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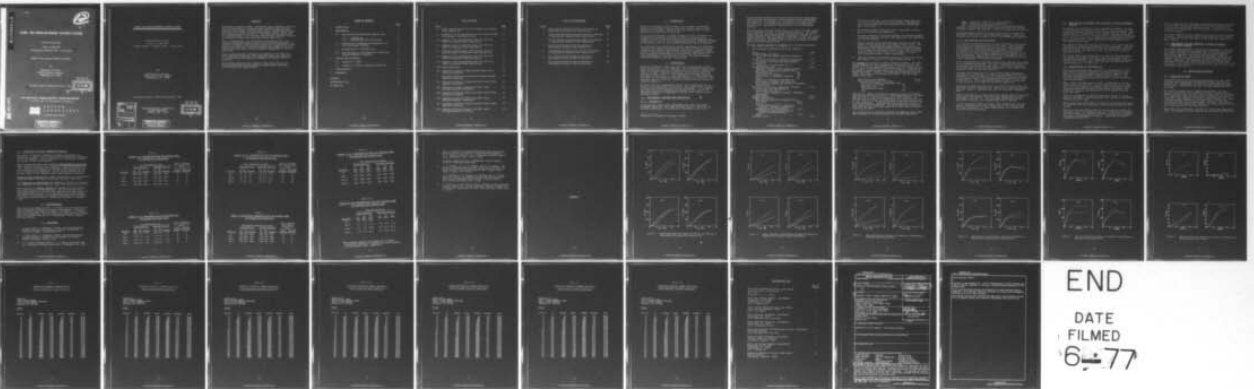
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FLAME - AND SMOKE-RETARDANT POLYMER SYSTEMS.(U)  
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# FLAME- AND SMOKE-RETARDANT POLYMER SYSTEMS

Third Quarterly Report

Issued: 23 May 1977

Period Covered: November 1976 — January 1977

Prepared Under Contract: N00024-76-C-5336

For

Department of the Navy  
Naval Sea Systems Command  
Washington, DC 20362

Leo Parts, Robert D. Myers and Norman F. May

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## ABSTRACT

Experimental neoprene rubber compositions were prepared to investigate the feasibility of reducing smoke formation and improving other fire performance characteristics with incorporated additives. Specimens were tested in an NBS smoke density chamber under flame and nonflame exposure conditions. The smoke optical density and the concentrations of CO, CO<sub>2</sub>, NO<sub>x</sub>, hydrocarbons, hydrogen chloride and hydrogen cyanide were monitored during these tests.

Ferric acetylacetonate by itself and in combination with poly(ammonium phosphate) reduced smoke optical density generated from neoprene foam under flame exposure conditions by approximately 10%. Cupric acetylacetonate, another candidate char formation catalyst evaluated in this work, catalyzed thermal degradation of the neoprene foam.

Ferric acetylacetonate catalyzed the formation of CO under non-flame exposure conditions. This catalytic effect was destroyed by incorporating poly(ammonium phosphate) with ferric acetylacetonate.

Crosslinking catalysts more effective than those used in the presently reported work are needed to reduce smoke formation from neoprene foam significantly.

## TABLE OF CONTENTS

	<u>Page</u>
1. INTRODUCTION	1
2. EXPERIMENTAL	1
2.1 Experimental Neoprene Foam Compositions	1
2.1.1 Preparation	1
2.1.2 Observations and Conclusions	4
2.2 Preparation of Neoprene Foam Specimens for Fire Performance Testing	5
2.3 Encapsulation of Phoschek P-30	5
2.4 Test Methods for the Formation of Smoke and Gaseous Combustion Products	6
3. RESULTS AND DISCUSSION	6
3.1 Formation of Smoke	6
3.2 Formation of Gaseous Combustion Products	9
4. ACKNOWLEDGMENT	9
5. REFERENCES	9
APPENDIX	14
DISTRIBUTION LIST	30
DD FORM 1473	31

LIST OF TABLES

<u>Table</u>	<u>Page</u>
I Smoke Optical Density Results Summary for Neoprene Foam Compositions	8
II Summary of CO Concentration Data for Selected Times for Neoprene Foam Compositions	10
III Summary of CO <sub>2</sub> Concentration Data for Selected Times for Neoprene Foam Compositions	10
IV Summary of NO <sub>x</sub> Concentration Data for Selected Times for Neoprene Foam Compositions	11
V Summary of Hydrocarbons Concentration Data for Selected Times for Neoprene Foam Compositions	11
VI Summary of HCl Concentration Data for Selected Times for Neoprene Foam Compositions	12
VII Summary of HCN Concentration Data for Selected Times for Neoprene Foam Compositions	12
VIII Sample Mass Data	21
IX Combustion Products Formed from BF Under Flame Exposure Conditions	22
X Combustion Products Formed from BF Under Nonflame Exposure Conditions	23
XI Combustion Products Formed from FSF-1 Under Flame Exposure Conditions	24
XII Combustion Products Formed from FSF-1 Under Nonflame Exposure Conditions	25
XIII Combustion Products Formed from FSF-2 Under Flame Exposure Conditions	26
XIV Combustion Products Formed from FSF-2 Under Nonflame Exposure Conditions	27
XV Combustion Products Formed from FSF-3 Under Flame Exposure Conditions	28
XVI Combustion Products Formed from FSF-3 Under Nonflame Exposure Conditions	29

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Smoke Optical Densities During the Burning of Experimental Neoprene Foam Compositions	7
2	Carbon Monoxide Concentrations During the Burning of Experimental Neoprene Foam Compositions	15
3	Carbon Dioxide Concentrations During the Burning of Experimental Neoprene Foam Compositions	16
4	NO <sub>x</sub> Concentrations During the Burning of Experimental Neoprene Foam Compositions	17
5	Hydrocarbons Concentrations During the Burning of Experimental Neoprene Foam Compositions	18
6	HCl Concentrations During the Burning of Experimental Neoprene Foam Compositions	19
7	HCN Concentrations During the Burning of Experimental Neoprene Foam Compositions	20

## 1. INTRODUCTION

Means for enhancing the fire safety of two polymeric materials, plasticized polyvinyl chloride (PVC) and neoprene, are investigated in this program. The preceding two reports (Ref. 1 and 2) summarized results obtained with PVC.

The successful approach to smoke retardance of PVC, entailing the incorporation of ferric and cupric acetylacetonates as char formation catalyst precursors, was evaluated on the neoprene foam rubber system. The use of poly(ammonium phosphate) (Phoschek P-30®)\* in conjunction with the metal acetylacetonates, was also explored.

The procedures for preparing the needed compositions are described. A method for encapsulating Phoschek P-30 into partially hydrolyzed ethylene-vinyl acetate copolymer was developed, to prevent coagulation of the neoprene rubber latex by the poly(ammonium phosphate). The encapsulation procedure is also described. The results of tests pertaining to the formation of smoke and gaseous degradation and combustion products are reported.

## 2. EXPERIMENTAL

Numerous formulations have been used for the preparation of neoprene foam (see Ref. 3 for examples). An isocyanide-modified foam, based on Neoprene Latex No. 357 produced by the duPont Company, was used as the base formulation because of its relatively good fire resistance (Refs. 4 and 5). This composition contains aluminum hydroxide as the filler. It has been shown in a recent investigation (Ref. 6) that aluminum hydroxide enhances char formation from neoprene, with a concomitant reduction of heat release during the thermal degradation.

Phoschek P-30 was incorporated into the latex formulation to enhance the fire resistance of the foam. This additive was expected to exert fire-inhibiting action by two mechanisms: (1) by supplying flame-inhibiting phosphorus-containing species, and (2) by forming a protective polyphosphate coating on the surface of the degrading polymer.

### 2.1 EXPERIMENTAL NEOPRENE FOAM COMPOSITIONS

#### 2.1.1 Preparation

In exploratory, small-scale experiments with fire- and smoke-retardant additives, the recipe reported in Ref. 4 served as the starting composition. The effects of incorporated fire- and

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\*Registered trademark of Monsanto Company.

smoke-retardant additives upon foam processing and curing characteristics were investigated. Processing and curing procedures were modified, as predicated by the effects of the incorporated FSR additives. Procedures were established for larger scale preparation of experimental compositions for fire performance testing.

The following is a brief description of the base foam (BF) preparation procedure (see Ref. 4 for additional details). One hundred sixty-seven and seven tenths grams of Neoprene Latex 357 (E. I. duPont Company, Elastomer Chemicals Department) of 60% total solids content was used. The quantities of the other reagents are given with reference to 100 parts of dry solids in the latex, or 166.7 parts of the water-based latex.

