

TECHNICAL REPORT
NATICK/TR-14/008



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ANALYSIS OF TWO METHODS FOR CHARACTERIZATION OF FLAME RESISTANT MILITARY FABRICS AND COMMERCIAL TEXTILE FIBERS: SIMULTANEOUS DSC-TGA AND PYROLYSIS GCMS

by
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April 2014

Final Report
June 2011 – November 2012

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**U.S. Army Natick Soldier Research, Development and Engineering Center
Natick, Massachusetts 01760-5020**

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1. REPORT DATE (DD-MM-YYYY) 04-04-2014		2. REPORT TYPE Final		3. DATES COVERED (From - To) June 2011-November 2012		
4. TITLE AND SUBTITLE ANALYSIS OF TWO METHODS FOR CHARACTERIZATION OF FLAME RESISTANT MILITARY FABRICS AND COMMERCIAL TEXTILE FIBERS: SIMULTANEOUS DSC-TGA AND PYROLYSIS GCMS				5a. CONTRACT NUMBER		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER AH98		
6. AUTHOR(S) Pearl W. Yip				5d. PROJECT NUMBER		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER 25091T; WBS 5821.3.13		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Natick Soldier Research, Development and Engineering Center ATTN: RDNS-WSC-M Kansas St., Natick, MA 01760-5020				8. PERFORMING ORGANIZATION REPORT NUMBER		
				NATICK/TR-14/008		
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)		
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT This report documents an effort to examine the usefulness of two methods for characterization of flammability and thermal stability of flame retardant (FR) polymeric fibers and textiles to support the transition of new FR polymers under development for military clothing and individual equipment and fabric shelters: (1) simultaneous differential scanning calorimetry (DSC) - thermal gravimetric analysis (TGA) and (2) pyrolysis gas chromatography (GC) - mass spectrometry (MS). A group of FR military fabrics, woven with different types of polymeric fibers to achieve fire protection as well as various other military requirements, were examined. Simultaneous DSC-TGA (SDT) enables study of thermal behaviors of these textiles/fibers beyond their decomposition temperature. Pyrolysis provides a means to break up the polymers into smaller, volatile fragments, which can subsequently be analyzed by GCMS. Individual industrial textile fibers were examined separately as well as in woven fabrics. Both SDT and pyrolysis GCMS were found to be valuable for characterizing military FR fibers and textiles and provide useful insights into FR military fabrics. Pyrolysis GCMS was able to isolate the FR chemical in rayon and cotton, at 200 °C, creating the potential to compare washed and unwashed materials and determine degradation from washing. SDT identified the degradation pathways in the materials.						
15. SUBJECT TERMS GC/MS POLYMERS POLYMER FIBERS PROTECTIVE CLOTHING FIBERS PYROLYSIS FIRE PROTECTION MASS SPECTROMETRY DSC/TGA FLAMMABILITY FLAME RETARDANT THERMAL PROPERTIES FABRICS IDENTIFICATION THERMAL STABILITY GAS CHROMATOGRAPHY WEAVING DECOMPOSITION TEMPERATURE QUALITATIVE ANALYSIS WOVEN FABRICS DIFFERENTIAL SCANNING CALORIMETRY THERMOGRAVIMETRIC ANALYSIS DSC(DIFFERENTIAL SCANNING CALORIMETRY) TGA(THERMOGRAVIMETRIC ANALYSIS)						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON	
a. REPORT	b. ABSTRACT	c. THIS PAGE			Pearl W. Yip	
U	U	U	UU	28	19b. TELEPHONE NUMBER (include area code) 508-233-4449	

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Preface

This report documents an effort to characterize a group of military flame resistant (FR) fabrics, woven with different types of polymeric fibers to achieve self-extinguishing properties, as well as various other military requirements using simultaneous differential scanning calorimetry (DSC) - thermal gravimetric analysis (TGA) and pyrolysis gas chromatography (GC) - Mass Spectrometry (MS). This work was performed by the Natick Soldier Research, Development and Engineering Center (NSRDEC) between June 2011 and November 2012, under Program Element Number AH98 and Work Unit Numbers 25091T and WBS 5821.3.13. Simultaneous DSC-TGA enables study of the thermal behaviors of textiles and fibers beyond their decomposition temperatures. Pyrolysis provides a means to break up the polymers into smaller, volatile fragments, which can subsequently be analyzed by GCMS. The ultimate goal of this work was to develop additional characterization methods to study flammability and thermal stability of FR polymeric fibers and textiles to support the transition of new FR polymers under development for military clothing and individual equipment and fabric shelters.

The author would like to thank Ms. Celia Powell for the materials, technical knowledge, and industrial contacts; without her assistance this project will not be possible. The author would also like to thank Dr. Phillip Gibson for his technical guidance.

ANALYSIS OF TWO METHODS FOR CHARACTERIZATION OF FLAME RESISTANT MILITARY FABRICS AND COMMERCIAL TEXTILE FIBERS: SIMULTANEOUS DSC-TGA AND PYROLYSIS GCMS

1 Introduction

Polymers are potent sources of fuel for a fire. The fire hazard of polymeric materials has long been a great concern due to their inherent flammability. It is known that most apparel (natural or man-made) fabrics will ignite and continue to burn after the source of ignition is removed. Synthetic polymers are often more flammable and release more heat than the polymers found in nature [1]. At the same time, polymeric materials are becoming stronger, tougher, and lighter. Their usages are increasing in all aspects of everyday life, including military clothing and shelters. Thus, higher demands are put on the industry to improve the flame-resistant (FR) properties of high performance polymer fibers and military textiles. Due to the increased use of incendiary weapons such as explosives and improvised explosive devices (IEDs), burn injuries on the battlefield have been more prevalent for military personnel in recent years [2]. Relevant testing parameters are necessary to relate experimental measurements to real-world military situations for protective clothing and fabric shelters. Part of the Army's effort to support FR materials research is to develop additional characterization methods to study the flammability and thermal stability of current and new FR polymers under development for clothing and individual equipment for soldiers.

This report documents an effort that examined two methods for their usefulness in characterizing FR military fabrics: (1) simultaneous differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) (SDC), and (2) pyrolysis gas chromatography (GC) and mass spectrometry (MS). A group of fielded military fabrics and their component fibers were tested using each method. This work was performed by the Natick Soldier Research, Development and Engineering Center (NSRDEC) between June 2011 and November 2012. The ultimate goal of this work was to develop additional characterization methods to study flammability and thermal stability of FR polymeric fibers and textiles to support the transition of new FR polymers under development for clothing and individual equipment.

Over several decades, many test methods were developed for evaluating the flammability and thermal stability of FR polymers. It was elucidated by previous researchers that "the flammability properties of a particular FR polymer are not inherent to the polymer; they are dependent on the test method and test condition" [3,4,5]. Thus when one FR polymer is claimed to be more thermally stable than another by a specific test measurement, the information or question the test method is addressing must be understood. How is thermal stability defined in the context of the test? Does it mean the polymer would decompose at a higher temperature? Does the FR polymer have less weight loss or higher residual weight? Would the flame be extinguished easily by the material? Or does this material have less total heat released? Since it is difficult to simulate a test that would replicate a real fire condition, whether a material is "FR" could vastly be in the eye of the evaluator and in the context of the test used.

It was reported that the flame-retardant additives for cellulose fibers (such as rayon, the majority fiber component in the Flame Resistant Army Combat Uniform (FRACU), and cotton, the fiber that is 50% of the ACU) often function by suppressing levoglucosan formation in those fibers. Levoglucosan, 1,6-anhydro- β -D-glucopyranose, a 6-carbon ring structure (Figure 1), is the major pyrolysis product (pyrolysate) of cellulose materials. It is the precursor of smaller flammable volatile fragments which contribute to cellulose combustion [6,7,8,9,10].

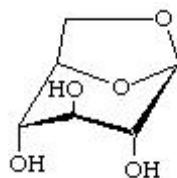


Figure 1. Chemical Structure of Levoglucosan.

FR rayon contains a phosphorus-based additive that is mixed in the viscous wood pulp solution (viscose dope) prior to the spinning process. The most successful commercial FR additive for rayon was reported to be based on an alkyl dioxaphosphorinane disulfide (Figure 2) [7]. It is believed that this branched compound with steric ring structure helps it stay durable in the rayon fiber after multiple launderings [8].

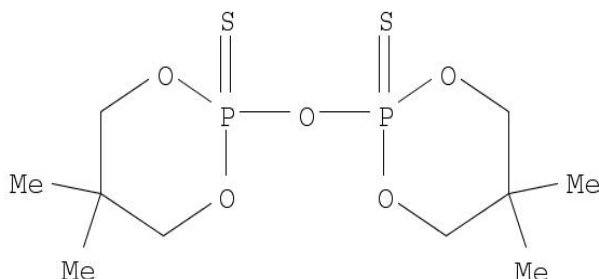


Figure 2. Dioxaphosphorinane Disulfide, Additive in Viscose Spinning Mixture for FR-Rayon.

