apple jelly samples were observed to be Newtonian while all thick apple jelly samples are non-Newtonian is not an irrelevant fact. It strongly supports the idea that whatever is causing the thick apple jelly to have a viscosity significantly higher than any pure water/DiEGME blend (usually by a factor of more than 1,000) is not simply some blend of highly viscous, miscible fluids, or other materials dissolved in the water/DiEGME solution. The material responsible for the greatly increased viscosity of thick apple jelly compared to blends of water and DiEGME appears by all the data thus far reported to be a bonafide thickener.

It should also be pointed out that some of the most common thickeners are metal salts of weak organic acids. Entire classes of lubricating greases are based on such thickeners. [20,21,22] Also, high molecular weight polymers in combination with (metal-containing) salts have been shown to cause extreme thickening, as invented and patented by J. A. Waynick. [23] The previous data showing that an extremely strong correlation exists between sodium, TBN, TGA residue, and viscosity, and its implication of a sodium salt of a weak acid in combination with some high molecular weight species, is perfectly consistent with the composition of well established, true thickeners. Therefore, the non-Newtonian behavior of thick apple jelly is not merely relevant but essential in any study aimed at determining the composition, chemistry, and formation dynamics of such materials.





**Figure 38. Viscosity vs. Shear Rate, AP-117 at 25°C**



**Figure 39. Viscosity vs. Shear Rate, AP-044 at 60°C**

## 10 LABORATORY SYNTHESIS OF APPLE JELLY

This section presents the results of efforts to synthesize apple jelly in the laboratory. Based on the laboratory analyses presented above, it was apparent that the steps to synthesize samples of both thick and thin apple jelly would have some differences. Since apple jelly is primarily water and DiEGME, we started with a 1:1 blend of these. It is noted that apple jelly in the field has varying amounts of these two liquids and the properties of the resulting apple jelly vary accordingly. No attempt was made to synthesize apple jelly-type materials using all observed water/DiEGME ratios since such work was outside the scope of this project.

### 10.1 Thin Apple Jelly

#### 10.1.1 FT-IR Properties of Thin Apple Jelly

Figure 40 shows the FT-IR spectra for water, DiEGME, and a 1:1 blend of the two. The spectra demonstrate that there are spectral differences between water and DiEGME, as expected. It also shows that the blend contains spectral characteristics of both.

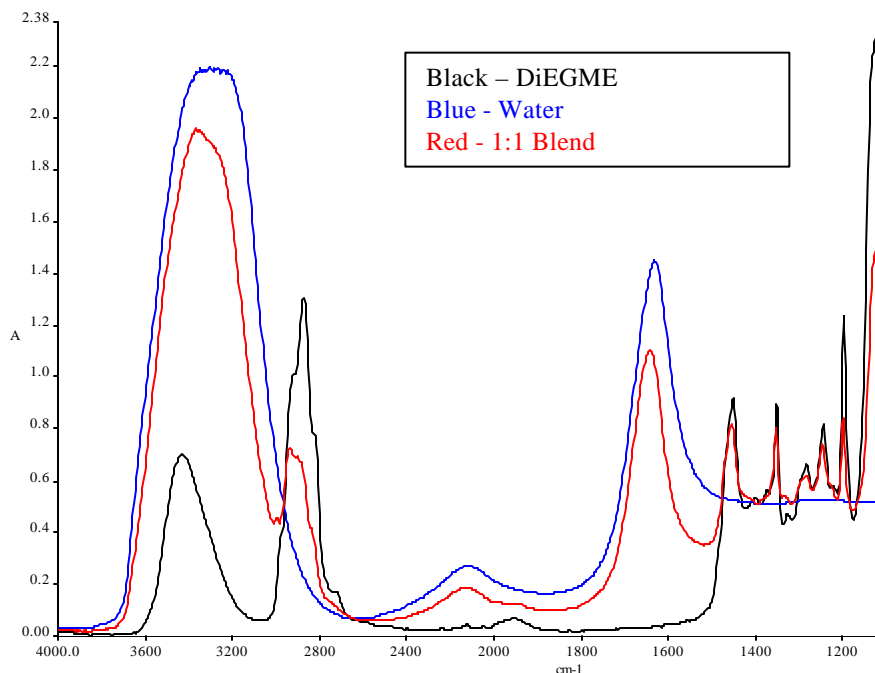


Figure 40. FT-IR Spectra of Water, DiEGME, and a 1:1 Blend of Each

Figure 41 shows the FT-IR spectra of a 1:1 blend of water and DiEGME; and, a sample of thin apple jelly. Notice that the two spectra are nearly equivalent, with only slight differences in some peaks due to concentration effects. The only significant difference between the two is the presence of a small peak in the apple jelly spectrum at  $1375\text{ cm}^{-1}$ . An enlargement of the region around  $1375\text{ cm}^{-1}$  is shown in Figure 42. Taken together Figures 40, 41 and 42 further demonstrate that thin apple jelly is mostly water and DiEGME with small amounts of various other compounds dissolved. Because, by definition, all thin apple jelly samples have viscosities that correspond to what is expected based on their water/DiEGME ratios, there does not appear to be significant levels of any thickener.

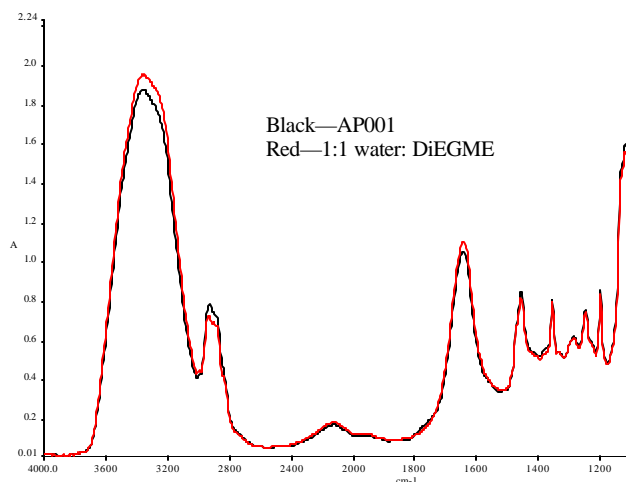


Figure 41. FT-IR Spectra of Water/DiEGME and a Representative Thin Apple Jelly

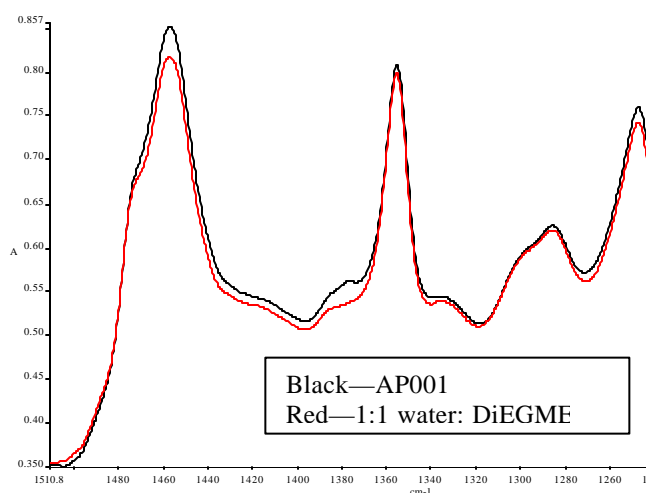


Figure 42. Enlarged Region of Spectra Shown in Figure

### ***10.1.2 Minor Constituents of Thin Apple Jelly***

It was well known that water/DiEGME is an aggressive solvent and will extract materials from its environment. While we knew that not all apple jelly is equivalent, we also knew that there were similarities in the samples we had received. Chief among these similarities was color (in most samples) and greatly increased conductivity (in all samples) compared to simple blends of water and DiEGME. Our analyses determined that minor constituents in thin apple jelly included sulfur compounds, nitrogen compounds, sodium, weak acids and bases, and various other ionic species. We assume that there are other unidentified species present as well. Because of these similarities, we knew that the primary source(s) of the minor constituents had to be common throughout the system. Our list of potential sources included the fuel, fuel additives, fuel filters, common elastomers, and fuel system surfaces.

Based on all the data thus far developed, we chose SDA for our first investigation of potential components in thin apple jelly. Specifically, this choice was made based on the following previously established facts:

- All thin apple jelly had conductivities higher than pure water/DiEGME blends. SDA is an additive blend intentionally formulated with a pronounced ionic character designed to enhance conductivity. On the other hand, corrosion inhibitor is only very slightly ionic ( $pK_a \sim 5$ ), while fuel components are virtually non-ionic.
- All thin apple jelly samples had both TAN and TBN values significantly higher than the virtually zero value expected for blends of pure water and DiEGME. SDA has a TAN of 24.1 and a TBN of 47.4. Conductivity improver, on the other hand has a TAN of 115.7 and a TBN of only 0.37, as expected for a di-carboxylic acid-based additive. JP-8 base fuel typically has very low TAN (well below the 0.015 max specification) and a TBN that is typically immeasurably low when using ASTM D2896.
- While the exact source of sodium in apple jelly had not yet been determined at this point in the investigation, SDA was the only commonly and consistently available

sodium-containing source introduced relatively close to the end user, albeit at a very low level.

- SDA contains nitrogen and sulfur, both determined to be present in thin apple jelly but not present in pure water/DiEGME blends. Corrosion inhibitor contains neither sulfur nor nitrogen. (All dimer acid corrosion inhibitors typically used in JP-8 contain essentially only carbon, hydrogen, and oxygen.) While JP-8 base fuel does contain small amounts of both nitrogen and sulfur, previous data showed neither element is concentrated in apple jelly compared to the fuel layer sample with which it was in equilibrium prior to sampling and analysis.
- Ion Chromatography data clearly indicated that certain SDA components were present in all apple jelly, thin and thick. While the identity of those indicated components were mostly unknown (except for the easily identifiable chloride), their presence was established. All attempts to characterize the presence of both base JP-8 components and corrosion inhibitor in apple jelly samples were unsuccessful since no anion peaks could be measured in the neat additive or base fuel.

Based on these points, SDA was the logical first choice for further investigations as to the role of the various potential minor constituents of thin apple jelly. At this point it should be pointed out that this reasoning does not imply that SDA is implicated as the only possible source of minor thin apple jelly constituents. We fully realized at this point in the investigation that it was not only possible but also likely that some of the minor constituents of thin apple jelly were derived from sources other than SDA. In fact, as shall be discussed later, the role of another source of minor thin apple jelly constituents proved to be not only significant, but also necessary. Nonetheless, the investigation had to begin with one potential source of minor thin apple jelly constituents. The overall data as described in the above points supported the choice of SDA. The previously cited work by Beal and Hardy [26] also pointed to a possible link between SDA and filter plugging in water saturated DiEGME/JP-8 blends. However, based on the data we had developed thus far in our study, it was unclear how this study related to the apple jelly formation process. While this work was interesting, and the methods used were rather ingenious, it did not significantly figure into our decision to look at SDA first.

### ***10.1.3 Extraction Properties of SDA***

#### **10.1.3.1 Extraction Properties of SDA into DiEGME**

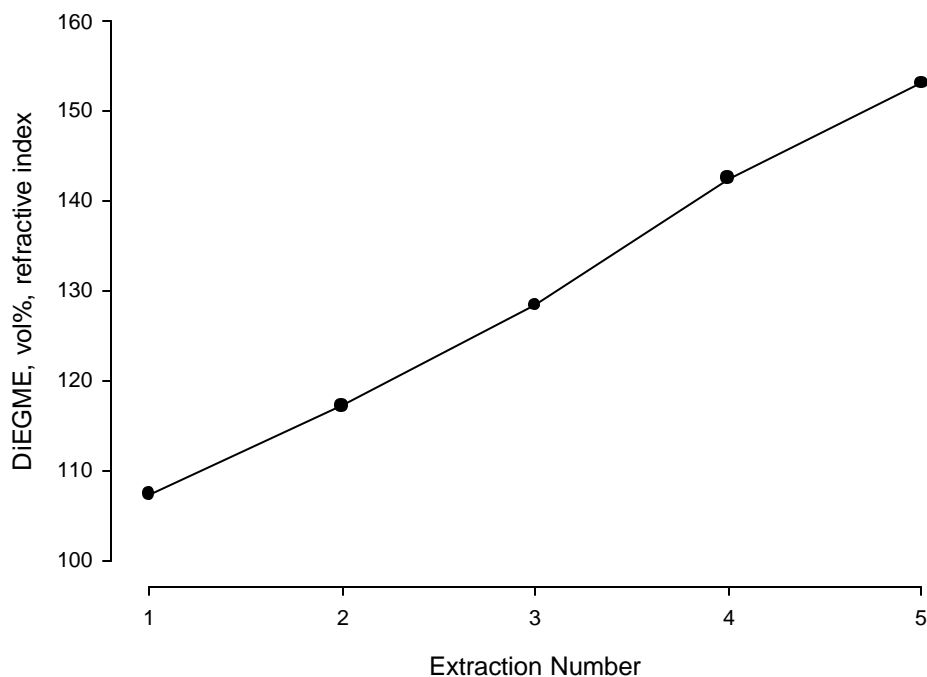
Previous data given in this report have shown apple jelly to be a complex set of materials with some common characteristics and some characteristics that vary from one type of apple jelly to another. In an attempt to characterize any potential role of SDA components in thin apple jelly, we thought it would be best to begin by determining some basic properties of SDA in blends of water and DiEGME. This preliminary work was necessary since such information could not be found in the open literature. To make such evaluations in all possible blends of water and DiEGME was clearly outside the scope of this project. As a starting point, we chose to determine the extraction properties of SDA in pure DiEGME. We fully understood that if SDA components have any role as minor apple jelly constituents, their extraction and solubility in blends containing both water and DiEGME would be the most relevant properties. However, since organic compounds (such as the major components of SDA) would likely be more soluble in DiEGME than water, the solubility and extraction properties of SDA in pure DiEGME were evaluated first as a foundational basis for later studies in water and in blends of DiEGME and water.

Since organic compounds are more soluble in DiEGME than water, the solubility and extraction properties of SDA in pure DiEGME were evaluated first. A 200-ml sample of DiEGME was thoroughly mixed with 25 ml of SDA in a separatory funnel and allowed to separate into two phases. The DiEGME phase was removed, a small sample was taken for testing, and the remainder was thoroughly mixed with another 25-ml portion of SDA. After allowing the two phases to separate, the DiEGME phase was again removed, a small portion was taken for testing, and the remainder was again thoroughly mixed with another 25-ml portion of SDA. This procedure was repeated until the extraction process had occurred 5 times. The portions of the DiEGME phases removed after each of the 5 extractions were tested for the following properties:

- % DiEGME by refractive index
- TAN
- TBN

- Total N
- Total S
- TGA residue
- Karl Fisher Water
- Sodium

Figure 43 shows the % DiEGME by refractive index (RI) for each extraction. We performed this analysis to determine the potential interaction of SDA components and DiEGME and their effect on the analysis. As indicated, the DiEGME concentration begins at around 107% and steadily increases with each extraction. Obviously, one would expect that the DiEGME content should never be more than 100%. The results obtained here demonstrate that compounds extracted into DiEGME will interfere with the refractive index measurements and cause erroneous results. This behavior is similar to the anomalous behavior observed in the thick apple jelly samples in Figure 9. However, none of the extracted DiEGME samples exhibited any thickening. The steady increase in RI readings does prove that SDA components are extracted into DiEGME and the amount increases with each extraction.



**Figure 43. DiEGME Content Following Successive Extractions of SDA with DiEGME**

Figure 44 shows the TAN and TBN as a function of extraction number. TAN increases steadily, reaching within the levels commonly observed in thin apple jelly samples. TBN remains at zero until the fourth extraction, then begins to increase. Note that, at least in pure DiEGME, extracted SDA components contribute far more to TAN than to TBN.

Figure 45 shows total nitrogen and sulfur levels as a function of extraction number. Both increase with sulfur being present in much greater levels. It must be noted that the much greater level of sulfur compared to nitrogen is not what is typically observed in the apple jelly samples.

Figure 46 shows the TGA residue as a function of extraction number. As seen, TGA residue increases almost linearly with extraction number. This is very similar to the relationship between TGA residue and viscosity previously shown in Figure 37. However, none of the five extracted DiEGME samples showed any evidence of thickening.

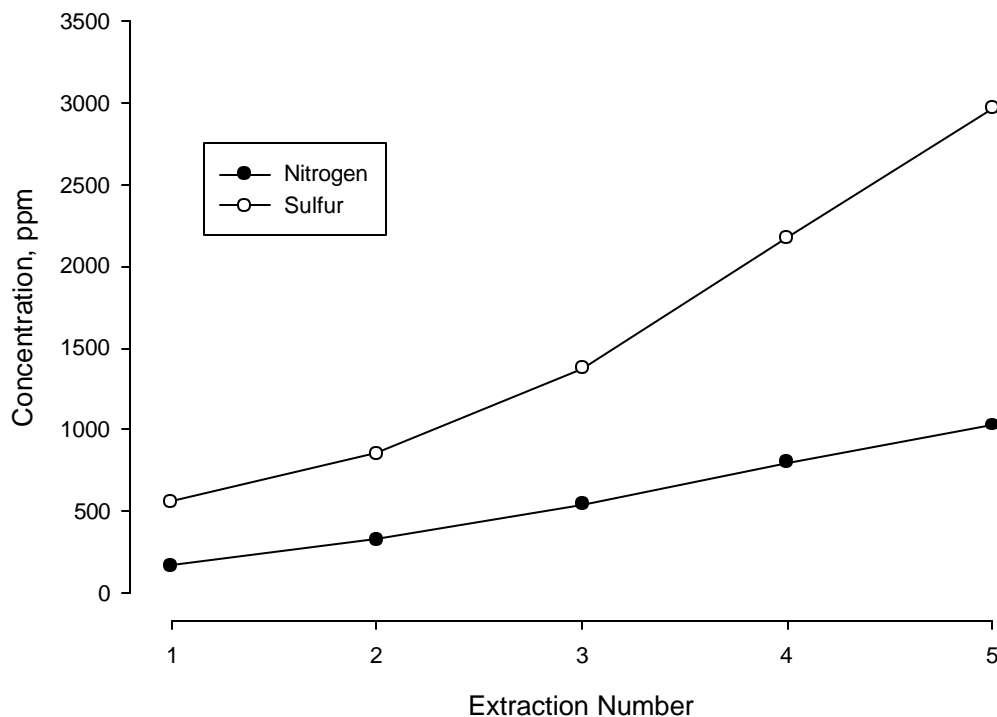
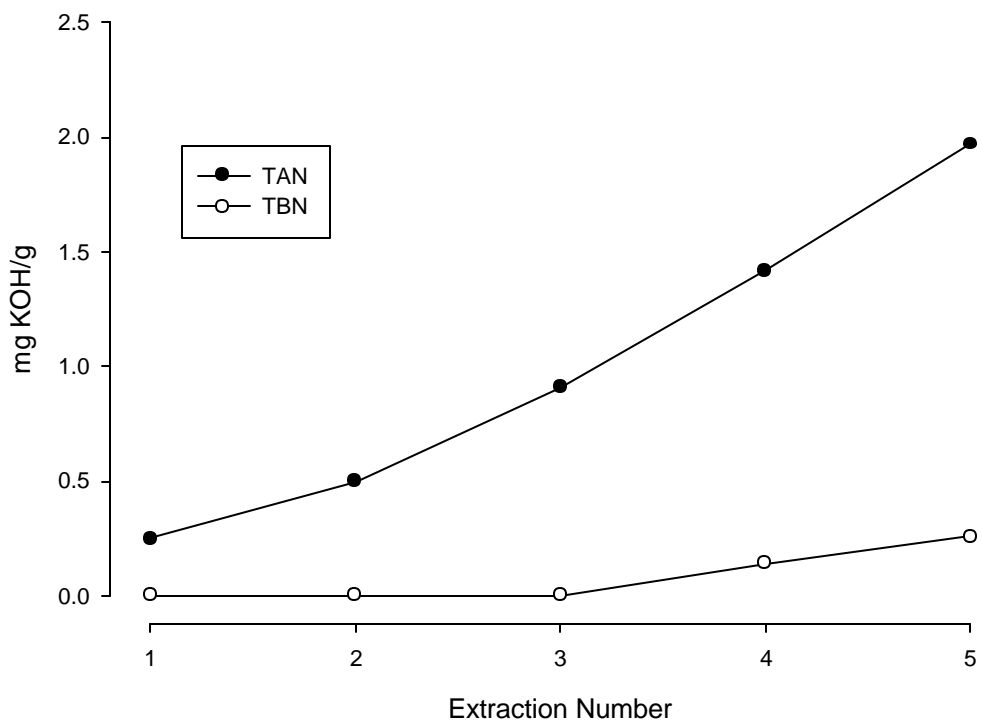
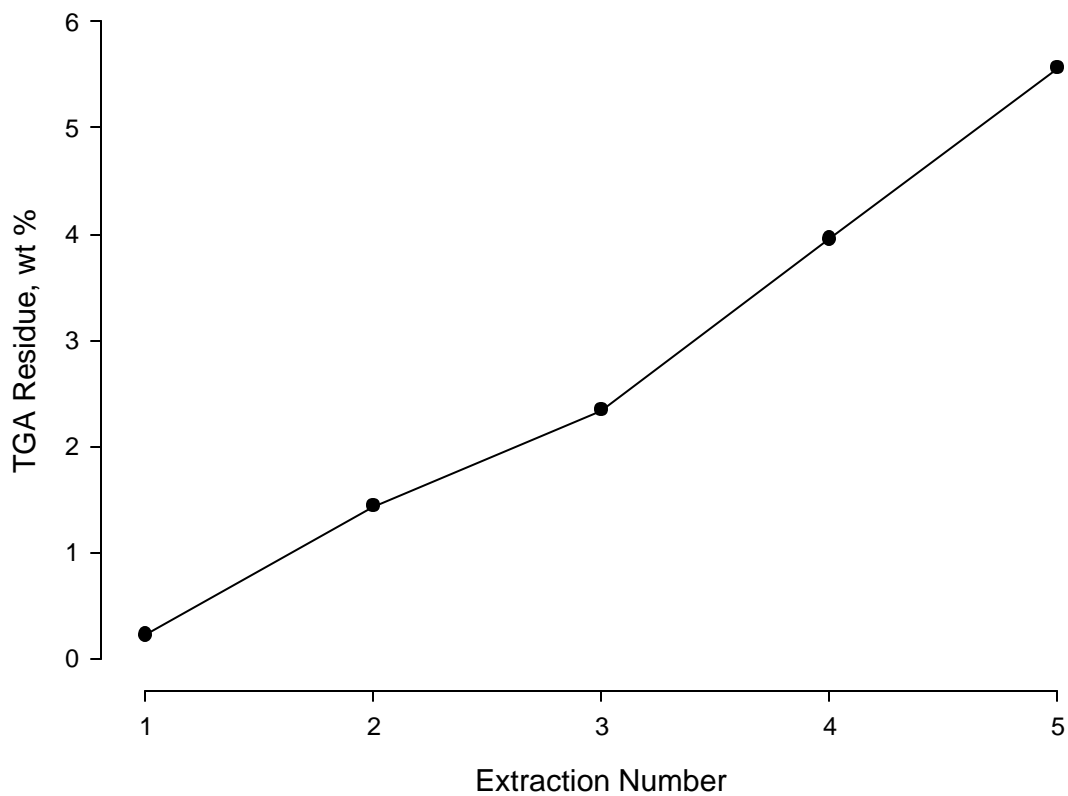


Figure 44. Total Acid and Base Numbers of DiEGME Following Successive Extractions of SDA





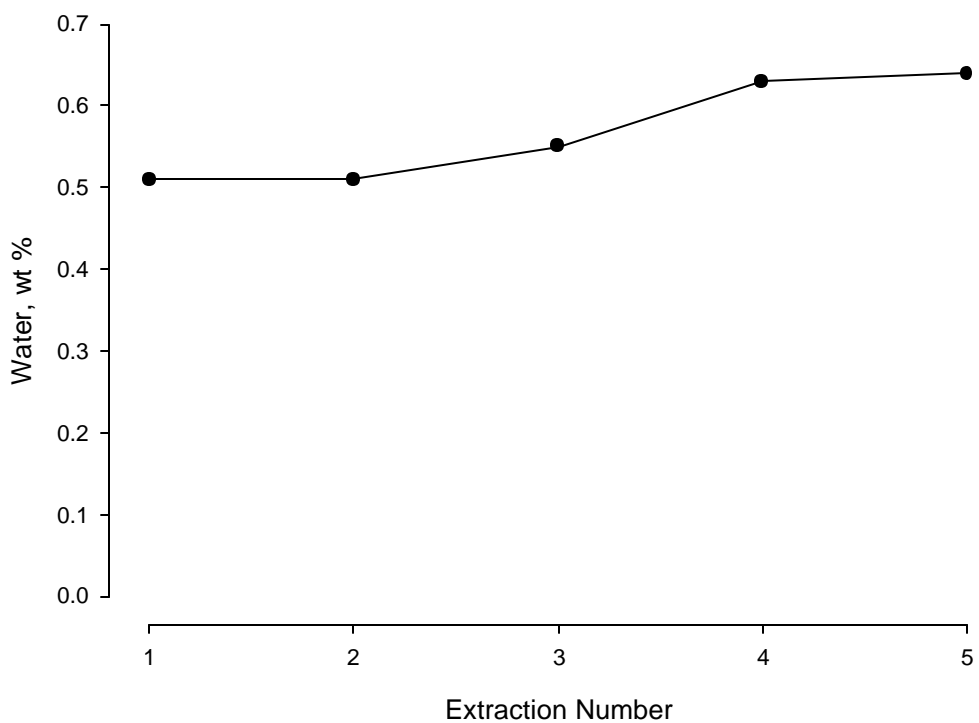
**Figure 45. Total Nitrogen and Sulfur in DiEGME Following Successive Extractions of SDA**



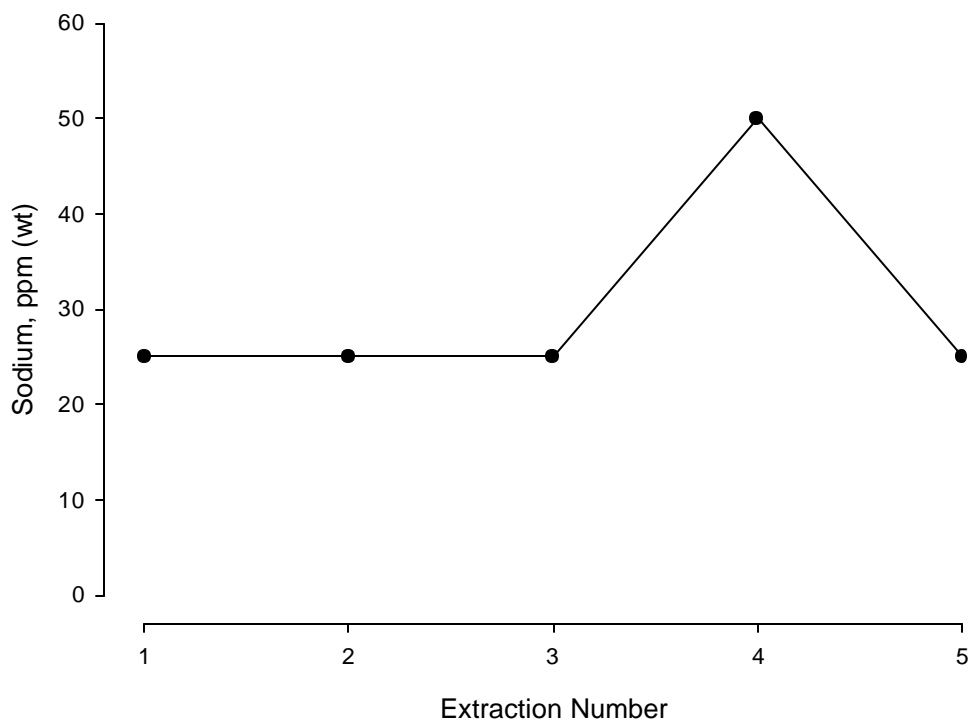
**Figure 46. TGA Residue of DiEGME Following Successive Extractions of SDA**

Figures 47 and 48 and show that total water and total sodium contents, respectively, did not vary significantly with extraction number. The sodium result is particularly interesting since it implies that the very small sodium content of neat SDA (about 33 ppm) does not progressively accumulate in DiEGME, and is therefore assumed to not be the source of the very large sodium contents of thick apple jelly. *[The apparent increase in sodium content for extraction #4 is an artifact of the digestion process used to prepare the samples for analysis. The process includes a 25X-dilution step. The results actually obtained from the analyzer were 1 ppm and 2 ppm. The small difference in these results was magnified by the 25X dilution and hence the apparent increase in the plotted results. The plotted results should be considered essentially equivalent.]*

In this experiment it should be noted that although equilibrium was established during each extraction, the composition of the DiEGME phase obviously changed with each extraction. This is due to the fact that SDA is a multi-component additive blend. With each extraction, a new portion of SDA is equilibrated with a DiEGME phase that has varying levels of SDA components, according to the equilibrium previously established. The next equilibrium will therefore be expected to be different than the previous one. Indeed, the data indicates that as certain SDA components begin to extract into DiEGME, their presence will further enhance the solubility of other SDA components. In subsequent discussions, the term “extraction of SDA” will be understood to mean extraction of various components of SDA. It is implicitly recognized from this point throughout the remainder of this report that SDA is a multi-component blend, and that data showing extraction and solubility of SDA is actually providing information on the simultaneous extraction and solubility of various components.



**Figure 47. Total Water in DiEGME Following Successive Extractions of SDA**



**Figure 48. Total Sodium in DiEGME Following Successive Extractions of SDA**

Additionally, after each extraction the DiEGME phase became more colored, going from light amber to dark amber to amber/brown. Because of the volume required, conductivity was measured only on the final extracted DiEGME phase. The conductivity was 23.4  $\mu\text{S}/\text{cm}$ . This is nearly 60 times greater than the previously determined conductivity of pure DiEGME of 0.39  $\mu\text{S}/\text{cm}$ . Also, it should be restated that this experiment was designed to establish foundational information concerning extraction equilibrium of DiEGME with neat SDA. In doing this work, we fully intended to follow it up with additional foundational studies of SDA extraction properties in pure water and in a few blends of water and DiEGME. In fact, this follow-up work is presented in the next two sections of this report. Also, in actual field applications where thin apple jelly forms, any SDA that is extracted into a phase containing DiEGME will most often do so while being dissolved in the JP-8 base fuel. Since the typical concentration of SDA in JP-8 is nominally 1 ppm, the solvent properties of base fuel have not been accounted for in this experiment. However, the solvent effects of JP-8 can be determined, as the following experiment shows.

To determine the solvency effect of the JP-8 base fuel, the final extracted DiEGME phase from the previous experiment was thoroughly mixed with some AP-003, a JP-8 fuel. The resulting DiEGME phase was separated, tested, and compared to the results before the JP-8 extraction. The results are provided in Table 8.