The base liquid compound was prepared by a five-step procedure:

1. The following components were first combined:

Water		1.00
Aquarex® WAQ, 30% active		1.2
(E. I. duPont Company, Elastomer Chemicals Dept.)		
Triton X-100, diluted to 30% active ingredient		3.00
(surfactant from Rohm and Haas Company)		
Dresinate 91		1.50
(surfactant from Hercules, Inc.)		
Thiocarbanilide, 33%		3.00
(Monsanto Company, Rubber Chemicals Dept.)		
Ball-milled dispersion prepared from:		
Thiocarbanilide	100	
10% Solution of Daxad 11	30	
10% Solution of ammonium caseinate	30	
Water	140	
Daxad 11 and ammonium caseinate are surfactants available from Dewey and Almy Chemical Company, and Sheffield Chemical, respectively)		
Tepidone, 23.5% active ingredient, prepared by diluting the 47% commercial composition with water		1.00
(E. I. duPont Company, Elastomer Chemicals Department)		
Diethanolamine		0.25
Pigment masterbatch		15.83
Ball-milled dispersion prepared from		
Zinc oxide	100.0	
Antox N	26.6	
(Antioxidant from E. I. duPont Co. Elastomer Chemicals Department)		
Marasperse N-22, 10% solution	12.7	
(surfactant from American Can Co. Chemical Products Division)		
Water	72.2	
Antimony oxide		4.40

The above components, except for antimony oxide, were agitated until a uniform mixture was obtained. Subsequently, the latter compound was incorporated and the mixture was again stirred well.

2. The blended mixture prepared in Step 1 was added to 166.7 parts of the latex with agitation.
3. Aluminum hydroxide, 20.00 parts (Hydral 710, Aluminum Company of America, Chemical Division) was added while the dispersion was stirred rapidly.
4. N-Methyldiethanolamine hydrochloride solution, 23.8%, was added to bring the pH into the range of 10.8 to 11.2. Subsequently, N-methyldiethanolamine (MDEA, Union Carbide Corporation) was added to bring the total quantity of MDEA to 7.35 grams per 100 grams of latex solid. The resulting suspension was allowed to age for at least two hours at room temperature before further processing.
5. Ammonium caseinate surfactant solution of 10% solids content was added at a ratio of 1 to 100 of latex solids.

The foaming was carried out with a Kitchen Aid mixer. The base liquid composition, whose preparation is described above, was frothed with air at high whipping speed for 8-10 seconds to produce a foam of approximately the desired density. Subsequently, the speed of agitation was reduced for further refining of the foamed dispersion. Finally, polymethylene polyphenyl isocyanate (PAPI) and sodium silicofluoride were added. The following amounts were used:

PAPI (from Upjohn Company)	14.00
Sodium silicofluoride dispersion	13.33
Ball-milled dispersion prepared from	
Sodium silicofluoride	100
Bentonite clay	2
10% solution of NaOH	5
Water	226

PAPI and the sodium silicofluoride dispersion were distributed rapidly over the entire surface. These ingredients were blended into the foam at the refining speed within 90 seconds. The blended foam, including all ingredients, was poured rapidly into a 5 in. x 10 in. x 4 in. mold, in which it was allowed to set up at room temperature. Approximately four hours were allowed for that stage of the preparation. The water-containing open-pore foam slab was then dried and cured for 12 hours in an oven maintained at 120-125°C.

The following fire- and smoke-retardant experimental foams (FSF) were prepared by the same procedure as the base formulation:

FSF-1, containing 5 phr ferric acetylacetonate  
FSF-2, containing 10 phr of Phoschek P-30  
FSF-3, containing both additives at the indicated concentrations. The FSR additives were incorporated into the frothed formulation before the addition of PAPI and sodium silicofluoride.

The encapsulated Phoschek P-30 contained 80 wt-% poly(ammonium phosphate) and 20 wt-% of the encapsulating polymer. The 10 phr Phoschek P-30 content of FSF-2 refers to poly(ammonium phosphate).

### 2.1.2 Observations and Conclusions

All additives incorporated into the base formulation affected the set-up and curing characteristics of the foam. Some candidate smoke-retardant compositions containing cupric acetylacetonate were prepared in the initial phase of the work. This additive accelerated the set-up at room temperature, allowing only 45 seconds for the final mixing as compared with 90 seconds for the base formulation. When it was learned that this additive also catalyzed thermal degradation of the neoprene polymer at the oven curing temperature, its use was abandoned.

Ferric acetylacetonate retarded both gellation and the rate curing at the elevated temperature. The FSF-1 formulation was mixed for four minutes before pouring into molds. Since twelve hours of curing time were needed for this formulation, other compositions were also cured for the same duration.

Phoschek P-30 lowered the pH of the frothed foams and greatly accelerated the gellation. To enable incorporation of this fire retardant, it was encapsulated into partially hydrolyzed ethylene-vinyl acetate polymer. The encapsulation procedure is reported in Section 2.3. With encapsulated Phoschek P-30, a mixing time of 60 seconds could be used for the FSF-2 composition.

In the FSF-3 composition, the effects of ferric acetylacetonate and Phoschek P-30 were counteractive. Ninety second final mixing time was used, which is identical with that for the base formulation (BF).

Higher concentrations of the surfactants Aquarex WAQ and Triton-100 were used in the present work than found optimal with the base formulation (see Ref. 4). The increased concentrations were essential to stabilize the frothed foam prior to set-up.

It was experienced during the small scale preparations that the ambient temperature must be at least 23°C in the room in which the foam is prepared. At lower temperatures, the frothed foam coalesced before setting up.

## 2.2 PREPARATION OF NEOPRENE FOAM SPECIMENS FOR FIRE PERFORMANCE TESTING

To prepare test specimens for fire performance testing, the quantities listed for the recipe in Section 2.1 were quadrupled. The mixing and frothing operations were conducted with a Hobart mixer, equipped with a 5-gallon bowl. The foamed foam was poured into 20 in. x 7.5 in. x 2.5 in. cardboard molds, lined with polyethylene film. 18 in. x 6 in. x 1 in. and smaller specimens were cut from the cured foam slabs, using a bandsaw that was equipped with a thin-edged, sharp blade.

The sample frothing and refining times were 13 sec and 10 minutes, respectively, during the preparation of test specimens. The mixing times, after the addition of PAPI and sodium silicofluoride, were as follows: BF, 120 sec; FSF-1, 360 sec; FSF-2, 90 sec, and FSF-3, 90 sec.

The set-up time in the molds was 1 to 4 hrs at room temperature. The samples were removed from the molds and air-dried at room temperature overnight. All samples were subsequently cured in an oven at  $\sim 121^{\circ}\text{C}$  for 12 hours. The densities of cured specimens ranged from 6.3 to 7.7 lbs/cu ft.

## 2.3 ENCAPSULATION OF PHOSCHEK P-30

The following procedure was used for the encapsulation of Phoschek P-30, to prevent coagulation of neoprene latex upon the addition of this phosphorus-containing fire retardant. This procedure was adapted from a patented process (Ref. 7), that utilizes partially hydrolyzed ethylene-vinyl acetate as the encapsulating material.

Three liters of reagent grade toluene, contained in a 4-liter beaker, was heated to  $85^{\circ}\text{C}$ . A water bath was used for the heating. Thirty-five grams of partially hydrolyzed ethylene-vinyl acetate polymer (60/40 E/VA, 44% hydrolyzed) was added. With continuous stirring, the dissolution of the polymer required about two hours.

After the polymer has completely dissolved, 250 ml of cottonseed oil was added. The solution was then allowed to equilibrate thermally with the bath.

Three hundred and fifty grams of Phoschek P-30 (sieved previously to  $>200 < 100$  mesh size) was added, while providing efficient agitation.

The water from the heating bath was drained and an ice-bath was prepared to cool the suspension rapidly. Samples of the suspension were removed at intervals and observed under the microscope, to monitor the encapsulation process. Encapsulation began at temperatures below  $40^{\circ}\text{C}$ .

When the capsules had formed and the temperature had been lowered to below 20°C, 50 grams of Mondur CB-75 isocyanate was added to form a crosslinked polymer surface-network. Stirring was continued at room temperature for 24-36 hours to attain the desired crosslink density.

After the preparation, the encapsulated Phoschek P-30 was allowed to settle. The supernatant solution was decanted. The capsules were washed twice, with agitation, using additional quantities with toluene.

#### 2.4 TEST METHODS FOR THE FORMATION OF SMOKE AND GASEOUS COMBUSTION PRODUCTS

An analysis system capable of continuous measurement of CO, CO<sub>2</sub>, NO<sub>x</sub>, total hydrocarbons and oxygen (Ref. 8) during the burning of polymers was used in conjunction with smoke measurements. This system, designed and constructed at Monsanto Research Corporation (MRC), is connected to the NBS-Aminco smoke density chamber, that is utilized for the burning of samples under controlled conditions. The apparatus and the test methods were described in some detail in a preceding quarterly report (Ref. 1). The sample sizes and the test conditions were identical with those specified in that report.

### 3. RESULTS AND DISCUSSION

#### 3.1 FORMATION OF SMOKE

The primary objective of the present program is to lower smoke formation from burning PVC and neoprene polymer compositions. Concomitantly, enhancement of other fire performance characteristics (e.g., reduction of the rate of flame propagation) will be sought.

