FINAL CONTRACTOR REPORT
NASA CONTRACT H-13035D

PRODUCTS OF COMBUSTION OF NON-METALLIC MATERIALS

prepared for
MARSHALL SPACE FLIGHT CENTER

prepared by
C L PERRY ASSOCIATES, PO BOX 4325, HUNTSVILLE, AL 35815


DISTRIBUTION COVER:

NASA/MSFC
Attn: (Mail codes shown below.)
Marshall Space Flight Center, AL 35812

Code: Number:
GP29-B/Campbell Original
LA20/Technology Utilization Office 1
CC01/Intellectual Property Counsel 1
CN22D/Repository 3
EH32/Caruso 2**
EM15-42/Manning 1
NASA Center for Aerospace Info. (CASI) 2**
Attn: Accessioning Dept.
800 Elkridge Landing Rd.
Linthicum Heights, MD 21090-2934

** One copy must be reproducible.
FINAL CONTRACTOR REPORT
NASA CONTRACT H-13035D

PRODUCTS OF COMBUSTION OF NON-METALLIC MATERIALS

prepared for
MARSHALL SPACE FLIGHT CENTER

prepared by
C L PERRY ASSOCIATES, PO BOX 4325, HUNTSVILLE, AL 35815
STATEMENT OF WORK

OBJECTIVE

The objective of this project is to evaluate methodologies for the qualitative and quantitative determination of the gaseous products of combustion of non-metallic materials of interest to the aerospace community. The goal is to develop instrumentation and analysis procedures which qualitatively and quantitatively identify gaseous products evolved by thermal decomposition and provide NASA a detailed system operating procedure.

APPROACH

The program is to be structured as a feasibility study. Combinations of test chambers and instrumentation systems shall be assessed for application to gaseous product evolution. It is anticipated that several instrumentation/sensor combinations may exhibit potential for qualitative/quantitative analysis of gaseous products by thermal decomposition. It is the goal of this study to identify optimum instrumentation. Following the identification of the optimum approach, development and testing of the system(s) shall be performed. Test plans shall be defined and the approved test plan completed for the optimization of instrumentation combinations.

TASK 1 - DETERMINATION OF OPTIMUM INSTRUMENTATION

For the study of thermal decomposition products of non-metallic materials, an assessment of government furnished test chambers and sensors such as portable mass spectrometers, fourier transform infrared spectrophotometers, thermogravimetric analyzers and data acquisition systems shall be made. An optimized configuration of instrumentation will be assembled and calibrated using known gas concentrations for system verification and qualification.

TASK 2 - ANALYTICAL METHODOLOGY DEVELOPMENT FOR DETECTION SYSTEM

A detailed stepwise operating procedure for the optimized system will be developed, tested for validity and reproducibility and documented.

TASK 3 - FINAL REPORT

A final report documenting the work accomplished, including identification of shortfalls or problem areas and recommendations shall be submitted.
INTRODUCTION

The concept for this study was formulated during the course of a previous study of the solid rocket motor thrust liner nozzle material by Dr. James Thompson at the Alabama A&M University (Contract NAS8-36299, January, 1989, entitled "Evolved Gases and Thermal Profiles - A Comparative study of Nozzle Materials"). In that study, the analysis of evolved gases was performed using mass spectrometry. The mass spectral data became rather complicated and difficult to interpret when more than one gaseous species was evolved. So the assignment of specific chemical compounds to the evolved gases was not as definite as would be desired.

Presently, the flammability of materials is determined by the test procedure specified in NHB 8060.1C. In the case of sheet stock, a sample measuring approximately 2 inches by 8 inches is ignited by an ignition source and the burning characteristics are observed. No information concerning the identity of gaseous or solid products is obtained.

TASK 1 - DETERMINATION OF OPTIMUM INSTRUMENTATION

Recently, the Chemistry Branch of the Materials and Processes Laboratory, purchased a thermogravimetric analyzer interfaced with a fourier transform infrared spectrophotometer. This instrument is used to determine the weight loss characteristics of materials while simultaneously recording infrared spectra of the gaseous products associated with the weight loss.

The instrumentation consists of a Perkin-Elmer Model TGA 7 Thermal Gravimetric Analyzer (TGA) interfaced via a heated transfer line to a System 2000 Fourier Transform Infrared Spectrophotometer (FTIR). Both systems operate under computer control and samples smaller than 50 milligrams are usually subjected to analysis.