<b>Table 8. Extraction of SDA into DiEGME – Effect of Fuel</b>		
Test	Before fuel extraction	After Fuel Extraction
Total N, ppm (wt)	1029	427
Total S (ppm)(wt)	2964	740
TAN, mg KOH/g	1.97	0.38
TBN, mg KOH/g	0.26	Nil
TGA Residue, %(wt)	5.6	1.3
KF Water, %(wt)	0.64	0.66

Nitrogen content decreased by 59% while sulfur content decreased by 75%. This suggests that the previous experiment overstated the tendency of sulfur to extract into DiEGME compared to nitrogen. This is important since it is directionally in agreement with results from actual apple jelly samples. TAN and TBN were both reduced as a result

of extraction with JP-8, the latter decreasing to non-detectable levels. This continues to suggest that any SDA components that extract into DiEGME-rich water bottoms may impact TAN much more than TBN although experimentation in water/DiEGME blends is required for further substantiation. TGA residue also dramatically decreased from 5.6% to 1.3%. This number is more in line with what is observed in actual thin apple jelly samples. It also implies that the cause of the increasing TGA residue in thick apple jelly is not primarily due to SDA. This is important since SDA does not meet the properties already established as likely attributes of the thickener present in thick apple jelly. Specifically, previous data indicates that the thickener in thick apple jelly contains sodium, but the information provided above suggests that the very small sodium content in SDA does not progressively extract into DiEGME. Previous data indicates that the thickener contains a weakly basic material that will titrate in the TBN procedure. However, data here suggest that the TBN contribution from SDA in DiEGME will be much less than the TAN contribution. Previous data indicates that the thickener does not contain nitrogen or sulfur, but SDA contains both, and both are extracted into DiEGME.

#### 10.1.3.2 Extraction Properties of SDA in Water

Once the extraction properties of SDA in DiEGME had been evaluated, the next logical step in determining foundational information on SDA extraction properties was to determine the extraction properties of SDA in water. Attempts were made to dissolve neat SDA in de-ionized water. Approximately 50 ml of de-ionized water was vigorously shaken with about 5 ml of SDA. The result was a turbid aqueous phase that mostly cleared over several days. The final aqueous phase was analyzed for total nitrogen and sulfur and results were 20 ppm (wt) and 60 ppm (wt), respectively. This result is not surprising since it is expected that most if not all, SDA components are substantially insoluble in water.

#### 10.1.3.3 Extraction Properties of SDA in DiEGME/Water Blends

Once the extraction properties of SDA in both DiEGME and water had been investigated, the next step was to investigate such behavior in blends with water and

DiEGME, since all submitted apple jelly samples contain both. As a first attempt to determine the solubility of SDA components in blends of DiEGME and water, about one milliliter of water was added to about 10 milliliters of the final extraction of the DiEGME phase from the experiment discussed in section 10.1.3.1. Immediately upon shaking, the water and DiEGME combined to form a clear phase and the SDA components that had been dissolved in the DiEGME separated out as a clear amber phase on top. Attempts to dissolve SDA into blends of DiEGME and water also failed, yielding only emulsions and precipitates.

#### ***10.1.4 Implications from Extraction Behavior of SDA***

The extraction experiments discussed in the preceding sections, especially those involving water are very interesting in light of what has already been observed in actual thin apple jelly. Specifically, the following items are known to be true about thin apple jelly samples:

- Ion chromatography indicates that SDA components are in thin apple jelly.
- Thin apple jelly does not appear to be an emulsion.
- Thin apple jelly does not form precipitates when either water or DiEGME is added.
- Thin apple jelly appears to be a stable solution.
- Thin apple jelly has at least 40% water.

Based on these observations, we next attempted to answer the question of how SDA components dissolve in the DiEGME/water matrix of thin apple jelly? It is clear from the foundational data discussed in section 10.1.3.1 that SDA components do have limited solubility in pure DiEGME. However, the presence of even minor amounts of water appears to prevent such SDA component solubilization from occurring. If SDA components are indeed present in thin apple jelly as the above points indicate, then some other factor must be involved. One factor that had not yet been considered was the base fuel itself.

### ***10.1.5 Effect of JP-8 Base Fuel***

#### 10.1.5.1 General Information

Our initial extraction studies concentrated on the potential effects and contributions of SDA. As discussed in section 10.1.2, SDA was examined first as a potential minor constituent based on a large amount of data that existed at that point in the investigation. However, it was recognized from the beginning that certain fuel components could also play a role. It was already known through the work of Wechter and Hardy [24,25,26] that methyl alcohol can extract polar materials from distillate fuels. The fuel compounds extracted by methyl alcohol are known to be rich in nitrogen and sulfur relative to the base fuel. Based on un-published work, it is also known that much of the material extracted from distillate fuels by methyl alcohol is in the heaviest boiling fraction of the fuel. Since methyl alcohol is the most similar to water of all simple alcohols, and since DiEGME is nearly as polar and slightly larger than methyl alcohol, it seems reasonable that DiEGME would also be able to extract components from distillate fuels such as JP-8. However, when a significant level of water is also present, the situation becomes more complex. Furthermore, if DiEGME can extract JP-8 components, what effect will such extracted materials have on subsequent extraction behavior of SDA?

#### 10.1.5.2 Effect on Extraction of SDA Components

To answer these questions, a set of experiments was conducted. First, portions of AP-003, a JP-8, were distilled via ASTM D86 until only the heaviest 5% remained. These distillations continued until about 50 ml of the heaviest 5% of the JP-8 were collected. A 47-ml portion was added to 10 ml of a 1:1 (v/v) blend of DiEGME and water and vigorously shaken in a separatory funnel. After the two phases separated, the DiEGME/water phase was distinctly yellow. A small portion of this yellow solution was removed for analysis. Then, a few milliliters of SDA were added to the separatory funnel and the contents were again shaken. After separation the DiEGME/water phase was orange. This DiEGME/water phase was removed, labeled as JAW1-75-1, and tested. A

similar experiment was performed using 47 ml of whole JP-8 (AP-003) instead of the heaviest 5% of the JP-8. After mixing with 10 ml of the 1:1 (v/v) blend of DiEGME and water, the DiEGME/water phase remained colorless.

The results of this experiment are given in Table 9. As seen, significantly more nitrogen and sulfur were extracted into the DiEGME/water phase from the heavy 5% fuel fraction than from the whole fuel. However, the most significant results are the effect that these heavy fuel components had on subsequent extraction of SDA. Both nitrogen and sulfur levels increased dramatically. Nitrogen was present at 253 ppm (wt) compared to the 20 ppm (wt) observed when SDA was added directly to water. Similarly, sulfur was present at 220 ppm (wt) compared with 60 ppm (wt) when SDA was added directly to water. The orange color is a clear indication that SDA components were extracted into the DiEGME/water blend. This is in stark contrast to the results obtained when trying to extract SDA directly into a 1:1 (v/v) blend of DiEGME and water. Also, it is noteworthy that 85% of the nitrogen in JAW1-75-1 was incorporated after extraction with SDA. Similarly, 60% of the sulfur is from SDA. This data proves that materials in JP-8 can extract into blends of DiEGME and water, even when water is present at 50%. If those extracted fuel components are sufficiently concentrated in the DiEGME/water, SDA components can and will be extracted as well, an effect that does not occur without the presence of the fuel components. It should be noted however that this work does not prove that the fuel components that solubilize SDA in DiEGME/water blends are limited to the heaviest 5%. In fact, it is assumed that components from other portions of the boiling range of the fuel may extract into water/DiEGME blends. Furthermore, the identity and relative effectiveness of the fuel components with respect to SDA extraction enhancement cannot be deduced from this data. The work to determine the identity of all the extracted fuel components and their individual effects on SDA component solubility is outside the scope of this project.

<b>Elemental Analysis</b>	<b>After extract with JP-8</b>	<b>After extract with heavy 5% of JP-8</b>	<b>After extract with heavy 5% of JP-8 and SDA (JAW1-75-1)</b>
N, ppm(wt)	3	40	253
S, ppm(wt)	13	90	220



At this point, some additional comments are useful regarding this experiment and the resulting data. When the SDA was added, the separatory funnel still contained the heaviest 5% fraction of the JP-8 (top phase) and the 1:1 (v/v) blend of water and DiEGME (bottom phase). Prior to this addition, the heavy fuel components in the top phase and the water/DiEGME blend in the bottom phase had established equilibrium. The introduction of the few milliliters of SDA obviously caused a shift in that equilibrium, as evidenced by the change in both color and chemical composition of the bottom water/DiEGME phase. Although a detailed analysis of the heavy 5% fraction of the JP-8 was not done, certain general characteristics are nonetheless known. Total nitrogen and sulfur levels of the heavy fuel fraction will be much higher than the whole fuel since it is well known that such components concentrate in the heaviest boiling fractions (as shown in this case in Table 11 in section 10.1.5.4). It has also been well established that the most aromatic components of kerosene-based jet fuels (and No. 2 diesel fuels) are concentrated in the heaviest boiling fractions. These materials are among the most polar and polarizable components in the whole jet fuel. Based on what is known about the composition of SDA, it is reasonable to expect SDA is going to be more soluble in the heaviest JP-8 fraction than in the whole JP-8. In fact, during this experiment as a preliminary check, a few milliliters of the heaviest 5% of the JP-8 was progressively admixed with some SDA in a small clear glass vial. There appeared no signs of phase separation or insolubility. In contrast, if SDA is added to a whole JP-8 in levels approaching 1%, the fuel becomes hazy, indicating that one or more components of SDA have exceeded their solubility limits in the fuel. Obviously, SDA (a mixture containing very polar organic compounds) is much more compatible (with respect to solubility) with the polar heavy boiling fraction of the fuel than with the whole (and relatively non-polar) fuel.

The increase in nitrogen and sulfur that occurred after addition of SDA is not likely to be an indication that the SDA forced additional heavy fuel components into the water/DiEGME phase while the SDA components rigorously remained totally in the heavy fuel phase. For such a scenario to occur would require that the heavy fuel components be more incompatible (with respect to solubility) with SDA than with a 1:1 blend of water/DiEGME, something that is clearly not so.

A far more likely explanation for the increase in nitrogen and sulfur in the water/DiEGME phase is the one previously given, namely that certain SDA components can begin to become solubilized in water/DiEGME blends once sufficient heavy fuel polars have been extracted. However, the idea of additional fuel components being extracted into the water/di/EGME phase is not entirely unreasonable. Once certain SDA components are extracted into the water/DiEGME phase, it is quite possible that the resulting water/DiEGME phase would be more attractive towards further extraction of certain heavy fuel components. In fact, it could be possible that once heavy fuel components have sufficiently extracted into the water/DiEGME phase, extraction of SDA components and further extraction of heavy fuel components could mutually enhance one another. However, further work would be required to establish this possibility.

Recall from Figure 42 that all thin apple jelly as sampled in this project has a peak at about  $1375\text{ cm}^{-1}$  in the infrared spectrum and that mixtures of water and DiEGME alone do not give that peak. Also recall that this is the only significant difference in the FT-IR spectra of thin apple jelly compared to water/DiEGME blends. The FT-IR spectrum of JAW1-75-1 is compared to the spectrum for a 1:1 blend of water and DiEGME in Figures 49, 50 and 51. Notice in these three figures that there are differences between the 1:1 blend and JAW1-75-1. The FT-IR spectra of JAW1-75-1 is compared to spectra for a thin apple jelly sample and a 1:1 (v/v) blend of DiEGME and water are given in Figures 52, 53, and 54. Notice in these three figures that JAW1-75-1 has the peak at  $1375\text{ cm}^{-1}$  that is characteristic of thin apple jelly. The spectra of JAW1-75-1 and AP-001 are virtually identical. Specifically, the behavior around  $1375\text{ cm}^{-1}$  has the same peak position and shape. It is also noteworthy that the matching of FT-IR spectra around  $1375\text{ cm}^{-1}$  was achieved, while not introducing any new peaks not present in AP-001.

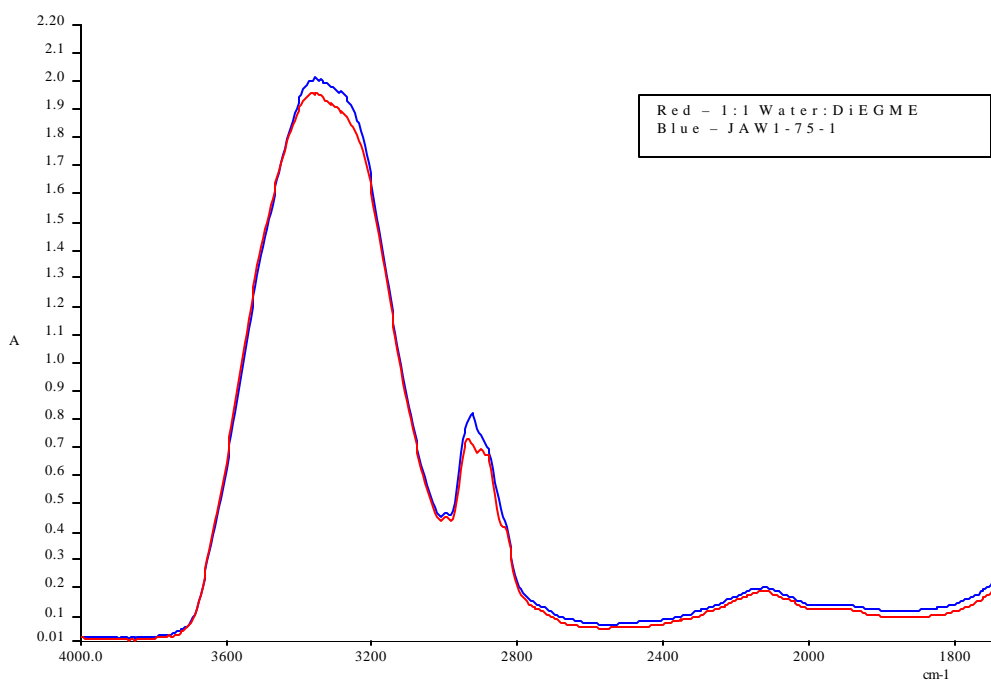


Figure 49. FT-IR Spectra of DiEGME/Water and JAW1-75-1 (1200 - 4000  $\text{cm}^{-1}$ )

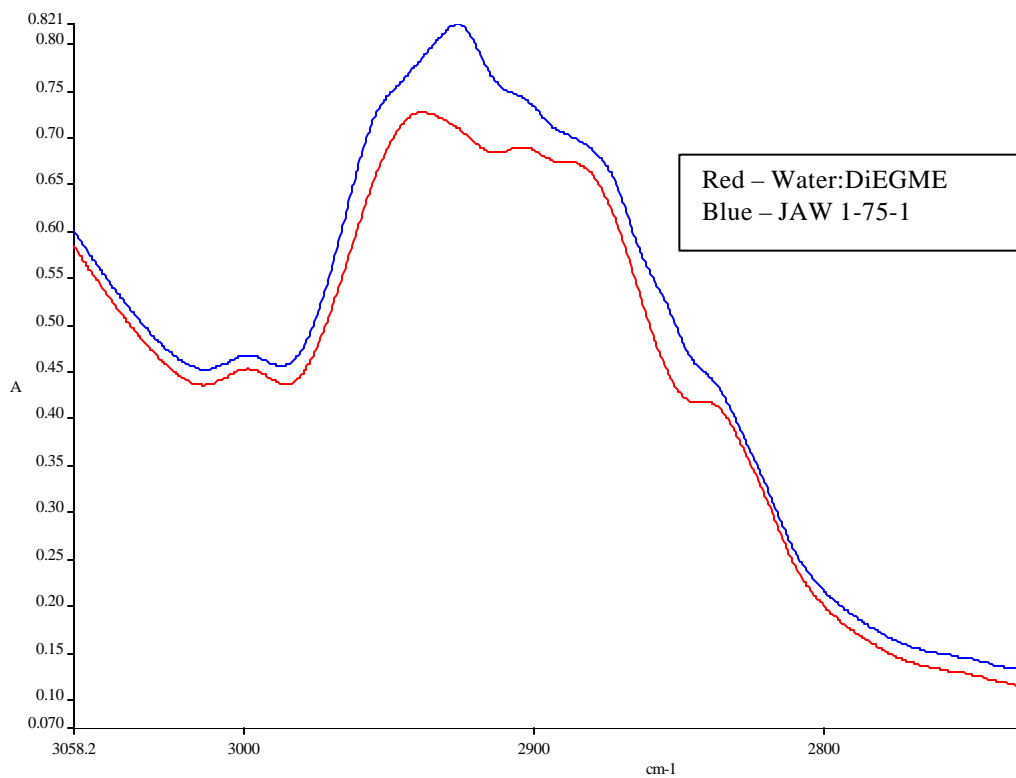


Figure 50. FT-IR Spectra of DiEGME/Water and JAW1-75-1 (2700 - 3058  $\text{cm}^{-1}$ )

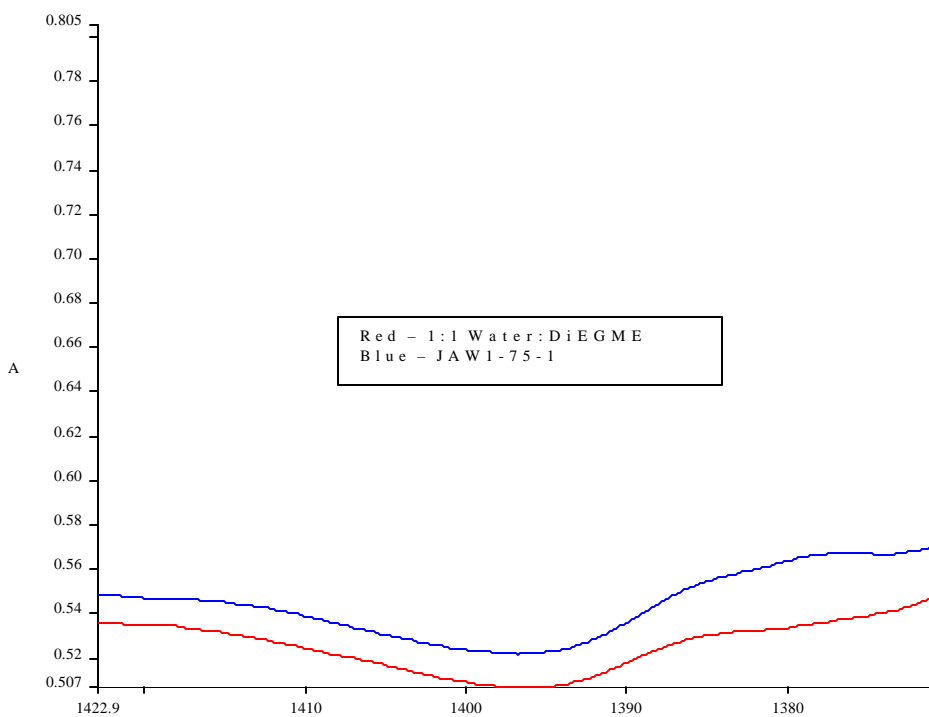


Figure 51. FT-IR Spectra of DiEGME/Water and JAW1-75-1 (1330 - 1420  $\text{cm}^{-1}$ )

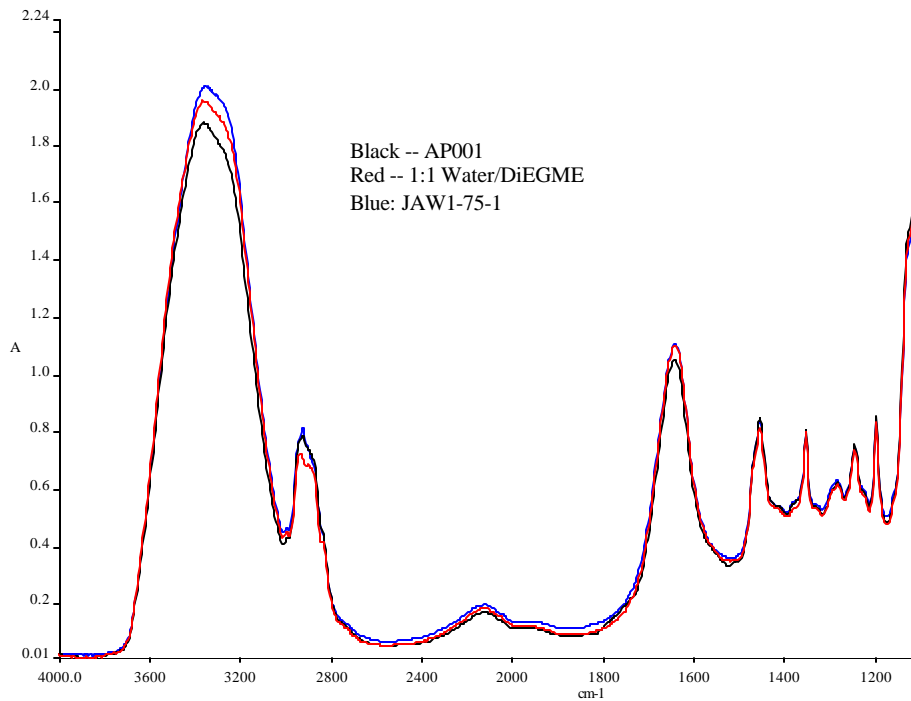


Figure 52. FT-IR Spectra of Thin Apple Jelly, DiEGME/Water, and JAW1-75-1 (1200 - 4000  $\text{cm}^{-1}$ )

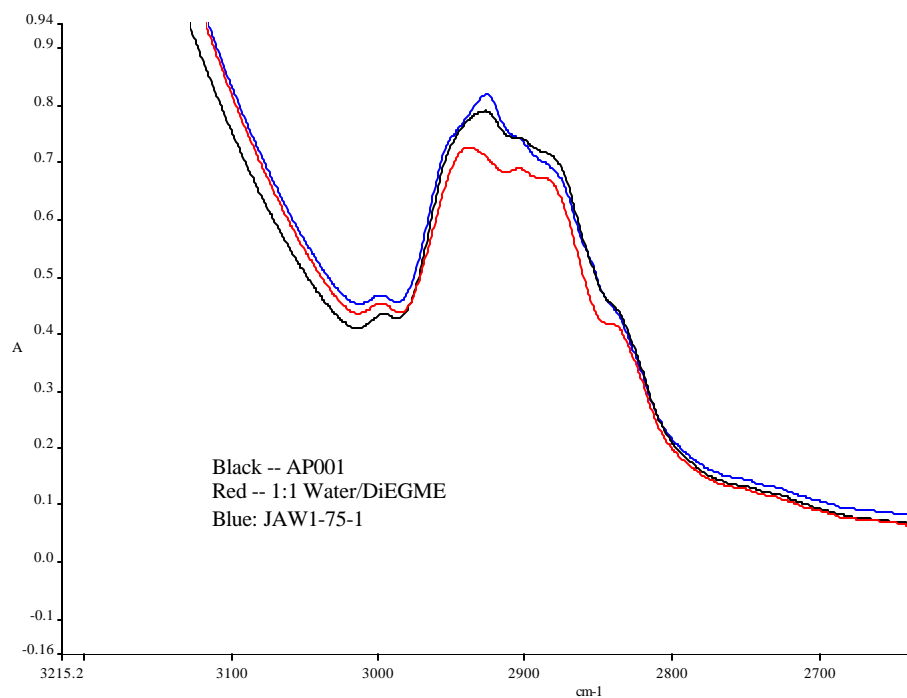


Figure 53. FT-IR Spectra of Thin Apple Jelly, DiEGME/Water, and JAW1-75-1 (2700 - 3215  $\text{cm}^{-1}$ )

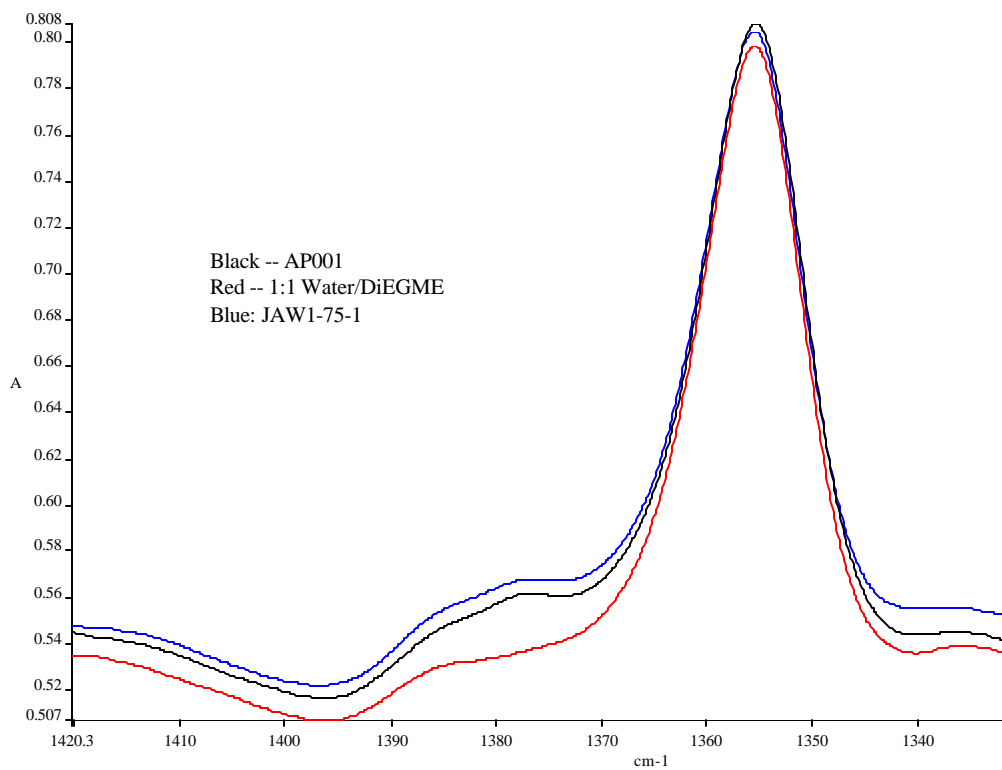


Figure 54. FT-IR Spectra of Thin Apple Jelly, DiEGME/Water, and JAW1-75-1 (1330 - 1420  $\text{cm}^{-1}$ )

### 10.1.5.3 Effect of Water Content

The previous experiment was repeated using a blend of two JP-8 fuels, AP-003 and AP-004. Portions of this fuel were repeatedly distilled via ASTM D86 to obtain the heaviest 10% portion. This distillation was continued until 200 ml of the heaviest 10% fraction of each JP-8 were collected. A 100-ml portion of this heaviest 10% fuel fraction was added to 10 ml of a 1:1 (v/v) blend of DiEGME and water in a separatory funnel. Another 100-ml portion was added to 10 ml of a 1:3 (v/v) blend of DiEGME and water in another separatory funnel. After vigorous shaking, the contents of both separatory funnels were allowed to separate into their two phases. The 1:3 DiEGME/water phase was yellow, but the 1:1 DiEGME/water phase was a darker shade of yellow.

A 10-ml portion of SDA was added to each separatory funnel and each was vigorously shaken. The 1:1 DiEGME/water solution separated from the fuel/SDA layer and was yellow. The 1:3 DiEGME/water layer did not separate from the fuel/SDA layer. Instead an emulsion and precipitate formed.

Results of analysis of the DiEGME/water layers from this experiment are given in Table 10. The general trends observed in the earlier experiment are seen in these data as well. Nitrogen and sulfur are extracted into the 1:1 DiEGME/water phase from the heavy 10% fraction of the JP-8, and subsequent extraction of SDA yields even larger amounts of nitrogen and sulfur. The levels of nitrogen and sulfur extracted into the 1:3 DiEGME/water phase from the heavy 10% fraction of JP-8 were somewhat less, but the subsequent enhancement to SDA extraction was not measured due to the absence of a distinct DiEGME/water phase. This result may indicate that the nitrogen and sulfur-containing species extracted from the 10% heaviest fuel fraction may not be responsible for the subsequent enhancement of SDA extraction. Whatever species in the heaviest fuel fraction are enhancing subsequent SDA extraction in the 1:1 DiEGME/water blend, they either are not being extracted into the 1:3 DiEGME/water blend, or they are no longer effective to improve clean SDA extraction with the additional concentration of water being present.

<b>Table 10. Composition of Thin Apple Jelly -- Effect of DiEGME/Water Ratio</b>			
<b>Elemental Analysis</b>	<b>1:1 after extract with heavy fuel</b>	<b>1:1 after extract with heavy fuel and SDA</b>	<b>1:3 after extract with heavy fuel</b>
N, ppm(wt)	13	107	8
S, ppm(wt)	175	403	114

#### 10.1.5.4 Effect of Fuel Composition

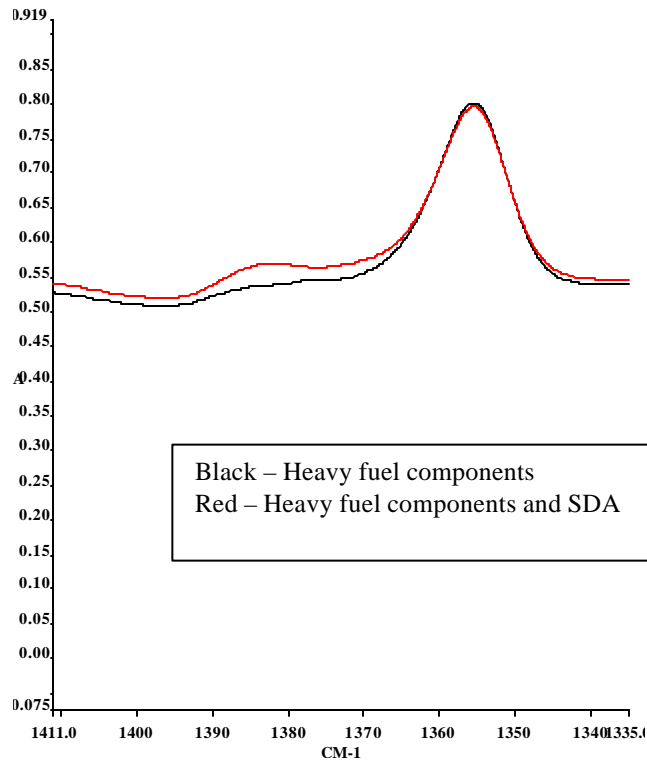
Table 11 compares results from the experiments using the 5% and 10% heaviest fuel fraction of JP-8, looking only at the effects on the 1:1 DiEGME/water blend. The 5% heaviest fraction was much higher in both nitrogen and sulfur compared to the 10% heaviest fraction. This is not surprising since it is well known that nitrogen and sulfur compounds in jet fuels are concentrated in the highest boiling fractions. (The nitrogen concentration in AP-003 and AP-004 is 27 ppm (wt) and 17 ppm (wt), respectively; sulfur is 219 ppm (wt) and 229 ppm (wt), respectively.) Further work would be needed to determine how fuel composition as a function of boiling range effects the solubility of SDA components in DiEGME/water blends. However, the fact that such enhancement occurs is proven.