The results of smoke optical density measurements with the neoprene compositions are presented in Figure 1 and in Table I. Ferric acetylacetonate by itself and in combination with Phoschek P-30 reduced the smoke optical density generated from neoprene foam under flame exposure conditions by approximately 10%. Under nonflame exposure conditions, the smoke optical density was slightly increased by the incorporated additives. The data for all neoprene foam compositions evaluated in this program indicate their high propensity for smoke formation.

More effective crosslinking catalysts for the thermal degradation stage are needed than those used in the presently reported work.

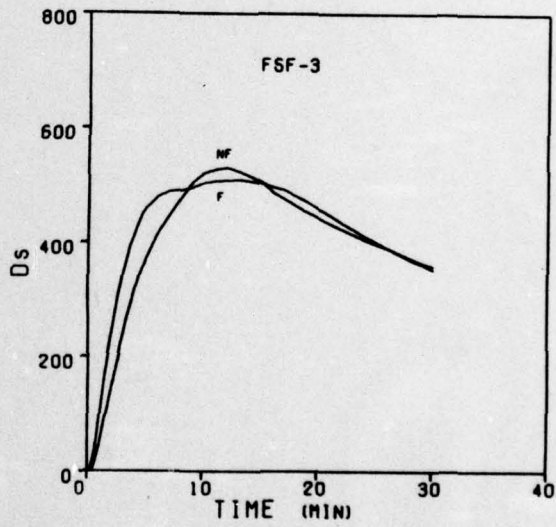
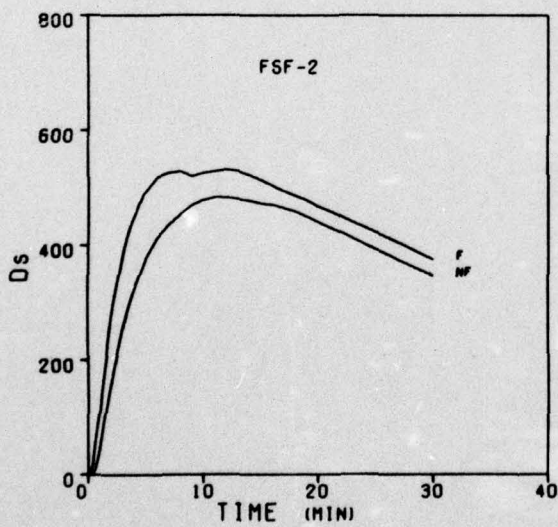
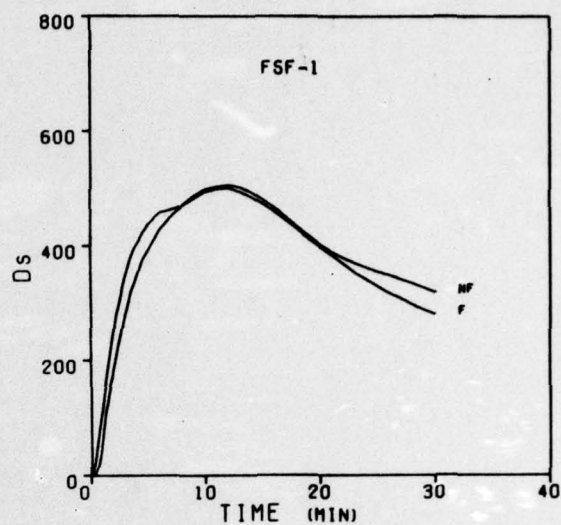
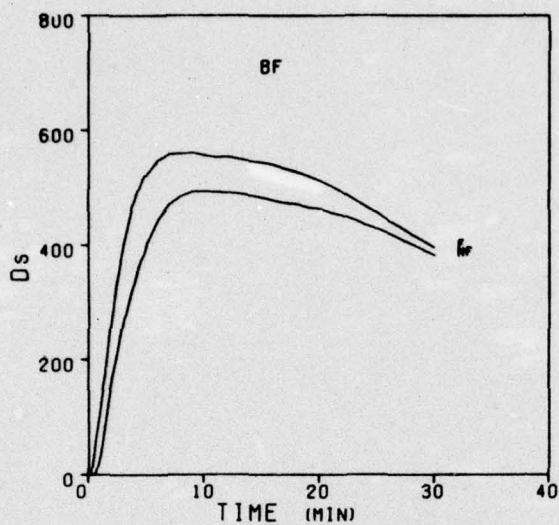


Figure 1. Smoke Optical Densities During the Burning of Experimental Neoprene Foam Compositions

Table I

SMOKE OPTICAL DENSITY RESULTS SUMMARY  
FOR NEOPRENE FOAM COMPOSITIONS<sup>a</sup>

<u>Material</u>	<u>Smoke Optical Density</u>						<u>Time to Maximum SOD (min)</u>	
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>			<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>		
BF	560	510	560	490	460	490	9	9
FSF-1 <sup>b</sup>	490	400	500	500	400	510	12	12
FSF-2 <sup>c</sup>	530	470	530	480	440	480	12	11
FSF-3 <sup>d</sup>	500	460	510	520	440	530	13	12

<sup>a</sup>Measurements conducted with 7.6 cm x 7.6 cm x 2.5 cm specimens in vertical orientation. Imposed energy flux in the center of the samples 2.5 watts/cm<sup>2</sup>. Foam density 7.0±0.7 lbs/cu ft.

<sup>b</sup>Contained 5 phr ferric acetylacetonate

<sup>c</sup>Contained 10 phr encapsulated Phoschek P-30

<sup>d</sup>Contained 5 phr ferric acetylacetonate and 10 phr encapsulated Phoschek P-30

### 3.2 FORMATION OF GASEOUS COMBUSTION PRODUCTS

The results of gaseous combustion products measurements are summarized in Tables II-VII, and presented graphically in Figures 2-7 in the Appendix. The following are the noteworthy findings pertaining to these measurements.

Ferric acetylacetonate alone increased carbon monoxide formation from neoprene foam under the conditions of nonflame exposure by ~40%. However, when this char formation catalyst precursor was used together with Phoschek P-30 in FSF-3, the catalytic effect for CO formation was destroyed. Ferric acetylacetonate had no adverse effect on CO formation under flame exposure conditions.

Carbon dioxide formation was slightly enhanced by the incorporated additives under both flame and nonflame exposure conditions.

The formation of hydrocarbons was reduced by ~45% by the incorporated additives in tests conducted under flame exposure conditions.

Small amounts of hydrogen cyanide were formed from the neoprene foam compositions used in this work. The incorporated poly-methylene polyphenyl isocyanate may have been the major source of this degradation product. It is noteworthy that ferric acetylacetonate, in the absence of Phoschek P-30, reduced the formation of hydrogen cyanide without a concomitant increase of nitrogen oxide.

### 4. ACKNOWLEDGMENT

The authors are indebted to Mr. J. T. Miller for assistance with smoke and gaseous combustion products measurements. They also wish to express appreciation to Miss K. A. Flayler for computerized data processing. The information provided about the preparation of neoprene foams by Dr. H. Burkheimer, duPont Company, was most helpful.

### 5. REFERENCES

1. L. Parts and C. A. Thompson, "Flame- and Smoke-Retardant Polymer Systems," First Quarterly Report on Contract N00024-76-C-5336, 15 September 1976.
2. L. Parts and C. A. Thompson, "Flame- and Smoke-Retardant Polymer Systems," Second Quarterly Report on Contract N00024-76-C-5336, 26 April 1977.
3. J. C. Carl, "Neoprene Latex," E. I. duPont de Nemours and Co., Elastomer Chemicals Department, Wilmington, Del., 1962.