1.1 Thermal Analysis – Simultaneous DSC-TGA (SDT)

DSC and TGA are two valuable and routinely used methods to study the thermal properties and stability of polymers. The measurements can be carried out in controlled conditions such as nitrogen or air environments, maximum temperature, and heating rate. The benefit of performing the DSC and TGA measurements simultaneously on the same sample, exposed to identical thermal treatment and environment, is that it simplifies the data interpretation. Additionally, it allows association of the heat flow events directly with the weight loss signal without having to worry about sample variation or instrumental differences. Another benefit of the SDT method is that the equipment was designed with a higher purge gas flow rate and a direct escape path for flushing out the pyrolysates. Such equipment design allows heat flow measurement up to and beyond the decomposition temperature of the sample without the concerns of contamination of the sample cell as in regular DSC equipment.

TGA and DSC are similar to other thermal tests, and while not representative of a real fire scenario, they allow a reproducible measurement of thermal properties of the polymers and a way to rate the materials quantitatively. Doing the TGA and DSC measurements together gives

a clear indication of the decomposition temperature of the polymer. A polymer which decomposes at a higher temperature is generally expected to have better FR effectiveness [4]. From all cases of measurements in literature, flame-retardant-treated rayon and cotton fibers have been found to have a lower thermal degradation temperature than the untreated fibers, but they also have a higher residual weight or less volatile matter evolved when heated [6].

1.2 Pyrolysis GCMS

Analytical pyrolysis is a useful tool for identifying polymeric materials which contain macromolecules that are too large to go through the GC column. During pyrolysis, the applied heat provides enough thermal energy to break the polymer into smaller fragments, and the volatile fragments can then be fed through and separated by the GC column [9,11]. The pyrolysis products are subsequently identified by the MS detector. The thermal degradation of a material during pyrolysis is caused by the dissociation of chemical bonds and the formation of free radicals. Depending on where the bond dissociation occurs, the fragments could be a mixture of oligomers (fragments with a few monomer units), monomers, and side groups. The free radical dissociation process usually starts by breaking the weakest bonds first. Because the thermal degradation mechanism of a polymer is largely an intramolecular event, a pyrolysis chromatogram of a fiber composed of two polymers would resemble the superposition of the chromatogram of the individual polymers [11]. Pyrolysis GCMS has been routinely used to study textile, polymers, and high performance fibers such as meta-aramid (Nomex[®]) and para-aramid (Kevlar[®], Twaron[®]) for decades. It is reported that pyrolysis measurements done at different temperatures would produce different pyrolysates or pyrolysates with different proportional amounts [12,13,14,15].

2 Materials

The military textiles tested were the regular nylon/cotton (NYCO) ACU, the FRACU, and Nomex® IIIa, and 100% Nomex® fabrics. Their constituent fibers are listed in Table 1, and the sources of those fibers are listed in Table 2.

Table 1. Military Fabrics with Fiber Content.

Fabric Type	Military Use	Cotton	Nylon	FR-Rayon	Para-Aramid	Meta-Aramid	P-140*
NYCO (generic)	ACU	50%	50%	-	-	-	-
Defender M	FRACU	-	10%	65%	25%	-	-
Nomex® IIIA	A2CU	-	-	-	5%	93%	2%
Indura®	Navy	100%**					

*Antistatic fiber

** Finished in fabric form with Proban® flame retardant (tetrakis-hydroxyphosphonium chloride)

Table 2. Individual Fibers and Fabrics Studied.

Fiber Type	Fiber/Fabric Brand	Source
Cotton	Not Applicable	Pharr Yarns, LLC
Nylon 6,6 (Invista)	Not Applicable	Pharr Yarns, LLC
FR-Rayon	Lenzing FR®	Lenzing AG
Untreated Rayon (Modal)	Lenzing Modal®	Lenzing AG
Para-Aramid	Kevlar®	E. I. DuPont de Nemours & Co.
Meta-Aramid	Nomex®	E. I. DuPont de Nemours & Co.
Para-Aramid	Twaron®	Teijin Limited
Polybenzimidazole	PBI®	PBI® Performance Products, Inc.

3 Methods

3.1 Thermal Analysis - SDT

The SDT instrument used in this experiment was manufactured by TA Instruments, Model Q600. The Q600 provides both the weight change and differential heat flow simultaneously on the same sample. The measurements were done mostly in nitrogen atmosphere from ambient to 600 °C. Some measurements were carried out up to 1000 °C, and some were done in air. The heating rate is 10 °C per min.

3.2 Pyrolysis GCMS

The pyrolysis component of the pyrolysis GCMS measurements was carried out with a CDS 5200 pyrolyzer, which is coupled to an Agilent GCMS instrument (GC Model 7890A, MS Model 5975C). A small amount of sample, usually less than 10 µg, was placed inside a quartz capillary tube, which was then inserted in the platinum coil in the pyroprobe. The platinum filament can be heated rapidly to a temperature high enough to pyrolyze the polymeric materials into volatile monomers or smaller fragments. The volatile pyrolysates (products of pyrolysis) were transferred to the gas chromatograph to be separated and eventually identified by the mass spectrometer. The pyrolysis was done under helium carrier gas at a flow rate of 54 mL/min. The pyrolysis temperature was set at 750 °C, and the interface temperature was set at 300 °C. The GC column was a DB-5ms low bleed, 5%-phenyl-methyl polysiloxane column (30 m x 0.25 mm x 0.25 µm), which is a general purpose column for GC. The GC temperature was initially held at 40 °C for 2 min, was programmed to 300 °C at 10 °C/min ramp, and then was held at 300 °C for 10 min. The mass spectra were measured using electron impact ionization energy of 70eV. The mass detector was scanned from 35 to 400 m/z at a rate of 4 scans per s. The data were searched using the NIST 2008 MS Library and the CDS's pyrolysis library.

4 Results and Discussion

4.1 Thermal Analysis –SDT

The measurements from the Q600 are usually very reproducible. Figures 3 and 4 show triplicate runs of NYCO and FRACU fabrics, respectively, conducted during this study that demonstrate the expected reproducibility.

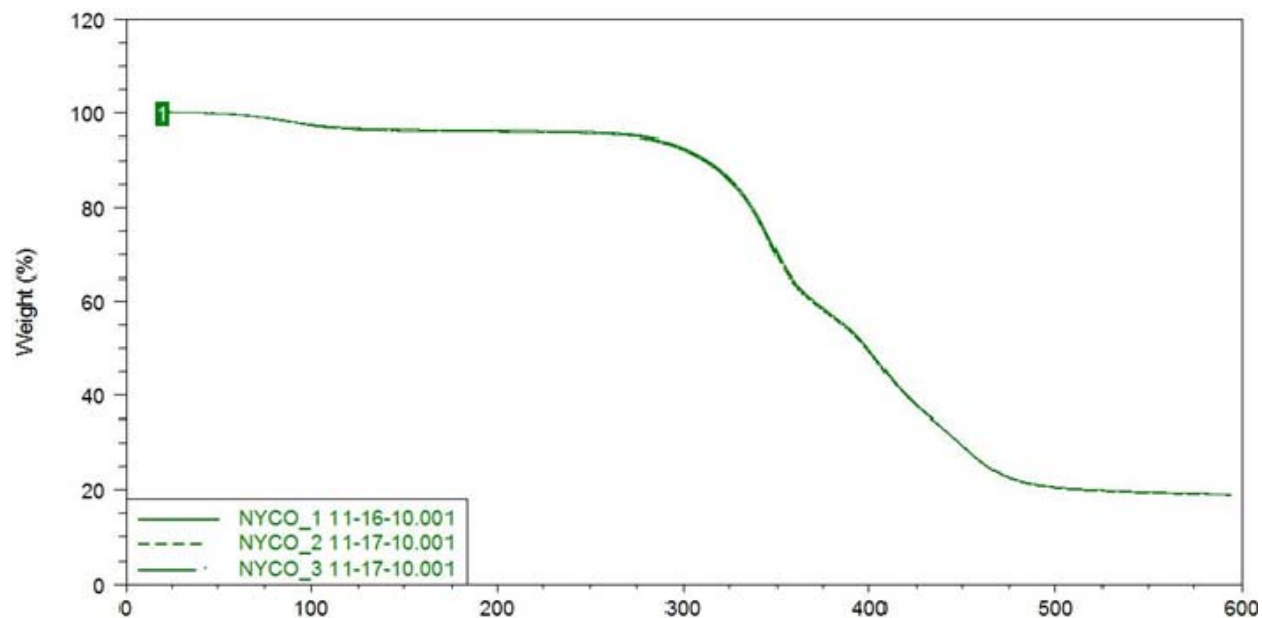


Figure 3. Triplicate SDT Runs of NYCO Fabric in Nitrogen Atmosphere from Ambient to 600 °C.