In fact, the concept that SDA extraction into polar fuel component-enriched water/DiEGME bottoms is a major part of the mechanism of dark thin apple jelly can be supported by a very simple observation from the field. Dark, thin apple jelly is almost exclusively observed in installations that use JP-8, and very seldom seen in installations that use JP-5. JP-5 typically can have a higher final boiling point compared to JP-8, owing to the difference in flash point and boiling point specifications between the two fuels. However, JP-5 does not, as a rule, contain SDA. Assume for a moment that the dark-color, conductivity-enhancing, and nitrogen-containing species in dark, thin apple jelly (compared to pure water/DiEGME blends) are due only to, or primarily to, heavy, polar fuel compounds, and absolutely not to any SDA components. Then one might expect dark, thin apple jelly to be at least as common in JP-5 as in JP-8, if not more so. In fact, such is not the case, JP-8 facilities are almost always the only locations where dark

thin apple jelly is found. In JP-5 facilities, the materials observed in receipt and issue F/S sumps are reported to be nearly always only slightly yellow in color, although DiEGME enrichment of those water bottoms does occur. While this fact does not of itself prove that SDA is a major source of the dark color, conductivity-enhancing, and nitrogen-containing species in dark thin apple jelly, it certainly makes it much less likely that heavy, polar fuel components are the primary or exclusive source of such species.

<b>Table 11. Composition of Thin Apple Jelly -- Effect of Fuel Composition</b>				
<b>Elemental Analysis</b>	<b>5% heaviest fuel (AP-003)</b>	<b>50/50 after 5% fuel and SDA</b>	<b>10% heaviest fuel (AP-003 + AP-004)</b>	<b>50/50 after 10% fuel and SDA</b>
N, ppm(wt)	165	253	30	107
S, ppm(wt)	1464	220	394	403

Finally, Figure 55 shows the FT-IR in the characteristic  $1375\text{ cm}^{-1}$  region of the 1:1 (v/v) blend of DiEGME/water after extraction with the 5% heaviest fuel fraction and with both the 5% heaviest fuel fraction and subsequent extracted SDA components. The spectra demonstrate that the characteristic peak at  $1375\text{ cm}^{-1}$  is primarily due to the extracted SDA components, not heavy fuel components.



**Figure 55. Effect of Heavy Fuel Components and SDA on 1:1 DiEGME/Water**

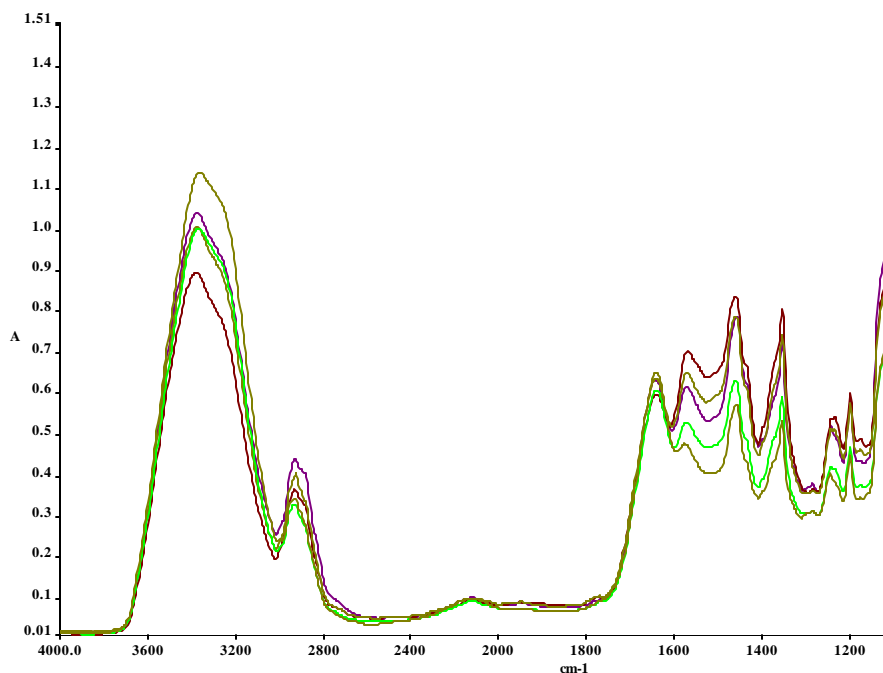


## 10.2 Thick Apple Jelly

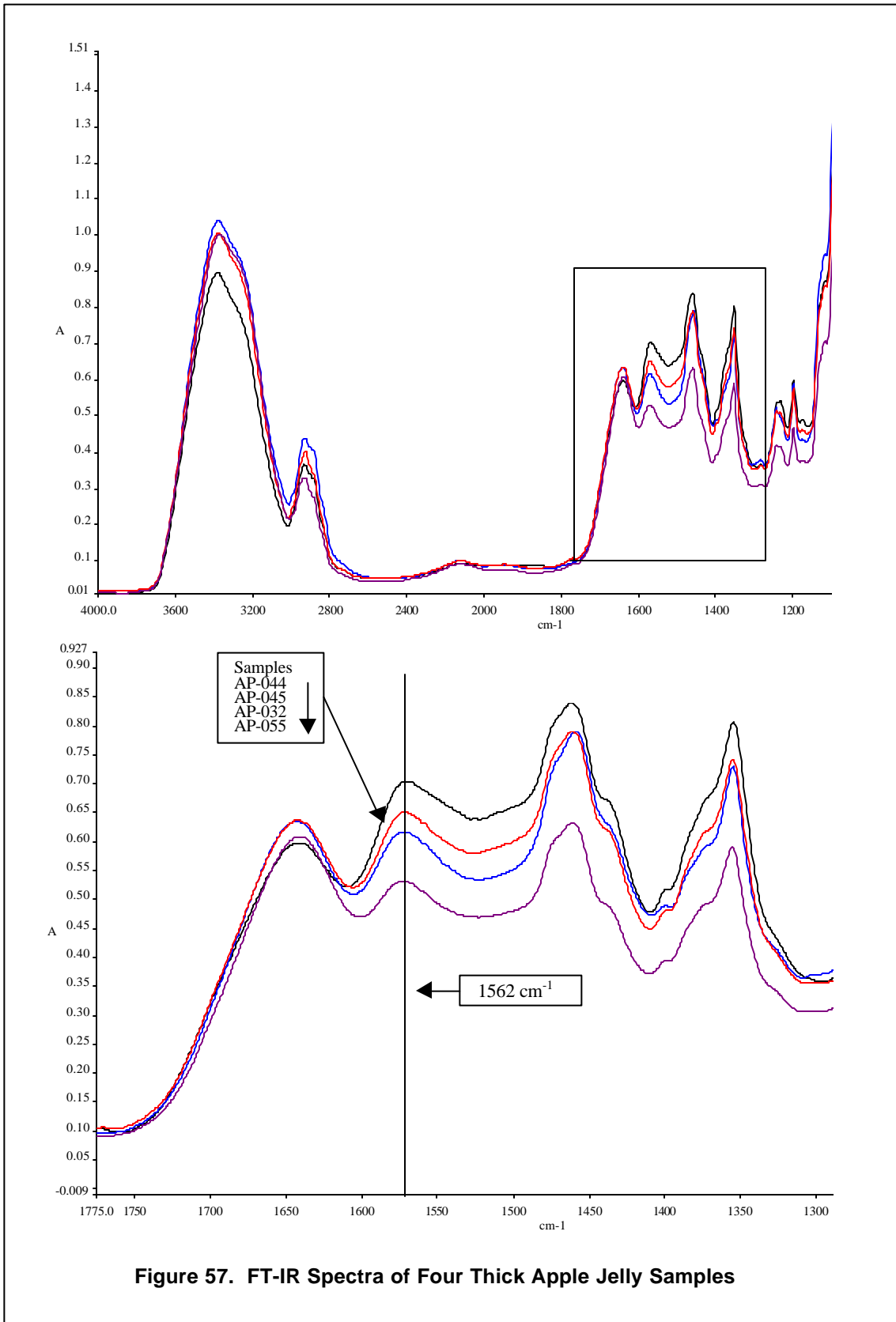
### 10.2.1 FT-IR Properties of Thick Apple Jelly

Other than sodium content, the most striking compositional difference between thick and thin apple jelly samples was found in the FT-IR spectra. All thick apple jelly samples have a prominent extra peak at about  $1572\text{ cm}^{-1}$ . This peak is absent in all thin apple jelly samples. Figure 56 shows the FT-IR spectra of five thick apple jelly samples. Figure 57 shows the FT-IR spectra of four labeled thick apple jelly samples with the region around  $1572\text{ cm}^{-1}$  magnified. The peak heights of these four apple jelly samples at  $1572\text{ cm}^{-1}$  are in the exact same ranking as their viscosities (see Table 12), with the least viscous sample having the lowest peak height.

Sample Number	Viscosity @ $25^{\circ}\text{C}$ , cP	Viscosity @ $60^{\circ}\text{C}$ , cP
AP-055	9,470	---
AP-032	>10,000	1,950
AP-045	>10,000	2,340
AP-044	>10,000	5,090

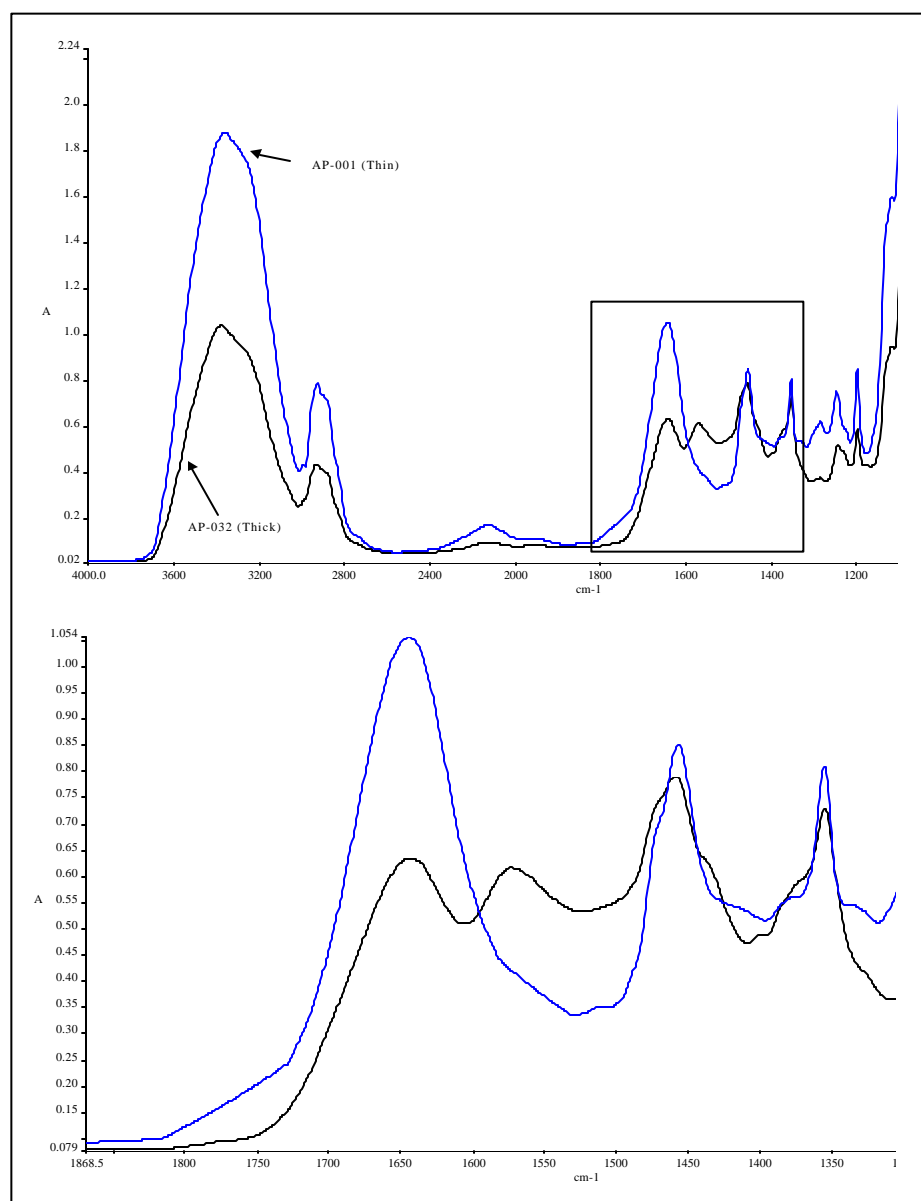


**Figure 56. FT-IR Spectra of Five Thick Apple Jelly Samples**

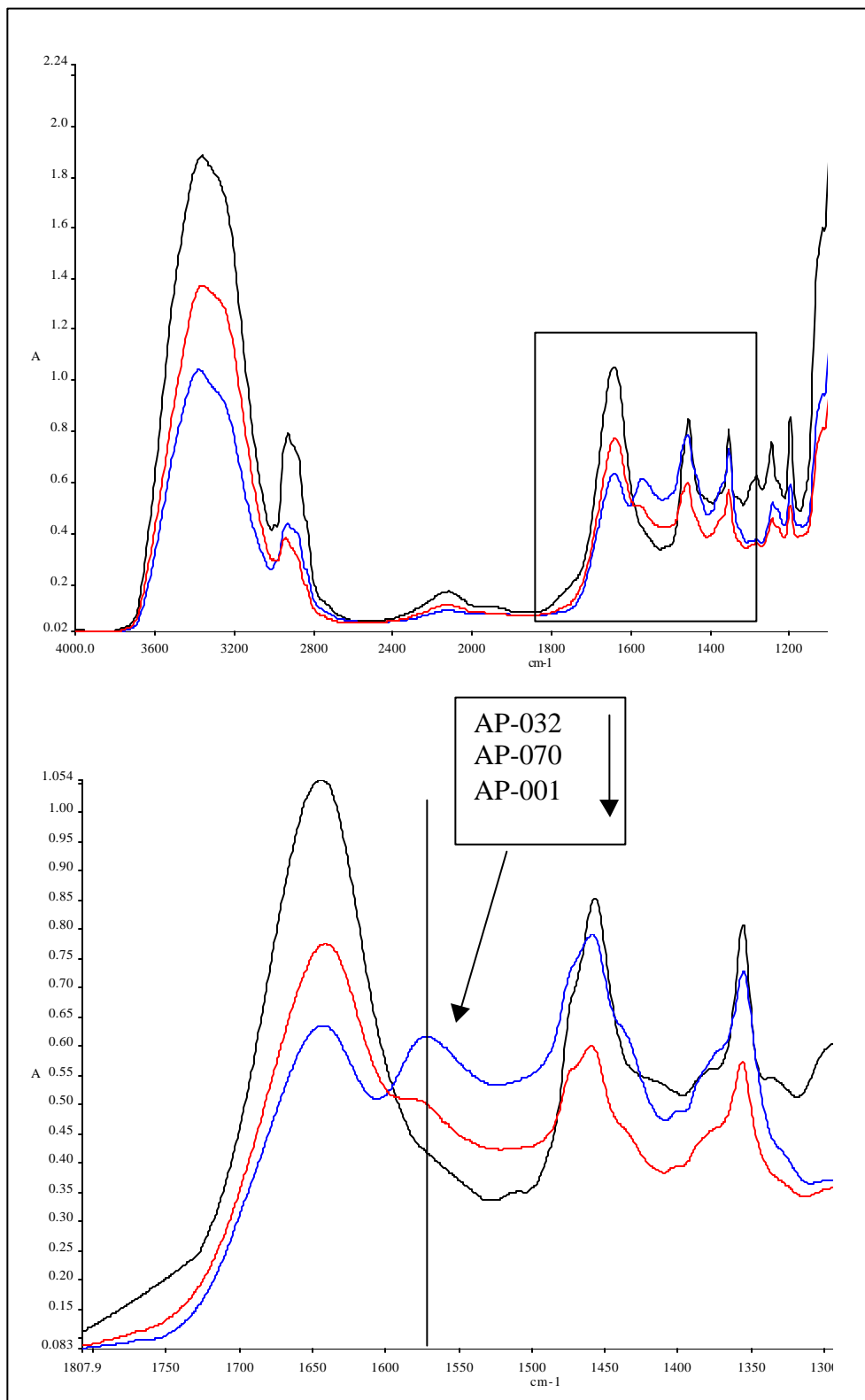


**Figure 57. FT-IR Spectra of Four Thick Apple Jelly Samples**

Figure 58 compares the FT-IR spectra of typical thin and thick apple jelly samples. The primary difference between these spectra is the peak at  $1572\text{ cm}^{-1}$  for the thick apple jelly sample. Figure 59 shows the FT-IR of three apple jelly samples, AP-001 (thin), AP-032 (thick), and AP-070 (moderately thick). The average Brookfield Viscosities of these samples are 4.6 cP, >10,000 cP, and 278 cP, respectively. As seen the peak heights at  $1572\text{ cm}^{-1}$  are ranked in the same order as the viscosities. In general, it was observed that the peak height at  $1572\text{ cm}^{-1}$  is roughly proportional to the average viscosity for the thick apple jelly samples we examined.



**Figure 58. FT-IR Spectra of Thin and Thick Apple Jelly Samples (with enlarged view)**



**Figure 59. FT-IR Spectra of Three Variably Viscous Apple Jelly Samples (with enlarged view)**

This information strongly suggests that the thickener is causing the FT-IR peak at  $1572\text{ cm}^{-1}$  in thick apple jelly samples. Also the size of this peak suggests that the concentration of the thickener must be at least about 1% of the total sample. The only other distinct, verified component in apple jelly that is within that concentration range is sodium. In solutions containing water, sodium must be present as sodium cations. Sodium cations are transparent to FT-IR, as are all mono-atomic cations. However, an anion must be associated with any sodium cations present in the apple jelly. It is well known that carboxylate anions adsorb in the infrared region around  $1570\text{ cm}^{-1}$ . Also, carboxylate anions would titrate as basic in the TBN procedure. Depending on the exact chemical structure of the carboxylate anion, it might contain little or no sulfur or nitrogen. All this information, taken together, suggests that the thickener could be a high molecular weight sodium carboxylate.

### ***10.2.2 Source of Sodium***

#### **10.2.2.1 Sodium from SDA**

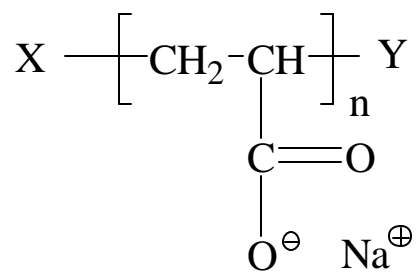
The only component of a fully additized JP-8 that contains sodium is SDA. The sodium content of neat SDA is about 33 ppm (wt). [27] It was determined in the DiEGME/SDA extraction experiment from section 10.1.3.1 that sodium from SDA does not accumulate in DiEGME as a result of successive extractions. That fact suggests that SDA is not the primary source of sodium in thick apple jelly. The small likelihood of SDA being the primary sodium source in thick apple jelly can be further demonstrated by a simple mass balance calculation that assumes the following:

- 100 grams of thick apple jelly (the maximum amount typically observed at each occurrence).
- 2% (wt) sodium in thick apple jelly.
- 33 ppm (wt) sodium in neat SDA.
- 1 ppm (wt) SDA in the JP-8.
- Assume all SDA sodium extracts into the apple jelly phase.

The last point above seems unlikely based on previous extraction data, but is assumed as a conservative estimate. Using these assumptions, one can calculate that it would require about 20 million gallons of JP-8 to provide sufficient SDA-sodium for 100 grams of thick apple jelly. Based on field operating data obtained from the site visits, this would require an R11 refueler to be used constantly with 1.6 refills per day for one year. This confirms that SDA is not the primary source of sodium in thick apple jelly, in agreement with the implications from the DiEGME/SDA extraction experiment discussed above. Clearly, the massive amount of sodium present in thick apple jelly must come from another source that has a much higher concentration of sodium.

#### 10.2.2.2 Sodium from Water-adsorbing Filter Media

Based on earlier reports and the information from the site visits, the one common factor in all occurrences of thick apple jelly was water-adsorbing filters. The thick apple jelly was always found just downstream of these filters. Every thick apple jelly sample, observed and collected during this study, occurred at a location where water-adsorbing filters were being used. The filter element currently in use by the Air Force is Facet Model GNG633SB. It is our understanding that other models and suppliers are approved but not currently in the Air Force inventory. The construction and materials used vary by manufacturer but the water-adsorbing material is usually a polyacrylate polymer, similar to the one shown in Figure 60. Some manufacturers use sodium polyacrylate, some use potassium polyacrylate. It is our understanding that the element currently in inventory use sodium polyacrylate. The degree and type of cross-linking in the polymer also varies with the manufacturer. We were unable to obtain specific compositional information from either of the two major water-adsorbing filter makers.



**Figure 60. Sodium Polyacrylate**

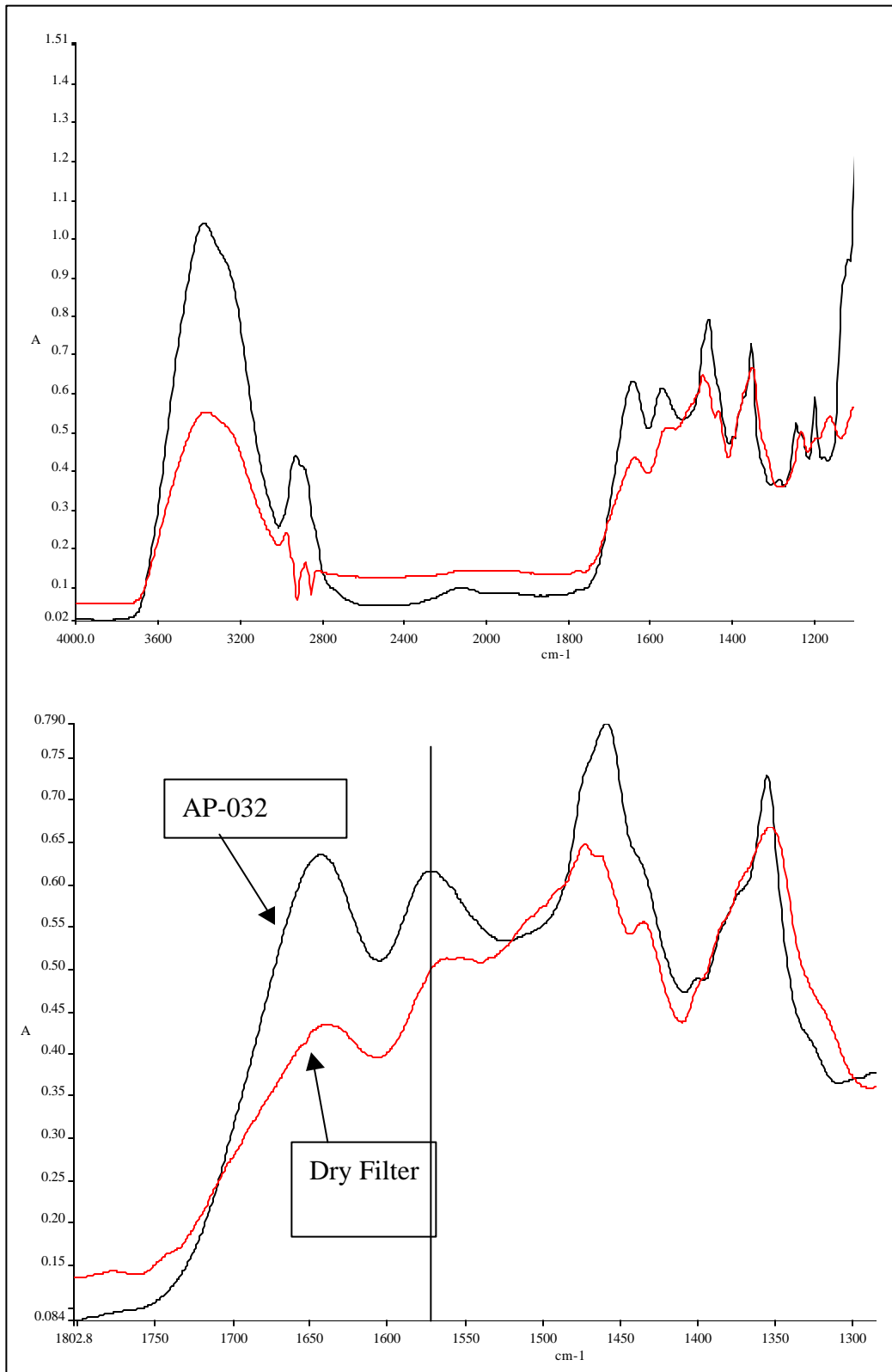
A new Facet GNG633SB filter was obtained and cut open. Each layer of the filter was separated and analyzed for sodium (acid digestion followed by ICP analysis). The results are given in Table 13 with the layers numbered from outermost to innermost. The white, somewhat fluffy layer, #2, has significant sodium, with less sodium in the innermost reticulated paper layer, #6. We were told, by the manufacturer, that layer #2 is the layer impregnated with the water-adsorbing polymeric material.

Filter Layer No.	1	2	3	4	5	6
Total Sodium, %(wt)	0.007	1.32	0.056	0.067	0.056	0.31

Sodium polyacrylate meets all the previously deduced properties of the thick apple jelly thickener. It contains about 24% (wt) sodium, contains no nitrogen, contains no sulfur, has a high molecular weight that should produce a residue on the TGA test, and many carboxylate groups that would test basic on the TBN test and should adsorb around  $1570\text{ cm}^{-1}$  in the infrared region. It should also be noted that based on the sodium content of the layer 2 material and the sodium content of pure sodium polyacrylate, the layer 2 material should contain about 5% sodium polyacrylate.

### ***10.2.3 FT-IR Properties of Water-adsorbing Filter Media***

The FT-IR spectrum of dry and wetted GNG633SB layer #2 material (hereafter referred to as water-adsorbing, filter media) was determined and compared to the FT-IR spectrum of thick apple jelly. Figure 61 shows the FT-IR of thick apple jelly sample AP-032 and the dry water-adsorbing filter media. The latter was obtained by placing the filter material directly on the ATR cell of the FT-IR. Although this is a less than optimum method of obtaining infrared spectra, and despite the low loading of sodium polyacrylate polymer on the media, the FT-IR of the dry filter media clearly shows a peak near  $1572\text{ cm}^{-1}$ . However, this peak is somewhat down field of  $1572\text{ cm}^{-1}$ . When the filter media was wetted with de-ionized water and placed on the ATR cell, the resulting FT-IR spectra provided better agreement, as shown in Figure 55. As seen, the wet filter media peak is now present as a distinct shoulder due to the greatly increased H-O-H bending mode adsorption at  $1650\text{ cm}^{-1}$ . Even so, the carboxylate peak has shifted to almost exactly  $1572\text{ cm}^{-1}$ .



**Figure 61. FT-IR Spectra of Thick Apple Jelly and Dry Layer #2**



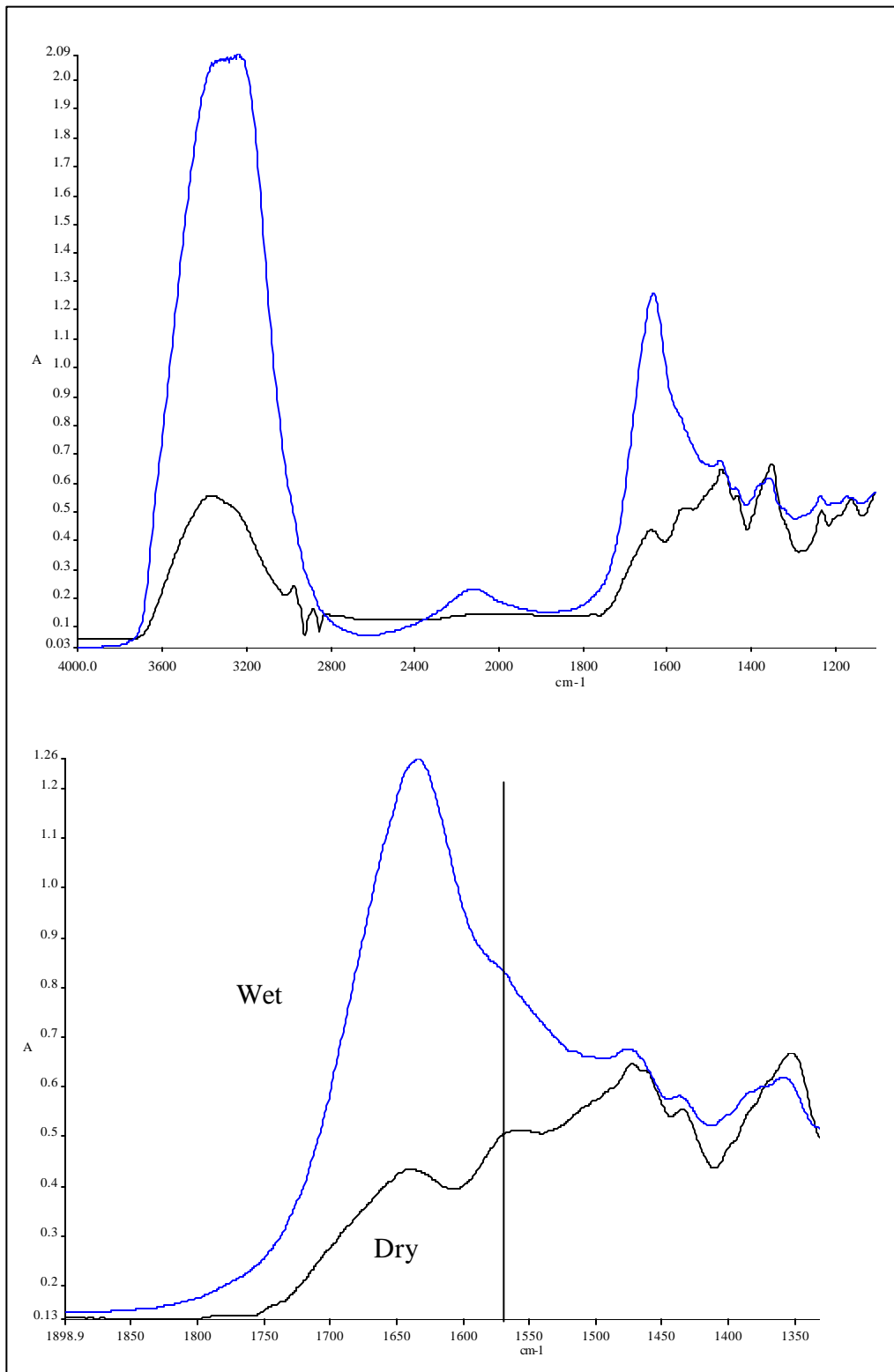


Figure 62. FT-IR Spectra of Wet and Dry Layer #2

As a final experiment a 90/10 (v/v) blend of DiEGME/water was mixed with small pieces of water-adsorbing, filter material. The pieces eventually swelled and the solution became somewhat thickened. The spent pieces of filter media were carefully removed, fresh pieces were added to the solution, and the mixing was repeated. Finally, a thickened, clear solution was obtained and labeled as JAW1-56-1. Although the viscosity was significantly less than that of a thick apple jelly, it was distinctly higher than the original DiEGME/water blend. Also, it should be noted that although nearly 800 ml of DiEGME/water blend was used, only about 50 ml of somewhat thickened solution resulted. The vast majority of the original blend was adsorbed into the pieces of filter media.