The TGA can be operated with various gaseous environments ranging from air and oxygen to inert environments such as nitrogen and argon. Samples can be heated at rates up to 200 degrees celsius per minute or held at fixed temperatures up to 1000 degrees celsius in a multi-ramp mode of operation. Data concerning the weight loss during heating are acquired by a digital computer and stored for further analysis. Gaseous products are sent to the FTIR via a transfer line heated to 250 degrees celsius to a micro-cell where transmittance spectra are obtained using a time resolved infrared (TRIR) computer program.

Since the TGA/FTIR instrumentation promises to be a powerful and self contained analytical system, it was chosen for evaluation over other systems, such as gas chromatography/mass spectrometry, which would require the development of a sampling technique and sampling hardware.
FINAL CONTRACTOR REPORT

TASK 2 - ANALYTICAL METHODOLOGY DEVELOPMENT

It was decided during preliminary testing to run the samples in the TGA at a heating rate of 20 degrees celsius per minute from room temperature (about 25 degrees celsius) up to the limit of the furnace, 1000 degrees celsius.

The FTIR was programmed for a 50 minute run to correspond with the TGA heating rate. Sixteen spectra were co-added at a resolution of 8 wavenumbers. A display of the infrared spectra from 4000 to 700 wavenumbers was automatically updated every sixteen scans, which presents a near real-time update of the spectral data. Both infrared and weight loss data are recorded on computer disk and are available for post-run data analysis.

A number of polymeric samples were chosen for analysis with the intent of testing a wide range of polymeric materials of interest to the aerospace community. These samples were subjected to the TGA/FTIR analysis and are presented here.

CALCIUM OXALATE

Since it is always best to proceed from the known to the unknown, a sample of calcium oxalate monohydrate, a well defined TGA sample, was chosen to verify the operation of the TGA/FTIR. All of the samples presented in this study are presented in the appendix to this report.

The weight loss graph from the TGA 7 for calcium oxalate monohydrate is on page A1 of the appendix. The graph is annotated with the well known reactions at each step in the decomposition of calcium oxalate. The upper box on page A2 of the appendix is a stacked plot of the infrared spectra from 4000 to 700 wavenumbers taken during the 50 minute run. It is easy to see the evolution of carbon dioxide near 2400 wavenumbers and carbon monoxide near 2200 wavenumbers. The evolution of water is not quite as obvious at 3700 wavenumbers.

Page A3 shows a slice in time at 25.6 minutes in the upper box and is annotated to indicate the products during the second step of the decomposition showing the evolution of carbon monoxide and its immediate oxidation to carbon dioxide. Water is also seen at about 3700 wavenumbers.

The lower box on page A3 contains the weight loss graph from the TGA 7 correlated with time and a Gram-Schmidt thermogram which is proportional the intensity of the infrared spectrum throughout the experiment.

ACTIVATED CHARCOAL

Since most of the samples in this study are expected to char to carbon followed by oxidation of the carbon to carbon monoxide and carbon dioxide, a sample of activated charcoal was analyzed to show the behavior of a charred sample. Page A4 in the appendix is the weight loss graph for activated charcoal. The first step at the beginning of the run is evolution of water adsorbed by the charcoal. The large step at about 540 degrees celsius is the actual oxidation of the charcoal. The stacked plot on page A5 shows the
evolution of carbon dioxide and a small amount of carbon monoxide. The broad band running at about 3000 wavenumbers is possibly an organic material which the charcoal has adsorbed. On page A6, the upper box contains the infrared spectrum at 31.9 minutes, showing the evolution of carbon monoxide and carbon dioxide plus water vapor near 3700 wavenumbers. The system seems to have a fairly long "memory" for water even though the transfer line from the TGA and the FTIR sample cell are both heated to 250 degrees celsius.

FIBERGLASS CIRCUIT BOARD

Page A7 is the weight loss graph for a fiberglass reinforced epoxy resin printed circuit board. From this weight loss graph we can see the about 55 percent of the board survived the heating to 1000 degrees celsius. This represents the fiberglass content of the board. There are two steps in the decomposition, one is very rapid at about 340 degrees celsius followed by a slow step out to about 740 degrees.