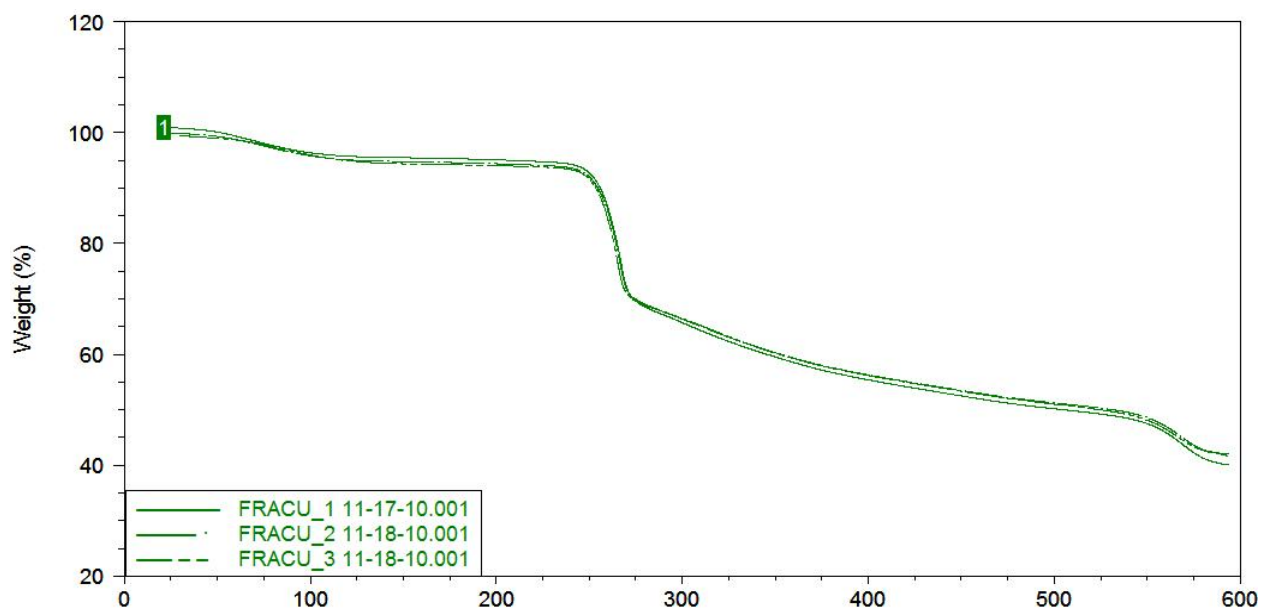


Figure 4. Triplicate SDT Runs of FRACU Fabric in Nitrogen Atmosphere from Ambient to 600 °C.

The thermal behavior of the four military textiles was measured with the Q600 in both nitrogen and air atmosphere, from room temperature to 1000 °C. The total weight loss percent and transition temperatures for both TGA and DSC are presented in Table 3. The TGA transition temperatures were taken from the peak maxima of the derivative weight curves where the samples exhibit the fastest weight loss. The DSC transition temperatures were taken at maxima or the minima on the heat flow curve. These temperatures indicate that some sort of physical change occurred in the sample while heated, such as glass transition, crystallization, melting, or decomposition. Table 4 contains the DSC and TGA measurements from the individual commercial fibers. Only the 600 °C measurements in both nitrogen and air environments are presented here. Different types of Kevlar® and Nomex® fibers, as well as Twaron®, raw cotton, and untreated rayon fibers, are also included in the comparison data. The thermal behavior of the various Kevlar® fibers was very similar. There was also no distinguishable difference between the Kevlar® and Twaron® fibers in the SDT results; both were para-aramid fibers processed differently from two different manufacturers. The graphical comparison of the SDT curves for these fibers in nitrogen are shown by weight and derivative weights in Figures 5 and 6, respectively, and the comparison SDT results in air are shown by weight and derivative weights in Figures 7 and 8, respectively.

Table 3. DSC and TGA Weight Loss of Military Fabrics in Nitrogen and in Air at Temperatures up to 1000 °C.

Fabric Sample	Weight Loss	Transition Temperatures (TGA)	Transition Temperatures (DSC)
Nitrogen			
NYCO	84.5%	365, 428 °C	252, 402, 490 °C
FRACU	70.5%	276, 591 °C	232, 277, 593 °C
Nomex® IIIa	52.4%	466, 587, 755 °C	425, 481, 722 °C
Nomex® 100%	52.4%	315, 462, 558, 752 °C	429, 479, 721 °C
Air			
NYCO	100%	338, 449, 557 °C	343, 449, 557 °C
FRACU	98.1%	275, 558, 762 °C	344, 578, 763 °C
Nomex® IIIa	99.3%	436, 595, 695 °C	441, 586, 595 °C
Nomex® 100%	100%	208, 438, 596, 680 °C	443, 589, 680 °C

Table 4. DSC and TGA Data of Individual Fibers in Nitrogen and in Air at Temperatures up to 600 °C.

Fiber Sample	Total Weight Loss	Temp of Max Derivative Wt. (%/°C)	Onset Decomp. Temperature (TGA peak)	Decomposition Temperature (DSC peak)
Nitrogen				
Nylon 6,6	98%	436 °C	413 °C	439 °C
Raw Cotton	82%	346 °C	314 °C	357 °C
FR-Cotton	60%	306 °C	292 °C	308 °C
Untreated Rayon	83%	337 °C	311 °C	337 °C
FR-Rayon	68%	259 °C	254 °C	261, 267 °C
Kevlar®	57%	577 °C	562 °C	579 °C
Kevlar® T970	60%	573 °C	557 °C	576 °C
Twaron®	63%	575 °C	562 °C	577 °C
Nomex®	43%	471 °C	432 °C	427 °C
PBI®	30%	475 °C	424 °C	477 °C
Air				
Nylon 6,6	98 %	452 °C	420 °C	439 °C
FR-Cotton	82 %	299 °C	285 °C	334 °C
Untreated Rayon	100%	324 °C	297 °C	358, 495 °C
FR-Rayon	87%	262 °C	254 °C	262, 296 °C
Kevlar®	44%	569 °C	526 °C	549 °C
Nomex®	47%	431 °C	410 °C	435 °C
PBI®	71%	582 °C	437 °C	566 °C

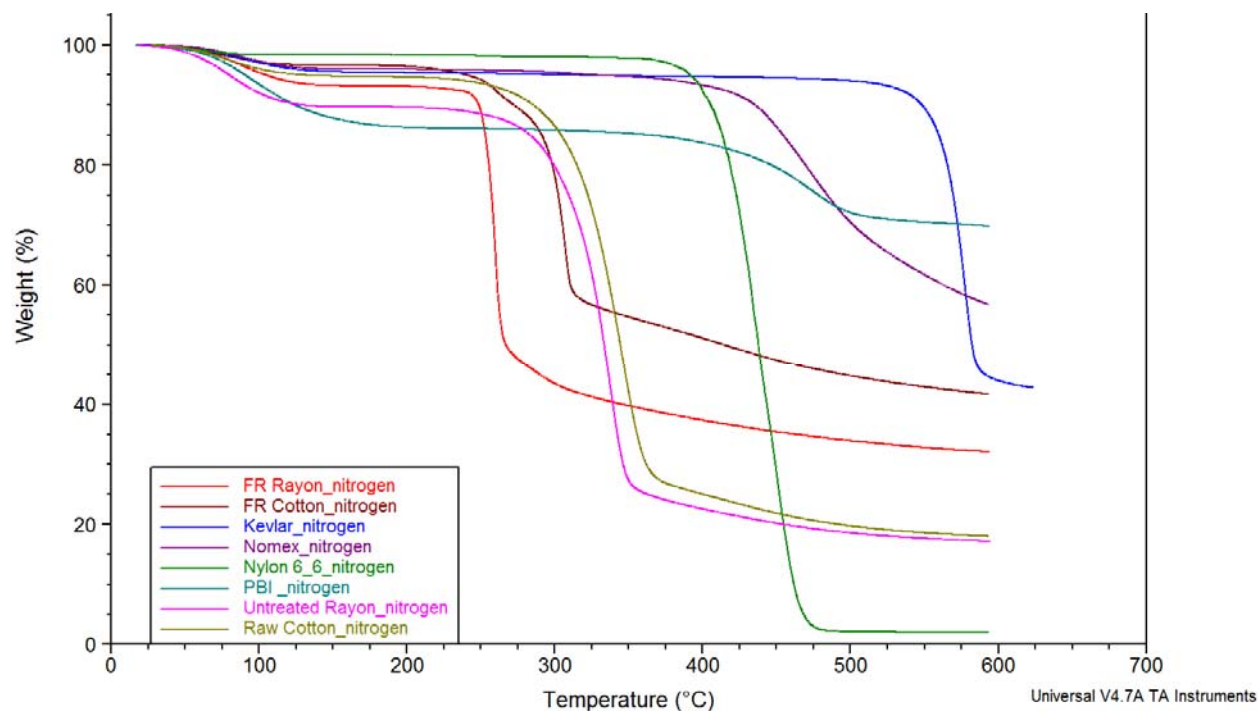


Figure 5. SDT Comparison of Thermal Stability of the Different Component Fibers Using Weight Verses Temperature Curves in Nitrogen from Room Temperature to 600 °C.