When the FT-IR of solution JAW1-56-1 was obtained, no peak at  $1572\text{ cm}^{-1}$  could be detected. However, considering the low sodium polyacrylate loading of the paper and the only mild thickening that was possible, the concentration of polymer in the blend was probably too low to detect by FT-IR. An ICP analysis of JAW1-56-1 detected only 900 ppm (wt) sodium, indicating about 3700 ppm (wt) of actual sodium polyacrylate. To indirectly determine the presence of carboxylate, a sample of JAW1-56-1 was acidified by addition of a few drops of concentrated HCl. The resulting neutral carboxylic acid groups should have extinction coefficients greater than that of the corresponding carboxylate anions. The FT-IR spectra of both the original and the acidified JAW1-56-1 are given in Figure 63. The distinct appearance of a small peak at around  $1730\text{ cm}^{-1}$  is clear evidence of a neutral carboxylic acid group. This provides indirect evidence that JAW1-56-1 does indeed contain carboxylate groups.

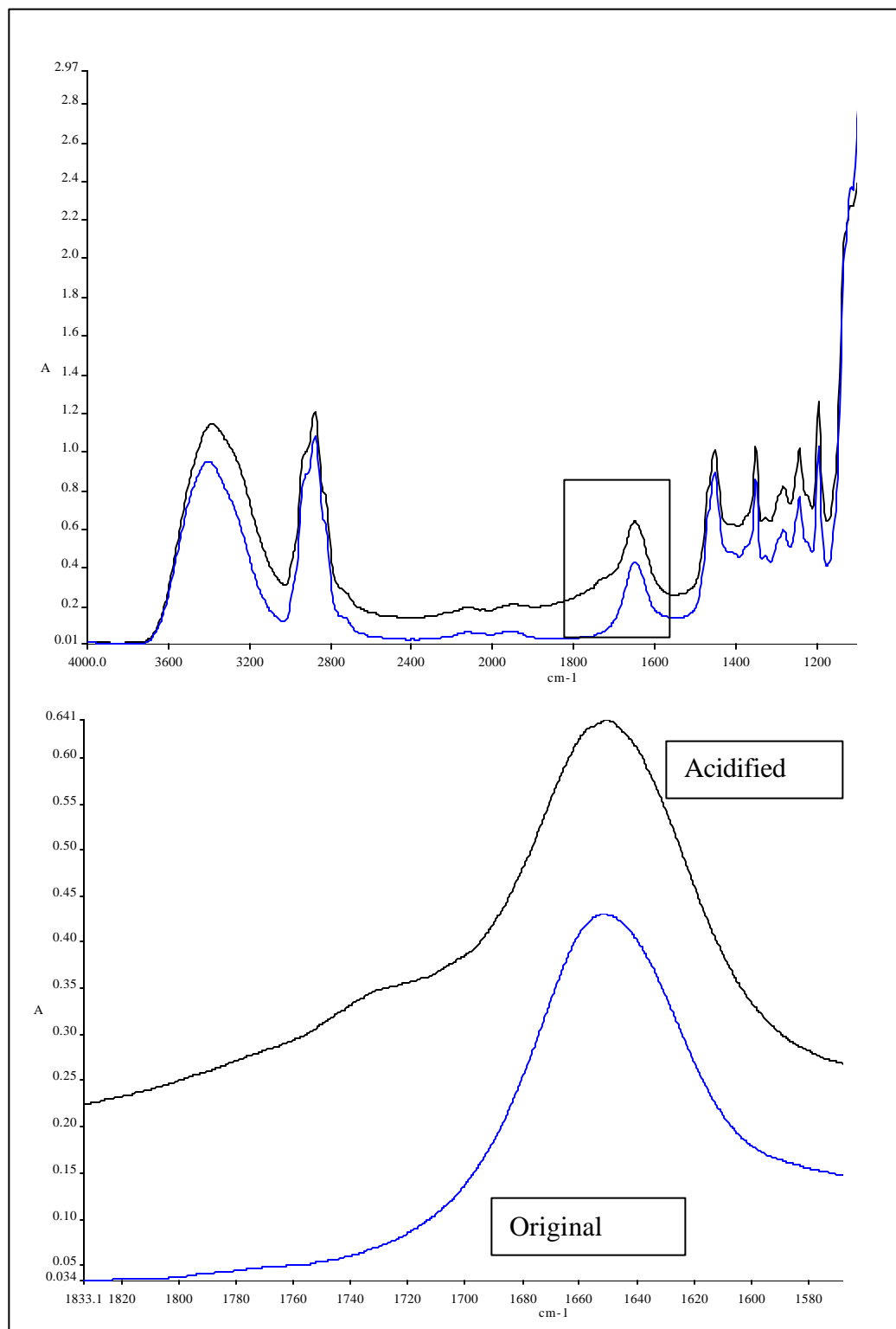


Figure 63. FT-IR Spectra of Original and Acidified Synthetic Apple Jelly JAW1-56-1

### 10.2.4 FT-IR Properties of Sodium Polyacrylate

A sample of generic sodium polyacrylate was obtained from a chemical supply house. Although this material is almost certainly not the exact same material used in current water-adsorbing filter media, it should be reasonably close. FT-IR spectra of this material are given in Figures 64 and 65. The characteristic carboxylate peak is prominently visible at around  $1570\text{ cm}^{-1}$ .

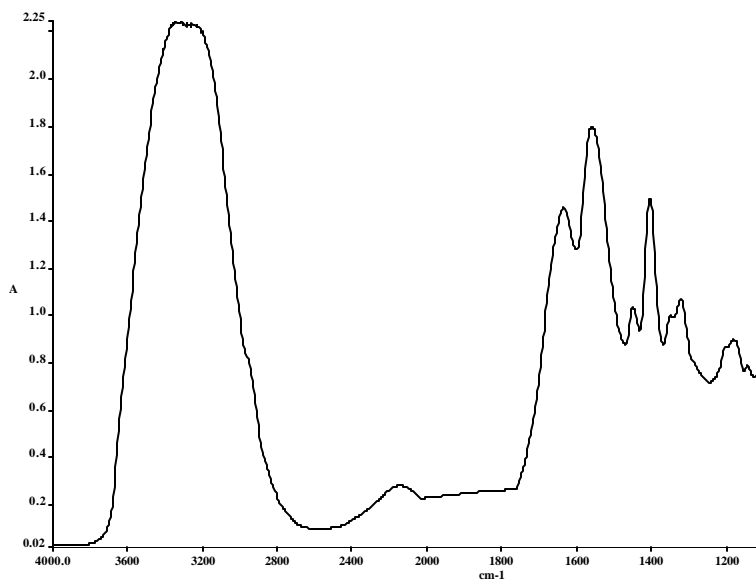


Figure 64. FT-IR Spectrum of Sodium Polyacrylate

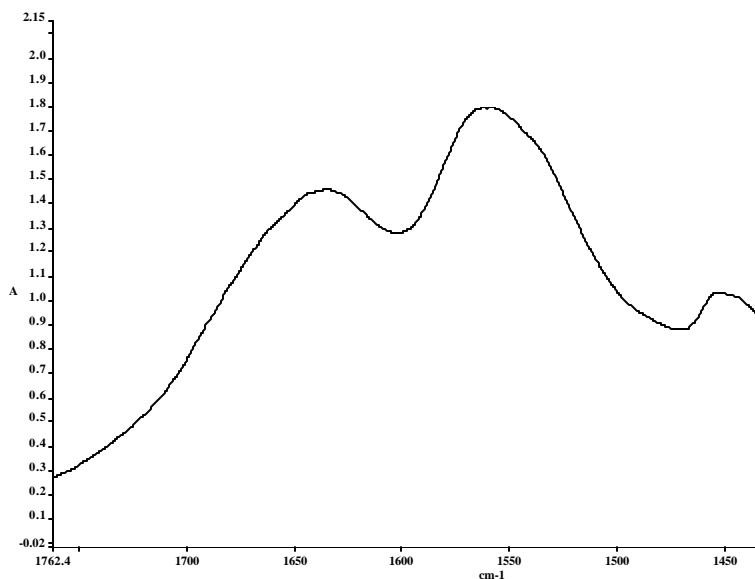


Figure 65. FT-IR Spectrum of Sodium Polyacrylate ( $1450\text{ to }1760\text{ cm}^{-1}$ )

### ***10.2.5 Effect of SDA on Thick Apple Jelly***

The thickened sample JAW1-56-1 from the previous section was clear in color. Sodium polyacrylate is a white powder. Yet, all the highly thickened apple jelly samples (viscosity at 25°C greater than 1,000 cP) we received were dark in color, had very high conductivity, and high nitrogen content. From our synthesis work with thin apple jelly, we knew that SDA components are extracted into the DiEGME/water matrix, and provide a major source of dark color, conductivity, and nitrogen. Ion chromatography data discussed earlier in this report indicate that SDA components are also present in thick apple jelly. Based on this information, we decided to investigate the interaction of SDA with our synthetic thick apple jelly.

A few milliliters of SDA were added to a glass vial of JAW1-56-1. The vial contents were carefully stirred, then the vial was inverted repeatedly to allow the SDA to flow through the entire body of the thickened blend of DiEGME and water. Almost immediately, the thickened blend of DiEGME and water became dark in color and began to contract. As the blend contracted, a clear fluid was squeezed out and to the top of the tightening gel. This clear fluid was poured off as it formed. After 15 minutes, the original blend of DiEGME and water had contracted to less than one-third of its original volume, and had become extremely gelatinous and sticky. The color was identical to the thickest of the apple jelly samples. This experiment was repeated several times with identical results.

The sample of JAW1-56-1 before and after treatment with the SDA was analyzed. Results are given in Table 14. The % DiEGME by RI was increased from 87.1% to 491%. The result of JAW1-56-1 before SDA is very close to the 90% value that is expected. The value after SDA treatment is consistent with the behavior already observed when SDA components are present. However, the magnitude of the % DiEGME level indicates concentrating of SDA components within the contracted gel structure. The dark color is also an indication that SDA components are concentrating within the JAW1-56-1 sample as it contracts. TGA residue of JAW1-56-1 before SDA treatment is consistent with the very low level of thickener that is present. The TGA residue after SDA treatment is consistent with the concentration of SDA components within gel. (Compare with data from the final extraction sample in Section 10.1.3.1.) Nitrogen and sulfur levels were not

run on the sample before SDA treatment since this sample (water, DiEGME, and material from the water adsorbing filter) will contain neither of those elements. Nitrogen and sulfur are greatly increased in the sample after SDA treatment. The sodium level increase from 600 ppm (wt) to 900 ppm (wt) is due to the contraction of the gel. The added sodium from SDA would be negligible, given the quantity that was added. The TAN and TBN of the sample after SDA treatment is much greater than the sample before treatment, as expected. As already observed in Section 10.1.3.1, the contribution of SDA to TAN is greater than its contribution to TBN. If the amount of sodium polyacrylate had been at levels sufficient to contribute 2% sodium instead of only 900 ppm (wt) sodium, the TBN would have been much greater. The 3.3% water of the gel after SDA treatment may be an indication that water was being preferentially squeezed out of the gel during contraction.

<b>Test</b>	<b>Before SDA</b>	<b>After SDA</b>
% DiEGME (R.I.)	87.1	491
TGA residue, % (wt)	0.6213	31.74
N, ppm(wt)	Not run	6719
S, ppm(wt)	Not run	11,399
Sodium, ppm(wt)	600	900
TAN, mg KOH/g	2.06	37.4
TBN, mg KOH/g	2.47	9.14
KF Water, %	11.8	3.3

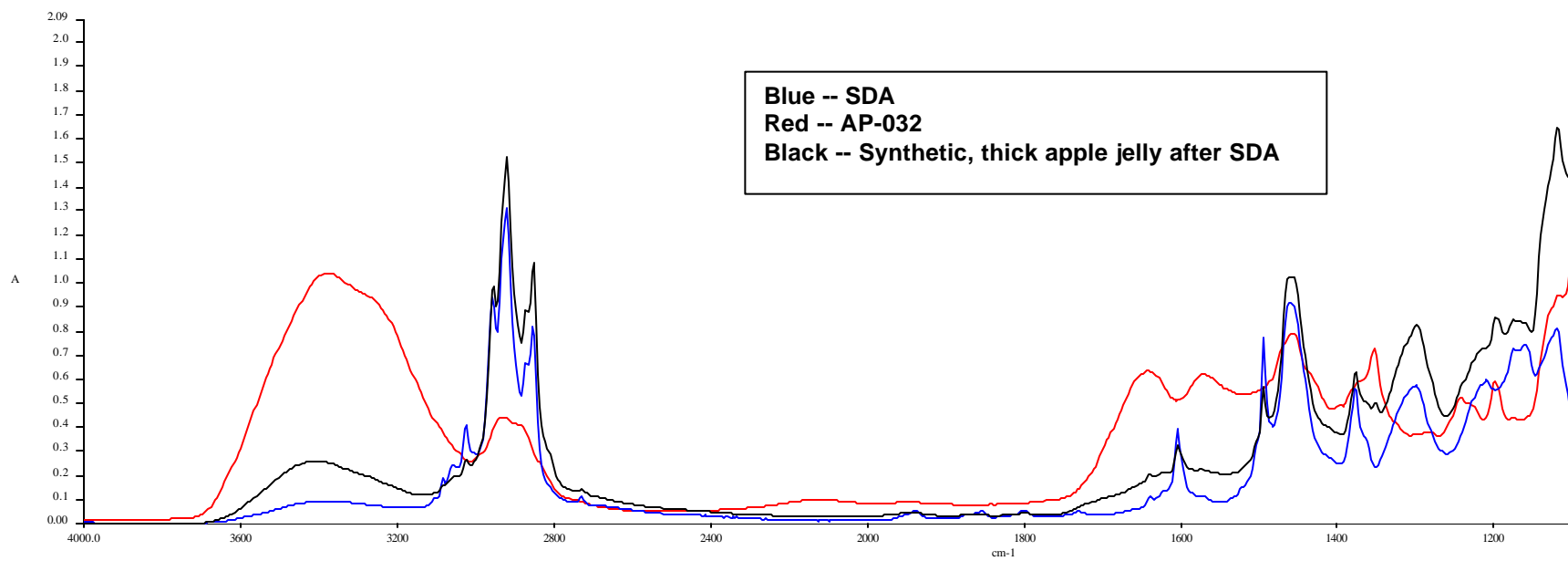
The much greater level of sulfur than nitrogen in the final gelled sample is likely the result of the absence of any JP-8 base fuel. As was observed earlier, when SDA was extracted into DiEGME, the amount of sulfur extracted was exaggerated compared to the amount of nitrogen. Since no JP-8 base fuel was present in the above experiment, all SDA sulfur-containing compounds were forced to reside within the gel structure instead of partitioning between the gel and a JP-8 phase. Hence, it is reasonable to assume that the sulfur level in the SDA treated JAW1-56-1 is higher than would be expected in a system where JP-8 was present.

In fact, enough is known about the composition of SDA to propose what is likely occurring with respect to the gel contraction and the fate of sulfur compounds. SDA sulfur is largely if not entirely due to the presence of dinonylnaphthylene sulfonic acid. Sulfonic acids are more than 100 times more acidic than carboxylic acids. As SDA is added to the

thickened DiEGME/water blend, the sulfonic acid will protonate available carboxylate groups from the sodium polyacrylate to form neutralized carboxylic acid groups and sodium dinonylnaphthylene sulfonate. Sodium dinonylnaphthylene sulfonate is not soluble in water but is quite soluble in petroleum based hydrocarbons including jet fuels, diesel fuels, and lube oils. If JP-8 were present during the partitioning of SDA into a sodium polyacrylate-thickened DiEGME/water blend (as would be the case in a water-adsorbing fuel filter system), the sodium dinonylnaphthylene sulfonate would preferentially go into the fuel phase. The protonated carboxylate groups would then be available to react with basic components known to exist in SDA. This would enhance extraction of these components from the SDA in the JP-8 into the forming, thickened DiEGME/water blend. The reaction of the polyacrylate groups with the sulfonic acid groups and the subsequent reaction with other basic groups from SDA are likely reasons for the gel contraction. As the polarity and counter ion structure of the polyacrylate anions are modified, the rheological effects from the thickener are likely to change. Although, this chemical model is reasonable and consistent with all the data and additive compositional information available, further experimental work would be required to establish it with complete certainty.

The FT-IR of the JAW1-56-1 sample after treatment with SDA (synthetic apple jelly) is shown and compared to that of a thick apple jelly in Figures 66, 67 and 68. A very slight indication of the characteristic peak at 1572 cm<sup>-1</sup> can be seen in the synthetic apple jelly. Unfortunately, the low level of sodium polyacrylate in the sample was not sufficient to produce a strong peak. Also, numerous other peaks were present in the synthetic apple jelly sample. These peaks are to be expected since there was no JP-8 to allow preferentially fuel-soluble components and reaction products to leave.

Also, the previous experiment did not allow heavy fuel components to concentrate in the DiEGME/water phase. This process has been previously shown to be critical to the formation of thin apple jelly, and there is nothing to prevent its occurrence in thick apple jelly. Such fuel components can be expected to enhance the solubility of SDA components in the DiEGME/water matrix of thick apple jelly. Nonetheless, it is interesting to note that an apparent strong interaction between the sodium polyacrylate and SDA components is a major driving force towards concentrating SDA components in thick apple jelly.



**Figure 66. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly**



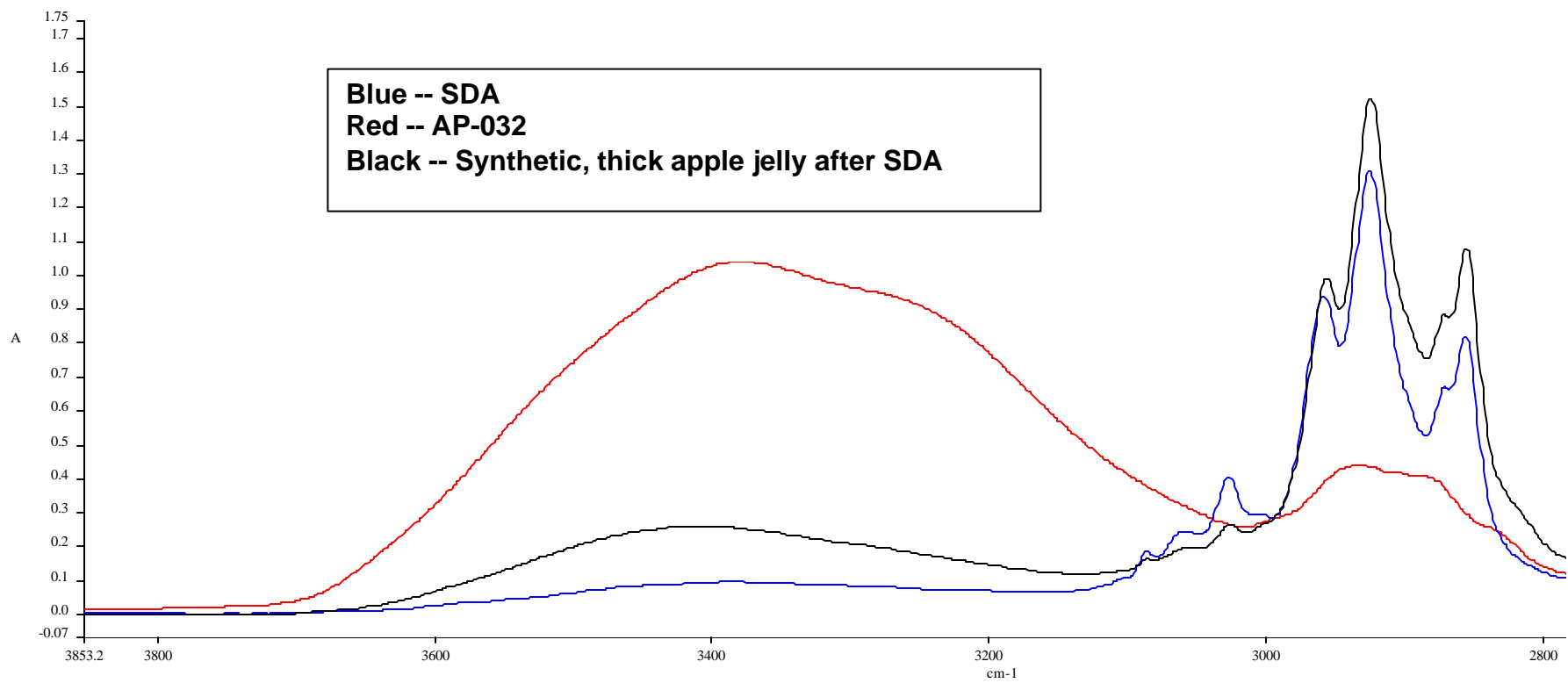
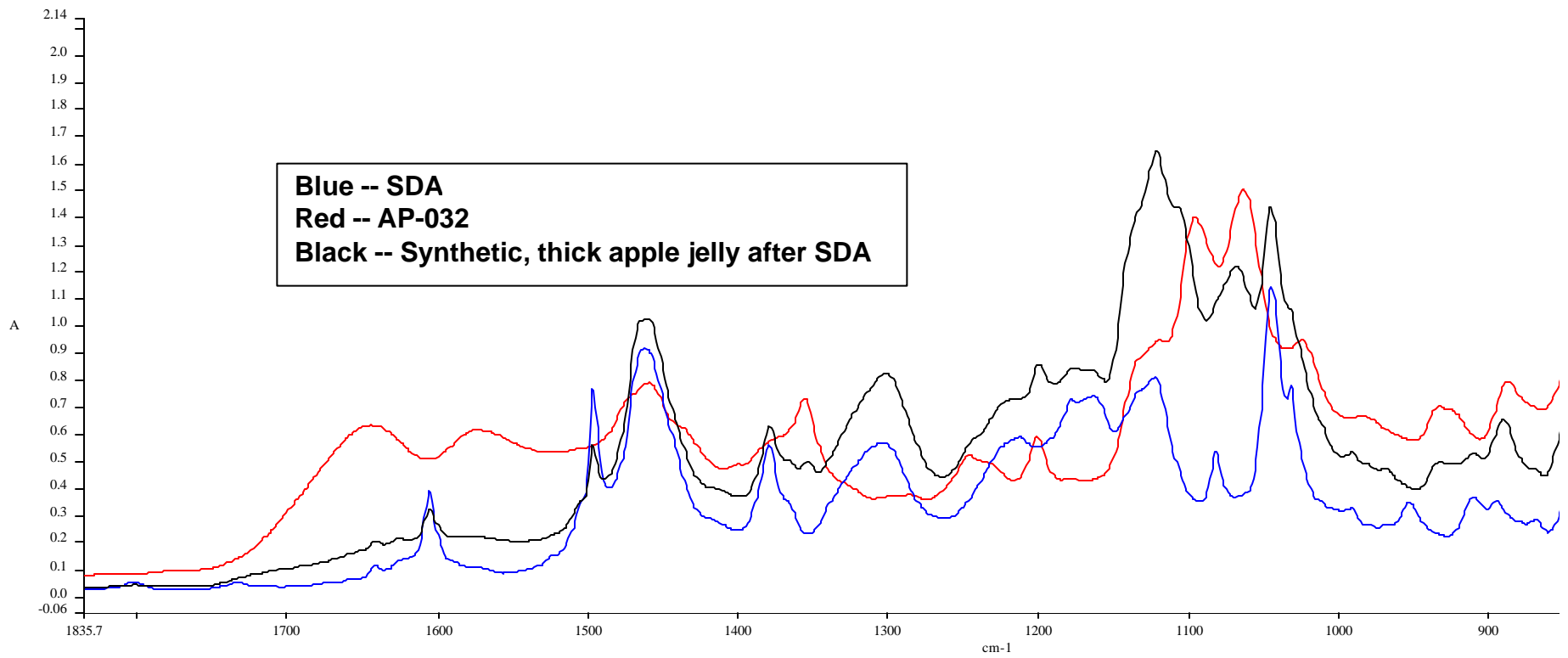


Figure 67. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly (2800 to 3800 cm<sup>-1</sup>)



**Figure 68. FT-IR Spectra of SDA, Thick Apple Jelly, and Synthetic Thick Apple Jelly (900 to 1800 cm<sup>-1</sup>)**

### 10.3 Effect of Water-adsorbing Filter Media on Thin Apple Jelly

Based on all the information discussed thus far, it appears that the same chemistry and dynamics that apply to thin apple jelly formation also will apply to thick apple jelly formation except for the effect caused by the sodium polyacrylate from the water-adsorbing filter media. As a final experiment, 100 ml of thin apple jelly AP-001 were stirred with pieces of the water-adsorbing filter media using a procedure similar to that described in the previous section regarding JAW1-56-1. The resulting blend was somewhat thickened, although not nearly to the level of a true thick apple jelly. Test results comparing AP-001 and the filter media-treated AP-001 are given in Table 15.

<b>Sample</b>	<b>AP-001</b>	<b>AP-001 + media</b>
Conductivity, pS/cm	1.06 x 10 <sup>11</sup>	1.31 x 10 <sup>11</sup>
Total N, ppm(wt)	265	249
Total S, ppm(wt)	338	180
TAN, mg KOH/g	0.56	1.14
TBN, mg KOH/g	0.96	4.70
Sodium, ppm(wt)	1575	1975

Conductivity increased significantly indicating that in the presence of a thin apple jelly composition, sodium polyacrylate will increase conductivity. This is interesting since the mobility of the polymeric polyacrylate anion should be low. The very high electrical charge of such anions may provide a compensating effect. Both TAN and TBN increased, but TBN increased the most, as expected. The very modest increase in sodium is an indication that not very much sodium polyacrylate was obtained from the media. This is as expected, given the inefficiencies that have been shown to exist in using this method to thicken DiEGME/water systems. Both sulfur and nitrogen levels dropped (not significantly so for nitrogen). The most likely explanation for this is that some of the nitrogen and sulfur-containing materials adsorbed onto the media filter pieces during the stirring. In actual field use, such adsorption would be negligible, since the adsorption capacity of the media would be quickly reached.

## 11 SUMMARY AND RECOMMENDATIONS

### 11.1 Summary

It is important to reiterate that we started this work with no constraints regarding what is and what is not apple jelly. We simply asked users in the field to submit samples of apple jelly whenever it was discovered. The samples we received varied in color, viscosity, and numerous other properties. We were told by some people that only thick apple jelly is really apple jelly, the rest is simply contaminated water bottoms. [We contend that all apple jelly is a form of contaminated water bottoms.] However, DESC instructed us to not limit our study because all the samples represented a potential problem for the user.

This work has demonstrated that apple jelly is a complex mixture. It begins with water and DiEGME. This mixture interacts with its environment, extracting and dissolving compounds from the materials with which it comes in contact.

Because apple jelly is such a complex and varied mixture, no one laboratory-synthesized apple jelly can represent all the possible compositions. Even one synthetic thin-apple jelly and one synthetic thick-apple jelly are not sufficient to represent all possible compositions.

In this work we started with apple jelly samples collected from throughout the DoD/Air Force fuel-distribution system. The majority of our samples came from fuel systems delivering JP-8 to aircraft. There were differences in the fuels, although all the fuels were kerosene jet fuels. All the fuels contained corrosion inhibitor, FSII, and SDA; although in varying amounts. The amount of water present in the various fuel systems differed greatly. All the fuel passed through filters, including particulate filters, filter/separators, and water-adsorbing filters. Other possible contaminants in the fuel systems included dirt, rust, paint, other fuels, elastomers, as well as myriad unknown contaminants. All these factors combined with water and DiEGME mixtures to form the several apple jelly samples we received from the field.

Other than FSII, this work focused on only one JP-8 additive, SDA. We did so because there was evidence that SDA (or its components) accounts for some of the properties of the apple jelly samples we analyzed. That is not an indictment of SDA as a cause of apple jelly. The SDA in the fuel is incorporated into the water/DiEGME just as other compounds in the fuel do. In a system without SDA, some form of apple jelly could still form given the presence of water and DiEGME. This other apple jelly would have some properties similar to our samples but would also have some properties different from our samples. The other additives in the fuel may also make some contribution to the various properties of apple jelly. The extent of those contributions was not thoroughly investigated, and would depend upon the other factors in the system. The same would be true if the water/DiEGME mixture formed in a diesel fuel system.

The work presented in this report explains the majority of the properties of the various apple jelly samples we received. We were able to demonstrate how thin and thick apple jelly, of the types we analyzed, could form. As mentioned above, all our samples came from somewhat similar fuel systems. Yet, there are differences and it is beyond the scope of this work to synthesize all possible combinations. As an example, our analyses showed sodium present in all the apple jelly samples. Possible sources include SDA, water-adsorbing filters, and also salt from the environment or even salt towers for drying fuel. We know that the majority of the sodium comes from the water-adsorbing filters because they are the most concentrated source. The relative amounts contributed by any other source are impossible to ascertain. Still, some conclusions are possible. As an example, thin apple jelly tends to be lower in sodium. This probably means it has had less exposure to water-adsorbing filters. Hence, it also has little or no thickener so its viscosity is low.

During the course of this project, two questions arose that we believe should be addressed at this point:

1. Why is apple jelly a problem in JP-8 but apparently not a problem in JP-5 or Jet A?

- The large majority of JP-5 and Jet A is stored in fixed-roof tanks, which reduces the influx of water.
- The Navy uses three stage filters on trucks
- JP-5 doesn't contain SDA so water/DiEGME bottoms in JP-5 systems will usually not appear dark, or as dark in color.
- Jet A does not routinely contain FSII. When Jet A is used, the additives (if used) are injected into Jet A at the skin of the aircraft.

2. Why has apple jelly been a problem in the US and the Pacific but less so Europe?

- In Europe, JP-8 is additized immediately prior to shipment to the bases so there is less chance of water/DiEGME mixtures forming.
- In Europe, JP-8 is stored in fixed-roof, cut-and-cover tanks, which reduces the amount of water in the fuel system.
- NATO tank design provides more effective removal of water/FSII from tank sumps.

The various properties of the apple jelly samples we analyzed are a result and a reflection of the various systems in which they were formed. It is a mistake to assume that there is only one type of apple jelly and that it is formed by only one means.