From the stacked plot on page A8, the rapid step shows a multiplicity of bands at about 18 minutes, which is the breakdown of the molecular structure of the resin. The resin peaks are altered, possibly partially charred. The large amount of carbon dioxide which follows is the oxidation of the char. Page A9 shows the spectrum of the sample at 18.4 minutes, which contains a band near 3000 wavenumbers indicative of the evolution of a hydrocarbon compound from the resin. The several bands from about 1600 wavenumbers down to 700 wavenumbers further confirm the presence of a hydrocarbon material. The spectrum at 31.8 minutes is much simplified due to the thermal decomposition of the resin.

CARBON FILLED FIBERGLASS EPOXY PANEL

Pages A10 through A14 show the weight loss and spectral information for a carbon filled fiberglass reinforced epoxy resin panel. The stacked plot shows the decomposition of the resin followed by the oxidation of the charred resin and the carbon filler. The spectrum on page A12 clearly shows the evolution of a hydrocarbon, possible the epoxy resin or a plasticizer. Page A13 shows the second step of the weight loss at 23.3 minutes and page A14 shows the long gradual loss with the evolution of carbon dioxide.

POLYIMIDE FILM (KAPTON)

The weight loss graph of a polyimide film is shown on page A15. The infrared transmittance spectrum of the original film is on page A16. The decomposition appears to occur in a single step leaving no residue. The stacked plot on page A18 shows that more than one compound is evolved during the course of heating.

CHLOROTRIFLUOROETHYLENE (KEL-F)

This polymer sample also appears to decompose in a single step. The weight loss graph is shown on page A21. The stacked plot on page A22 shows that a small amount of what may be a plasticizer is gradually lost before the onset of the major weight loss. Page A23 is an infrared spectrum during the major weight loss. Very little carbon dioxide is evolved.
POLYCARBONATE (LEXAN)

This polycarbonate sample shows a two step thermal decomposition. From the stacked plot on page A25 it can be seen that the first step is probably a breakup of the polymer chain with charring, followed by the burnoff of the charred material. The material evolved during the charring is shown in the infrared spectrum on page A26.

POLYMETHYL METHACRYLATE

This material appeared to boil during the decomposition. The weight loss graph on page A27 shows a single rapid step. The stacked plot on page A28 shows very little carbon dioxide evolution. The infrared spectrum shown in the top box on page A29 shows a material which proves to be methyl methacrylate by comparison to the spectrum on page A30, which is an authentic sample of methyl methacrylate treated in the same manner as the polymer, i.e., it has gone through the same thermal analysis as the polymer. The conclusion is that polymethyl methacrylate decomposes upon heating back to the monomer.

POLYETHYLENE TEREPTHALATE (MYLAR)

This sample decomposes in two steps and appears to be filled with about 10% of an inert material as shown on page A31. The infrared spectrum of the polymer film is shown on page A32. The stacked plot on page A33 shows the evolution of a small amount of a carbonyl compound (about 1800 wavenumbers, from 20 to 25 minutes) during the initial decomposition. The reason for the ever increasing bands from 1600 wavenumbers out to 700 wavenumbers is not understood. A white "smoke" was deposited on the furnace walls during the decomposition.

NYLON 66

This material appears to decompose to a simpler form accompanied by charring and the evolution of carbon dioxide and carbon monoxide. The weight loss graph, stacked plot and a spectrum at 25.78 minutes are shown on pages A36 through A38.

POLYPHENYLENESULFIDE

The weight loss curve (page A39) for this material changes slope around 780 degrees Celsius. The stacked plot on page A40 shows the evolution of an organic compound (page A41) with charring followed by the continued evolution of carbon dioxide and carbon monoxide. Page A42 shows that the principle product in the latter stages of heating is carbon dioxide with a smaller amount of water. A yellowish-black deposit formed on the furnace during the decomposition.

CARBON RESISTOR

What was thought to be a carbon resistor turned out to have only about 25% volatile material as shown on the weight loss graph on page A43. The stacked plot on page A44 shows the evolution of a tiny amount of an organic compound, probably a binder, with a gradual increase in carbon dioxide and carbon monoxide. The spectrum at 23.9 minutes on page A45 appears to be that of an unsaturated hydrocarbon.
RED SILICONE RUBBER

The weight loss graph for the material on page A46 shows that this rubber compound is heavily filled as it looses about 45% of its total weight. The stacked plot (page A47) shows the major compound being evolved to be a siloxane. The spectrum on page A48 is consistent with dimethyl siloxane.