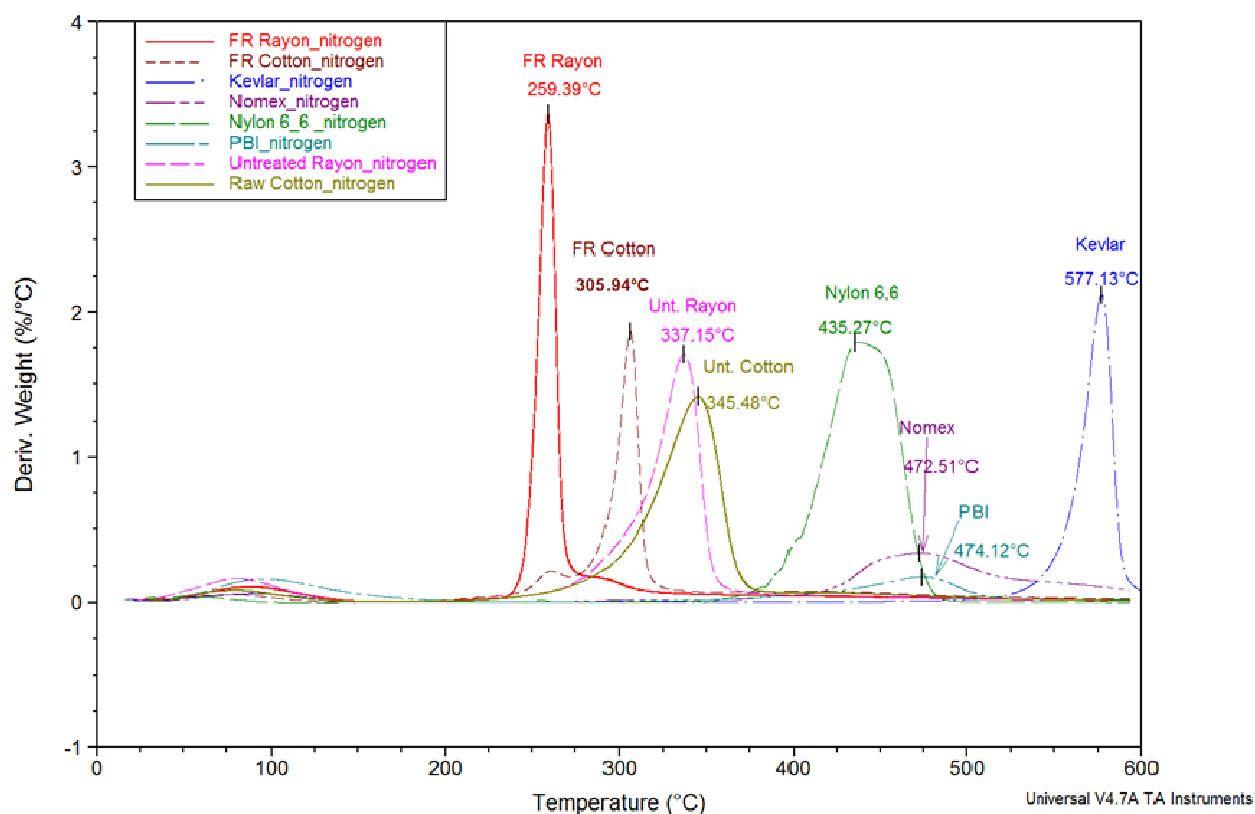


Figure 6. SDT Comparison of Thermal Stability of the Different Component Fibers Using Derivative Weight Verses Temperature Curves in Nitrogen from Room Temperature to 600 °C.

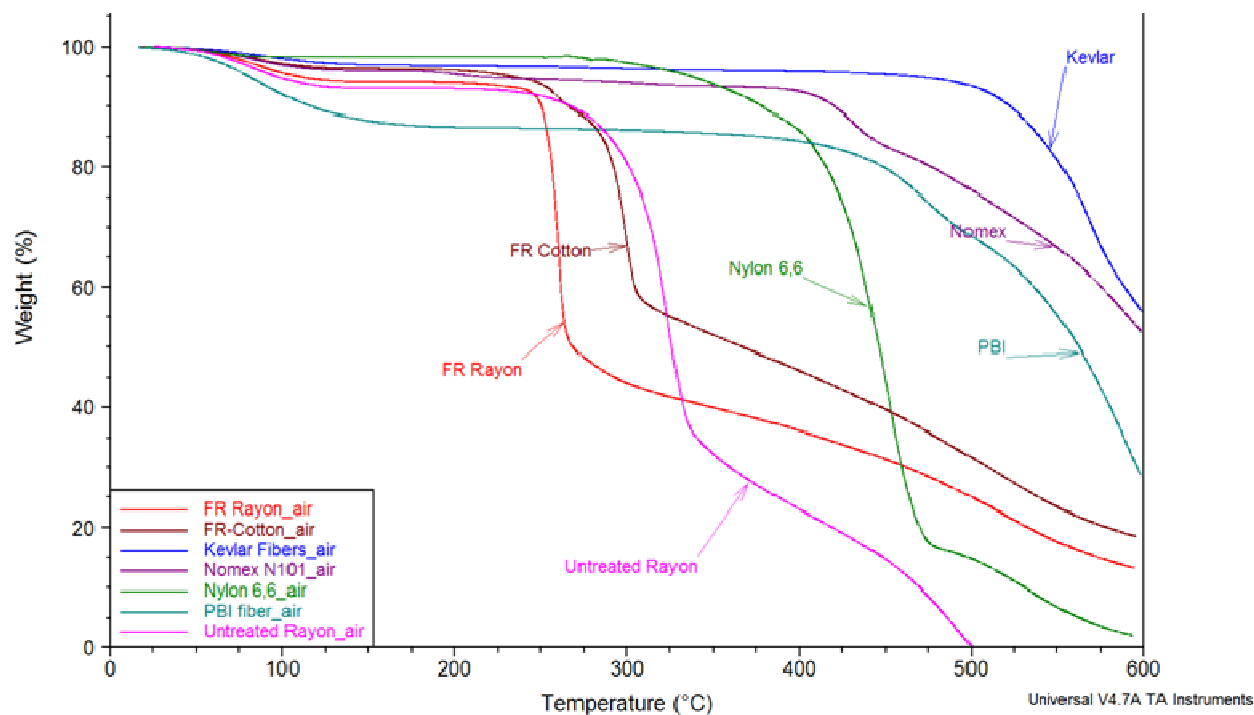


Figure 7. SDT Comparison of Thermal Stability of the Different Component Fibers Using Weight Versus Temperature Curves in Air from Room Temperature to 600 °C.

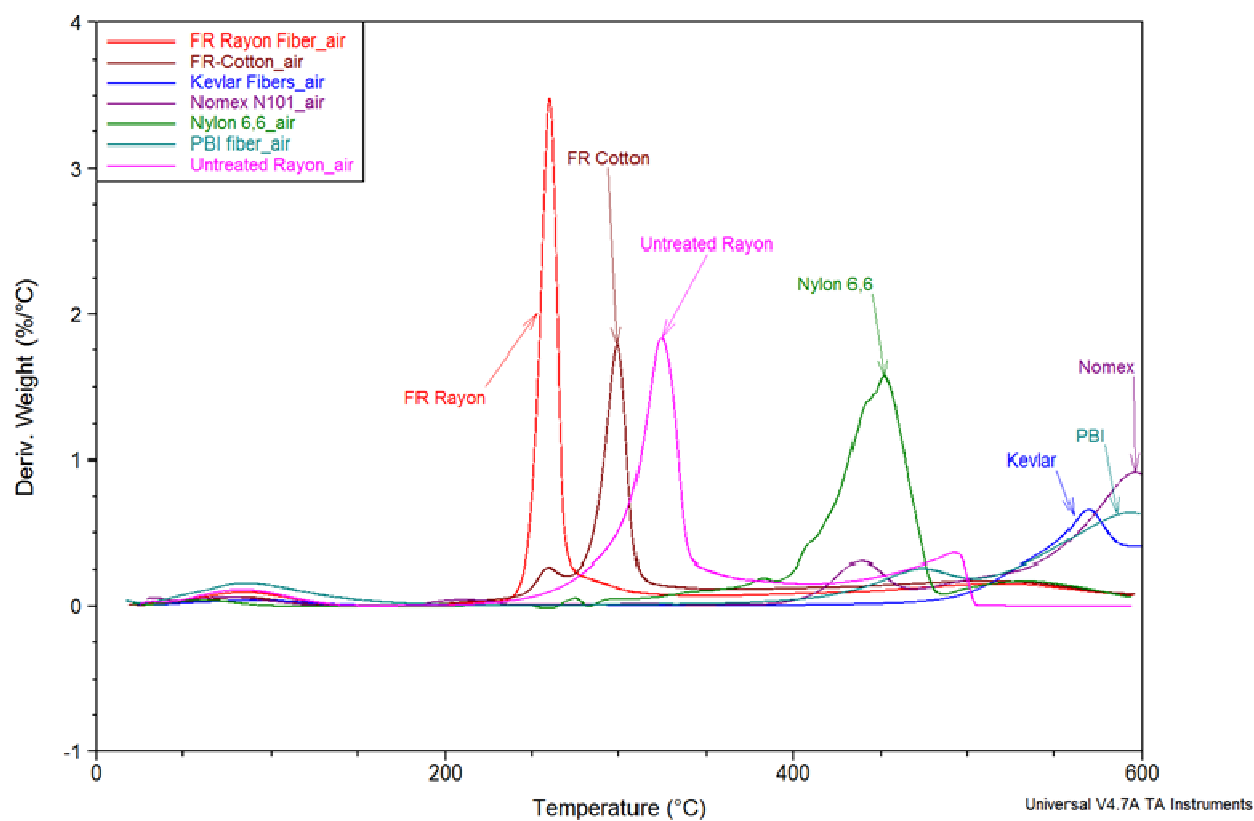


Figure 8. SDT Comparison of Thermal Stability of the Different Component Fibers Using Derivative Weight Versus Temperature Curves in Air from Room Temperature to 600 °C.