Table II

SUMMARY OF CO CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS

<u>Material</u>	<u>CO Concentration (ppm)</u>						<u>Time to Maximum CO Concentration (min)</u>	
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>			<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>		
BF	1050	2000	2600	490	1400	2100	30	30
FSF-1	1200	2100	2700	900	2000	2900	30	30
FSF-2	900	1800	2300	550	1600	2200	30	30
FSF-3	930	1800	2300	680	1650	2100	30	30

Table III

SUMMARY OF CO<sub>2</sub> CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS

<u>Material</u>	<u>CO<sub>2</sub> Concentration (%)</u>						<u>Time to Maximum CO<sub>2</sub> Concentration (min)</u>	
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>			<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>		
BF	0.56	1.15	1.89	0.22	0.58	1.00	30	30
FSF-1	0.80	1.65	2.43	0.42	1.02	1.61	30	30
FSF-2	0.63	1.34	2.03	0.25	0.69	1.15	30	30
FSF-3	0.68	1.44	2.16	0.38	0.94	1.37	30	30

Table IV  
SUMMARY OF NO<sub>x</sub> CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS

<u>Material</u>	<u>NO<sub>x</sub> Concentration (ppm)</u>						<u>Time to Maximum NO<sub>x</sub> Concentration (min)</u>	
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>			<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>		
BF	12	19	25	1.8	3.6	5.4	30	30
FSF-1	11	18	23	2.2	3.7	5.3	30	30
FSF-2	14	23	30	3.8	6.4	8.7	27	30
FSF-3	14	23	30	3.7	7.7	10.1	30	30

Table V  
SUMMARY OF HYDROCARBONS CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS

<u>Material</u>	<u>Hydrocarbons Concentration (ppm)</u>						<u>Time to Maximum Hydrocarbons Concentration (min)</u>	
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>			<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>	<u>10 min</u>	<u>20 min</u>	<u>Maximum</u>		
BF	3700	5600	6600	2800	3200	3300	30	30
FSF-1	3300	3800	3900	3400	3700	3800	20	17
FSF-2	2200	2800	2900	2000	2700	2700	27	21
FSF-3	2600	3500	3800	2300	2700	2700	27	21

Table VI

SUMMARY OF HCl CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS

<u>Material</u>	<u>HCl Concentration (ppm)</u>					
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>		
	<u>5</u> <u>min</u>	<u>15</u> <u>min</u>	<u>30</u> <u>min</u>	<u>5</u> <u>min</u>	<u>15</u> <u>min</u>	<u>30</u> <u>min</u>
BF	930	2100	1550	1100	2100	1950
FSF-1	1050	2300	1550	1100	2000	1500
FSF-2	1500	1600	1550	1150	2300	2300
FSF-3	950	2000	1450	800	1650	1600

Table VII

SUMMARY OF HCN CONCENTRATION DATA FOR SELECTED TIMES  
FOR NEOPRENE FOAM COMPOSITIONS<sup>a</sup>

<u>Material</u>	<u>HCN Concentration</u>					
	<u>Flame Exposure</u>			<u>Nonflame Exposure</u>		
	<u>5</u> <u>min</u>	<u>15</u> <u>min</u>	<u>30</u> <u>min</u>	<u>5</u> <u>min</u>	<u>15</u> <u>min</u>	<u>30</u> <u>min</u>
BF	3.3	16.8	31.8			
FSF-1	1.5	1.7	9.1	1.8	2.3	5.9
FSF-2	3.2	8.4	36.8	4.6	16.6	40.2
FSF-3	4.6	37.6	61.3	2.2	36.9	53.6

<sup>a</sup>The hydrogen cyanide was formed from nitrogen-containing components, including the polyisocyanate, in the neoprene rubber composition.

4. Anon., "Standard Isocyanate-Modified Neoprene Foam Using Neoprene Latex 357," SD-2311, Second Revised Edition, E. I. duPont de Nemours & Co., Elastomer Chemicals Department, Wilmington, Del., July 4, 1976.
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APPENDIX

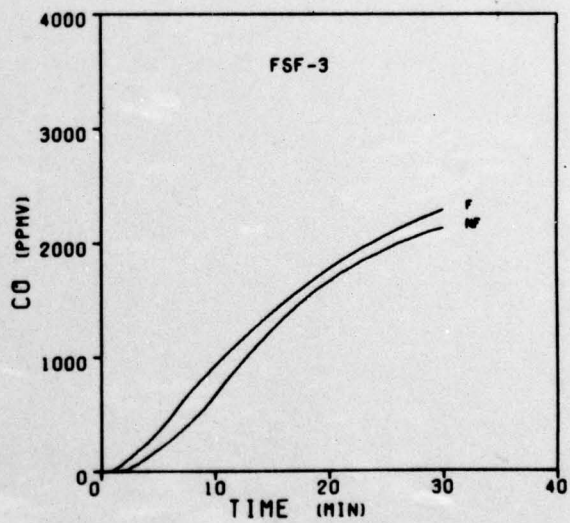
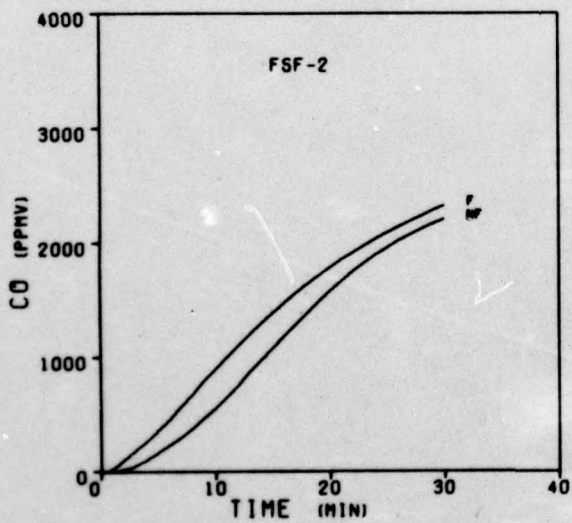
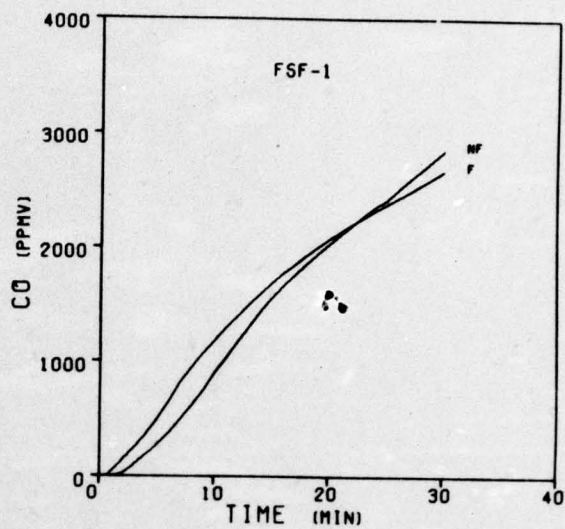
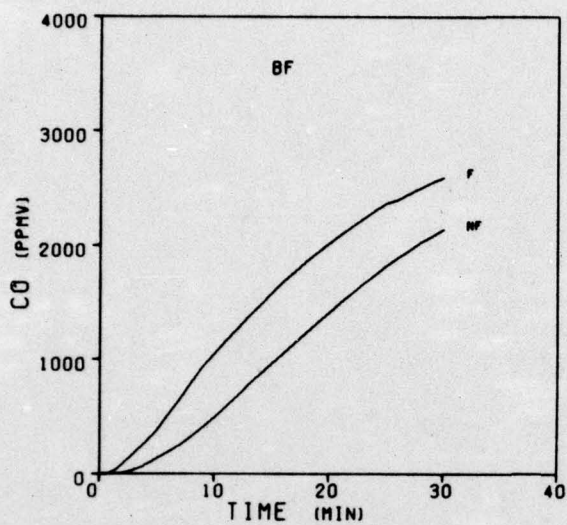


Figure 2. Carbon Monoxide Concentrations During the Burning of Experimental Neoprene Foam Compositions

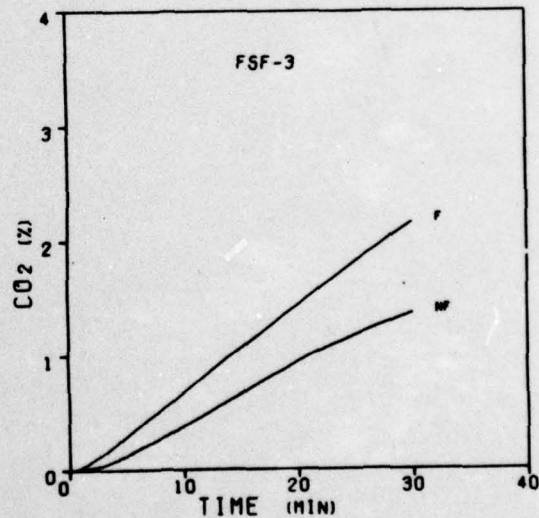
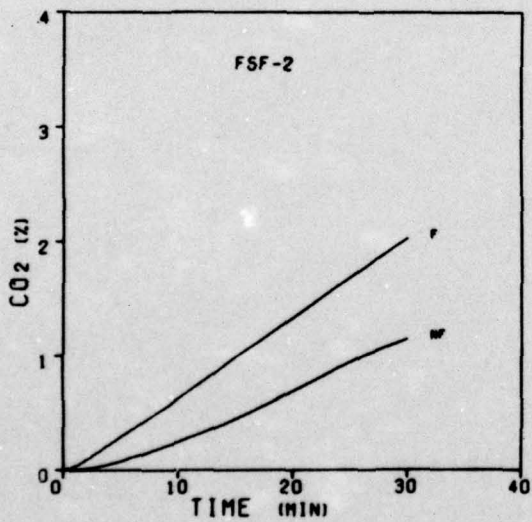
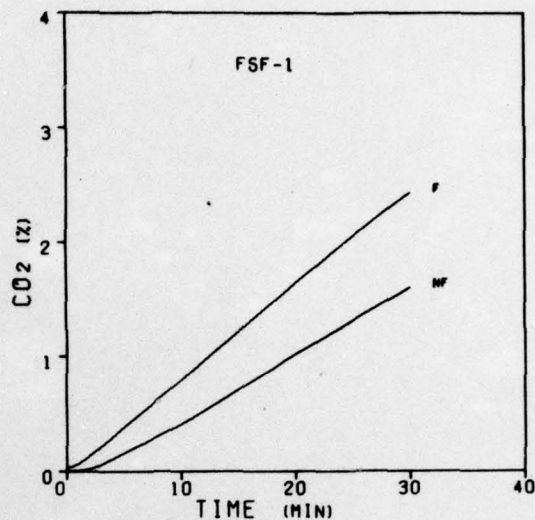
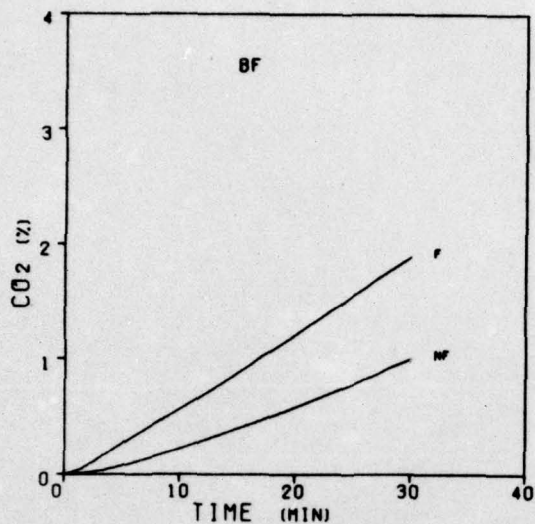