SRM NOZZLE MATERIAL

This material which is a woven carbon fabric impregnated with an epoxy resin has a gradual evolution at low temperature (page A49) followed by a faster decomposition. The stacked plot on page A50 also shows this early evolution followed by increasing evolution of carbon dioxide with small amounts of carbon monoxide. The spectrum at 33.99 minutes (page A51) shows the evolution of carbon dioxide, carbon monoxide and water.

POLYETETRAFLUOROETHYLENE

This material begins decomposing near 600 degrees celsius and is completely decomposed by about 670 degrees celsius as seen in the weight loss graph on page A52. A spectrum of the original material is shown on page A53, a rather simple spectrum showing the C-F vibrations. The stacked plot on page A54 shows a very sharp and simple decomposition, apparently yielding only a single product. This product, whose spectrum is shown on page A55, turns out to be carbonyl fluoride. A white solid material condensed on the furnace wall. It was not possible to obtain a spectrum of this solid.

POLYETETRAFLUOROETHYLENE-POLYETHYLENE

This material whose trade name is TEFZEL (DuPont trade name) also decomposes in a single step as shown in the weight loss graph on page A56 except for the tiny loss between 600 degrees and 640 degrees celsius. The stacked plot on page A57 shows a decomposition to a simpler compound which persists to the end of the heating cycle. The spectra on pages A58 and A59 illustrate this simplification. A white solid material condensed on the furnace wall and the spectrum of this condensate, shown on page A60, is far from simple.

TYGON TUBING

Tygon tubing is a heavily plasticized polyvinyl chloride material. The weight loss graph (page A61) shows three different stages of decomposition. The stacked plot on page A62 seems to bear this out. The spectrum on page A63 at 11.9 minutes is that of the plasticizer which closely resembles the spectrum of dioctyl phthalate shown on page A66. The spectrum on page A64 contains a fascinating feature in the extremely rapid vibrations around the 3000 wavenumber region. By the time of the spectrum on page A65, it appears that the main product is carbon dioxide.
VELCRO PILE

This material had a very thick adhesive backing, so presents a dual problem in sorting out the decomposition of the adhesive from the decomposition of the actual polymeric material. The weight loss graph on page A67 is deceptively simple in that only one or two stages of decomposition are apparent. The stacked plot on page A68 can be interpreted as showing at least four stages of decomposition as shown by some spectral features rising and falling at different rates and times. The spectrum on page A69 shows carbon dioxide, carbon monoxide, a possible carbonyl, and hydrocarbon bands. A computer search would certainly be helpful in determining the nature of this material.

POLYVINYLIDENE FLUORIDE-HEXAFLUOROPROPYLENE

This material, marketed under the trade name VITON, is a popular gasket and seal material. The weight loss graph on page A70 shows a two step weight loss. The spectrum on page A72, at 25.6 minutes, shows the evolution of a compound with several structural features. Less than 3 minutes later, at 28.3 minutes, the spectrum has simplified to a carbon dioxide band at 2400 wavenumbers with a sharp spike near 1000 wavenumbers and a broad spike near 700 wavenumbers. Near the end of the heating cycle, the band at 1000 wavenumbers has broadened considerably as shown on page A74.
The analytical method used for this study requires that the analyst be completely familiar with the operation of the hardware and software systems of the Perkin-Elmer model 2000 FTIR and the Perkin-Elmer model TGA 7. A working knowledge of infrared spectral interpretation is also necessary. Installation of infrared spectral search software along with gas phase spectral search libraries would be almost an absolute essential. Assuming that that the above is accomplished, the following is a narrative of the analytical procedure. This procedure is also shown in the vendor manual "SYSTEM 2000 TG-IR INTERFACE" in chapter 2:

On the TGA 7, power up the system according to the vendor procedure. Set up the parameters for a heating rate of 20 degrees celsius per minute, from 25 degrees to 1000 degrees celsius, holding at 1000 degrees at the end of the run. Zero the balance, and place a sample in the sample pan and record the sample weight. Identify the sample and prepare to start the experimental run.