The results of this study (presented in Tables 3 and 4 and in Figures 5, 6, 7, and 8) agree with the existing data discussed in Section 1.1 that FR-treated rayon and cotton fibers had been found to have a lower thermal degradation temperature than untreated fibers, despite the expectation that polymers which decompose at higher temperatures generally have better fire retardancy, but also had a higher residual weight or less volatile matter evolved when heated. Both the FR-rayon and FR-cotton fibers had lower decomposition temperatures and left with a higher percent weight residual. It can also be observed that the total area of the endothermic peaks (i.e., those pointing downward) of the treated rayon is larger than that for the control (Figure 9). Although SDT is meant for high temperature measurements from 200 up to 1400 °C, weight change due to moisture loss, associated with the endothermic peak in the heat flow curve, was observed at about 100 °C. The TA Instruments graphs use downward-pointing peaks to indicate transitions caused by endothermic reactions in the DSC curve and upward-pointing peaks to indicate exothermic transitions (as shown in Figures 9, 10, and 11).

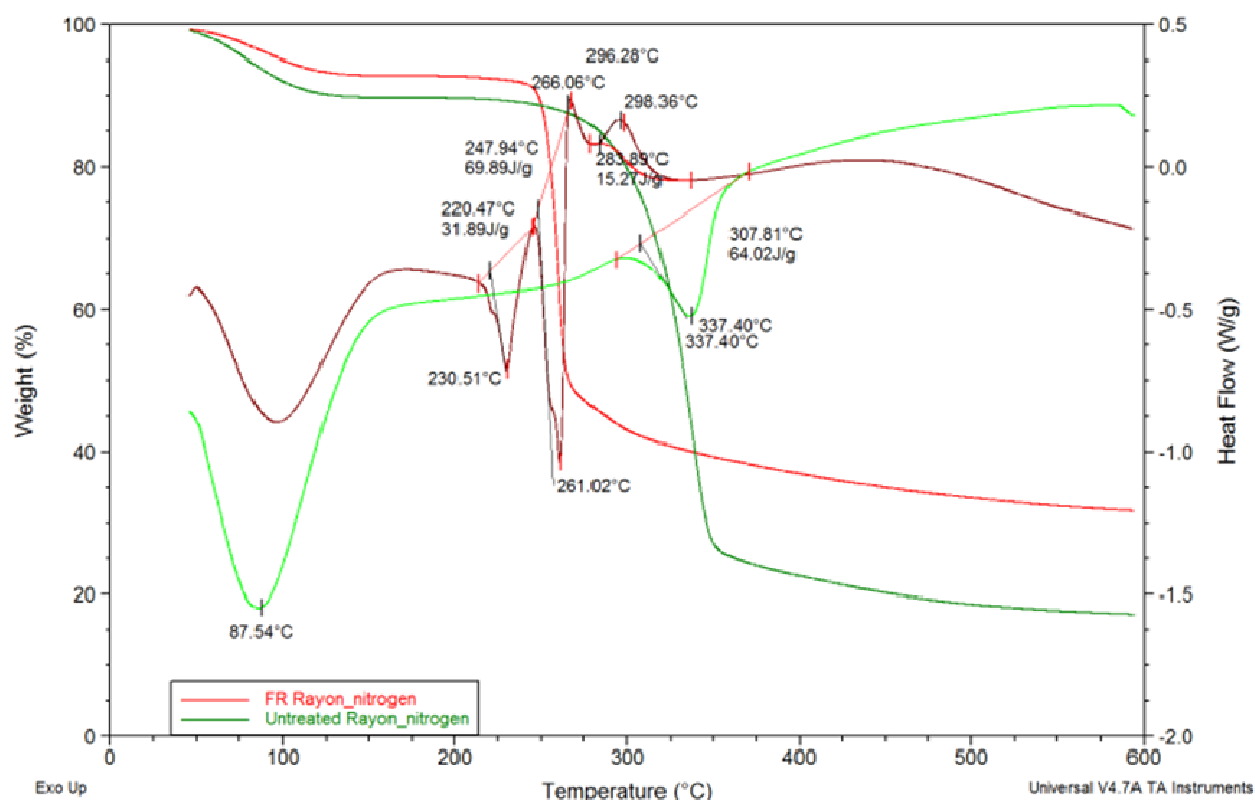


Figure 9. Thermograms of Untreated and FR-Treated Rayon with SDT Measurements.

From the SDT measurements, it is difficult to differentiate one type of FRACU from another, including the commercial FR additive for rayon, whether it is made with different weave patterns or prints or whether it has been washed (Figure 10).

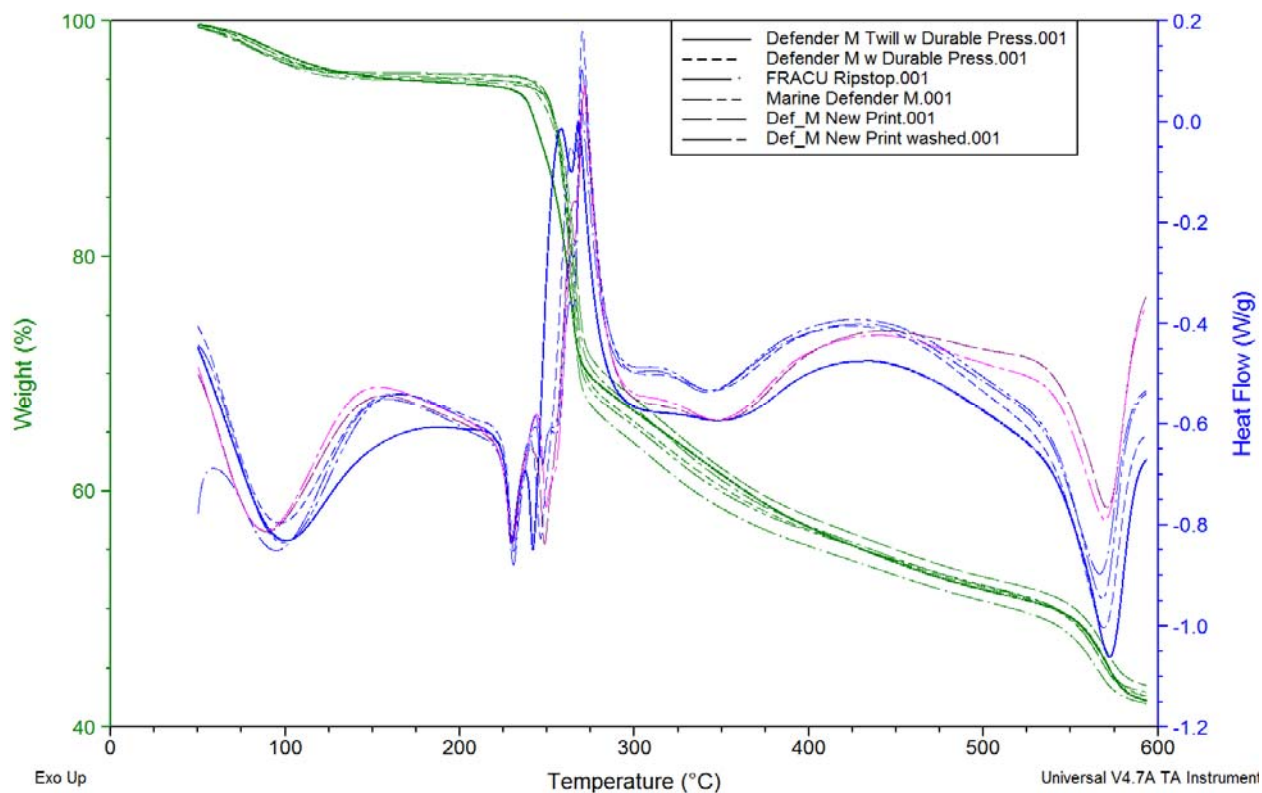


Figure 10. SDT Thermograms of Various FRACU Fabrics.

One of the purposes of studying the component fibers separately from the textile itself is to allow comparison of the thermal behaviors of the individual component fibers to the final manufactured textile, where the fibers are mixed intimately together. It is interesting to note that the FRACU, consisting of 65% FR-rayon, 25% Kevlar®, and 10% Nylon 6,6, when tested as a fabric, did not show any thermal behavior contribution from the nylon fiber, as shown in Figure 11, when compared with other component fibers in the FRACU.

Many effective FRs contain phosphorus and nitrogen. It was proposed that nitrogen interacts with the phosphorus-based flame retardant to catalyze dehydration and carbonization of cellulose, and thus enhances the effectiveness of the FR mechanism [6, 7]. This may explain why the nitrogen-containing nylon fiber does not have the same thermal behavior in the FRACU fabric.