Figure 3. Carbon Dioxide Concentrations During the Burning of Experimental Neoprene Foam Compositions

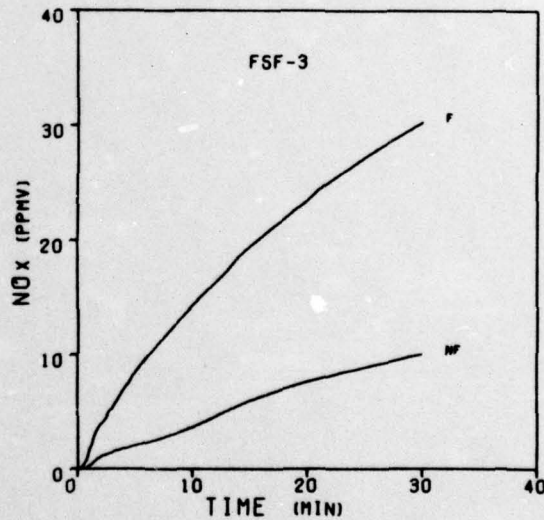
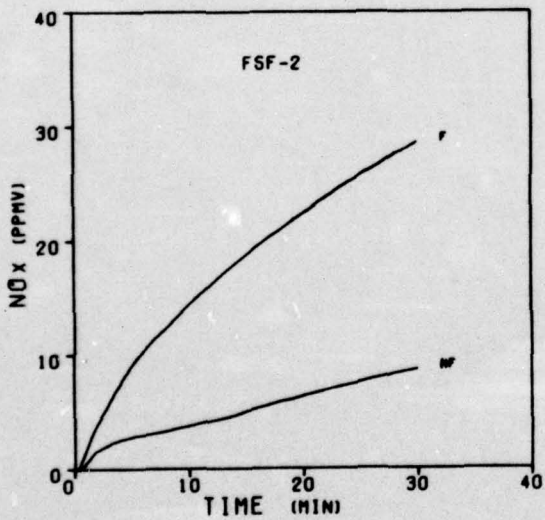
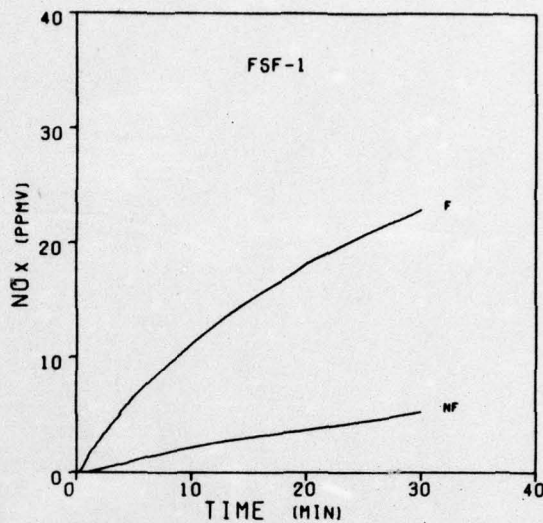
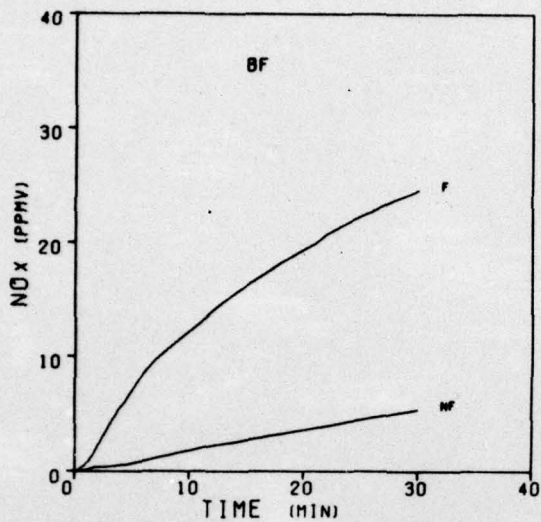


Figure 4. NO<sub>x</sub> Concentrations During the Burning of Experimental Neoprene Foam Compositions

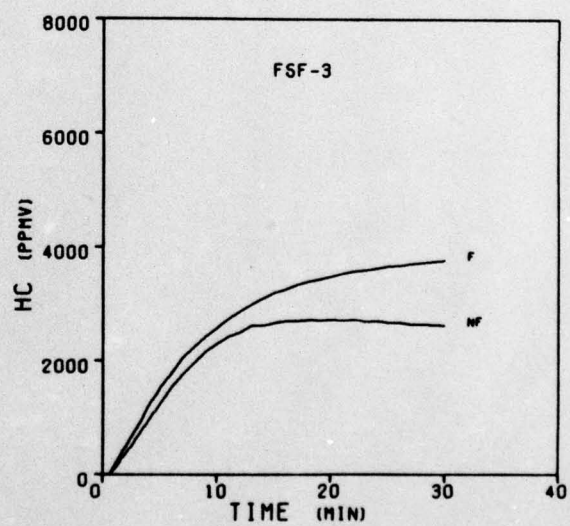
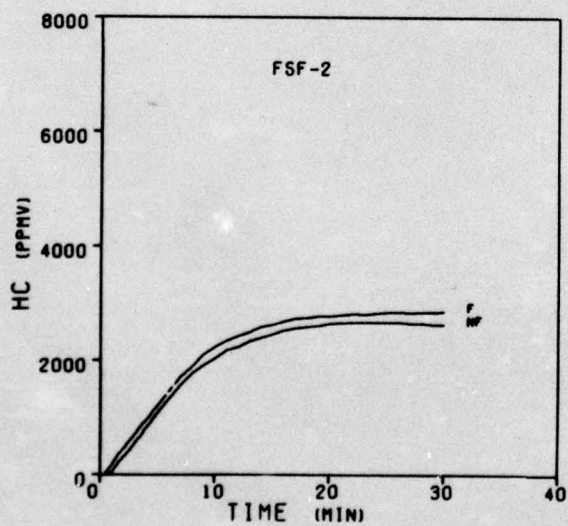
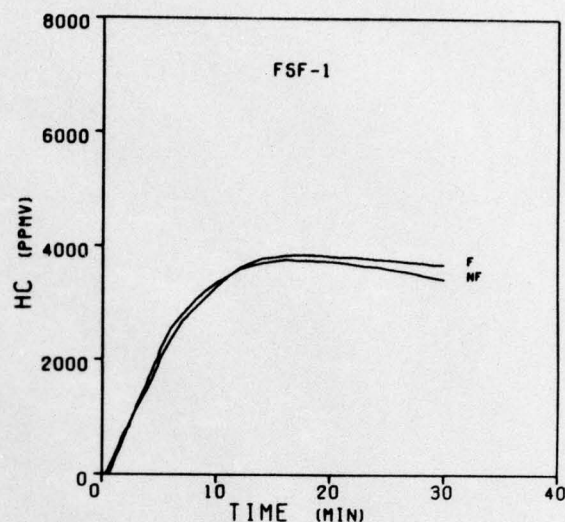
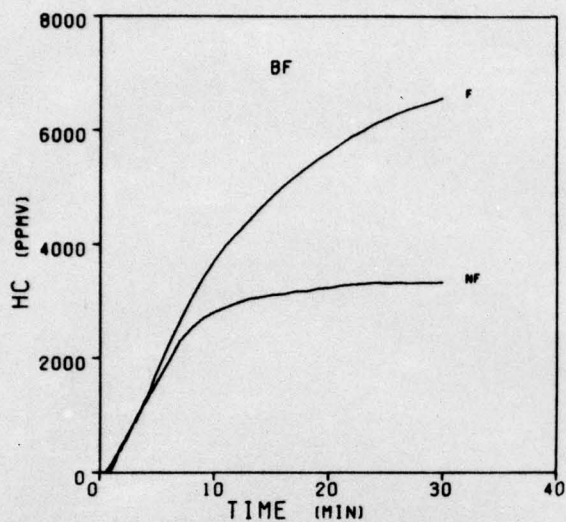