The only effective way to eliminate this problem contaminant (whether you call it apple jelly or contaminated water bottoms) is to eliminate either the DiEGME, or the water, or both. In fact, simply eliminating the water may not solve all problems since the DiEGME remains a potentially damaging compound if it is not properly mixed into the fuel.

## 11.2 Findings From The Field Investigations

An enormous amount of information was collected during the site visits and assembled into the report from C4e. [28] Selected information from each of the sites is included in this report. The information included herein is directly related to the issues fuel receipt, handling, storage, and delivery. Other information may have been obtained at some sites and is included in the full report from C4e. The conclusions and recommendations listed below are those derived only from the information included in this report.

- Floating roof tanks and ineffective geodesic domes allow water into the fuel system.
- The self-cleaning tank design found at some sites eases tank-cleaning at the expense of fuel quality. In this type of system, the contaminants in the fuel are “pushed downstream” where they can potentially cause other problems.
- Many of the product recovery sump-draining systems currently in use are ineffective.
- Improved sump and low point draining practices are needed at numerous bases.
- The sump draining practices at terminals storing additized JP-8 need improvement to reduce the amount of water in contact with FSII-treated fuel.
- Many filtration systems currently in use are not designed for additized turbine fuels.
- Old DoD standard filter separator vessels and NSN4330-00-983-0998 coalescer elements are ineffective in removing water from JP-8. Old DoD vessels are still used in Oshkosh R-11s, MH-2C hosecarts, and in some fill stands, hydrant, and receipt filter separators.
- Some additives impact the ability of filtration systems to remove water and thus aid in the formation of apple jelly.
- On the occasions when tests were conducted in the field, apple jelly samples exhibited extremely high conductivity.
- There have been very few apple jelly reports where JP-8+100 is in use.
- The use limits for FSII range from 0.07% to 0.20% by volume.
- Bases with water problems often request high final FSII dosage.

- Some bases reported that reducing amounts from 0.18% range to 0.10% to 0.12% range seemed to reduce apple jelly frequency.
- The Air Force-Authorized single compartment blending procedure for FSII contributes to problems by allowing poorly mixed additives (slugs), non-additized fuel, and over-additized fuel to enter the system.
- There is obsolete additive-injection equipment at some locations.
- Work must be done to optimize injection point of additives (e.g., upstream from pump and in turbulent flow conditions). This will improve the mixing of additives into the fuel.
- Additive dilution procedures are inadequate for some current additive injection equipment.
- Use of an injection quill to the center of pipeline flow is preferable to injection at interior pipe surface.
- Additive injection can complicate downstream water removal by reducing the interfacial surface tension of the water.
- Additives are injected prior to coalescence/water removal at some terminals, thereby increasing the possibility of water/FSII interaction early in the delivery system. This also increases the chance of apple jelly formation earlier in the fuel delivery system.
- Many terminals use vacuum truck removal of water bottoms only once every 3-6 months (some don't remove water bottoms at all).
- Apple jelly seems to appear most often at times of temperature change; either warm to cold or cold to warm. Sharp temperature drops are often accompanied by storms, allowing rainwater entry into floating roof tanks and ineffective geodesic dome tanks. It appears in some cases that warming temperatures reduce the viscosity of thick apple jelly in R-11 water absorption cartridges allowing it to drain into the filter sump.
- Almost all high-viscosity apple jelly samples were found in sumps of R-11 F/S equipped with water absorption media cartridges; all thick apple jelly samples were found downstream of a water-adsorbing filter.
- The rapid increase in apple jelly reports corresponds to expanded use of water absorption filters in all AF R-11's during 1999-2000.



### 11.3 Findings From the Laboratory Studies

The following specific conclusions may be drawn from the laboratory work reported herein, based on the analysis of the samples we received.

- Apple jelly appears in several types; including thin and thick; and, light and dark.
- Thin apple jelly has a viscosity within the range of DiEGME and water blends, i.e., less than 7.5 to 10 cP at 25°C. Thick apple jelly has a viscosity greater than the range of DiEGME and water blends.
- Regarding dark, thin apple jelly:
  - The color ranges from dark amber to dark brown or black
  - It is over 95% (vol) DiEGME and water in various proportions.
  - The DiEGME to water ratio is usually greater than or equal to 1.
  - It contains higher molecular weight (heavy ends) components extracted from the fuel.
  - It contains components from SDA.
  - Components extracted from SDA are a primary cause of the dark color.
- Regarding light, thin apple jelly:
  - The color ranges from clear to light amber.
  - It is over 95% (vol) a mixture of DiEGME and water.
  - The DiEGME to water ratio is usually less than 1.
  - It contains higher molecular weight (heavy ends) components extracted from the fuel.
  - It contains fewer components from SDA than dark, thin apple jelly.
  - It is probably often not regarded as apple jelly when observed in the field.
- Regarding dark, thick apple jelly:
  - The color ranges from light brown to dark brown or black.
  - The DiEGME to water ratio is usually greater than 1.
  - It contains higher molecular weight (heavy ends) components extracted from the fuel.
  - It contains significant levels of sodium polyacrylate thickener, from water-adsorbing filters.
  - It contains significant levels of components from SDA.

- Regarding light, thick apple jelly:
  - The color ranges from light amber to dark amber.
  - The DiEGME to water ratio is usually greater than or equal to 1.
  - It contains higher molecular weight (heavy ends) components extracted from the fuel.
  - It contains lower levels of sodium polyacrylate thickener from water-adsorbing filters than dark, thick apple jelly.
  - It contains lower levels of components from SDA than dark, thick apple jelly.
- Regarding the conductivity of apple jelly:
  - The conductivity ranges from  $3 \times 10^{10}$  to  $8 \times 10^{11}$  pS/m.
  - The increased conductivity of apple jelly compared to DiEGME and water blends is due to species dissolved in the water/DiEGME matrix. Possible species include components from SDA and other additives, other dissolved ions from the fuel system, such as sodium, and materials carried in by external water (rain and runoff).
  - Thick apple jelly tends to have higher conductivity compared to thin apple jelly; but some thin apple jelly can have a very high conductivity.
- Regarding sodium content in apple jelly:
  - In thin apple jelly found in systems that do not use water-adsorbing filters, the sodium comes primarily from SDA and other low sodium sources. Only the thick samples are high in sodium content. There were no observed exceptions to this.
  - In thin apple jelly formed in systems that do use water-adsorbing filters, the sodium may come from the sodium polyacrylate, but the level of the sodium polyacrylate will not be sufficiently high to either cause noticeable thickening or be measurable by FTIR
  - In thick apple jelly, the sodium comes primarily from sodium polyacrylate thickener in the water-adsorbing filters.
- Regarding the acid number and base number of apple jelly:
  - In thin apple jelly, the TAN and TBN are due to fuel components and SDA components.

- In thick apple jelly, the TAN and TBN are due to SDA components and sodium polyacrylate thickener.
- Sodium polyacrylate accounts for most of the TBN in thick apple jelly.
- Regarding the total nitrogen content in apple jelly:
  - The total nitrogen content is primarily due to SDA components extracted into the apple jelly.
  - Sodium polyacrylate thickener greatly enhances the extraction of SDA components into the apple jelly. This explains the moderate correlation between nitrogen and viscosity, as well as the moderate correlation between nitrogen and sodium.
- Regarding the total sulfur content in apple jelly:
  - Sulfur in apple jelly comes primarily from fuel sulfur; and possibly, but to a far lesser extent, sulfur-containing components of SDA.
  - Most of the SDA sulfur-containing components do not extract from the fuel into the DiEGME and water in either thin or thick apple jelly.
  - SDA sulfur has much lower propensity to extract into apple jelly than SDA nitrogen.
- Regarding other properties of apple jelly:
  - The pH of apple jelly is usually near neutral (i.e., 5-8).
  - Chlorine levels in apple jelly are from components of SDA, and other sources such as dissolved salts.
  - Chlorine and certain other components of SDA can concentrate in apple jelly to levels greater than in neat SDA.
  - The role of corrosion inhibitor in apple jelly is unclear.
- Regarding the mechanism of formation of thin apple jelly:
  - It is a dynamic and continuous process.
  - DiEGME partitions from the fuel into water bottoms.
  - Fuel components extract into the DiEGME/water bottoms to the extent allowed by the DiEGME concentration.
  - SDA components extract into the DiEGME/water bottoms to the extent allowed by the DiEGME concentration and the concentration of fuel components. As SDA components extract into the fuel component-enriched, water/DiEGME bottoms, this may further enhance the extraction of fuel components, which, in

turn, may further enhance the extraction of SDA components. The exact nature of this interaction has not been completely defined.

- Regarding the mechanism of formation of thick apple jelly:
  - It is a dynamic and continuous process.
  - All thin apple jelly formation factors apply.
  - It requires contact with water adsorption filters.
  - As sodium polyacrylate concentrates in DiEGME/water in the filter, SDA extraction is enhanced, further contracting and thickening the gelled structure.
  - Shearing, from passing through the filter, further disperses the thickener.

#### **11.4 Additional Findings**

- Process factors that enhance the formation of apple jelly:
  - Splash blending of additives, especially DiEGME and SDA.
  - Wet DiEGME.
  - Poor mixing of DiEGME and/or SDA before storage tanks and/or filter/coalescer units.
  - Addition of DiEGME and SDA too close to each other in the fuel system.
  - Use of water adsorption filter units.
  - Sudden drops in ambient temperature.
  - Leaking fuel storage tanks.
- Regarding microbial aspects of apple jelly formation:
  - Microbes will grow in the presence of apple jelly. Several samples were either received with evidence of growth or growth appeared in the sample after it was received. The Air Force has also reported finding microbial growth in apple jelly samples with DiEGME concentration as high as 80%.
  - Microbial growth will likely contribute some material, in the form of metabolites, to the apple jelly matrix.
  - Microbial growth is not a required precursor for apple jelly formation. Many of the samples received had no visible signs of microbial growth and never displayed any. Also, apple jelly samples that had no visible microbial colonies

when received, but eventually had some growth at the fuel/water interface, exhibited no increase in viscosity.

## **11.5 Recommendations**

The following recommendations are given as possible means to reduce or eliminate the formation of apple jelly in fuel systems. In forming these recommendations, no consideration was given to cost or feasibility of implementation. Those factors should be considered and applied by DESC and its customers.

1. Replace DiEGME with an alternative, less-problematic FSII, assuming this is chemically possible.
2. The problems of water intrusion and removal are critical. Re-engineer fuel facilities, equipment, and processes to address water problems.
3. If possible, JP-8 should pass through a water removal process prior to injection of FSII.
4. Move additive injection (especially FSII) as far forward in the logistics system as possible.
5. Store additized JP-8 in tanks with fixed roofs or effective geodesic domes and an effective sump draining / product recovery system.
6. Fuel service personnel must maintain an active sump draining process. Sumps should be drained after each receipt, prior to issue, and following dramatic temperature drops.
7. Suction lines in large storage tanks should be moved so they do not draw fuel from the sump.
8. Inventory levels should be sufficient to allow adequate settling time for receipt tanks.
9. Filter vessels built to old DoD design should be modified or replaced (including vehicles and hose carts) to conform to API/IP 1581 standards. This will help remove water that is currently passing through some of the older systems.
10. Use of NSN 4330-00-987-0998 coalescer elements should be discontinued.

11. Reduce or eliminate splash blending of additives (especially FSII) wherever possible.
12. All additive injection should be done in such a way to allow sufficient pipeline mixing of the additives. In pipeline delivery systems, inject a second additive only after sufficient pipeline mixing of the previously injected additive.
13. Add DiEGME only at the skin of the aircraft or downstream of all filtering. This will not eliminate possible problems if there is water in the aircraft fuel tanks. Contaminated water bottoms will form and must be removed from the aircraft fuel tanks.

### **11.6 Possible Additional Work**

The follow list of possible additional work is considered interesting and would provide some additional information about apple jelly.

- Investigate alternative FSII compounds with regard to apple jelly formation.
- Further define the relationship between heavy fuel components, SDA components and other fuel additives.
- Further define the relationship between percent water and extraction of heavy fuel/SDA components.
- Evaluate the role of corrosion inhibitor additive.
- Perform liquid chromatography-mass spectroscopy analyses as additional verification of the presence of additive components in apple jelly.
- Conduct additional syntheses using sodium polyacrylate and a mechanical homogenizer to better understand the role of shearing and the role of sodium polyacrylate on SDA component incorporation into apple jelly.
- Further refine the methods for measuring water and DiEGME content of apple jelly, especially thick apple jelly. Develop an FT-IR method for this analysis.

## 12 ABBREVIATIONS AND ACRONYMS

AST	above ground storage tank
ASTM	American Society for Testing and Materials
BS&W	bottom sediment & water
EGME	ethylene glycol monomethyl ether
FARP	Forward Area Refueling Point
F/S	filter/separator
FSII	fuel system icing inhibitor
FT-IR	fourier transform infrared
DiEGME	diethylene glycol monomethyl ether
ICP	inductively coupled plasma spectroscopy
JPTS	jet propellant thermally stable
KMP	Kinder/Morgan Pipeline
NATO	North Atlantic Treaty Organization
NRL	Naval Research Laboratory
NSN	National Stock Number
SDA	static dissipator additive
TEPPCO	Texas Eastern Pipeline Products Company
TGA	thermogravimetric analysis

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**APPENDIX A**  
**SAMPLING INSTRUCTIONS**



## **Procedure For Taking And Sending Samples Of Apple Jelly And Associated Jet Fuel**

The following steps explain the system that will be used in taking and sending samples of apple jelly and associated jet fuel. This information also explains the containers to be used in taking such samples, how such containers are obtained, and what information needs to be documented and sent concerning the samples. Please read the entire document. If after doing so, you have any questions please contact Andy Waynick at [jwaynick@swri.org](mailto:jwaynick@swri.org) or by phone at 210-522-6844.

1. Whenever an apple jelly occurrence at any facility is observed, a sample of the apple jelly is to be taken and placed directly into the appropriate 500 ml glass sample container. Details concerning this container are given in step 4, below. If possible, the apple jelly sample should fill the glass container at least four fifths to the top (i.e. at least about 400 ml of apple jelly). If you can fill the glass container almost to the top, do so. However, leave a small amount of head space for volume expansion in case the sample temperature rises during shipping to Southwest Research Institute (SwRI). When taking samples, please observe the following guidelines:
  - a. Immediately upon noticing any apple jelly formation and just before taking any apple jelly samples, take the temperature of the apple jelly by direct insertion of a thermometer. Do NOT agitate or stir the apple jelly during the process of taking the temperature.
  - b. Place the apple jelly in the glass container with a minimum amount of agitation or stirring.
  - c. When taking the sample of apple jelly, try to obtain ONLY the apple jelly material, and not any fuel or colored (brown, reddish-brown, amber, etc) thin liquid that may also be present with the apple jelly. The final apple jelly sample in the glass container should, as much as possible, appear as one single phase, not two phases.
  - d. Along with the glass container, send a brief document that states when the apple jelly was first observed (date and time), where it was observed, where it was taken (if different than the location first observed), temperature of apple jelly just prior to taking it, appearance of the apple jelly when the sample was taken. The description of the apple jelly appearance should include color, general comments about its thickness, and whether the sample in the container consisted of one single phase at the time it was packaged and shipped.
2. If less than 300 ml of apple jelly is available for sampling, use one or more of the 125 ml (4 ounce) glass containers. Details concerning these containers are given in step 4, below. Depending on the quantity of apple jelly available, you may use up to three of the 125 ml glass bottles. Fill as many as the first two of of the 125 ml glass containers nearly full, allowing a small amount of head space for volume expansion in case the sample temperature rises during shipping to Southwest Research Institute (SwRI). If a third 125 ml glass container is required, place the remaining apple jelly in it. Obviously, if there is enough apple jelly to completely fill three 125 ml glass containers, you should not use the 125 ml glass containers but instead you should place the entire apple jelly sample in a 500 ml glass container as described above in step 1. When taking samples, please observe the same guidelines listed above in step 1a-d.

3. Whenever apple jelly is observed and a sample is taken, if at all possible a one gallon sample of the fuel from which the apple jelly has apparently formed should also be taken. The one-gallon sample should be taken in a 1-gallon epoxy-lined steel can as described in step 4, below. Care should be taken to obtain a fuel sample that is as representative as possible of the fuel from which the apple jelly was formed. For instance, taking a sample of fuel from a tank days after the apple jelly was formed in a downstream filter/coalescer, and after the fuel tank has turned over one or more times is not acceptable as a method of taking a representative fuel sample. Other than insuring that the fuel sample is representative of what the apple jelly came from, no other special procedures are required when sampling the fuel except for normal safety practices.
4. Samples should be taken as described above in steps 1 and 2 and sent to SwRI using specific containers and packaging boxes sent to you by SwRI. Each location that needs to send samples of apple jelly and fuel will be sent a complete ensemble of containers and matching packaging boxes containing the following items:
  - a. One 500 ml wide-mouth glass bottle with plastic teflon-coated cap.
  - b. One packaging box for shipping the 500 ml glass bottle.
  - c. Three 125 ml (4-ounce) wide-mouth glass bottles with plastic polyethylene lined cap.
  - d. One packaging box for shipping three of the 125 ml wide-mouth glass bottles. Please note that although this packaging box can accommodate as many as six of the 125 ml wide-mouth glass bottles, you will normally need only up to three of the six available spaces. The other three spaces will remain empty under normal sampling and shipping circumstances.
  - e. One steel, epoxy-lined 1-gallon can.
  - f. One packaging box for shipping the 1-gallon, epoxy-lined steel can.
5. Based on prior history of apple jelly occurrences and current problems, the following locations will be automatically sent a complete ensemble of containers and matching packaging boxes as listed in step 4, above. Each of the below locations should receive their ensemble of containers and boxes by February 15, 2001.
  - a. Grand Forks AFB, ND
  - b. Otis ANG, MA
  - c. Tinker AFB, OK
  - d. McConnell AFB, KS
  - e. Barksdale, AFB, LA
  - f. Osan Air Base, Korea
  - g. Pease ANG, NH
  - h. Maine ANG, Bangor, ME
  - i. Misawa AB, Japan
  - j. Maxwell AFB, AL
  - k. Shaw AFB, SC
  - l. Kadena AB, Japan
  - m. Yokota AB, Japan

6. If any of the locations listed in step 5, above, observe any apple jelly, they should take samples of the apple jelly and fuel (if possible) in accordance with the steps above. An appropriate person at that location should immediately call Andy Waynick, SwRI, at 210-522-6844 to inform him that the samples are being sent. Once this call is made and the samples are taken and appropriately packaged, they should be sent by overnight express mail. Remember to include the information taken when the samples were obtained, as described in step 1d, above. Use only an overnight express mail carrier that can trace undelivered shipments. Once we know that the samples are on the way to SwRI, we will send replacement containers and boxes to replace the ones that were used to take and send the apple jelly and fuel samples. Apple jelly and fuel samples should be sent to the following address:

Southwest Research Institute  
6220 Culebra Road  
Building 99  
San Antonio, Texas 78238-5166  
Attn: Joe Garza/Andy Waynick/Apple Jelly

7. Locations other than those listed above will need to phone Andy Waynick at SwRI, 210-522-6844 if apple jelly is found. During this phone call you will need to identify who you are, where you are including complete mailing address, and a brief description of the apple jelly (what it looks like, where it was observed). Once this call is made and received, a complete ensemble of containers and boxes will be immediately overnight express mailed to you at the address given during your phone call. Once you receive the containers and boxes, take your samples in accordance with the above steps. If time and operations make it impossible to wait until the containers and boxes arrive, use whatever temporary container(s) is available to take the apple jelly and fuel samples. However, use the same precautions and procedures as outlined in the above steps. All apple jelly and fuel samples taken in temporary containers while waiting for the official containers to arrive should be tightly sealed to prevent evaporation and/or contamination. Under no circumstances should apple jelly samples be placed in a container with any exposed metal, either in the container itself or in the cap. When the official ensemble of containers and boxes arrive, carefully transfer the apple jelly and fuel samples to the appropriate official containers, in accordance with the above steps. Be especially careful to cause as little agitation of the apple jelly as possible when transferring it from the temporary container to the official one (or ones). Samples should be sent to the same address as given in step 6, above. Remember to include the information taken when the samples were obtained, as described in step 1d, above.

**If any apple jelly situation occurs that does not appear to fit within the guidelines given in the above steps, please call Andy Waynick at 210-522-6844 to determine what procedures should be followed in sampling and sending the appropriate samples.**





**APPENDIX B**  
**LISTING OF ALL SAMPLES**



<b>Apple Jelly Project Samples</b>			
<b>Sample ID</b>	<b>Sample Matrix (Jet-A/JP-8/JP-5/other)</b>	<b>Type of Sample (Fuel/Apple Jelly/ DIEGME/other)</b>	<b>Base Name</b>
AP-001	N/A	AJ (Apple Jelly)	Maine ANG
AP-002	N/A	AJ	Maine ANG
AP-003	JP-8	Fuel	Ft. Sam Houston
AP-004	JP-8	Fuel	Camp Bullis
AP-005	JP-8	Fuel	Maxwell AFB
AP-006	JP-8	AJ	Maxwell AFB
AP-007	JP-8	AJ	Maxwell AFB
AP-008	JP-8	AJ	Maxwell AFB
AP-009	JP-8	AJ	Maxwell AFB
AP-010	JP-8	AJ	NFARS
AP-011	JP-8	AJ	NFARS
AP-012	JP-8	AJ	NFARS
AP-013	JP-8	Fuel	NFARS
AP-014	N/A	AJ	Ellsworth AFB
AP-015	N/A	AJ	Ellsworth AFB
AP-016	N/A	AJ	Ellsworth AFB
AP-017	N/A	AJ	Ellsworth AFB
AP-018	N/A	AJ	Ellsworth AFB
AP-019	N/A	AJ	Ellsworth AFB
AP-020	N/A	AJ	Ellsworth AFB
AP-021	N/A	AJ	Ellsworth AFB
AP-022	N/A	AJ	Ellsworth AFB
AP-023	N/A	AJ	Ellsworth AFB
AP-024	JP-8	Fuel	Fairchild AFB
AP-025	JP-8	Fuel	Fairchild AFB
AP-026	JP-8	Fuel	Fairchild AFB
AP-027	JP-8	Fuel	Fairchild AFB
AP-028	JP-8	AJ	Fairchild AFB
AP-029	JP-8	AJ	Fairchild AFB
AP-030	JP-8	AJ	Fairchild AFB
AP-031	JP-8	AJ	Fairchild AFB
AP-032	JP-8	AJ	Grand Forks AFB
AP-033	JP-8	AJ	Grand Forks AFB
AP-034	JP-8	AJ	Grand Forks AFB
AP-035	N/A	AJ	Barksdale AFB
AP-036	N/A	Fuel	Barksdale AFB
AP-037	N/A	AJ	McConnell AFB
AP-038	N/A	AJ	McConnell AFB
AP-039	N/A	AJ	McConnell AFB
AP-040	N/A	AJ	McConnell AFB
AP-041	N/A	AJ	McConnell AFB
AP-042	N/A	AJ	McConnell AFB
AP-043	N/A	AJ	McConnell AFB
AP-044	N/A	AJ	McConnell AFB
AP-045	N/A	AJ	McConnell AFB
AP-046	JP-8	Fuel	Shaw AFB
AP-047	JP-8	Fuel	Shaw AFB

<b>Apple Jelly Project Samples</b>			
<b>Sample ID</b>	<b>Sample Matrix (Jet-A/JP-8/JP-5/other)</b>	<b>Type of Sample (Fuel/Apple Jelly/ DIEGME/other)</b>	<b>Base Name</b>
AP-048	JP-8	AJ	Shaw AFB
AP-049	JP-8	AJ	Shaw AFB
AP-050	JP-8	AJ	Shaw AFB
AP-051	JP-8	AJ	Shaw AFB
AP-052	JP-8	AJ	Shaw AFB
AP-053	JP-8	AJ	Shaw AFB
AP-054	JP-8	AJ	Shaw AFB
AP-055	N/A	AJ	Delaware ANG
AP-056	N/A	AJ	Beale AFB
AP-057	N/A	AJ	New York ANG
AP-058	N/A	AJ	Maine ANG
AP-059	N/A	AJ	Maine ANG
AP-060	JP-8	AJ	Eglin AFB
AP-061	JP-8	AJ	Eglin AFB
AP-062	N/A	AJ	Maine ANG
AP-063	N/A	Fuel	Maine ANG
AP-064	N/A	Fuel	Maine ANG
AP-065	N/A	AJ	McGuire AFB
AP-066	N/A	AJ	McGuire AFB
AP-067	N/A	AJ	McGuire AFB
AP-068	N/A	AJ	McGuire AFB
AP-069	N/A	AJ	Shaw AFB
AP-070	N/A	AJ	Travis AFB
AP-071	JP-8	Fuel	Atlantic Product Services
AP-072	JP-8	Fuel	Atlantic Product Services
AP-073	-	Stadis 450 Static Dissipator Additive	Exxon Mobil Refining & Supply
AP-074	-	Deicer - DIEGME	Exxon Mobil Refining & Supply
AP-075	N/A	AJ	Barksdale AFB
AP-076	N/A	AJ	Barksdale AFB
AP-077	-	Nalco 5403 Jet Corrosion Inhibitor	Exxon Mobil Refining & Supply
AP-078	JP-8	Fuel	Edwards AFB
AP-079	JP-8	AJ	Edwards AFB
AP-080	JP-8	AJ	Edwards AFB
AP-081	JP-8	AJ	Edwards AFB
AP-082	JP-8	AJ	Edwards AFB
AP-083	JP-8	AJ	Edwards AFB
AP-084	N/A	AJ	Misawa AB
AP-085	N/A	Fuel	Misawa AB
AP-086	N/A	AJ	Misawa AB
AP-087	N/A	Fuel	Misawa AB
AP-088	N/A	AJ	Misawa AB
AP-089	JP-8	Fuel	Shaw AFB
AP-090	JP-8	AJ	Shaw AFB
AP-091	JP-8	Fuel	Beale AFB
AP-092	JP-8	Fuel	Beale AFB
AP-093	JP-8	AJ	Edwards AFB

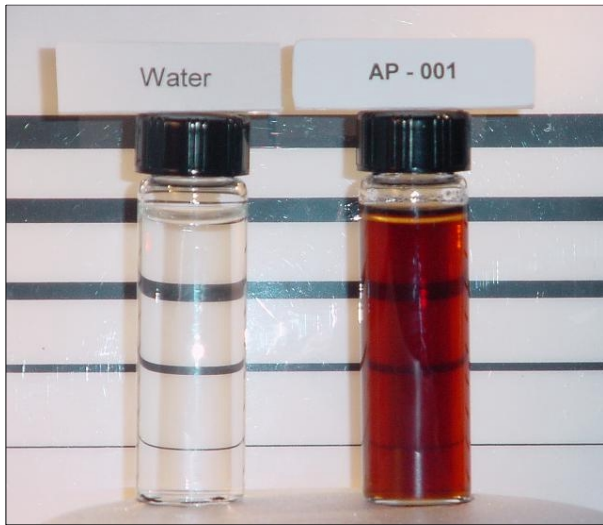
<b>Apple Jelly Project Samples</b>			
<b>Sample ID</b>	<b>Sample Matrix (Jet-A/JP-8/JP-5/other)</b>	<b>Type of Sample (Fuel/Apple Jelly/ DIEGME/other)</b>	<b>Base Name</b>
AP-094	JP-8	AJ	Edwards AFB
AP-095	JP-8	AJ	Edwards AFB
AP-096	JP-8	AJ	Edwards AFB
AP-097	JP-8	AJ	Edwards AFB
AP-098	JP-8	AJ	Edwards AFB
AP-099	JP-8	AJ	Edwards AFB
AP-100	N/A	Absorption Media Element	Edwards AFB
AP-101	N/A	Filter Media Element	Edwards AFB
AP-102	N/A	AJ	Shaw AFB
AP-103	N/A	AJ	McConnell AFB
AP-104	N/A	Filter Media Element	N/A
AP-105	JP-8	Fuel	Lajes Field
AP-106	JP-8	Fuel	McGuire AFB
AP-107	JP-8	Fuel	McGuire AFB
AP-108	JP-8	AJ	Lajes Field
AP-109	JP-8	Fuel	Lajes Field
AP-110	JP-8	AJ	Lajes Field
AP-111	JP-8	Fuel	Lajes Field
AP-112	N/A	AJ	Boise ANG
AP-113	N/A	AJ	Yokota
AP-114	N/A	AJ	Yokota
AP-115	N/A	AJ	Yokota
AP-116	N/A	Filter Media Element	Yokota
AP-117	N/A	AJ	Edwards AFB
AP-118	N/A	AJ	Dover AFB
AP-119	JP-8	Fuel	Misawa AB
AP-120	JP-8	AJ	Misawa AB
AP-121	JP-8	AJ	Misawa AB
AP-122	JP-8	AJ	Eglin AFB
AP-123	JP-8	AJ	Eglin AFB
AP-124	JP-8	AJ	Eglin AFB
AP-125	JP-8	AJ	Eglin AFB
AP-126	JP-8	AJ	Eglin AFB
AP-127	JP-8	AJ	Eglin AFB
AP-128	JP-8	AJ	Eglin AFB
AP-129	N/A	Filter Media Element	McCord AFB
AP-130	JP-8	Fuel	Dover AFB
AP-131	JP-10	AJ	Williams International
AP-132	JP-10	Fuel	Williams International
AP-133	N/A	AJ	Elmendorf AFB
AP-134	JP-8	AJ	Life Flight of Maine
AP-135	JP-8	Fuel	Life Flight of Maine
AP-136	JP-5	AJ	Elmendorf AFB
AP-137	JP-5	Filter Media Element	Elmendorf AFB
AP-138	JP-8	Fuel	ANG CRTC
AP-139	JP-8	AJ	Charleston AFB