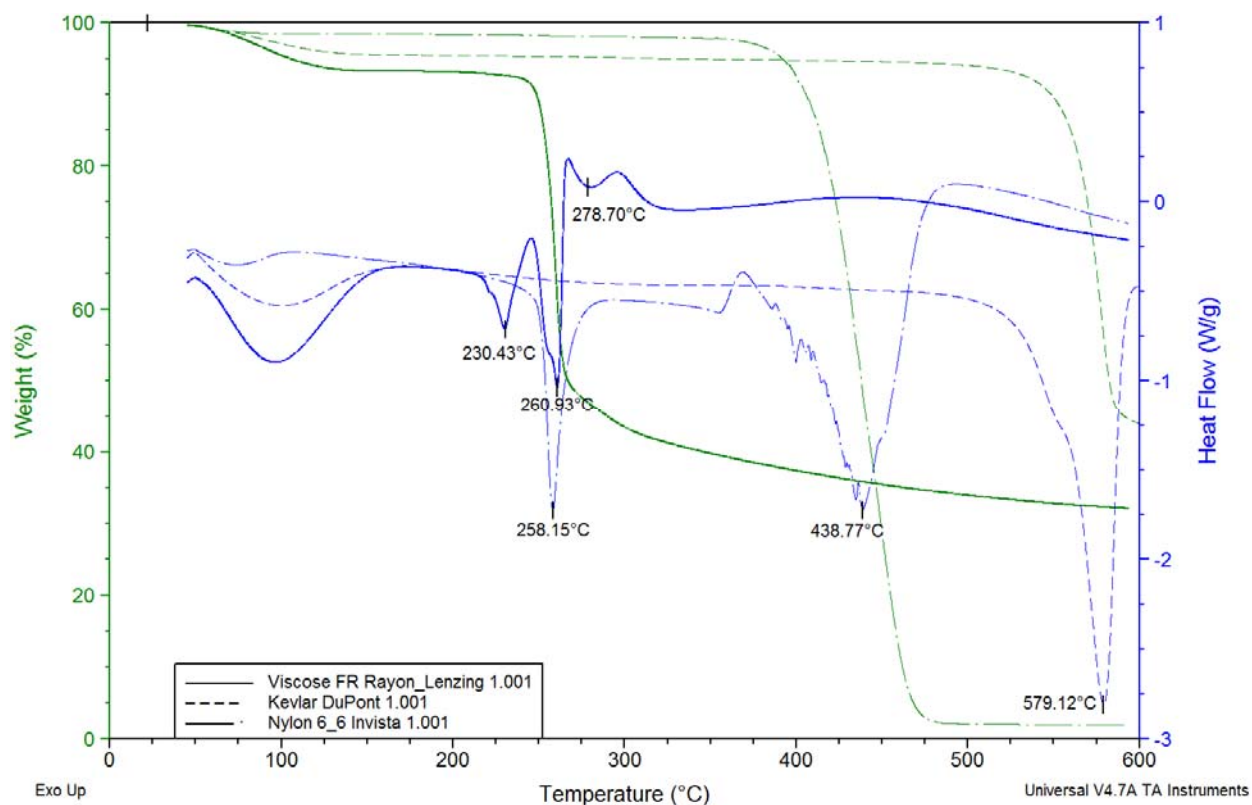


Figure 11. SDT Thermograms of All the Component Fibers in FRACU Fabric.

4.2 Pyrolysis GCMS

The pyrogram, or the chromatogram of the pyrolysis products, of a material recorded at the same pyrolysis temperature and with the same GCMS parameters will give the same characteristic “finger print” of peaks pertained to the material. Examples of this found during this study can be seen in Figure 12.

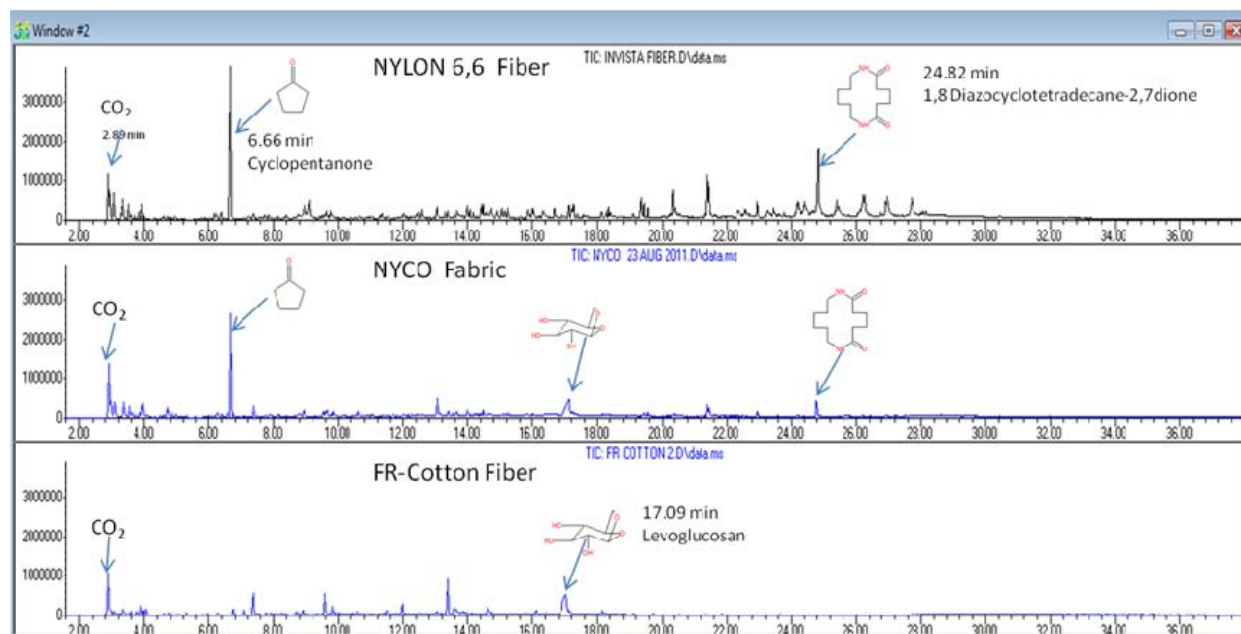


Figure 12. Pyrograms of Nylon 6,6 Fiber, FR-Cotton Fiber and NYCO Fabric

Various types of Kevlar® (129, 49, and KM2D) and Nomex® (N101 and N303) fibers were pyrolyzed and analyzed by the pyrolysis GCMS method. When these pyrograms were lined up on top of each other, it was very easy to differentiate the Kevlar® and Nomex® fibers just by visualization. These two fibers were made up with monomers of the same weight except different orientations; Kevlar® is the *para*-aramid and Nomex® is the *meta*-aramid. One would not be able to differentiate them by mass alone. It is fortunate that the *para* and the *meta* fragmentations of these fibers turn out to have slightly different polarity and thus result in different retention times in the GC column. Also it is noted that benzene diamine is a more preferable fragment for Nomex® fiber than for Kevlar® (Figure 13).

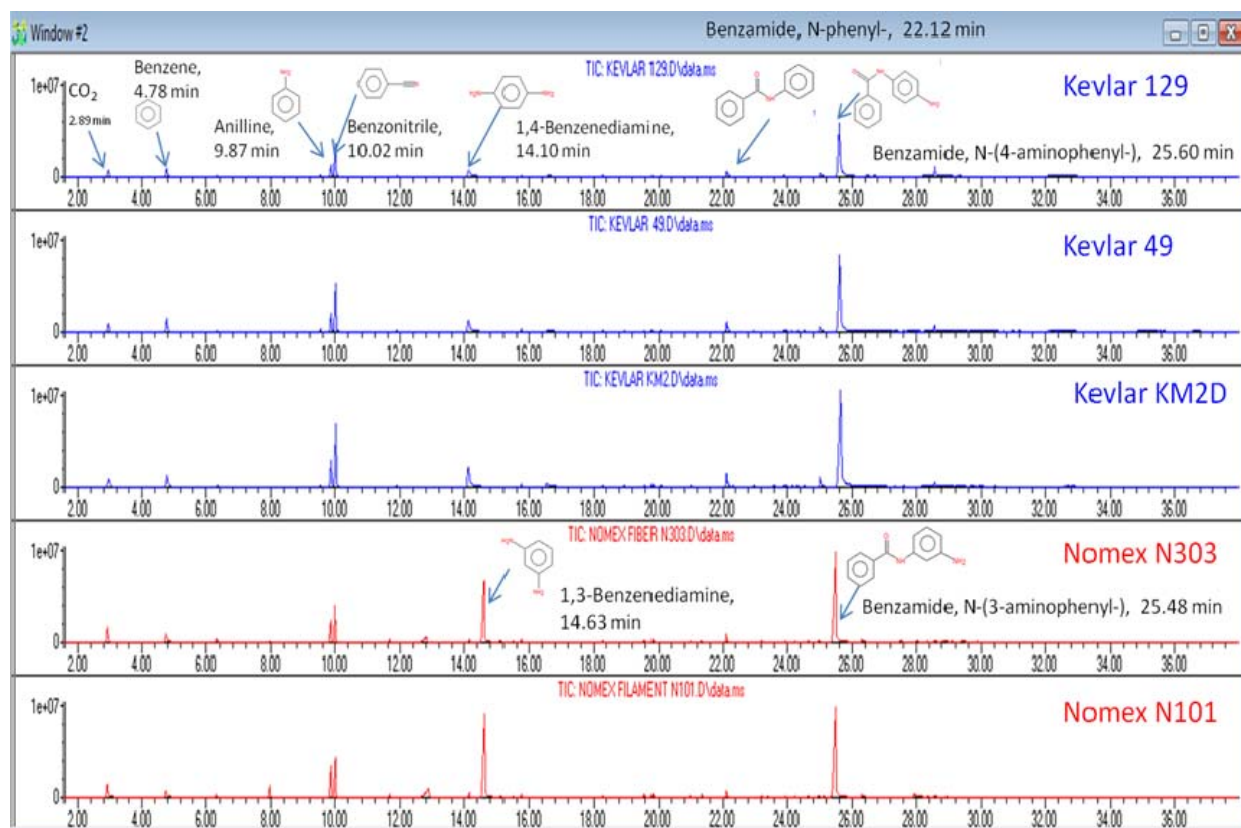


Figure 13. Comparative Analyses of the Kevlar® and Nomex® Pyrograms.