Figure 5. Hydrocarbons Concentrations During the Burning of Experimental Neoprene Foam Compositions

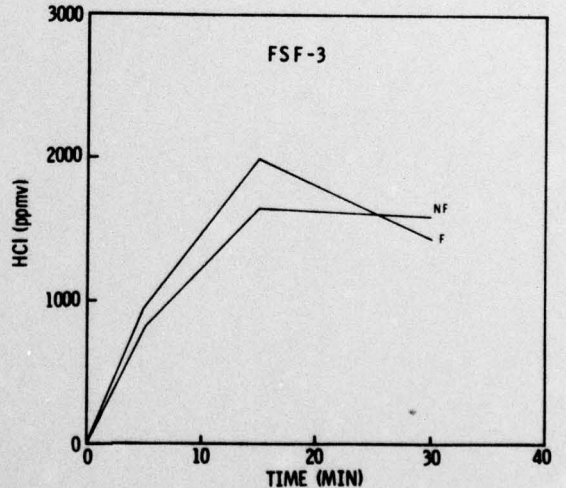
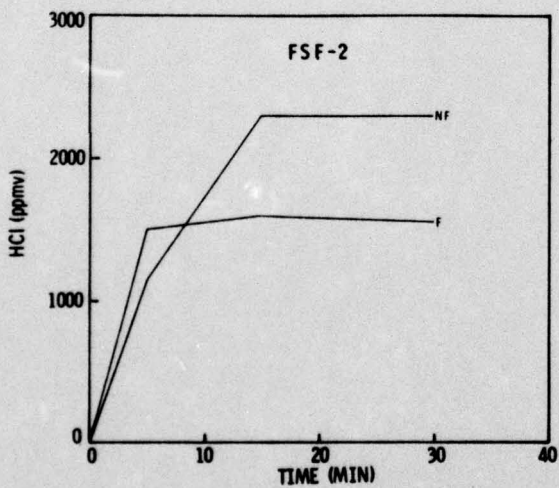
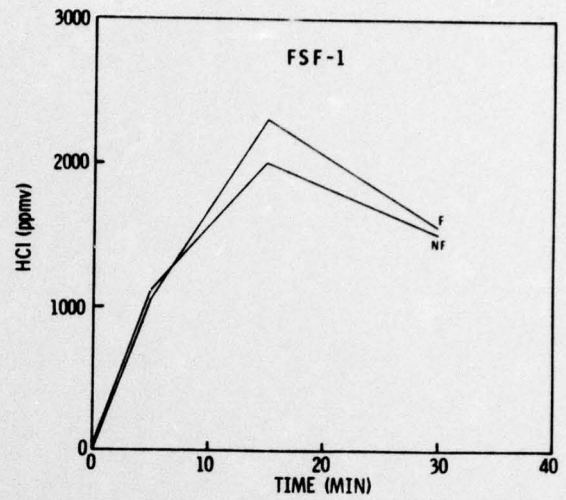
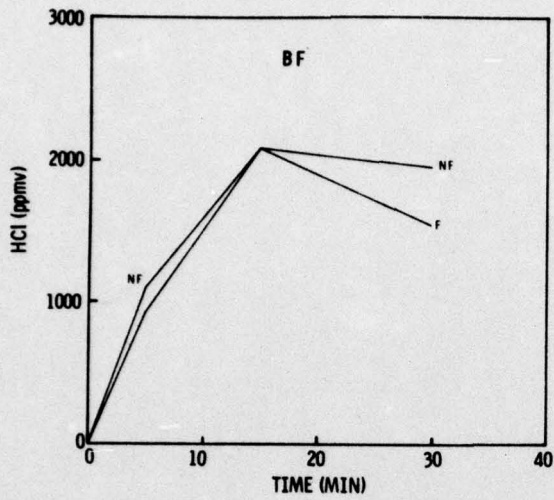


Figure 6. HCl Concentrations During the Burning of Experimental Neoprene Foam Compositions

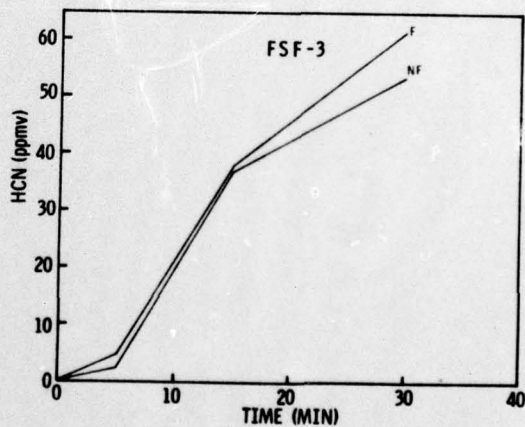
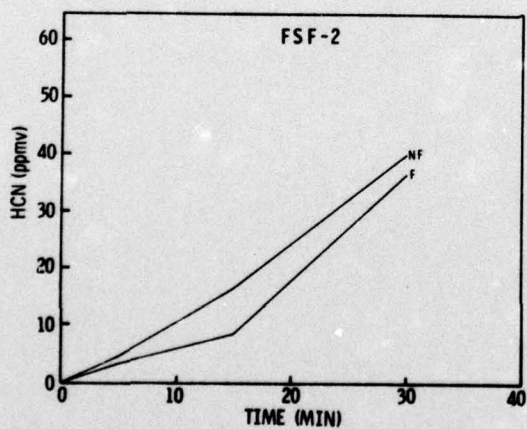
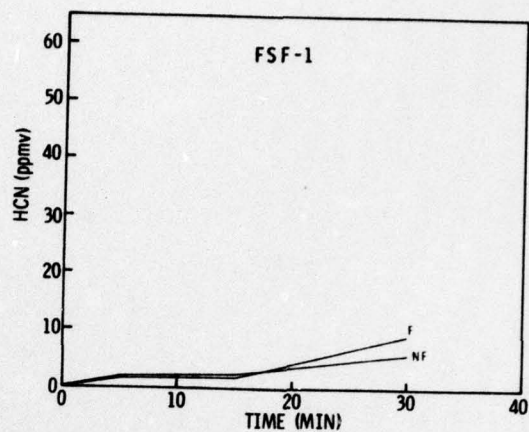
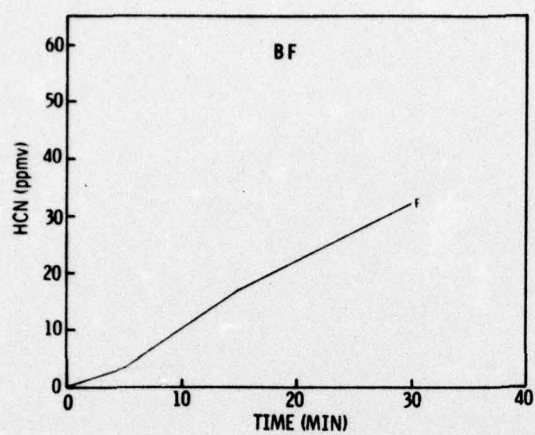


Figure 7. HCN Concentrations During the Burning of Experimental Neoprene Foam Compositions

Table VIII

SAMPLE MASS DATA

<u>Material</u>	<u>Sample Mass (g)<sup>a</sup></u>		<u>Consumed Mass (g)<sup>b</sup></u>	
	<u>Flame Exposure</u>	<u>Nonflame Exposure</u>	<u>Flame Exposure</u>	<u>Nonflame Exposure</u>
BF	17.67	18.14	12.73	11.22
	17.77	18.15	13.22	13.19
FSF-1	14.89	15.82		
	15.94	14.83		
FSF-2	15.47	14.92	11.61	9.99
	15.60	16.74	12.07	10.41
FSF-3	16.08	14.77	10.66	10.31
	14.12	14.71	9.79	9.72

<sup>a</sup>The dimensions of foam specimens were 7.6 cm x 7.6 cm x 2.5 cm.

<sup>b</sup>The chars formed from some specimens could not be recovered completely. Therefore, the data in the consumed mass column are incomplete.