**APPENDIX C**  
**PHOTOGRAPHS OF FIRST 69 SAMPLES**



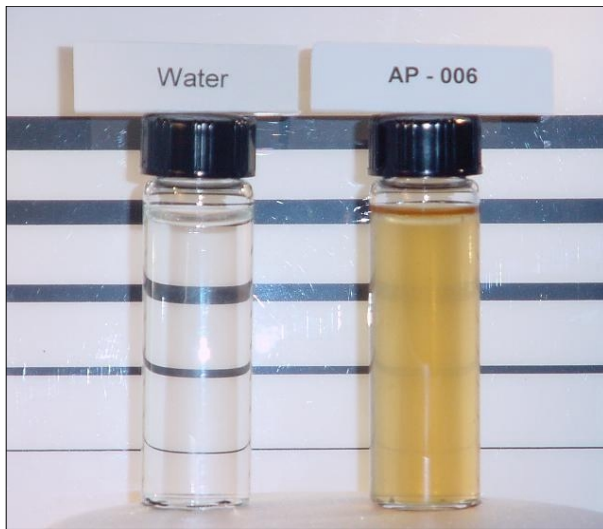




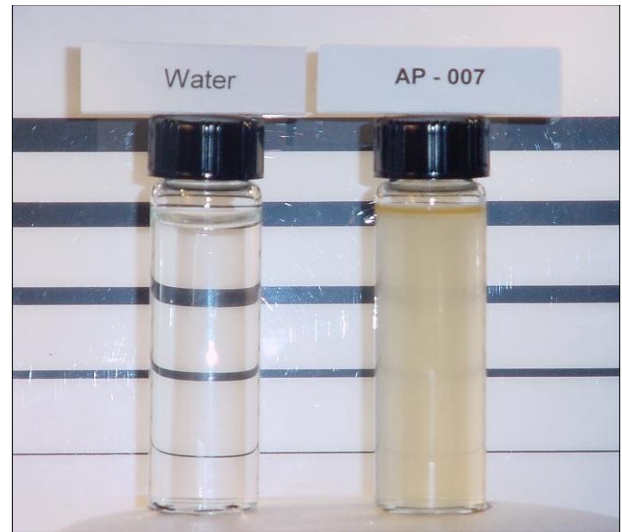
**Figure C-1. Apple Jelly Sample AP-001**



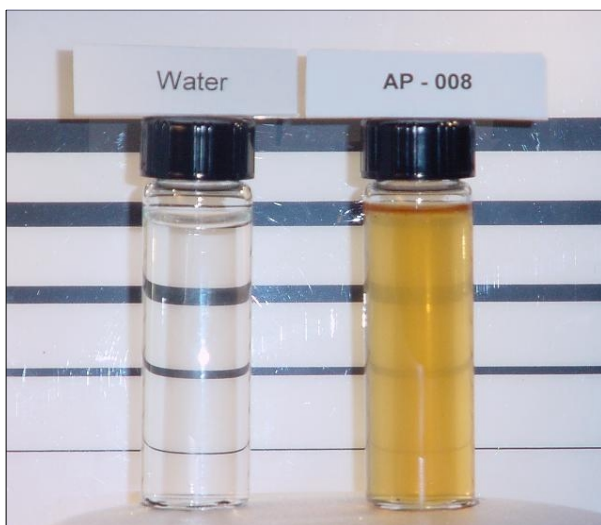
**Figure C-2. Apple Jelly Sample AP-002**



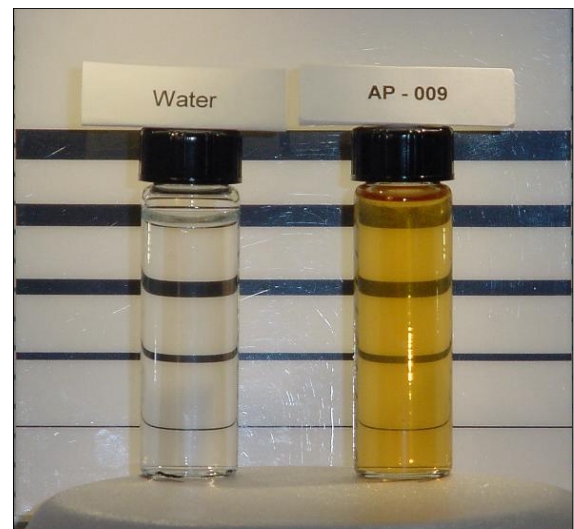
**Figure C-3. Apple Jelly Sample AP-006**



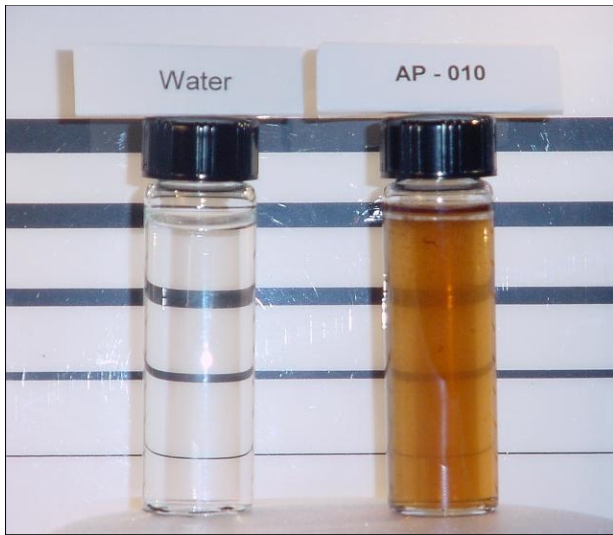
**Figure C-4. Apple Jelly Sample AP-007**



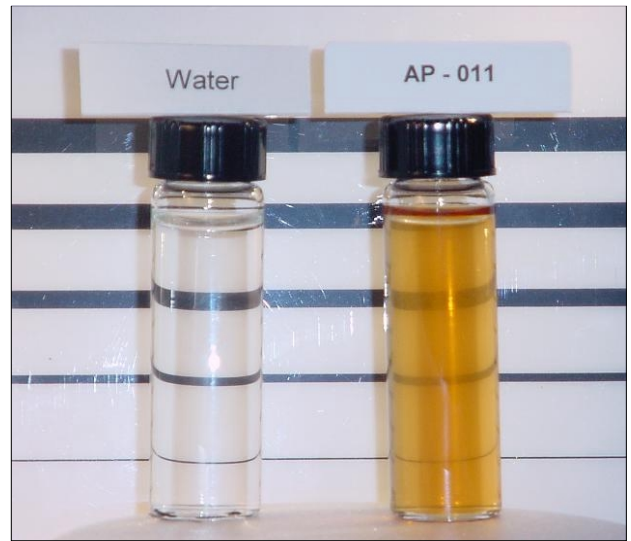
**Figure C-5. Apple Jelly Sample AP-008**



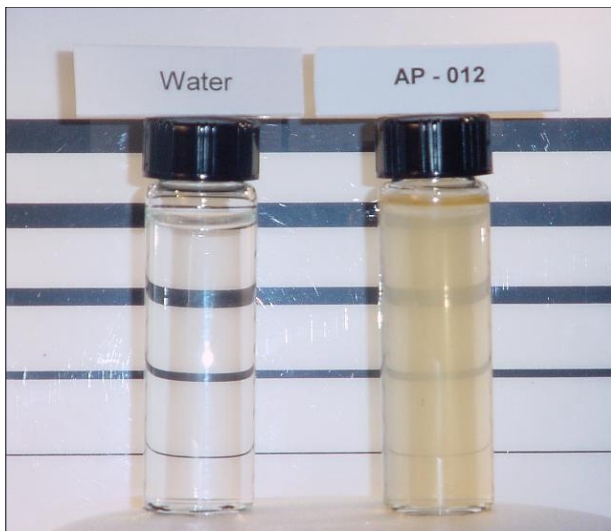
**Figure C-6. Apple Jelly Sample AP-009**



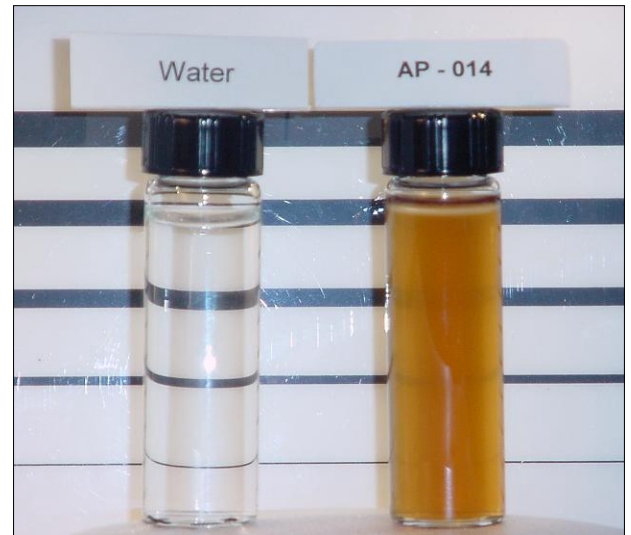
**Figure C-7. Apple Jelly Sample AP-010**



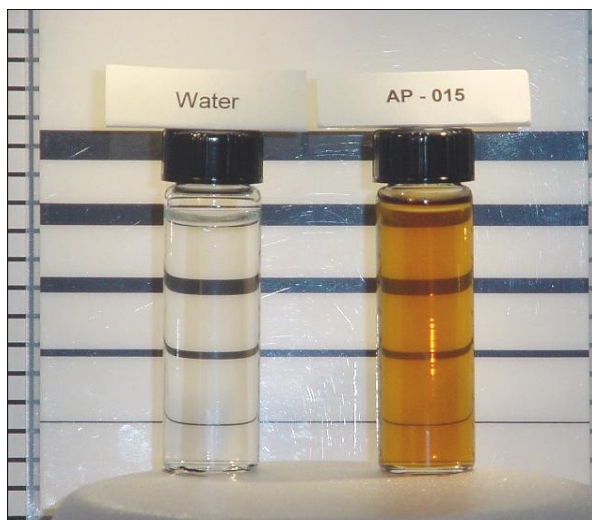
**Figure C-8. Apple Jelly Sample AP-011**



**Figure C-9. Apple Jelly Sample AP-012**



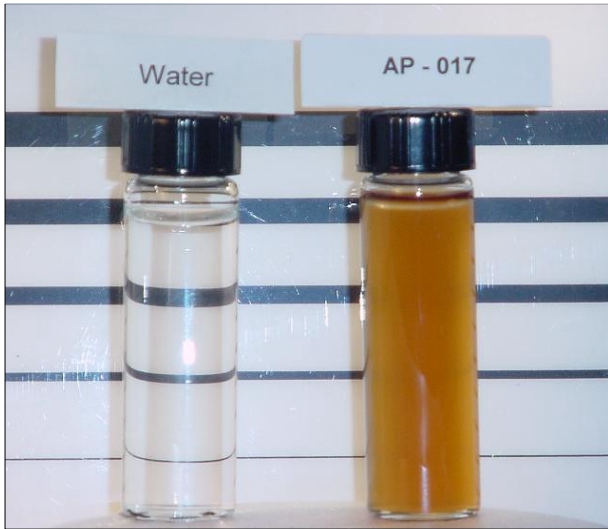
**Figure C-10. Apple Jelly Sample AP-014**



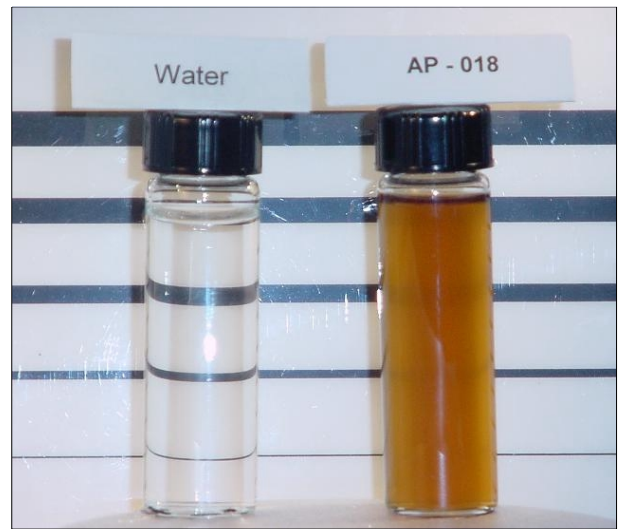
**Figure C-11. Apple Jelly Sample AP-015**



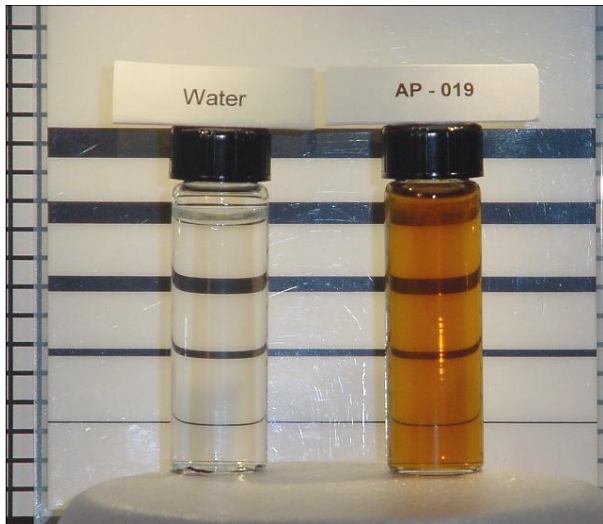
**Figure C-12. Apple Jelly Sample AP-016**



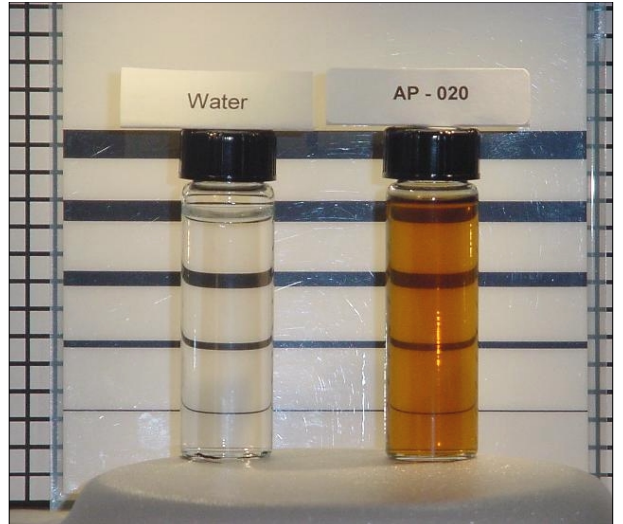
**Figure C-13. Apple Jelly Sample AP-017**



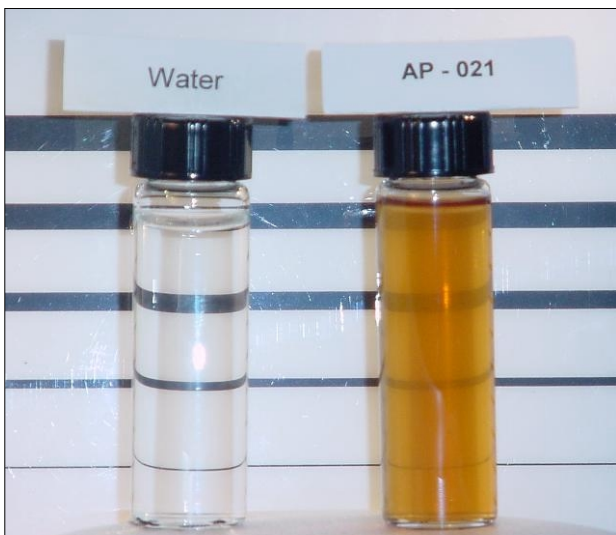
**Figure C-14. Apple Jelly Sample AP-018**



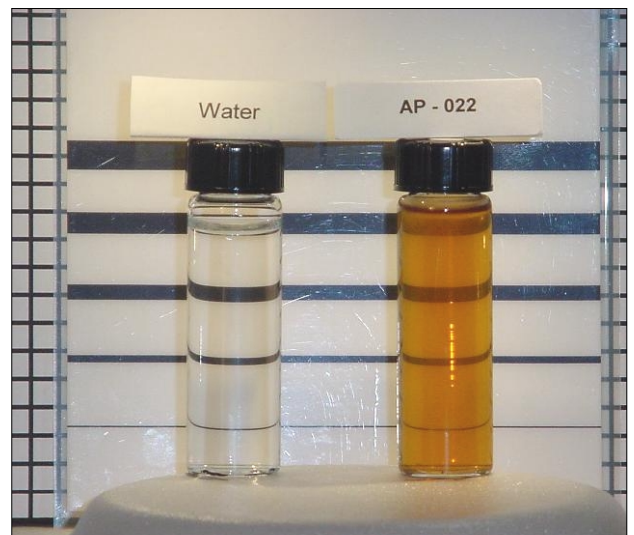
**Figure C-15. Apple Jelly Sample AP-019**



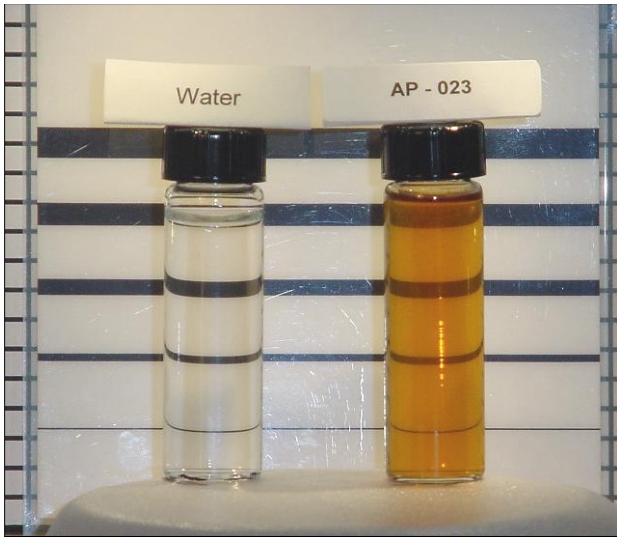
**Figure C-16. Apple Jelly Sample AP-020**



**Figure C-17. Apple Jelly Sample AP-021**



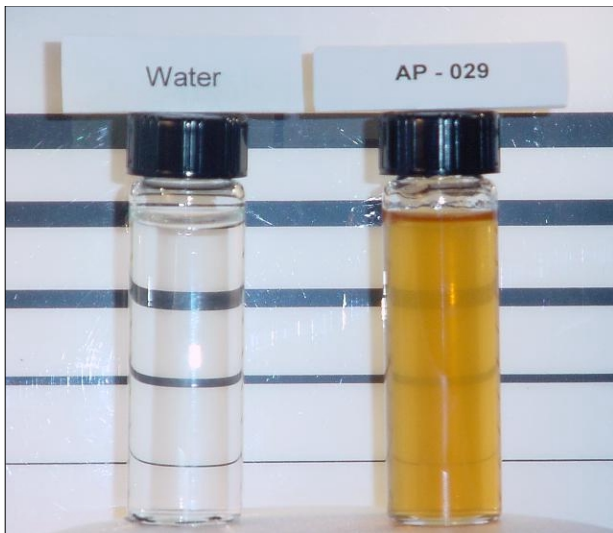
**Figure C-18. Apple Jelly Sample AP-022**



**Figure C-19. Apple Jelly Sample AP-023**



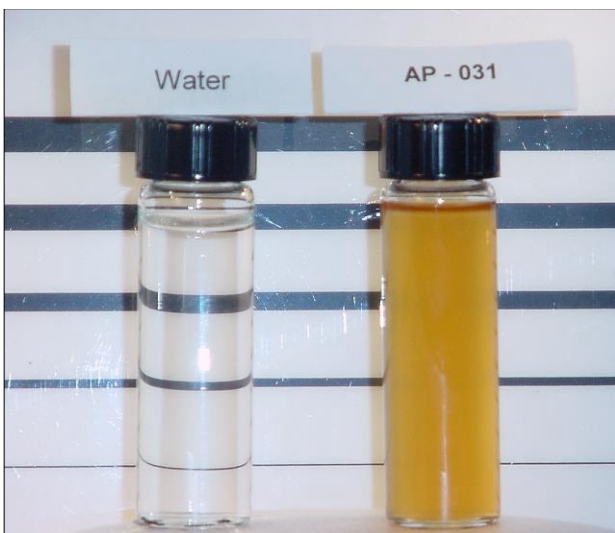
**Figure C-20. Apple Jelly Sample AP-028**



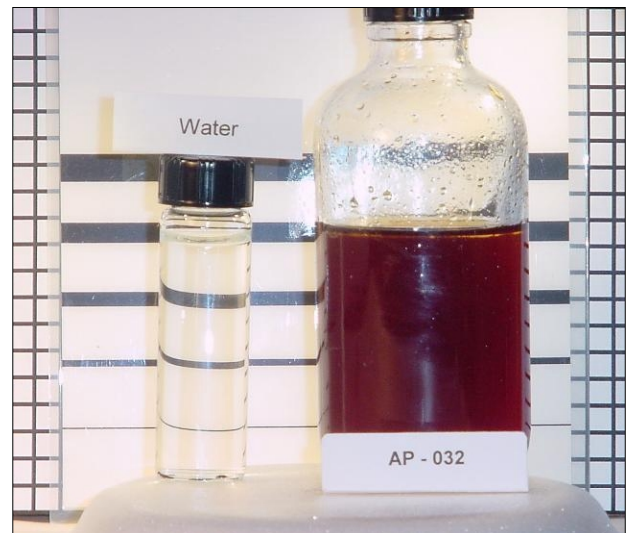
**Figure C-21. Apple Jelly Sample AP-029**



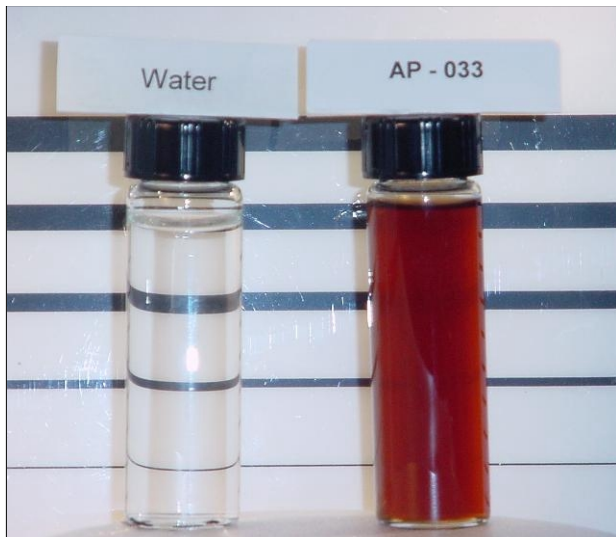
**Figure C-22. Apple Jelly Sample AP-030**



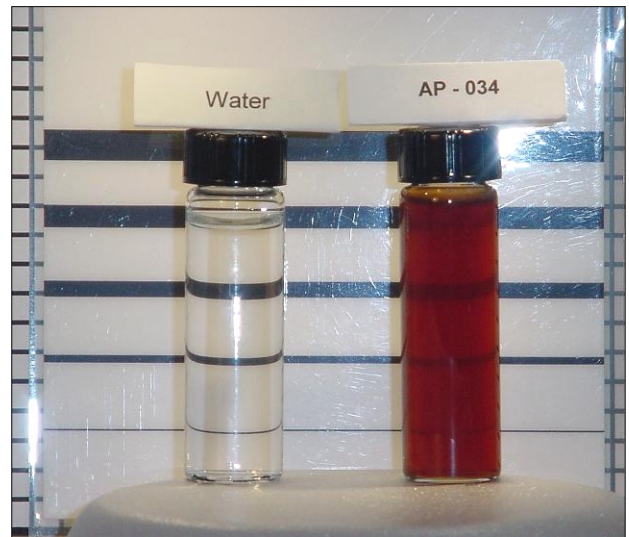
**Figure C-23. Apple Jelly Sample AP-031**



**Figure C-24. Apple Jelly Sample AP-032**



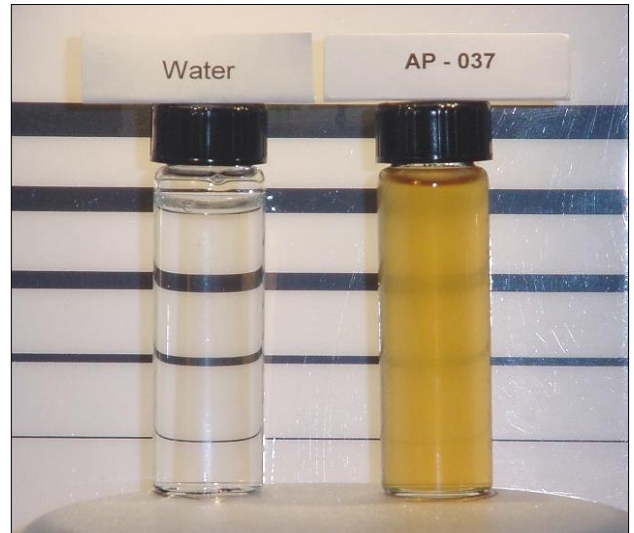
**Figure C-25. Apple Jelly Sample AP-033**



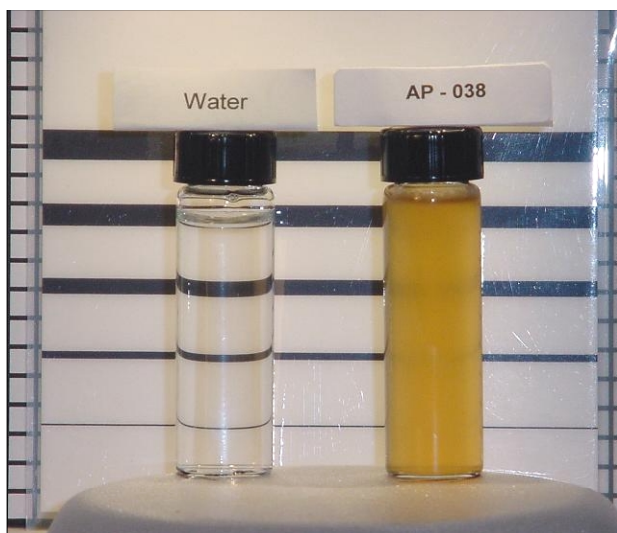
**Figure C-26. Apple Jelly Sample AP-034**



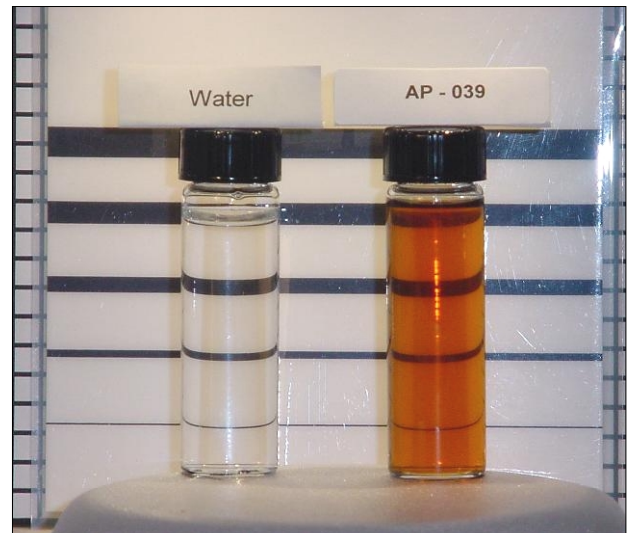
**Figure C-27. Apple Jelly Sample AP-035**



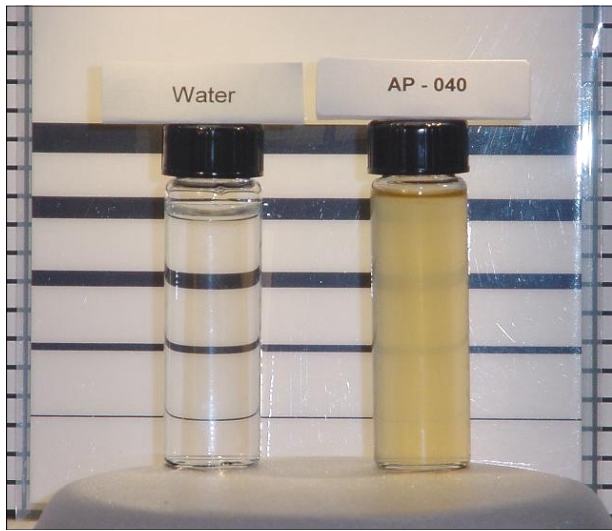
**Figure C-28. Apple Jelly Sample AP-037**



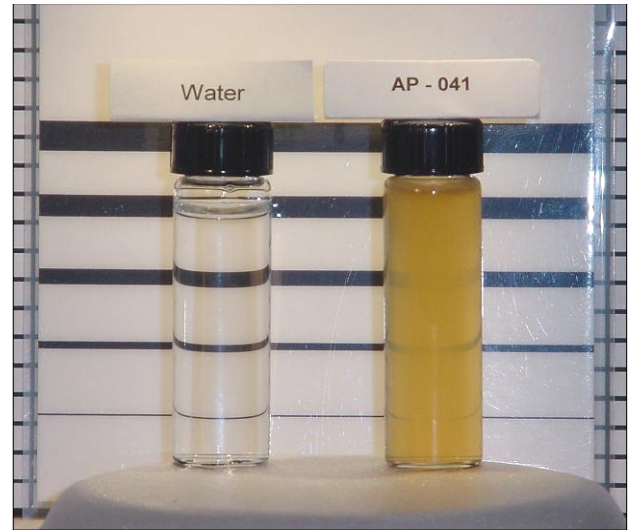
**Figure C-29. Apple Jelly Sample AP-038**



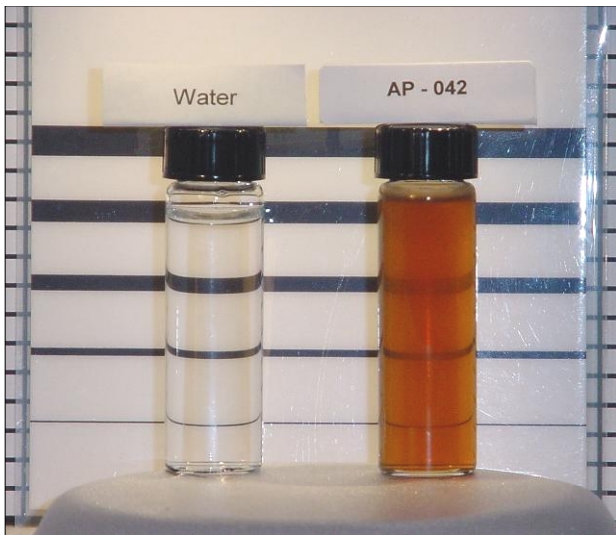
**Figure C-30. Apple Jelly Sample AP-039**



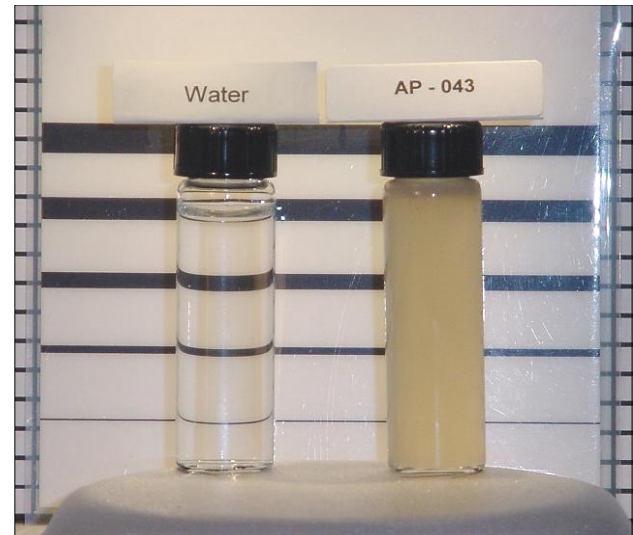
**Figure C-31. Apple Jelly Sample AP-040**