On the FTIR 2000, fill the MCT detector dewar with liquid nitrogen. Start up the computer. When the DOS prompt appears, enter the IRDM program by typing IRDM followed by ENTER. When the IRDM program appears display the INSTRUMENT menu and click on SETUP INSTRUMENT. Select the MCT detector and click on OK. Display the INSTRUMENT menu and click on SCAN MODE. Set the resolution to 8.0 wavenumbers, J-stop to 8.0, OPD velocity to 2.0 cm/s and click on OK. In the APPLICATIONS menu, click on TRIR.

When the TRIR application appears, click on CONFIGURE and verify the destination of the DATA and SPECTRA files, and verify that the SPECTRUM RANGE is 4000 to 700, BACKGROUND SCANS is 8 and GS THRESHOLD is 150. Click on OK. Display the FILE menu and click on OPEN METHOD if you wish to use the method used in this study. Under OPEN METHOD, select TRIR and click on OK. Open the DATA COLLECTION parameters dialog and on the first line enter the name of the file in which you want to store your data and click on OK.

Verify that the TGA 7 is ready to run. In the TRIR software, click on the METHOD menu and click on BACKGROUND. READY TO RUN BACKGROUND is displayed. Click on CONTINUE. In the METHOD menu click on START RUN. The START RUN dialog is displayed. Click on TRIGGER. Immediately select START RUN on the TGA 7. The FTIR computer is now recording and displaying the spectral information during the course of the heating run on the TGA 7. The weight loss of the sample is also being recorded and displayed on the TGA computer. The weight loss data can be combined with the infrared data after the run by following the vendor instructions.
CONCLUSIONS, SHORTFALLS AND RECOMMENDATIONS

There is no doubt that the combination of fourier transform infrared spectroscopy with thermogravimetric analysis offers a powerful tool to the understanding of the thermal decomposition of polymeric materials. However, a highly skilled infrared spectroscopist is required to obtain the maximum potential of the tandem system. A casual comparison of spectral data from this system to conventional liquid-phase infrared spectra can lead to erroneous results because gas-phase spectra usually have finer detail than the liquid phase spectra.

Very few of the degradation products evolved during this study were identifiable. The few that were identified were done by running a sample of the suspected material through the system, end to end, and comparing the resultant spectrum with the unknown spectrum. Such was the case in identifying methyl methacrylate as a product of the decomposition of polymethyl methacrylate and the identification of a phthalate ester as the plasticizer in Tygon tubing. The identification of carbonyl fluoride from tetrafluoroethylene was done by comparison to a gas phase spectrum.

The lack of a library of gas-phase spectra and a library search program are serious shortfalls in this TGA/FTIR system. Both are available from the vendor.

Further work is required to establish a method for obtaining quantitative information. Perhaps a method as simple as supplying known concentrations of premixed gases to the system would suffice to obtain quantitative data. It is doubtful that this method will be able to detect trace quantities of evolved materials. Semi-quantitative estimates can be made from spectral intensity information.

The availability of qualitative data, which serves to identify the evolved gases, is a giant step forward from the combustion tests of the past, which only show that a material burns or doesn't burn. Whether this method can replace the traditional flammability test is anybody's guess. It is understood that a flammability test can produce vastly different results depending on the geometry of the test chamber and the presence or absence of humidity.
Stacked plot from 0.00 to 50.00 mins

CALCIUM OXALATE IN AIR
Sample Information:

Sample ID: ACTIVATED CHARCOAL
Operator ID: CLP

Parameters:
- Final Temp: 1000 C
- Start Temp: 30 C
- Scanning Rate: 20 C/min.
- Y Range: 100 %
- Sample Weight: 4.033569 mg.

Conditions:
- End Condition: Hold at Final
- Load Temp: 20 C
- Go to Temp Rate: 200 C/min.
- Event 1 Time: 0 min.
- Event 2 Time: 0 min.
- Delay Time: 0 min.

Date: Sep 29, 1995 11: 23am
Scanning Rate: 20.0 C/min
Sample Wt: 4.034 mg Path: \PERRY\ File: A CHARCOAL CLP

Perkin-Elmer TGA7
ACTIVATED CHARCOAL

Spectrum at 31.9719 mins

Weight loss

Gram-Schmidt thermogram
--- = fiberglass circuit board (pcboard)

Sample Information:
Sample ID: fiberglass circuit board
Operator ID: CLP

Parameters:
Final Temp: 1000 °C
Start Temp: 30 °C
Scanning Rate: 20 °C/min.
Y Range: 100 %
Sample Weight: 29.50881 mg.