Table IX

COMBUSTION PRODUCTS FORMED FROM BF  
UNDER FLAME EXPOSURE CONDITIONS

MATERIAL BF  
SAMPLE MASS AVG 17.7082  
IMPOSED EXPOSURE CONDITION FLAMING  
FLUX 2.5 W/CM<sup>2</sup>; VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	OS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NOX (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	115.	19.	0.03	150.	0.7	20.56
2.	274.	91.	0.08	515.	2.3	20.54
3.	399.	187.	0.14	874.	4.0	20.42
4.	478.	280.	0.21	1279.	5.6	20.37
5.	522.	386.	0.27	1773.	7.0	20.33
6.	548.	523.	0.33	2229.	8.5	20.21
7.	559.	653.	0.39	2646.	9.7	20.17
8.	560.	800.	0.45	3053.	10.6	20.08
9.	561.	939.	0.51	3413.	11.4	20.04
10.	556.	1052.	0.56	3712.	12.2	19.87
11.	553.	1163.	0.62	3985.	13.0	19.80
12.	554.	1268.	0.68	4201.	14.0	19.66
13.	550.	1378.	0.75	4412.	14.8	19.51
14.	546.	1481.	0.81	4627.	15.6	19.52
15.	544.	1585.	0.88	4828.	16.3	19.50
16.	540.	1683.	0.95	5017.	16.9	19.47
17.	533.	1772.	1.02	5183.	17.6	19.34
18.	527.	1860.	1.08	5341.	18.2	19.18
19.	520.	1942.	1.15	5489.	18.8	19.12
20.	512.	2021.	1.21	5625.	19.4	18.90
21.	502.	2097.	1.28	5764.	20.0	18.87
22.	492.	2167.	1.35	5904.	20.8	18.85
23.	480.	2237.	1.42	6002.	21.3	18.76
24.	468.	2307.	1.48	6116.	21.8	18.66
25.	456.	2371.	1.55	6214.	22.3	18.46
26.	442.	2400.	1.62	6297.	22.8	18.35
27.	431.	2454.	1.69	6376.	23.3	18.26
28.	418.	2505.	1.76	6437.	23.8	18.17
29.	407.	2555.	1.83	6505.	24.2	18.06
30.	395.	2593.	1.89	6565.	24.6	17.94

Table X

COMBUSTION PRODUCTS FORMED FROM BF  
UNDER NONFLAME EXPOSURE CONDITIONS

MATERIAL BF  
SAMPLE MASS AVG 18.1455  
IMPOSED EXPOSURE CONDITION NONFLAMING  
FLUX 2.5 W/CM<sup>2</sup>. VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NOX (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	36.	0.	0.00	56.	0.1	20.79
2.	169.	11.	0.01	497.	0.4	20.78
3.	271.	38.	0.03	662.	0.4	20.69
4.	338.	83.	0.05	1243.	0.5	20.68
5.	402.	139.	0.07	1590.	0.7	20.67
6.	445.	196.	0.10	1939.	0.9	20.65
7.	471.	257.	0.13	2312.	1.1	20.56
8.	485.	329.	0.16	2532.	1.3	20.41
9.	493.	410.	0.19	2698.	1.6	20.40
10.	493.	493.	0.22	2811.	1.8	20.45
11.	492.	592.	0.26	2891.	2.0	20.37
12.	491.	684.	0.29	2974.	2.2	20.35
13.	489.	783.	0.32	3038.	2.4	20.30
14.	486.	874.	0.36	3072.	2.6	20.26
15.	482.	965.	0.39	3108.	2.7	20.25
16.	477.	1057.	0.43	3121.	2.9	20.19
17.	473.	1145.	0.47	3165.	3.1	20.13
18.	471.	1233.	0.50	3174.	3.3	20.12
19.	465.	1324.	0.54	3219.	3.4	20.09
20.	463.	1409.	0.58	3234.	3.6	20.01
21.	455.	1498.	0.62	3274.	3.8	19.95
22.	450.	1581.	0.66	3285.	4.0	19.94
23.	443.	1666.	0.70	3301.	4.1	19.89
24.	434.	1742.	0.74	3320.	4.3	19.86
25.	427.	1818.	0.78	3314.	4.5	19.86
26.	419.	1890.	0.83	3316.	4.7	19.82
27.	409.	1957.	0.87	3310.	4.9	19.80
28.	400.	2023.	0.92	3316.	5.0	19.71
29.	390.	2081.	0.96	3323.	5.2	19.63
30.	381.	2138.	1.00	3332.	5.4	19.49

Table XI  
COMBUSTION PRODUCTS FORMED FROM FSF-1  
UNDER FLAME EXPOSURE CONDITIONS

PATRIAL FSF-1  
 SAMPLE MASS AVG 15.4184  
 IMPOSED EXPOSURE CONDITION FLAMING  
 FLUX 2.5 W/CM<sup>2</sup>, VERTICAL  
 DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
 -----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NO <sub>x</sub> (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	117.	36.	0.07	284.	1.4	20.80
2.	253.	145.	0.14	761.	2.8	20.75
3.	347.	250.	0.22	1157.	4.2	20.67
4.	403.	366.	0.31	1525.	5.5	20.52
5.	438.	499.	0.39	2010.	6.6	20.35
6.	459.	649.	0.47	2374.	7.6	20.15
7.	464.	819.	0.55	2695.	8.5	20.11
8.	470.	951.	0.63	2899.	9.4	20.10
9.	483.	1074.	0.71	3095.	10.2	19.96
10.	495.	1187.	0.80	3299.	11.2	19.78
11.	500.	1298.	0.88	3476.	12.0	19.60
12.	501.	1402.	0.97	3628.	12.8	19.38
13.	494.	1505.	1.05	3715.	13.6	19.45
14.	485.	1602.	1.14	3796.	14.3	19.26
15.	473.	1694.	1.22	3823.	14.9	19.13
16.	459.	1778.	1.30	3854.	15.6	18.99
17.	444.	1857.	1.39	3857.	16.2	18.86
18.	429.	1935.	1.47	3857.	16.8	18.77
19.	411.	2008.	1.56	3850.	17.4	18.64
20.	397.	2078.	1.65	3835.	18.2	18.53
21.	383.	2146.	1.73	3823.	18.8	18.41
22.	368.	2208.	1.81	3816.	19.2	18.33
23.	353.	2270.	1.90	3797.	19.8	18.13
24.	341.	2331.	1.97	3782.	20.2	18.04
25.	329.	2387.	2.06	3774.	20.7	17.94
26.	317.	2441.	2.13	3752.	21.2	17.80
27.	308.	2495.	2.22	3733.	21.7	17.64
28.	297.	2553.	2.29	3718.	22.0	17.50
29.	288.	2613.	2.36	3691.	22.4	17.40
30.	280.	2674.	2.43	3687.	22.9	17.40

Table XII

COMBUSTION PRODUCTS FORMED FROM FSF-1  
UNDER NONFLAME EXPOSURE CONDITIONS

MATERIAL FSF-1  
SAMPLE MASS AVG 15.3274  
IMPOSED EXPOSURE CONDITION NONFLAMING  
FLUX 2.5 W/CM<sup>2</sup>, VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NOX (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	76.	1.	0.01	238.	0.1	20.92
2.	190.	31.	0.02	706.	0.3	20.90
3.	283.	109.	0.06	1217.	0.5	20.85
4.	350.	197.	0.11	1671.	0.8	20.64
5.	393.	284.	0.17	2168.	1.1	20.52
6.	429.	384.	0.21	2570.	1.3	20.44
7.	452.	498.	0.26	2807.	1.6	20.42
8.	471.	619.	0.31	3030.	1.8	20.34
9.	487.	748.	0.37	3219.	2.0	20.27
10.	499.	905.	0.42	3373.	2.2	20.13
11.	504.	1037.	0.47	3485.	2.4	20.15
12.	506.	1177.	0.53	3606.	2.6	20.08
13.	501.	1307.	0.59	3665.	2.8	19.90
14.	493.	1438.	0.65	3721.	2.9	19.85
15.	480.	1560.	0.71	3752.	3.0	19.90
16.	466.	1668.	0.77	3780.	3.2	19.89
17.	449.	1767.	0.84	3762.	3.3	19.80
18.	432.	1860.	0.89	3761.	3.4	19.79
19.	416.	1950.	0.97	3754.	3.5	19.74
20.	401.	2034.	1.02	3743.	3.7	19.76
21.	388.	2119.	1.08	3716.	3.9	19.68
22.	378.	2200.	1.14	3686.	4.0	19.63
23.	369.	2282.	1.20	3659.	4.2	19.58
24.	361.	2360.	1.26	3647.	4.3	19.47
25.	354.	2418.	1.31	3611.	4.5	19.41
26.	348.	2515.	1.37	3576.	4.6	19.32
27.	341.	2599.	1.43	3549.	4.8	19.26
28.	334.	2681.	1.49	3502.	4.9	19.19
29.	326.	2763.	1.54	3456.	5.1	19.09
30.	318.	2857.	1.61	3433.	5.3	18.98