Multi-step pyrolysis GCMS measurements were performed on a group of fibers and textiles. It was found that pyrolyzing the materials at different temperatures can provide interesting and valuable additional information. The thermal desorption technique at low temperature can separate volatiles from the material well before it reaches pyrolytic conditions. Figures 14 and 15 show the pyrograms from the multi-step pyrolysis GCMS experiment collected for NYCO and FRACU. It was noticed that the FR chemicals were a major and the only peak in FRACU fabric at 200 °C. Subsequently, the washed and pre-washed FRACU were tested to see if this method could detect any difference in the fabric after washing. Previously, at the regular 750 °C pyrolysis temperature, the disparity could not be differentiated; however, at the low, thermal desorption temperature of 200 °C, it was repeatedly found that the FR peak was significantly lower in the fabric after five launderings (Figure 16). Since it was believed that the FR treatment for the FRACU fabric is “inherent,” in that it could not be washed off during laundering, it was suggested that perhaps the rayon fibers themselves were damaged and came off the fabric during laundering. Experiments were then designed to test the FR-rayon fiber by itself without being through any laundering process to see if the FR chemical was able to be thermally desorbed off the un-laundered FR-rayon fiber at temperatures lower than 200 °C. As shown in Figure 17, an increasing amount of FR was detected with increased temperature from 120 to 200 °C. It seems that this FR can be baked off the fiber by heat lower than 200 °C, and thus is not as stable and inherent as is commonly supposed. It should be noted that the FR peak was the only peak observed in the pyrolysis chromatograms at these low temperatures, between 125 °C to 200 °C, and no other volatiles were detected in this region. In addition, as a control, the untreated rayon fiber was also tested at 200 °C, and no FR peak was observed (as would be expected).

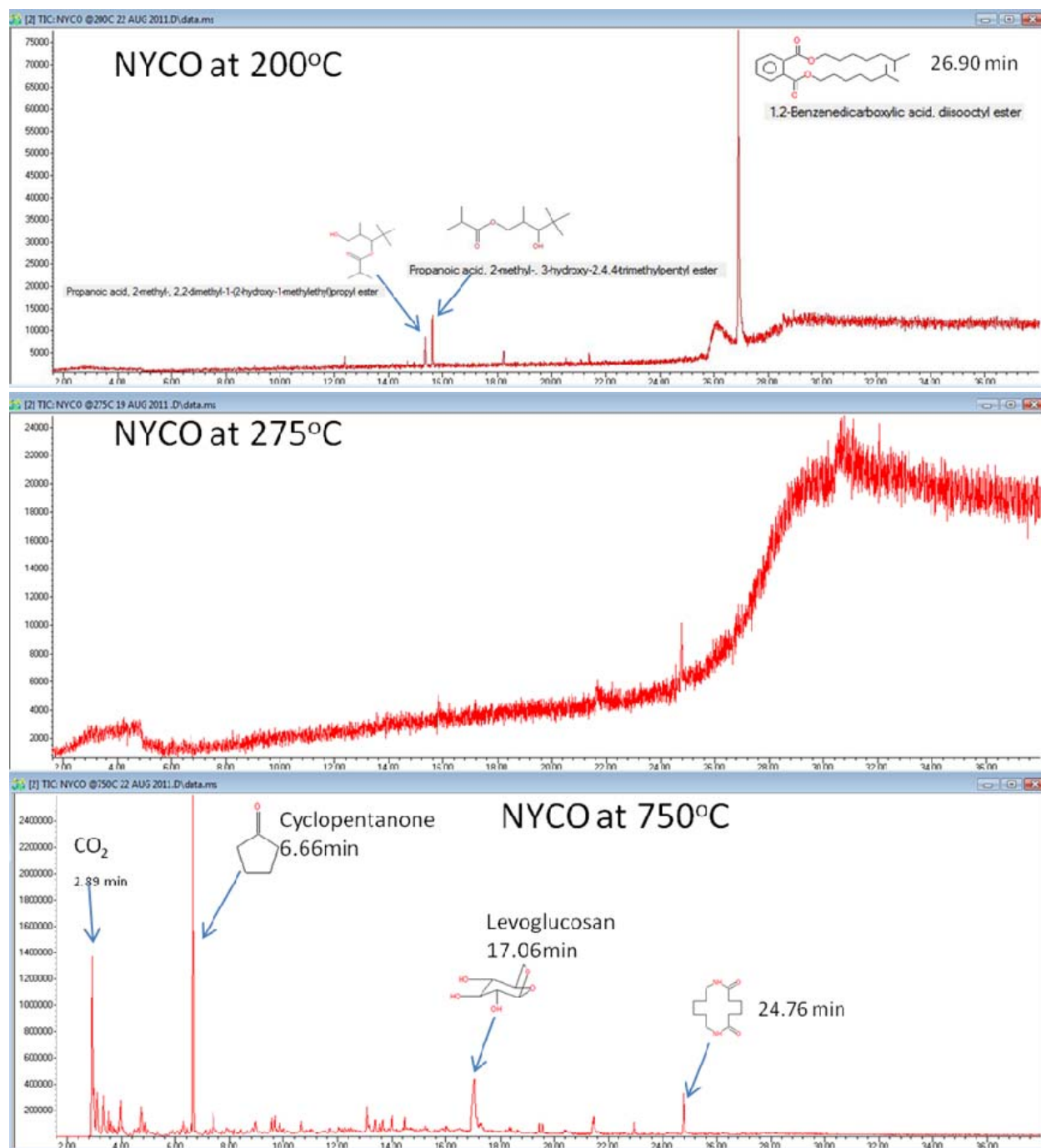


Figure 14. Pyrograms of Multi-Step Pyrolysis GCMS of NYCO Fabric at 200, 275, and 750 °C.

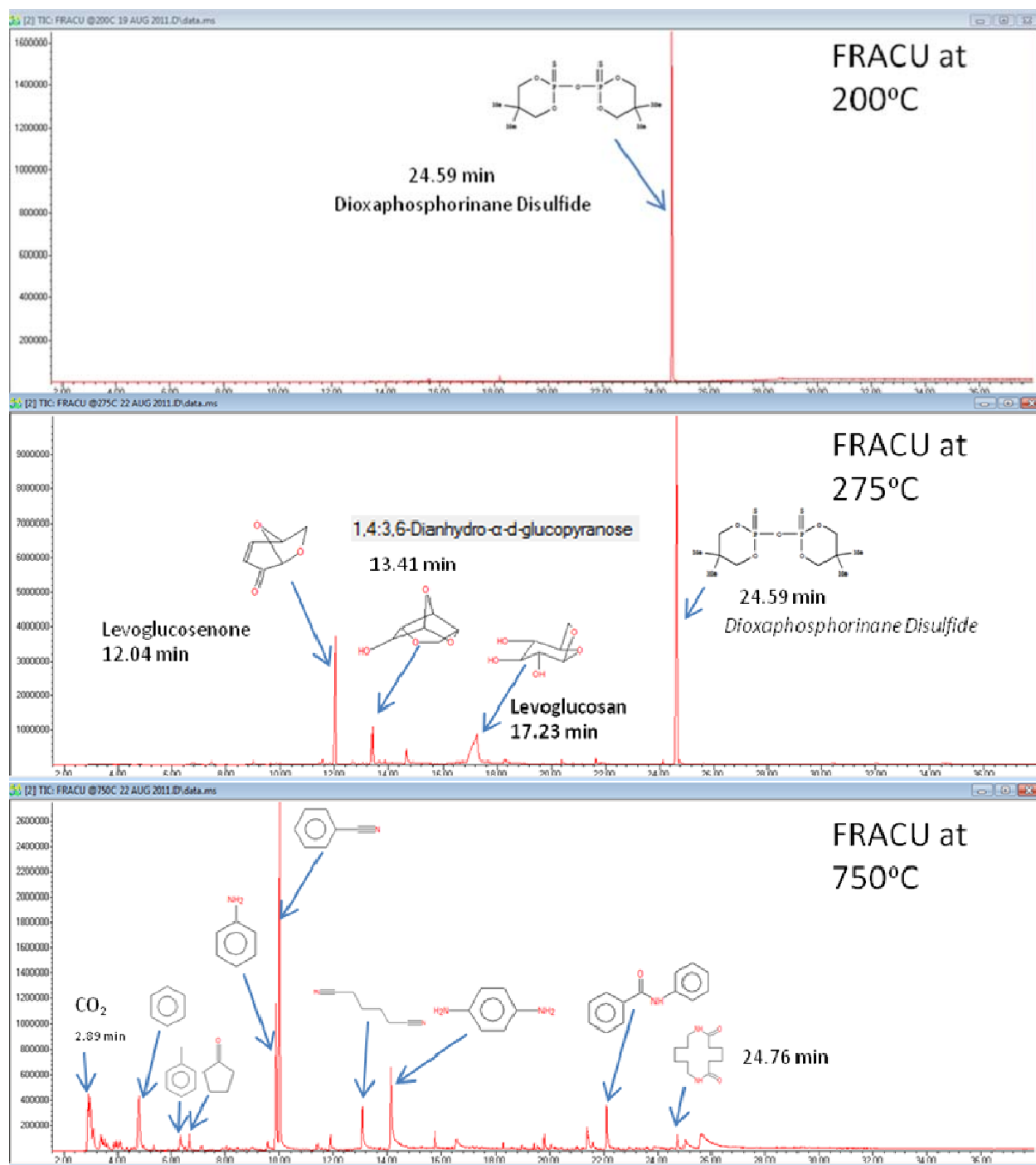


Figure 15. Pyrograms of Multi-Step Pyrolysis GCMS of FRACU Fabric at 200, 275, and 750 °C.