**Figure C-32. Apple Jelly Sample AP-041**



**Figure C-33. Apple Jelly Sample AP-042**



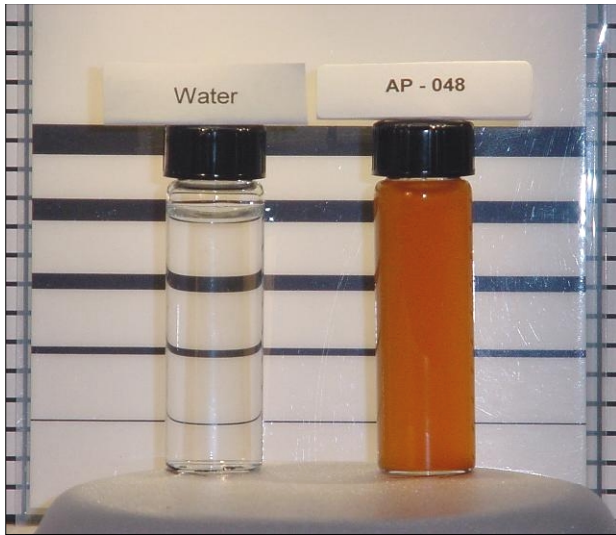
**Figure C-34. Apple Jelly Sample AP-043**



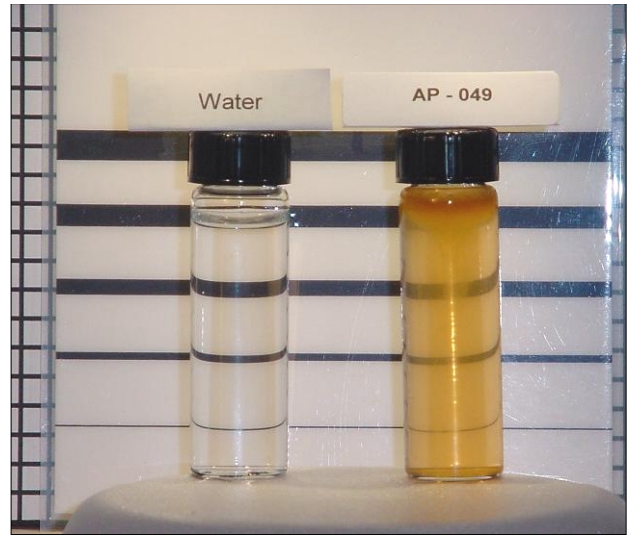
**Figure C-35. Apple Jelly Sample AP-044**



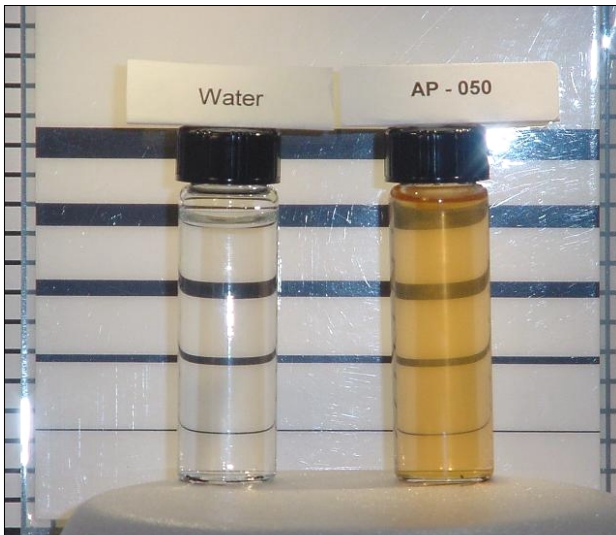
**Figure C-36. Apple Jelly Sample AP-045**



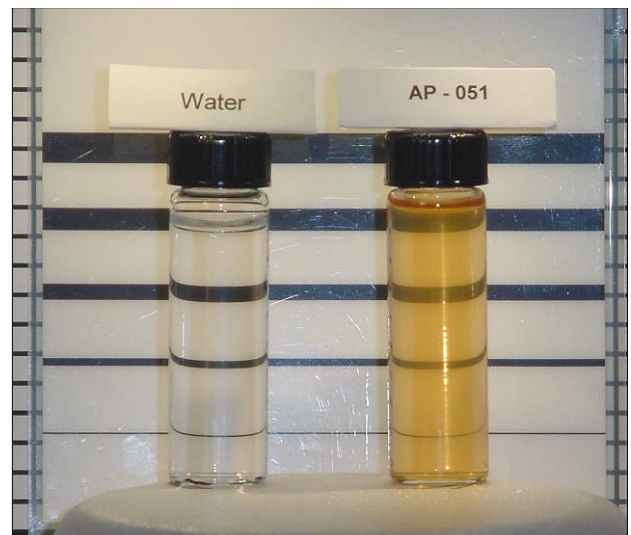
**Figure C-37. Apple Jelly Sample AP-048**



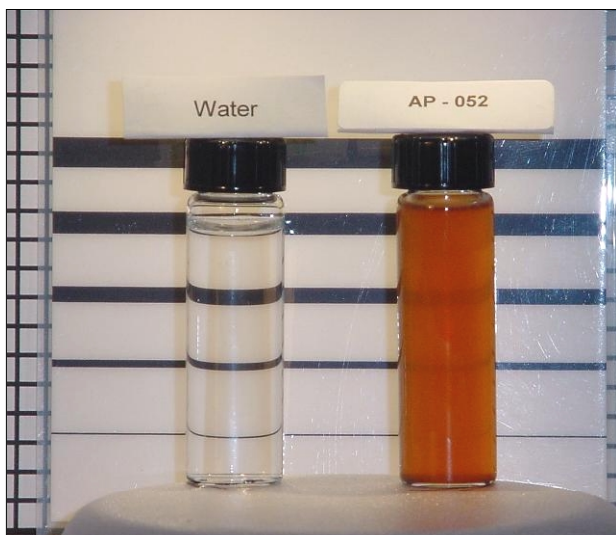
**Figure C-38. Apple Jelly Sample AP-049**



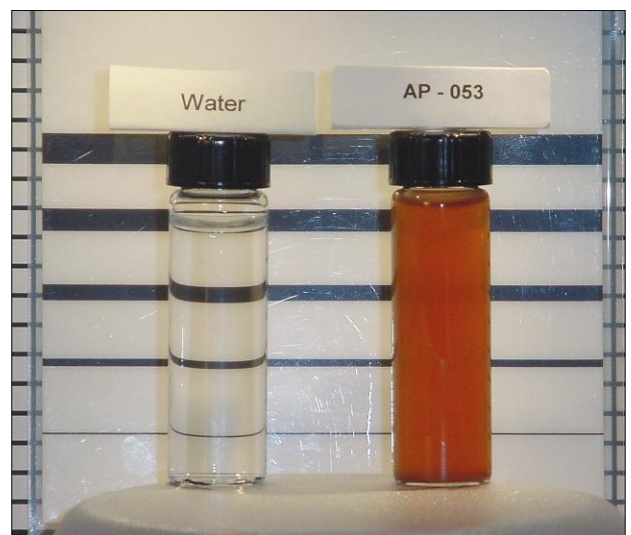
**Figure C-39. Apple Jelly Sample AP-050**



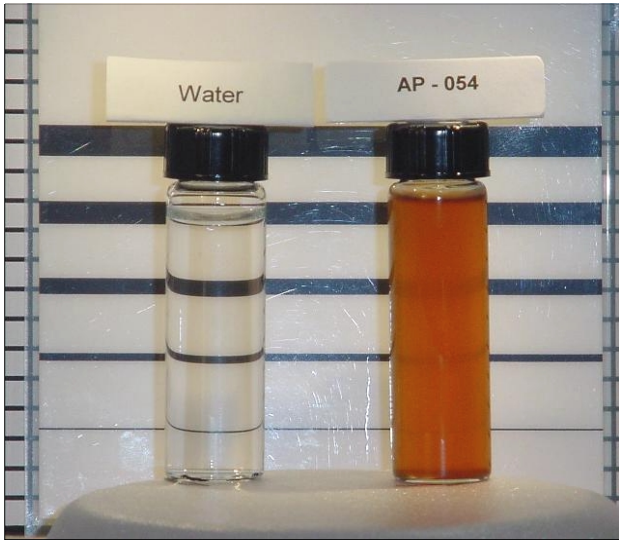
**Figure C-40. Apple Jelly Sample AP-051**



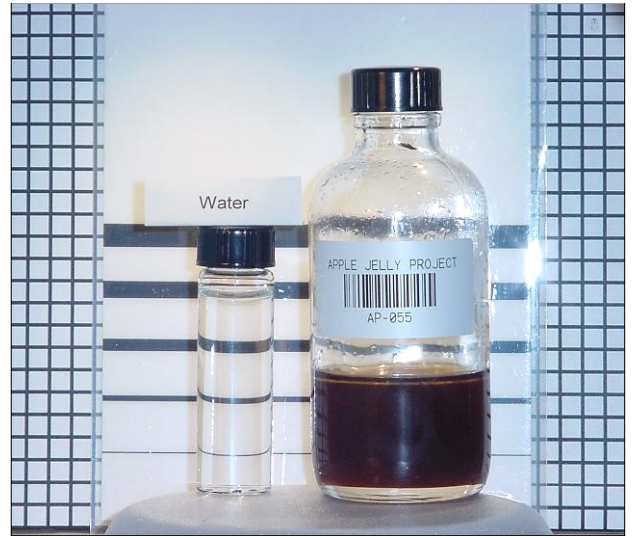
**Figure C-41. Apple Jelly Sample AP-052**



**Figure C-42. Apple Jelly Sample AP-053**



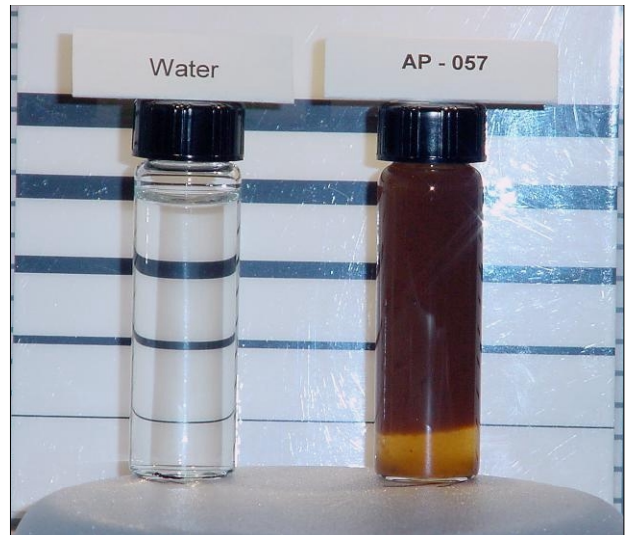
**Figure C-43. Apple Jelly Sample AP-054**



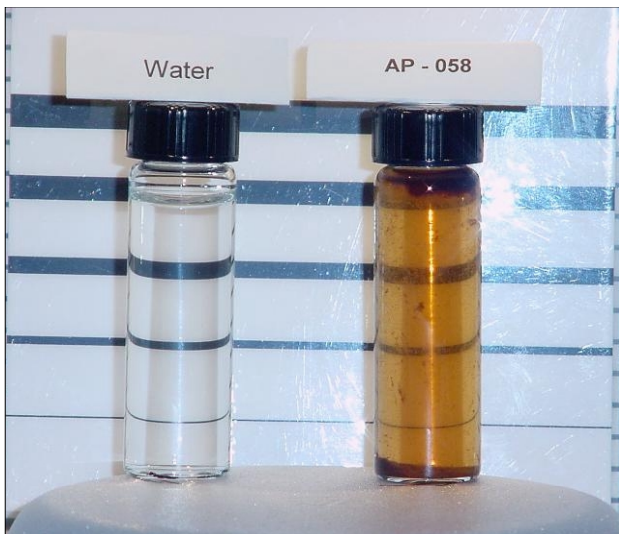
**Figure C-44. Apple Jelly Sample AP-055**



**Figure C-45. Apple Jelly Sample AP-056**



**Figure C-46. Apple Jelly Sample AP-057**

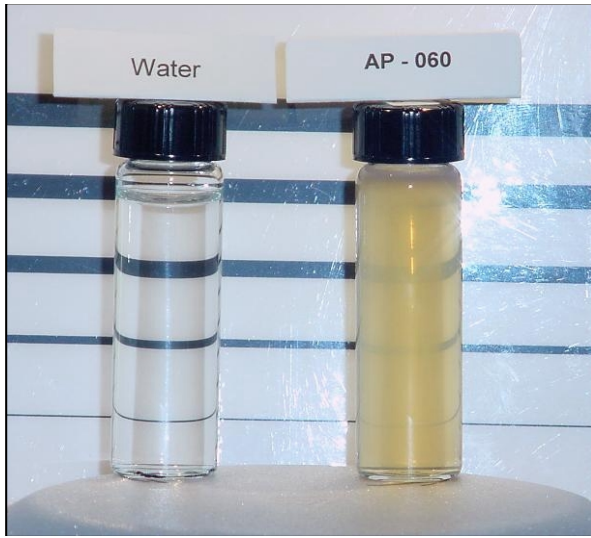


**Figure C-47. Apple Jelly Sample AP-058**

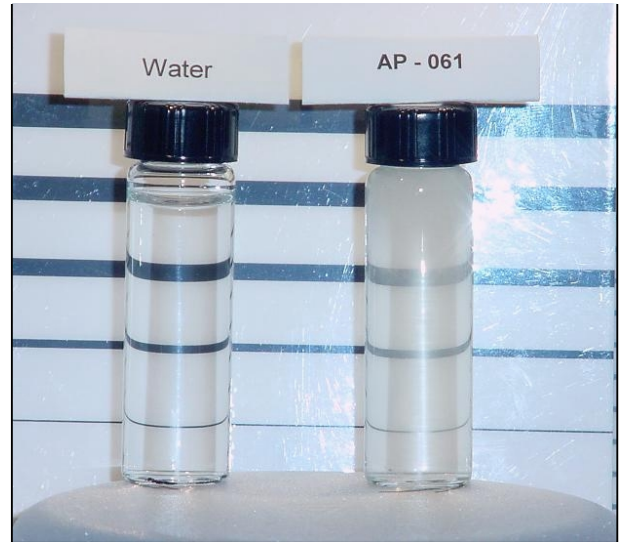


**Figure C-48. Apple Jelly Sample AP-059**

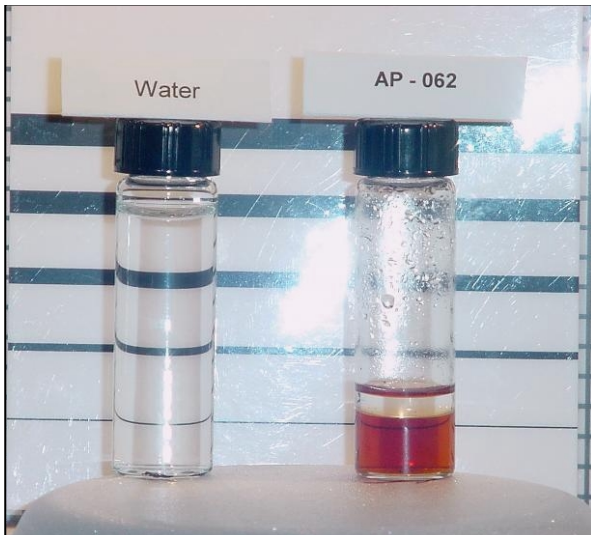




**Figure C-49. Apple Jelly Sample AP-060**



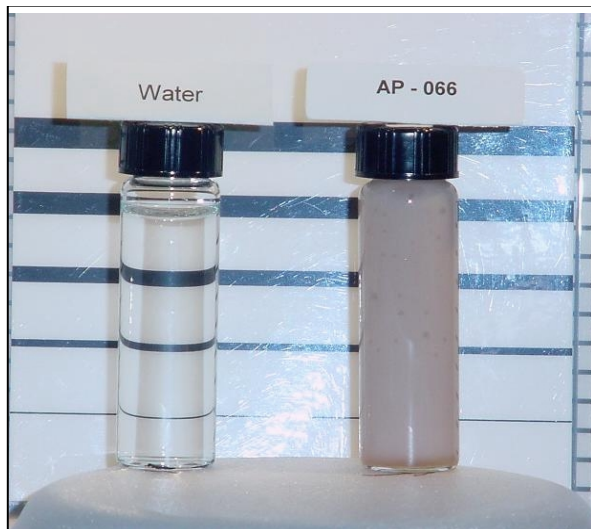
**Figure C-50. Apple Jelly Sample AP-061**



**Figure C-51. Apple Jelly Sample AP-062**



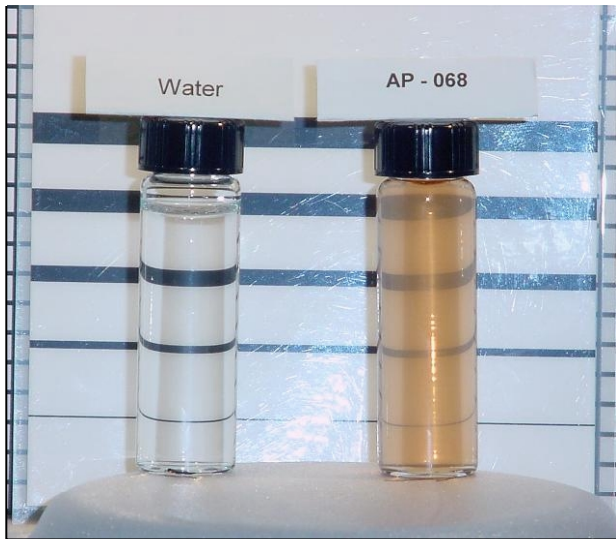
**Figure C-52. Apple Jelly Sample AP-065**



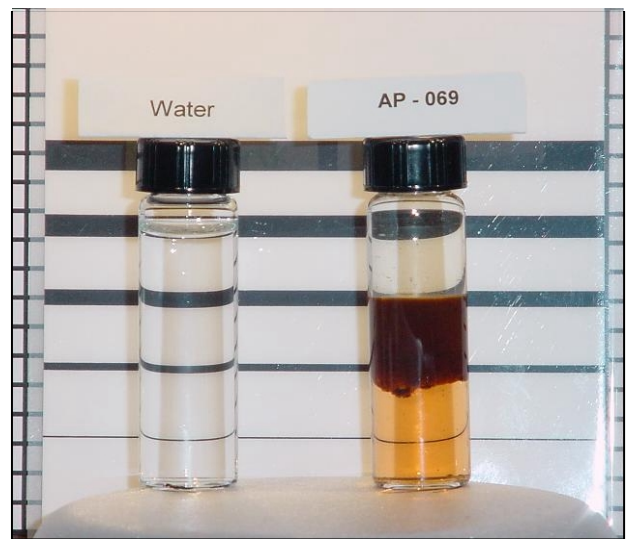
**Figure C-53. Apple Jelly Sample AP-066**



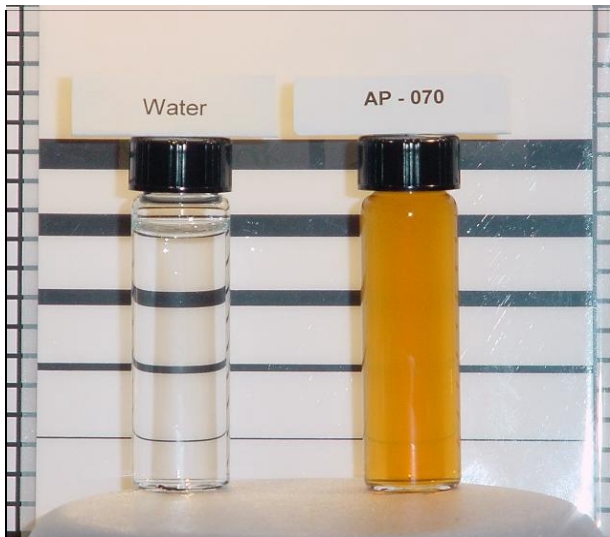
**Figure C-54. Apple Jelly Sample AP-067**



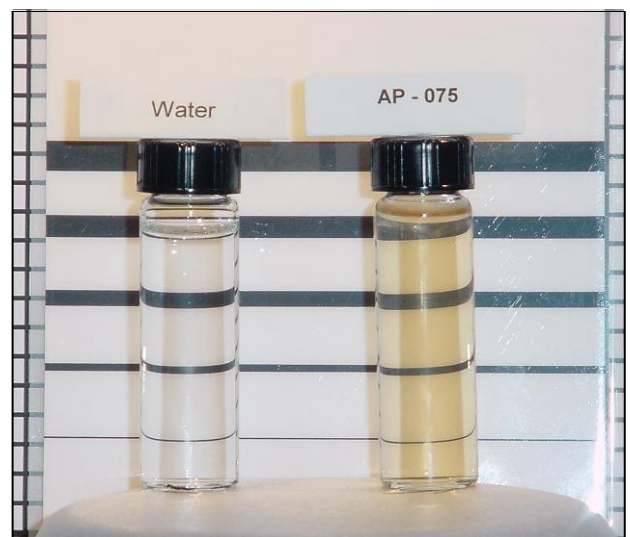
**Figure C-55. Apple Jelly Sample AP-068**



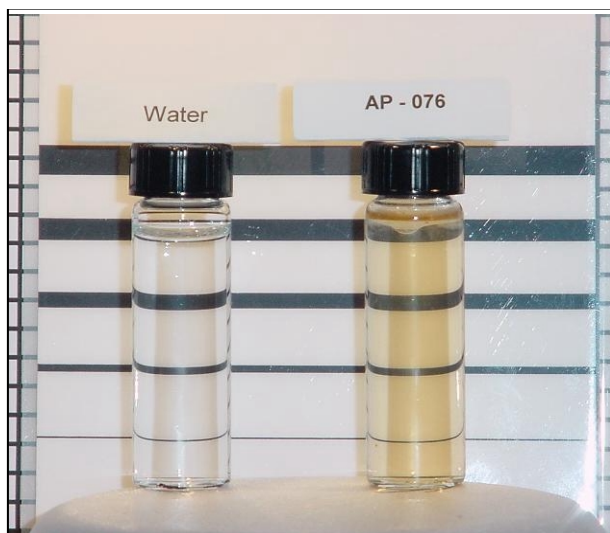
**Figure C-56. Apple Jelly Sample AP-069**



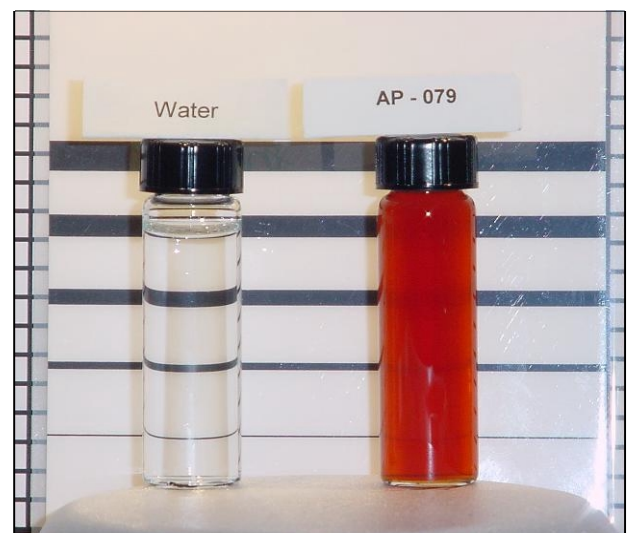
**Figure C-57. Apple Jelly Sample AP-070**



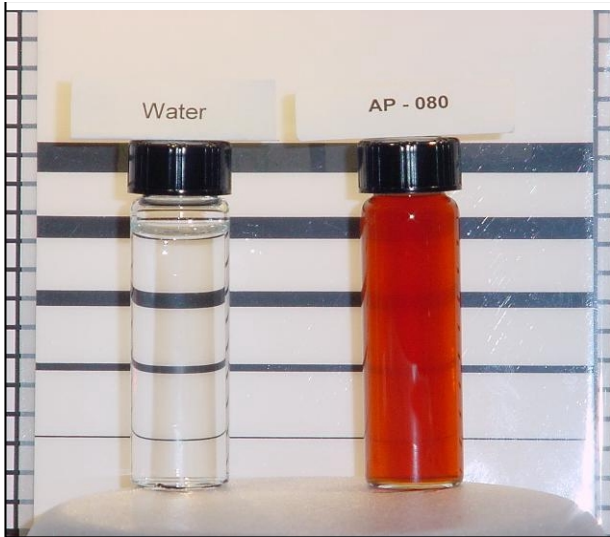
**Figure C-58. Apple Jelly Sample AP-075**



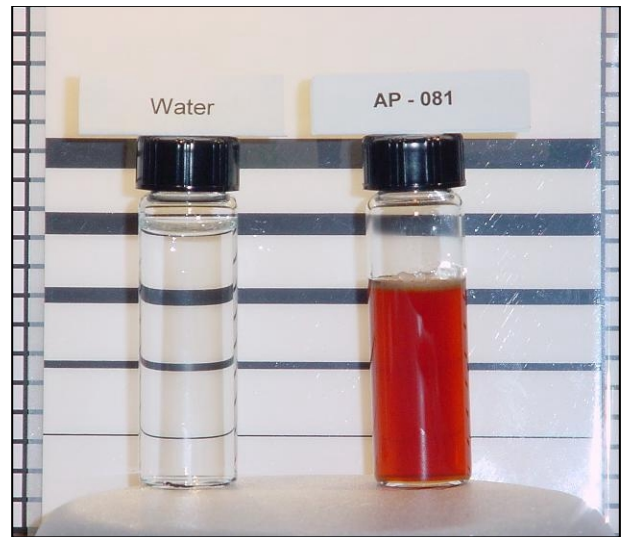
**Figure C-59. Apple Jelly Sample AP-076**



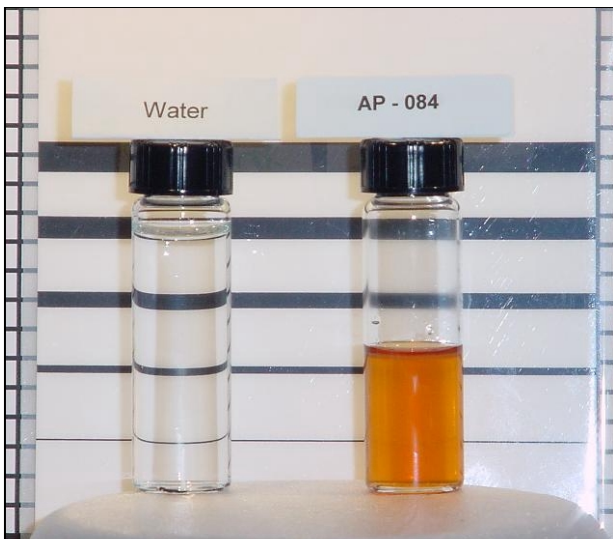
**Figure C-60. Apple Jelly Sample AP-079**



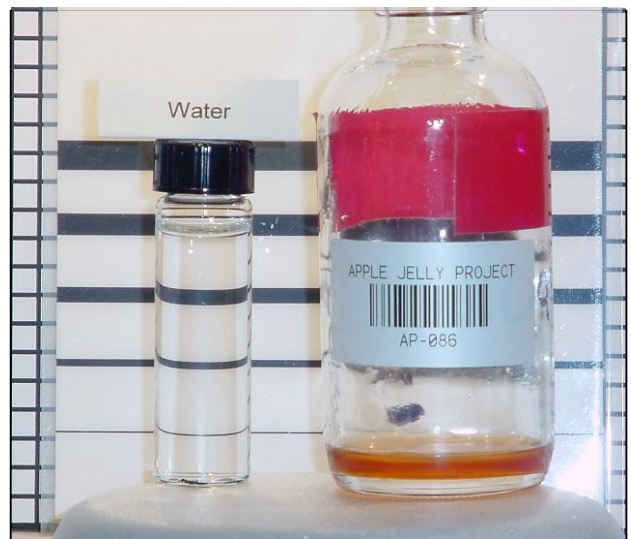
**Figure C-61. Apple Jelly Sample AP-080**



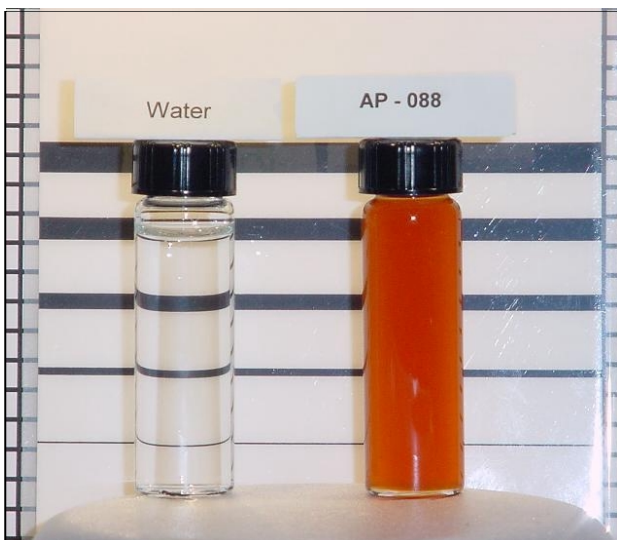
**Figure C-62. Apple Jelly Sample AP-081**



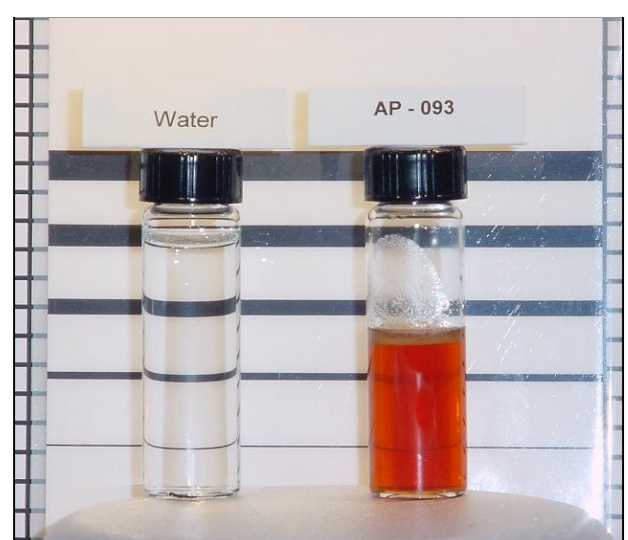
**Figure C-63. Apple Jelly Sample AP-084**