Conditions:
End Condition: Load Temp
Load Temp: 30 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Sep 06, 1995 11:45am
Scanning Rate: 20.0 °C/min
Sample Wt: 29.509 mg Path: \perry\"n
Perkin-Elmer TGA7
Sample Information:
Sample ID: black carbon fiberglass epoxy
Operator ID: clp

Parameters:
Final Temp: 1000 °C
Start Temp: 25 °C
Scanning Rate: 20 °C/min.
Y Range: 100 %
Sample Weight: 16.707 mg.

Conditions:
End Condition: Hold at Final
Load Temp: 20 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Sep 25, 1995 2:30pm
Scanning Rate: 20.0 C/min
Sample Wt: 16.707 mg Path:\perry\Files: BLKEPOI CLP

Perkin-Elmer TGA7
TGA7 Method: Kapton

Sample Information:
Sample ID: Kapton film
Operator ID: EIP

Parameters:
Final Temp: 1000 °C
Start Temp: 30 °C
Scanning Rate: 20 °C/min.
Y Range: 100 x
Sample Weight: 3.028355 mg.

Conditions:
End Condition: Load Temp
Load Temp: 30 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Aug 22, 1995 4:03pm
Scanning Rate: 20.0 °C/min
Sample Wt: 3.028 mg Path: \perry\calc-kapton.nota

Perkin-Elmer TGA7
Sample Information:
Sample ID: ctef kil-f
Operator ID: CLP

Parameters:
Final Temp: 1000 C
Start Temp: 30 C
Scanning Rate: 20 C/min.
Y Range: 0-100%
Sample Weight: 42.3136 mg.

Conditions:
End Condition: Load Temp
Load Temp: 30 C
Go to Temp Rate: 200 C/min.
Event 1 time: 0 min.
Event 2 time: 0 min.
Delay Time: 0 min.

Date: Aug 30, 1995 09: 26am
Scanning Rate: 20.0 C/min
Sample Wt: 42.314 mg Path: \perry\ File: KELF CLP

Perkin-Elmer TGA7
POLYMETHYL METHACRYLATE

Stacked plot from 0.00 to 50.00 mins
Sample Information:
Sample ID: mylar film 9/20/95
Operator ID: clp

Parameters:
Final Temp: 1000 °C
Start Temp: 25 °C
Scanning Rate: 20 °C/min
Y Range: 100 %
Sample Weight: 2.325699 mg

Conditions:
End Condition: Hold at Final
Load Temp: 25 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Sep 20, 1995 3:17pm
Scanning Rate: 20.0 °C/min
Sample Wt: 2.326 mg Path: \perry\perkin.elmer tga7
File: mylar1.clp
Sample Information:
Sample ID: nylon 66 pure
Operator ID: clp

Parameters:
Final Temp: 1000 °C
Start Temp: 20 °C
X Range: 100 °C/min.
Sample Weight: 28.5135 mg.

Conditions:
End Condition: Load Temp
Load Temp: 30 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Temperature (°C)

Date: Aug 17, 1995 10:45am
Scanning Rate: 20.0 °C/min
Sample Wt: 28.514 mg Path: \perry\File: nylon66.clr.cp

Perkin-Elmer TGA7
TGA7 Method: polysulfide

Sample Information:
Sample ID: polysulfide small sample
Operator ID: clp

Parameters:
Final Temp: 1000 °C
Start Temp: 23 °C
Scanning Rate: 20 °C/min.
Y Range: 100 %
Sample Weight: 17.39878 mg

Conditions:
End Condition: Hold at Final
Load Temp: 20 °C
Go to Temp Rate: 200 °C/min.
Event 1 Timer: 0 min.
Event 2 Timer: 0 min.
Delay Time: 0 min.