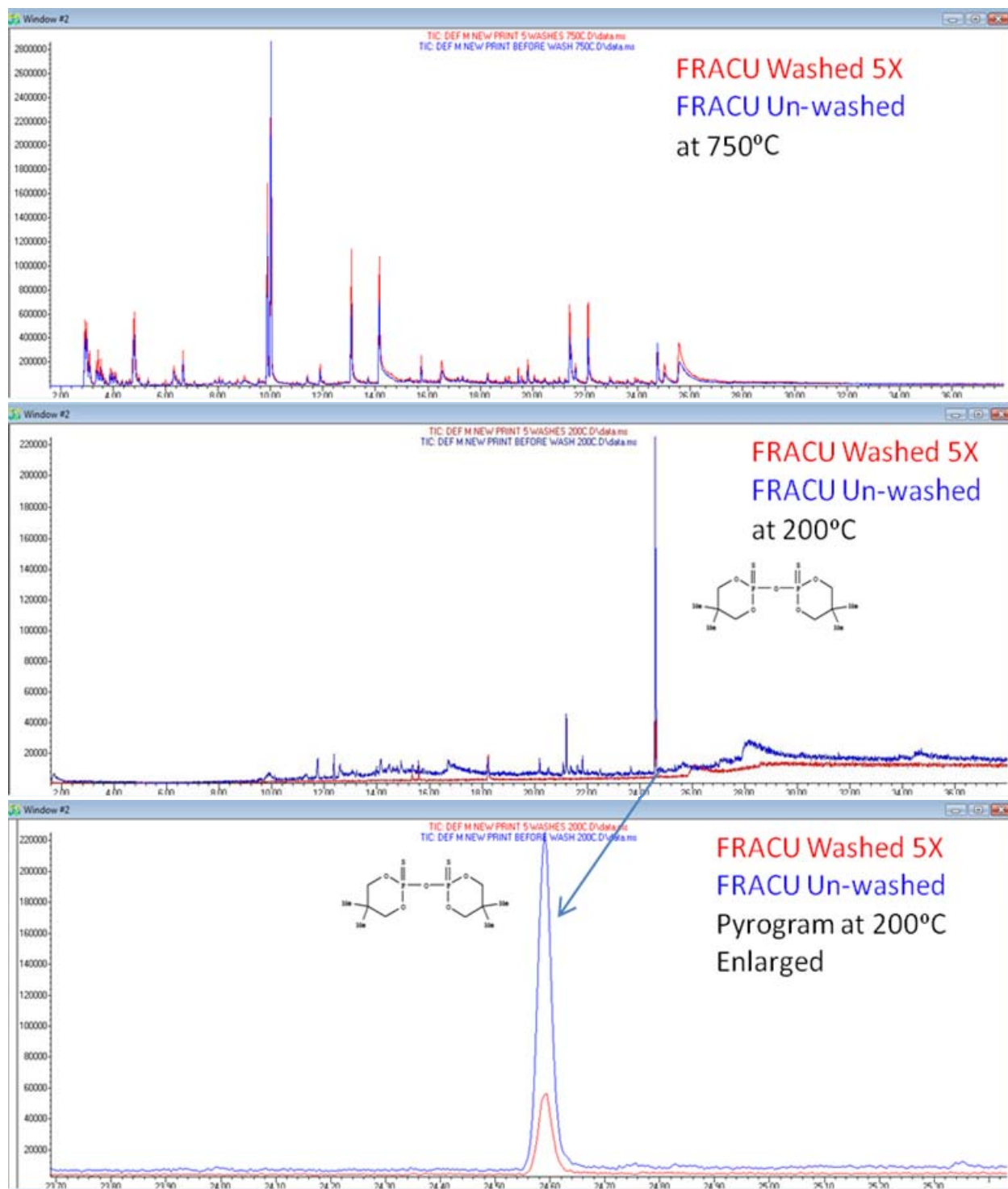


Figure 16. Pyrograms of Multi-Step Pyrolysis GCMS of the Washed and Unwashed Fabric at 200 and 750 °C.

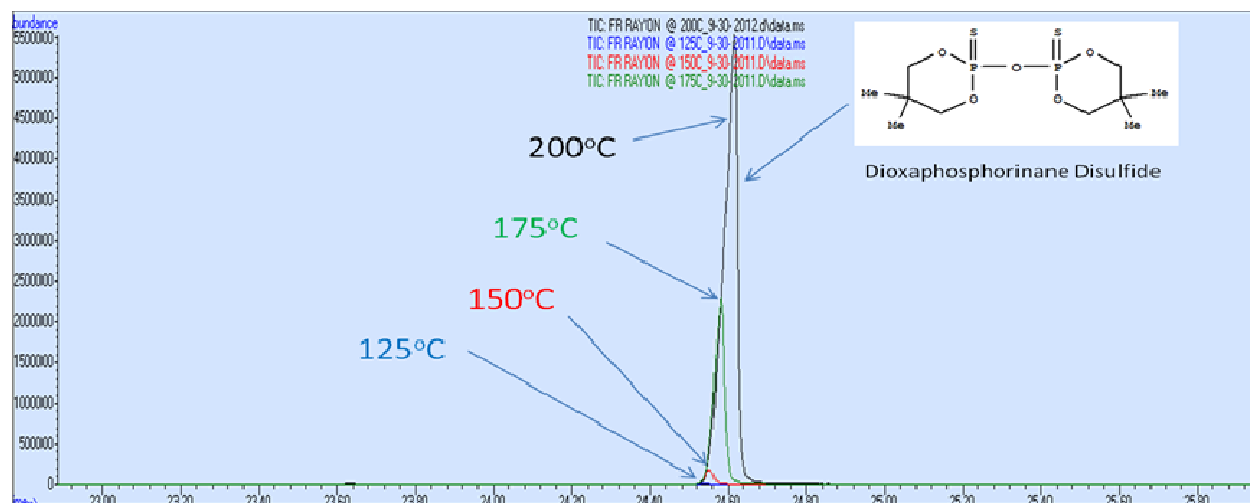


Figure 17. Pyrograms of Multi-Step Pyrolysis GCMS of the FR-Rayon Fibers at 125, 150, 175, and 200 °C.

5 Conclusions

The measurements obtained during this study using the TA Instruments Q600 and a CDS 5200 pyrolyzer coupled with an Agilent GC 7890A and MS 5975C show that SDT and pyrolysis GCMS, respectively, are valuable thermal methods and identification tools for characterizing military FR fibers and textiles. They provide very useful insights into understanding FR military fabrics.

The pyrolysis GCMS was able to isolate the FR chemical (Dioxaphosphorinane Disulfide) in the FRACU, which suppresses the formation of levoglucosan in the FR-treated rayon fibers, as evidenced by a major and the only peak in unwashed FRACU fabric at 200 °C shown in Figure 16. At the initial, regular 750 °C pyrolysis testing temperature, the disparity could not be differentiated; however, it was repeatedly found that the Dioxaphosphorinane Disulfide peak was significantly lower in the fabric after five launderings at the low thermal desorption temperature of 200 °C. Based on these findings, NSRDEC performed another study, subsequent to the one reported here, that more extensively compared washed and pre-washed FRACU using pyrolysis GCMS at 200 °C, and clear repeatable differences were observed. The findings here and their confirmation in the subsequent study signify potential for learning more about the effect of washing on FR cellulose fibers and identifying the number of times that a FRACU can be washed and still remain effective as an FR uniform.

The major gas detected during pyrolysis of the fibers and fabrics was CO₂; however, that was mainly due to the limited range set for the mass spectrometer to not scan for masses below 35 m/z, to help minimize air and water contamination of the detector. Different testing parameters will be explored in the future to gain further understanding of the complex pyrolysis and degradation behavior of polymer films, fibers, blends, and FR treatments.

Unlike pyrolysis GCMS, SDT was unable to isolate Dioxaphosphorinane Disulfide and component fibers. However, SDT was useful in identifying the degradation pathways in FR military fabrics and fibers.

This document reports research undertaken at the U.S. Army Natick Soldier Research, Development and Engineering Center, Natick, MA, and has been assigned No. NATICK/TR- 14/008 in a series of reports approved for publication.

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