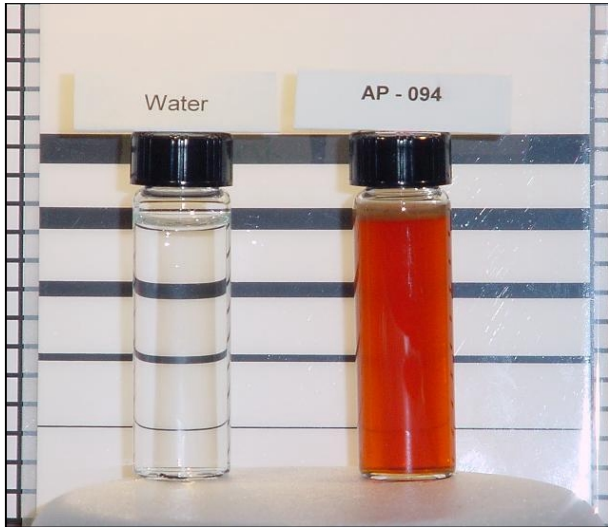
**Figure C-64. Apple Jelly Sample AP-086**



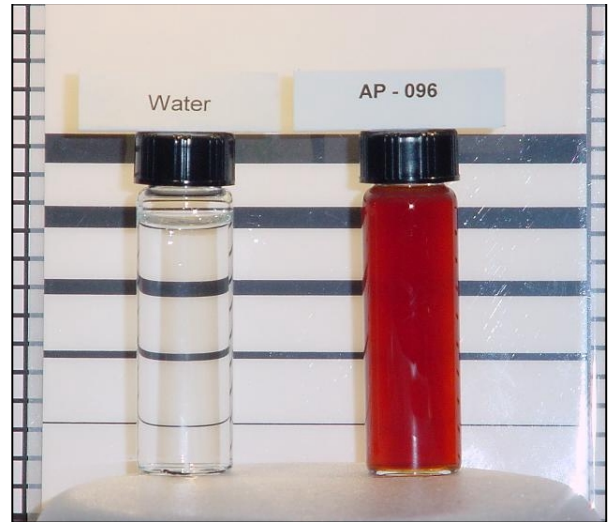
**Figure C-65. Apple Jelly Sample AP-088**



**Figure C-66. Apple Jelly Sample AP-093**



**Figure C-67. Apple Jelly Sample AP-094**



**Figure C-68. Apple Jelly Sample AP-096**



**Figure C-69. Apple Jelly Sample AP-102**

**APPENDIX D**  
**RESULTS OF COMPOSITIONAL ANALYSES**



Sample ID	Type of Sample (Fuel/Apple Jelly/DIME/other)	TAN used for plotting (mg KOH/g)	TAN D 664				TBN used for plotting (mg KOH/g)	TBN D 2896		FTIR	NITROGEN D4629 (PPM)	SULFUR D5453 (PPM)	Density (g/cm <sup>3</sup> )	Surface Tension (dyne/cm <sup>2</sup> )	Interfacial Tension D971 (dyne/cm <sup>2</sup> )	Conductivity @ 25C (µS/cm)	Color	% di-EGME (by R.I.)	KF Water, %
			Standard Method		Modified Method			Standard Method	Modified Method										
			Inflection End- Point (mg KOH/g)	Buffer End- Point (mg KOH/g)	Inflection End Point (mg KOH/g)	Buffer End- Point (mg KOH/g)		Inflection End-Point (mg KOH/g)	Inflection End-Point (mg KOH/g)										
AP-001	AJ	0.56	0.59	NA	0.71	NA	0.96	1.10 / 0.70	1.06		265	338	1.0455	32.44		1060	3	55.8	42.0
AP-001-F	Fuel Layer										20	325	0.8040	25.35					
AP-002	AJ	0.45	0.45 / 0.44	NA	0.41 / 0.41	NA	0.62	0.56	0.68		182	148	1.0405	33.88		909	3	48.0	47.0
AP-002-F	Fuel Layer										21	284	0.8067	26.10					
AP-003	Fuel										27	219	0.8191	26.61					
AP-004	Fuel										17	229	0.8191	26.25					
AP-005	Fuel										6	182	0.8093	22.15					
AP-006	AJ	5.35	did not dissolve	NA	5.35	NA	5.30	5.45	5.15		389	223	1.0456	38.30		1320	2	49.0	51.1
AP-006-F	Fuel Layer										18	217	0.8096	25.86					
AP-007	AJ	0.24	0.21	NA	0.27	NA	0.26	no TBN	0.26		70	70	1.0200	43.64		1320	1	24.3	72.7
AP-007-F	Fuel Layer										19	221	0.8100	25.32					
AP-008	AJ	1.29	1.14	NA	1.43	NA	0.77	0.73	0.80		345	280	1.0408	37.59			2	49.0	48.6
AP-008-F	Fuel Layer										28	219	0.8099	25.79					
AP-009	AJ		duplicate of 008					duplicate of 008			318	236	1.0408	31.03			2	49.0	52.7
AP-009-F	Fuel Layer										20	234	0.8098	20.47					
AP-010	AJ	0	no inflection	no TAN	no inflection	no TAN			no TBN		205	167	1.0411	37.28			3	48.6	49.7
AP-010-F	Fuel Layer										15	514	0.8011	25.20					
AP-011	AJ	0.35	0.38	NA	0.31	NA	0.54		0.54		167	167	1.0429	37.98		293	2	53.8	46.4
AP-011-F	Fuel Layer										18	58	0.8198	26.44					
AP-012	AJ	0.05	0.06	0.08	0.04	0.05	0		no TBN		28	17	1.0177	42.67			1	21.8	74.9
AP-013	Fuel										2.5	36	0.8196	23.81					
AP-014	AJ	0.77	0.75	NA	0.79	NA	0.7		0.70		354	173	1.0401	38.35		394	2	48.4	50.0
AP-014-F	Fuel Layer										16	228	0.7993	25.81					
AP-015	AJ		duplicate of 014					duplicate of 014			332	175	1.0401	39.28			2	48.0	51.9
AP-016	AJ	0.76	0.74	NA	0.77	NA	0.66		0.66		271	71	1.0402	35.49		394	2	48.0	50.2
AP-016-F	Fuel Layer										13	221	0.7995	25.42					
AP-017	AJ	0.8	0.81	NA	0.78	NA	0.64		0.64		287	144	1.0402	31.15		396	2	48.0	49.6
AP-017-F	Fuel Layer										15	234	0.7996	25.81					
AP-018	AJ	1.09	1.07	NA	1.11	NA	1.08		1.08		439	309	1.0407	34.18		592	2	48.0	51.4
AP-019	AJ		duplicate of 018					duplicate of 018					1.0406	36.37		585	2	48.0	50.4
AP-019-F	Fuel Layer										15	189	0.7998	25.66					
AP-020	AJ		duplicate of 018					duplicate of 018			371	441	1.0406	38.08			2	48.0	51.2
AP-020-F	Fuel Layer										19	195	0.7998	23.98					
AP-021	AJ	1.4	1.43	NA	1.36	NA	1.87		1.87		482	405	1.0429	36.71		994	2	49.9	48.1
AP-022	AJ		duplicate of 021					duplicate of 021					1.0428	37.32			2	49.9	50.9
AP-023	AJ		duplicate of 021					duplicate of 021			493	296	1.0427	34.01			2	49.9	50.8
AP-023-F	Fuel Layer										20	195	0.7994	21.13					
AP-024	Fuel										27	373	0.8110	26.13					
AP-025	Fuel		duplicate of 024					duplicate of 024			15	349	0.8110	24.00					
AP-026	Fuel		duplicate of 024					duplicate of 024			14	358	0.8110	22.27					
AP-027	Fuel		duplicate of 024					duplicate of 024			15	361	0.8110	26.08					
AP-028	AJ	0.58	0.55	NA	0.61	NA	0.38		0.38		193	214	1.0413	39.62		408	2	50.3	49.5
AP-028-F	Fuel Layer										24	293	0.8244	25.57					
AP-029	AJ		duplicate of 028					duplicate of 028					1.0409	39.52			2	48.8	48.7
AP-029-F	Fuel Layer										17	293	0.8181	25.88					
AP-030	AJ	0.72	0.71	NA	0.73	NA	0.39		0.39		350	144	1.0416	38.93		433	2	50.3	48.0
AP-030-F	Fuel Layer										15	312	0.8326	25.69					
AP-031	AJ	0.52	0.52	NA	0.51	NA	0.36		0.36		240	226	1.0406	34.45		454	2	48.8	50.2
AP-031-F	Fuel Layer										15	301	0.8184	26.00					
AP-032	AJ	29.67	did not dissolve	NA	29.67	NA	53.73		53.73		5476	353	insufficient sample			4700	3	91.1	32.2
AP-033	AJ	4.85	5.09	NA	4.6	NA	5.47		5.47		1721	1402	1.0440	35.30		4530	3	42.7	56.1
AP-034	AJ		duplicate of 033					duplicate of 033					1.0440	35.37			3	42.4	58.9
AP-035	AJ	0.15	0.13	NA	0.16	NA	0		no TBN		52	86	1.0199	45.84		415	1	23.6	73.5
AP-035-F	Fuel Layer										14	320	0.7945	23.27					
AP-036	Fuel										23	307	0.7945	25.35					

AP-037	AJ	1.67	1.68	NA	1.65	NA	0.76		0.76		489	235	1.0442	34.62		1490	2	54.2	46.2
AP-038	AJ	1.59	1.59	NA	1.59	NA	0.64		0.64		545	237	1.0446	34.76		1390	2	55.4	44.6
AP-038-F	Fuel Layer										17	772	0.8032	25.91					
AP-039	AJ	5.15	4.98	NA	5.31	NA	4.83		4.83		2251	705	1.0504	36.62		3300	3	53.4	47.3
AP-039-F	Fuel Layer										21	481	0.8037	25.91					
AP-040	AJ	0.57	0.58 / 0.55	NA	0.54 / 0.59	NA	1.12		1.12		124	117	1.0391	39.98		571	1	46.2	54.9
AP-040-F	Fuel Layer										26	674	0.8032	26.08					
AP-041	AJ		1.50	NA	1.03	NA	1.27		1.27		487	504	1.0325	39.13		1520	2	35.7	60.8
AP-041-F	Fuel Layer										21	514	0.8050	25.86					
AP-042	AJ	1.18	1.15	NA	1.20	NA	0.98		0.98		244	246	1.0429	37.54		489	3	52.6	48.5
AP-042-F	Fuel Layer										25	618	0.8060	23.57					
AP-043	AJ	14.98	did not dissolve	NA	14.86/15.1	NA	15.73		15.73		376	34	1.0224	40.37		7010	1	21.8	78.8
AP-044	AJ	60.08	did not dissolve	NA	60.08	NA	73.21		73.21		1711	372	too thick	41.45		6460	3	100.0	46.0
AP-044-F	Fuel Layer																		
AP-045	AJ	79.33	did not dissolve	NA	79.33	NA	51		51.00		1875	210	too thick	39.74			3	95.4	50.0
AP-046	Fuel										21	377	0.7979	25.05					
AP-047	Fuel										22	381	0.7980	25.79					
AP-048	AJ	3.06	2.86	NA	3.26	NA	0.84		0.84		750	439	1.0493	40.37		6140	2	49.1	51.7
AP-048-F	Fuel Layer																		
AP-049	AJ	0.61	0.59	NA	0.62	NA	0		no TBN		152	43	1.0401	40.18			2	48.4	50.9
AP-050	AJ		duplicate of 049						duplicate of 049				1.0402	40.32		683	2	48.0	50.3
AP-051	AJ		duplicate of 049						duplicate of 049				1.0401	40.42			2	48.0	50.6
AP-052	AJ	3.24	3.35	NA	3.13	NA	1.1		1.10		743	459	1.0492	39.13		6420	3	47.7	52.5
AP-052-F	Fuel Layer										31	388	0.7981	25.61					
AP-053	AJ		duplicate of 052						duplicate of 052				1.0493	38.10			3	47.7	51.2
AP-053-F	Fuel Layer												0.7984	25.86					
AP-054	AJ		duplicate of 052						duplicate of 052				1.0493	35.49			3	48.0	50.9
AP-054-F	Fuel Layer												0.7983	23.93					
AP-055	AJ	63.14	did not dissolve	NA	63.14	NA	55.34		55.34		2482	214	too thick	41.23			3	75.9	
AP-056	AJ	39.36	did not dissolve	NA	39.36	NA	39.26		39.26		1032	249	1.0902	35.45		7440		60.1	57.5
AP-057	AJ	2.85	3.39	NA	2.30	NA	1.7		1.70		87	53	1.0359	34.57				40.7	52.8
AP-058	AJ	0.46	0.49	NA	0.42	NA	0.61		0.61		146	126	1.0417	37.88			2	49.9	49.0
AP-058-F	Fuel Layer																		
AP-059	AJ		duplicate of 058						duplicate of 058				1.0415	38.30			2	49.9	52.8
AP-059-F	Fuel Layer												0.8034	26.27					
AP-060	AJ	1.75	1.39	NA	2.10	NA	2.39		2.39		125	180	1.0421	37.86		823	1	47.7	52.8
AP-061	AJ	0.23	0.15	NA	0.30	NA	0.52		0.52		11	3	1.0083	38.49			1	11.7	95.0
AP-062	AJ		insufficient sample						insufficient sample		305	454	fficient sample	ufficient sample			3	57.1	42.1
AP-063	Fuel												0.8075	ufficient sample					
AP-064	Fuel		duplicate of 063						duplicate of 063		19	449	0.8075	ufficient sample					
AP-065	AJ	0.49	0.55	NA	0.42	NA	1.94		1.94		79	154	1.0382	37.28		816	2	42.7	54.2
AP-065-F	Fuel Layer																		
AP-066	AJ		did not dissolve	NA	4.12	NA	4.73		4.73		459	116	0.8783	ND					75.9
AP-066-F	Fuel Layer										13	204	0.8036	24.47					
AP-067	AJ	2.36	0.80	NA	3.92	NA	3.94		3.94		138	69	1.0110	41.71			1	10.6	90.0
AP-068	AJ	0.15	0.14	NA	0.16	NA	0		no TBN		24	25	1.0384	40.10			1	45.9	53.6
AP-069	AJ		insufficient sample						insufficient sample		131	49	0.7995	25.83				143.7	55.5
AP-070	AJ	26.87	did not dissolve	NA	26.87	NA	29.00		29.00		607	448	1.0758	41.67		8060	2	50.3	60.0
AP-071	Fuel										14	675	0.8001	24.76					
AP-072	Fuel										14	670	0.7997	23.54					
AP-073	Stadis 450	24.14	24.14				47.41		47.41				-	-					
AP-074	Additive												-	-					
AP-075	AJ	0.24	0.25	NA	0.22	NA	0		no TBN		118	187	1.0239	42.76			1	26.8	71.8
AP-075-F	Fuel Layer										17	288	0.7937	25.30					
AP-076	AJ		duplicate of 075						duplicate of 075				1.0244	32.47			1	27.9	73.4
AP-076-F	Fuel Layer										15	261	0.7937	25.15					
AP-077	Additive												-	-					
AP-078	Fuel										28	1208	0.8240	26.71					
AP-079	AJ	20.83			20.83		18.9		18.9		1537	538	1.0629	39.47				52.6	54.0











Sample ID	Sample Type	Ag (ppm)	AL (ppm)	B (ppm)	Ba (ppm)	Ca (ppm)	Cd (ppm)	Cr (ppm)	Cu (ppm)	Fe (ppm)	K (ppm)	Mg (ppm)	Mn (ppm)	Mo (ppm)	Na (ppm)	Na x 25 (ppm)	Ni (ppm)	P (ppm)	Pb (ppm)	Si (ppm)	Sn (ppm)	Sb (ppm)	Sr (ppm)	Ti (ppm)	V (ppm)	Zn (ppm)	
AP-001	AJ		<1	<1		<1			<1	6	2	<1	<1	<1	63	1575	5	<1	<1	<1						<1	
AP-001-F	Fuel Layer	<1	<1	<1	<1	2	<1	<1	<1	<1	<1	1	<1	<1	5		<1	<1	<1	<1	<1	1	<1	<1	<1	1	
AP-002	AJ		<1	<1		<1			<1	<1	1	<1	<1	<1	21	525	<1	<1	<1	<1	<1	<1				<1	
AP-002-F	Fuel Layer	<1	<1	<1	<1	3	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	2	<1	<1	<1	<1	2	
AP-003	Fuel	<1	<1	1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	
AP-004	Fuel																										
AP-005	Fuel	<1	<1	2	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	1	<1	<1	<1	<1	<1	
AP-006	AJ		<1	<1		<1			<1	4		<1	<1	<1	32	800	<1	<1	<1	<1	<1					<1	
AP-006-F	Fuel Layer																										
AP-007	AJ		<1	<1		<1			<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1					<1	
AP-007-F	Fuel Layer																										
AP-008	AJ		2	1		<1			<1	<1	2	<1	<1	<1	9	225	<1	<1		<1	<1					<1	
AP-008-F	Fuel Layer																										
AP-009	AJ																										
AP-009-F	Fuel Layer																										
AP-010	AJ		<1	<1		<1			<1	<1	2	1	<1	<1	22	550	<1	<1		<1	<1					<1	
AP-010-F	Fuel Layer																										
AP-011	AJ		<1	<1		<1			<1	<1	1	<1	<1	<1	5	125	<1	<1		<1	<1					<1	
AP-011-F	Fuel Layer																										
AP-012	AJ		<1	<1		<1			<1	<1	<1	<1	<1	<1	1	25	<1	<1	<1	<1	<1						
AP-013	Fuel	3	<1	9	<1	<1	1	<1	<1	<1	<1	<1	1	<1	<1		<1	<1	<1	<1	1	<1	1	<1	<1	<1	
AP-014	AJ		<1	<1		<1			<1	<1	<1	<1	<1	<1	3	75	<1	<1	<1	<1	<1						
AP-014-F	Fuel Layer	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	
AP-015	AJ																										
AP-016	AJ		<1	<1		36			<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1					4	
AP-016-F	Fuel Layer	<1	<1	11	<1	<1	<1	<1	<1	<1		<1	<1	<1	5		<1	1	<1	<1	<1	<1	<1	<1	<1	<1	
AP-017	AJ									1	<1	<1			4	100		<1									
AP-017-F	Fuel Layer																										
AP-018	AJ		1	<1		<1			<1	<1	<1	<1	<1	<1	7	175	<1	<1	<1	<1	<1						
AP-019	AJ																										
AP-019-F	Fuel Layer																										
AP-020	AJ																										
AP-020-F	Fuel Layer																										
AP-021	AJ									<1	3	<1			24	600		<1									
AP-022	AJ																										
AP-023	AJ																										
AP-023-F	Fuel Layer																										
AP-024	Fuel	<1	<1	8	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	3	<1	<1	<1	<1	<1	
AP-025	Fuel																										
AP-026	Fuel																										
AP-027	Fuel																										
AP-028	AJ		1	<1		44			<1	1	1	1	<1	<1	6	150	<1	<1	<1	<1	<1					4	
AP-028-F	Fuel Layer	<1	<1	6	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	1	<1	<1	<1	<1	<1	<1	
AP-029	AJ																										
AP-029-F	Fuel Layer																										
AP-030	AJ									<1	<1	<1			4	100		<1									
AP-030-F	Fuel Layer																										
AP-031	AJ									<1	1	<1			6	150		<1									
AP-031-F	Fuel Layer																										
AP-032	AJ		1	<1		<5			<1	<1	5	<1	<1	<1	355	8875	<1	<1	<1	6	<1					<1	

AP-033	AJ		<1	1		<1			<1	<1	10	3	<1	<1	87	2175	<1	<1	<1	<1	<1					
AP-034	AJ																									
AP-035	AJ		<1		<1	<1			<1	<1	<1	<1	<1	<1	4	100	<1	<1	<1	<1	<1					<1
AP-035-F	Fuel Layer	<1	<1	3	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	1	<1	<1	<1	<1
AP-036	Fuel	<1	<1	7	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	1	<1	<1	<1	<1	<1
AP-037	AJ		<1		<1	<1			<1	<1	1	<1	<1	<1	22	550	<1	<1	<1	<1	<1					<1
AP-038	AJ		<1	<1		<1			<1	<1	1	<1	<1	<1	20	500	<1	<1		<1						<1
AP-038-F	Fuel Layer	<1	<1	4	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
AP-039	AJ		<1	<1		<1			<1	<1	3	<1	<1	<1	55	1375	<1	<1		<1						
AP-039-F	Fuel Layer	<1	<1	3	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
AP-040	AJ		1			<1			<1	<1	<1	<1	<1	<1	7	175	<1	<1		<1						<1
AP-040-F	Fuel Layer	<1	<1	1	<1	<1	<1	<1	<1	<1	<1	4	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
AP-041	AJ		<1	<1		<1			<1	<1	<1	<1	<1	<1	9	225	<1	<1		<1						<1
AP-041-F	Fuel Layer	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	3	<1	<1	<1	<1	<1	<1
AP-042	AJ		<1	<1		<1			<1	<1	1	<1	<1	<1	10	250	<1	<1	<1	<1						<1
AP-042-F	Fuel Layer																									
AP-043	AJ		<1	<1		<1			<1	1	<1	<1	<1	<1	181	4525	<1	<1	<1	<1	<1					<1
AP-044	AJ		<1	<1		<1			<1	4	1	<1	<1	<1	875	21875	<1	<1	<1	<1	<1					<1
AP-044-F	Fuel Layer	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	4	<1	<1	<1	<1	<1	1
AP-045	AJ		1	<1		<5			<1	6	3	<1	<1	<1	982	24550	<1	<1	<1	6	<1					<1
AP-046	Fuel	<1	<1	6	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	1	<1	<1	2	<1	<1	<1	<1	<1
AP-047	Fuel																									
AP-048	AJ		<1	<1		<1			<1	2	5	13	<1	<1	109	2725	<1	<1	<1	<1						<1
AP-048-F	Fuel Layer	<1	<1	<1	<1	<1	<1	<1	<1	1	<1	<1	<1	<1	8		<1	3	<1	<1	<1	<1	<1	<1	<1	<1
AP-049	AJ		<1	<1		<1			<1	<1	<1	1	<1	<1	12	300	<1	<1	<1	<1						<1
AP-050	AJ		<1	<1		<1			<1	<1	7	16	<1	<1	143	3575	<1	<1	<1	<1						<1
AP-051	AJ																									
AP-052	AJ																									
AP-052-F	Fuel Layer																									
AP-053	AJ																									
AP-053-F	Fuel Layer																									
AP-054	AJ																									
AP-054-F	Fuel Layer																									
AP-055	AJ		1	<1		<5			<1	<1	3	<1	<1	<1	413	10325	<1	6	<1	6	<1					<1
AP-056	AJ		<1	<1		<1			<1	<1	2		<1	<1	263	6575	<1	<1	<1	<1	<1					<1
AP-057	AJ		67	<1		9			<1	27	3		<1	<1	8	200	<1	<1	<1	<1	<1					<1
AP-058	AJ		1	<1		<1			<1	1	3		<1	<1	25	625	<1	<1	<1	<1	<1					<1
AP-058-F	Fuel Layer	<1	<1	19	<1	6	<1	<1	<1	1		1	<1	<1	12		<1	2	<1	<1	1	<1	<1	<1	<1	2
AP-059	AJ		1	<1		<1			<1	1	2		<1	<1	19	475	<1	<1	<1	<1	<1					<1
AP-059-F	Fuel Layer																									
AP-060	AJ		<1	1		<5			<1	55	<1	<1	1	2	29	725	10	<1	<1	<1						<1
AP-061	AJ		<1	<1		<5			<1	<1	<1	<1	<1	<1	9	225	<1	<1	<1	<1						<1
AP-062	AJ		<1	1		<5			<1	<1	15	3	<1	<1	56	1400	<1	<1	<1	<1						<1
AP-063	Fuel			7																						
AP-064	Fuel																									
AP-065	AJ		<1	1		<5			<1	<1	2	<1	<1	<1	36	900	<1	<1	<1	<1						<1
AP-065-F	Fuel Layer	<1	<1	5	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	1		<1	2	<1		<1	<1	<1	<1	<1	<1
AP-066	AJ		<1	<1		<5			<1	1	2	<1	<1	<1	124	3100	<1	<1	<1	<1						<1
AP-066-F	Fuel Layer																									
AP-067	AJ		<1	<1		<5			<1	<1	2	<1	<1	<1	62	1550	<1	<1	<1	<1						<1
AP-068	AJ		1	<1		<5			<1	<1	1	<1	<1	<1	3	75	<1	<1	<1	6	<1					<1
AP-069	AJ		<1	<1		<5			<1	<1	1	1	<1	<1	12	300	<1	<1	<1	5	<1					<1

AP-070	AJ																									
AP-071	Fuel	<1	<1	2	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1		<1	<1	<1	<1	<1	<1	<1	<1	<1	
AP-072	Fuel																									
AP-073	Additive																									
AP-074	Additive																									
AP-075	AJ									<1	3		<1		12	300				<1						
AP-075-F	Fuel Layer																									
AP-076	AJ																									
AP-076-F	Fuel Layer																									
AP-077	Additive																									
AP-078	Fuel																									
AP-079	AJ									<1	2		<1		237	5925				<1						
AP-080	AJ									1	2	<1			234	5850				<1						
AP-081	AJ									<1	1	<1			295	7375				<1						
AP-082	AJ									2	3		<1		917	22925										
AP-082-F	Fuel Layer																									
AP-083	AJ																									
AP-083-F	Fuel Layer																									
AP-084	AJ																									
AP-085	Fuel																									
AP-086	AJ																									
AP-087	Fuel																									
AP-088	AJ									<1	4	<1			717	17925				<1						
AP-089	Fuel																									
AP-090	AJ																									
AP-091	Fuel																									
AP-092	Fuel																									
AP-093	AJ																									
AP-093-F	Fuel Layer																									
AP-094	AJ									<1	3	<1			232	5800				<1						
AP-094-F	Fuel Layer																									
AP-095	AJ																									
AP-095-F	Fuel Layer																									
AP-096	AJ									<1	2	<1			336	8400				<1						
AP-097	AJ																									
AP-097-F	Fuel Layer																									
AP-098	AJ									<1	2	<1			389	9725				<1						
AP-098-F	Fuel Layer																									
AP-099	AJ																									
AP-099-F	Fuel Layer																									
AP-100	Filter Media																									
AP-101	Filter Media																									
AP-102	AJ																									
AP-103	AJ									<1	1	<1			12	300				<1						
AP-103-F	Fuel Layer																									
AP-104	Filter Media																									
AP-105	Fuel																									
AP-106	Fuel																									
AP-107	Fuel																									
AP-108	unknown									2	4		<1		813	20325				1						
AP-109	unknown																									
AP-110	unknown									2	6		<1		641	16025				1						





% DIEGME in H <sub>2</sub> O	Viscosity @ 25°C, cP
0	1.08
10	1.38
20	1.83
30	2.40
40	3.17
50	4.19
60	5.04
70	5.61
80	5.62
90	4.92
100	3.56

TGA

SAMPLE ID	FILE #	TEMP AT WATER CUT OFF	TGA							
			Residue	Water	DIEGME	#3	#4	#5	#6	#7
AP-002	0.065	100	0.542	16.35	83.1					
AP-002	0.067	100		13.26	86.16					
AP-002	0.13		0.3279	53.65	45.88	0.1322	0.002175	0.0523	0.01692	0.03977
AP-006	0.132		0.4092	55.44	42.84	0.1143	0.1061	0.1774	0.07103	0.8342
AP-032	0.133		5.772	10.34	8.676	8.345	1.918	1.868	1.587	2.375
AP-035	0.135		5.772	43.44	13.67	18.83	2.74	4.739	3.686	7.138
AP-043	0.134		1.455	73.88	21.14	0.2995	0.1204	0.4741	0.107	2.5
AP-044	0.136		9.124	38.55	11.46	18.18		7.509	5.345	9.816
AP-045	0.137		8.155	34.58	17.48	20.34		5.855	4.312	9.233
AP-055	0.138		6.496	38.43	25.63	14.44		3.854		2.434
AP-056	0.139		4.182	60.46	23.94	0.8332	0.04828	1.598	0.4499	7.196
AP-060	0.141		0.5507	49.93	49.03	0.03644	0.01498	0.09813	0.04241	0.2866
AP-067	0.142		0.6513	93.73	4.681	0.08032	0.03281	0.1539	0.04896	0.6169
AP-070	0.143		3.976	60.34	19.69	7.87	0.351	2.772	1.798	3.193
AP-080	0.146		7.43	22.88	24.58	27.95		5.622	4.067	7.446
AP-088										
AP-108	0.131		9.189	41.79	12.88	11.64	4.926	7.215	4.801	7.527
AP-110	0.129		9.145	27.71	22.09	9.147	1.934	14.13	6.09	9.606
AP-110 WAX (From Acid Digestion)	0.145		5.96	53.95	5.589	2.444	7.274		24.75	
AP-117										
STATIS 450 AP-073	0.167		2.683	52.91	6.625	7.917		26.76		3.067
1.3 g STADIS 450/6.0g NALCO 5403	0.288	100	44.75	21.76	33.49					
1.3 g STADIS 450/6.0g NALCO 5403	0.289		0.939	51.13		2.203	1.894	28.61		15.21
NALCO 5403 AP-077	0.168		0.6372	50.54		1.436	5.992	26.88		14.5
SYNETHIC APPLE JELLY	0.29	100	0.921881	53.58	45.5					
JAW-1-56-1	0.258	100	0.06213	10.39	89.55					
JAW-1-52-2	0.257		31.74	32.02		11.72	6.871	5.822	11.8	
JAW-1-60-1B	0.261	100	0.2247	11.69	88.06					
JAW-1-61-1B	0.262	100	1.478	11.32	87.2					
JAW-1-61-2B	0.264	100	2.342	18.14	79.5					
JAW-1-62-1T	0.265	100	3.952	26.72	69.3					
JAW-1-62-2T	0.266	100	5.563	18.22	76.2					
JAW-1-73-1	0.286	100	1.337	23.03	75.62					
JAW-1-73-2	0.287	100	27.33	69.35	69.35					

SAMPLE ID	TEMP AT				
	FILE #	OFF	WATER	DIEGME	RESIDUE
<b>100% WATER</b>	<b>0.056</b>	<b>100</b>	<b>99.82</b>	<b>0</b>	0.18
10% DIEGME/90% WATER	0.055	160	87.19	12.56	0.25
20% DIEGME/80% WATER	0.06	200	80.57	19.31	0.12
30% DIEGME/70% WATER	0.063	184	72.54	27.27	0.19
40% DIEGME/60% WATER	0.078	100	8.54	91.21	0.25
50% DIEGME/50% WATER	0.061	158	50.61	49.62	0
50% DIEGME/50% WATER	0.062	100	10.74	89.09	0.17
70% DIEGME/30% WATER	0.077	100	11.52	88.26	0.22
80% DIEGME/20% WATER	0.076	100	10.4	89.38	0.22
100% DIEGME	0.053	100	5.764	94.17	0.066
100% DIEGME	0.064	100	5.4034	94.4966	0.1