Date: Sep 25, 1995 11:08am
Scanning Rate: 20.0 C/min
Sample Wt: 17.399 mg Path:\perry\File: POLSULI CLP

Perkin-Elmer TGA7
Sample Information:
Sample ID: srn nozzle material
Operator ID: clp
Parameters:
Final Temp: 1000 C
Start Temp: 30 C
Scanning Rate: 20 C/min.
Y Range: 100 x
Sample Weight: 16.22617 g.
Conditions:
End Condition: Load Temp
Load Temp: 30 C
Go to Temp Rate: 200 C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Sep 07, 1995 10: 23am
Scanning Rate: 20.0 C/min
Sample Wt: 16.226 mg Path: \perry\
Sample Information:

Sample ID:
Operator ID:

Parameters:
Final Temp: 1000 C
Start Temp: 50 C
Scanning Rate: 20 C/min.
Y Range: 100 %
Sample Weight: 26.42619 mg.

Conditions:
End Condition: Load Temp
Load Temp: 25 C
Go to Temp Rate: 200 C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Jul 07, 1995 3:51pm
Scanning Rate: 20.0 C/min
Sample Wt: 26.426 mg Path: test
File: 103212bc
Sample Information:
Sample ID: tefzel rod
Operator ID: clp

Parameters:
Final Temp: 1000 °C
Start Temp: 25 °C
Scanning Rate: 20 C/min.
Y Range: 100 %
Sample Weight: 26.7405 mg.

Conditions:
End Condition: Hold at Final Load Temp: 25 °C
Go to Temp Rate: 200 C/min.
Event 1 Time: 0 min.
Delay Time: 0 min.

Date: Sep 13, 1995 11:30am
Scanning Rate: 20.0 C/min
Sample Wt: 26.741 mg Path: \perry\ File: TFF7F12 CLP

Perkin-Elmer TGA7
Sample Information:
Sample ID: tygon tubing
Operator ID: EJG
Parameters:
Final Temp: 1000 C
Start Temp: 30 C/min.
Y Range: 100%
Sample Weight: 38.0272 mg.
Conditions:
End Condition: Load Temp
Load Temp: 30 C
Go to Temp Rate: 200 C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Perkin-Elmer TGA7

Date: Aug 23, 1995 10:33 am
Scanning Rate: 20.0 C/min
Sample Wt: 38.023 mg Path: \perry\ File: TYGN7 CLP
Sample Information:
Sample ID: velcro pile w/adhesive
Operator ID: cip

Parameters:
Final Temp: 1000 C
Start Temp: 30 C
Scanning Rate: 20 C/min.
Y Range: 100%
Sample Weight: 15.26196 mg.

Conditions:
End Condition: Load Temp
Load Temp: 30 C
Go To Temp Rate: 200 C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.
Delay Time: 0 min.

Date: Aug 29, 1995 1:45pm
Scanning Rate: 20.0 C/min
Sample Wt: 15.262 mg Path: \perry\File: VPile CLP

Perkin-Elmer TGA7
Sample Information:
Sample ID: viton nipple seal
Operator ID: clp
Parameters:
Final Temp: 1000 °C
Start Temp: 30 °C
Scanning Rate: 20 °C/min.
Y Range: 100 %
Sample Weight: 31.66911 mg.
Conditions:
End Condition: Hold at Final
Load Temp: 20 °C
Go to Temp Rate: 200 °C/min.
Event 1 Time: 0 min.
Event 2 Time: 0 min.

Date: Sep 26, 1995 3:34pm
Scanning Rate: 20.0 °C/min
Sample Wt: 31.669 mg
Path: \perry\f170\viton\n
Perkin-Elmer TGA7
VITON NIPPLE SEAL

Spectrum at 26.3740 mins

Gram-Schmidt thermogram

Weight loss
PRODUCTS OF COMBUSTION OF NON-METALLIC MATERIALS

Cortes L. Perry

C L Perry Associates
P. O. Box 4325
Huntsville, AL 35815

NASA/ Marshall Space Flight Center
EH-32
Marshall Space Flight Center, AL 35812

This is the final report on a study to devise a method and instrumentation to determine the gaseous products of combustion of non-metallic materials. The instrumentation consists of a Thermogravimetric Analyzer delivering a gaseous product to a Fourier Transform Infrared Spectrophotometer (TGA/FTIR). Several non-metallic materials are studied and the weight-loss graphs and corresponding infrared spectra are presented.

The method presented appears to be a valuable tool in the study of the thermal degradation of non-metallic materials.