Table XIII

COMBUSTION PRODUCTS FORMED FROM FSF-2  
UNDER FLAME EXPOSURE CONDITIONS

MATERIAL FSF-2  
SAMPLE MASS AVG 15.5350  
IMPOSED EXPOSURE CONDITION FLAMING  
FLUX 2.5 W/CM<sup>2</sup>, VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NO <sub>x</sub> (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	109.	27.	0.04	173.	1.7	20.70
2.	272.	104.	0.09	455.	4.0	20.61
3.	383.	186.	0.16	706.	5.8	20.57
4.	451.	269.	0.23	963.	7.5	20.47
5.	494.	363.	0.29	1217.	9.1	20.40
6.	519.	461.	0.36	1473.	10.4	20.56
7.	526.	577.	0.43	1724.	11.5	20.47
8.	529.	680.	0.49	1905.	12.4	20.36
9.	520.	800.	0.56	2106.	13.5	20.25
10.	526.	902.	0.63	2240.	14.4	20.23
11.	529.	1006.	0.70	2350.	15.4	20.17
12.	532.	1104.	0.77	2428.	16.3	20.08
13.	529.	1206.	0.84	2499.	17.1	19.85
14.	522.	1299.	0.92	2591.	18.0	19.84
15.	513.	1391.	0.99	2630.	18.7	19.83
16.	503.	1477.	1.06	2693.	19.5	19.69
17.	494.	1560.	1.13	2736.	20.3	19.60
18.	486.	1639.	1.20	2754.	20.9	19.56
19.	478.	1713.	1.26	2777.	21.7	19.45
20.	467.	1786.	1.34	2783.	23.4	19.36
21.	459.	1853.	1.41	2804.	24.8	19.26
22.	449.	1916.	1.48	2819.	26.1	19.17
23.	440.	1977.	1.55	2813.	27.0	19.11
24.	431.	2034.	1.62	2832.	27.8	19.04
25.	422.	2089.	1.69	2846.	28.5	18.96
26.	413.	2140.	1.76	2850.	29.1	18.86
27.	404.	2189.	1.83	2840.	29.8	18.80
28.	394.	2237.	1.90	2854.	27.5	18.76
29.	384.	2283.	1.96	2850.	28.0	18.72
30.	375.	2325.	2.03	2857.	28.6	18.57

Table XIV

COMBUSTION PRODUCTS FORMED FROM FSF-2  
UNDER NONFLAME EXPOSURE CONDITIONS

MATERIAL FSF-2  
SAMPLE MASS AVG 15.8328  
IMPOSED EXPOSURE CONDITION NONFLAMING  
FLUX 2.5 W/CM<sup>2</sup>, VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NOX (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	49.	0.	0.01	62.	0.6	20.86
2.	164.	16.	0.01	320.	1.7	20.77
3.	256.	54.	0.03	557.	2.2	20.64
4.	322.	107.	0.05	825.	2.6	20.68
5.	378.	169.	0.08	1097.	2.8	20.66
6.	414.	235.	0.11	1341.	3.0	20.54
7.	436.	302.	0.15	1579.	3.2	20.53
8.	454.	381.	0.18	1779.	3.4	20.49
9.	470.	468.	0.22	1916.	3.6	20.38
10.	479.	555.	0.25	2040.	3.8	20.28
11.	484.	654.	0.29	2181.	4.0	20.11
12.	483.	753.	0.33	2244.	4.2	20.10
13.	480.	877.	0.37	2339.	4.5	20.11
14.	477.	977.	0.41	2403.	4.7	19.97
15.	472.	1079.	0.45	2458.	5.1	19.89
16.	470.	1183.	0.50	2525.	5.4	19.87
17.	464.	1286.	0.54	2568.	5.6	19.84
18.	458.	1385.	0.59	2593.	5.9	19.85
19.	449.	1486.	0.65	2610.	6.1	19.71
20.	439.	1578.	0.69	2651.	6.4	19.57
21.	430.	1667.	0.75	2658.	6.6	19.50
22.	421.	1749.	0.80	2665.	6.9	19.50
23.	411.	1825.	0.85	2670.	7.1	19.48
24.	401.	1893.	0.90	2667.	7.4	19.43
25.	292.	1957.	0.94	2672.	7.6	19.35
26.	383.	2018.	0.99	2669.	7.9	19.29
27.	373.	2068.	1.03	2653.	8.0	19.26
28.	364.	2119.	1.07	2650.	8.3	19.28
29.	355.	2163.	1.11	2626.	8.5	19.28
30.	346.	2203.	1.15	2630.	8.7	19.25

Table XV

COMBUSTION PRODUCTS FORMED FROM FSF-3  
UNDER FLAME EXPOSURE CONDITIONS

MATERIAL FSF-3  
SAMPLE MASS AVG 15.1045  
IMPOSED EXPOSURE CONDITION FLAMING  
FLUX 2.5 W/CM<sup>2</sup>, VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NO <sub>x</sub> (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	97.	13.	0.03	147.	1.7	20.81
2.	239.	83.	0.08	495.	3.9	20.63
3.	344.	167.	0.15	826.	5.3	20.56
4.	415.	251.	0.22	1207.	6.9	20.50
5.	461.	357.	0.30	1527.	8.5	20.39
6.	481.	471.	0.37	1778.	9.8	20.28
7.	490.	603.	0.45	2062.	11.0	20.16
8.	492.	727.	0.52	2247.	12.1	20.05
9.	497.	832.	0.60	2431.	13.2	19.98
10.	505.	935.	0.68	2584.	14.3	19.93
11.	508.	1035.	0.76	2737.	15.4	19.77
12.	509.	1128.	0.83	2869.	16.3	19.61
13.	510.	1222.	0.91	2989.	17.3	19.50
14.	507.	1311.	0.99	3103.	18.6	19.37
15.	504.	1397.	1.07	3195.	19.5	19.33
16.	497.	1481.	1.14	3259.	20.3	19.21
17.	492.	1562.	1.22	3345.	21.2	19.13
18.	481.	1636.	1.29	3398.	21.9	18.99
19.	471.	1710.	1.37	3443.	22.7	18.85
20.	459.	1780.	1.44	3493.	23.4	18.82
21.	448.	1844.	1.52	3542.	24.5	18.77
22.	435.	1907.	1.59	3577.	25.2	18.56
23.	424.	1964.	1.67	3601.	25.9	18.47
24.	412.	2017.	1.75	3631.	26.5	18.37
25.	402.	2072.	1.82	3672.	27.3	18.24
26.	391.	2121.	1.89	3680.	27.9	18.17
27.	381.	2166.	1.96	3713.	28.5	18.06
28.	371.	2208.	2.03	3731.	29.1	17.95
29.	363.	2249.	2.09	3755.	29.7	17.88
30.	353.	2289.	2.16	3773.	30.3	17.75

Table XVI

COMBUSTION PRODUCTS FORMED FROM FSF-3  
UNDER NONFLAME EXPOSURE CONDITIONS

MATERIAL FSF-3  
SAMPLE MASS AVG 14.7400  
IMPOSED EXPOSURE CONDITION NONFLAMING  
FLUX 2.5 W/CM<sup>2</sup>. VERTICAL  
DIMENSIONS 7.6 X 7.6 X 2.54

AVERAGE  
-----

TIME (MIN)	DS	CO (PPMV)	CO <sub>2</sub> (%)	HC (PPMV)	NO <sub>x</sub> (PPMV)	O <sub>2</sub> (%)
0.	0.	0.	0.00	0.	0.0	21.00
1.	47.	1.	0.01	96.	0.3	20.74
2.	146.	12.	0.02	381.	1.1	20.69
3.	238.	59.	0.05	668.	1.5	20.71
4.	319.	125.	0.09	958.	1.8	20.64
5.	373.	196.	0.13	1253.	2.1	20.54
6.	417.	268.	0.18	1542.	2.3	20.49
7.	448.	354.	0.23	1770.	2.7	20.36
8.	478.	451.	0.28	1966.	3.0	20.34
9.	506.	551.	0.33	2168.	3.3	20.27
10.	522.	677.	0.38	2302.	3.7	20.27
11.	528.	806.	0.44	2425.	4.1	20.23
12.	531.	920.	0.50	2505.	4.6	20.16
13.	524.	1033.	0.55	2619.	5.1	20.23
14.	515.	1142.	0.61	2625.	5.6	20.07
15.	506.	1247.	0.67	2667.	6.0	20.05
16.	487.	1345.	0.72	2697.	6.3	20.03
17.	476.	1437.	0.78	2701.	6.7	19.93
18.	464.	1522.	0.83	2718.	7.0	19.87
19.	453.	1598.	0.88	2712.	7.4	19.81
20.	443.	1663.	0.94	2718.	7.7	19.82
21.	433.	1735.	1.00	2706.	7.9	19.79
22.	424.	1790.	1.04	2718.	8.2	19.64
23.	415.	1850.	1.09	2693.	8.4	19.58
24.	406.	1897.	1.13	2703.	8.7	19.57
25.	398.	1944.	1.17	2670.	8.9	19.45
26.	391.	1993.	1.21	2669.	9.2	19.41
27.	382.	2028.	1.25	2643.	9.4	19.38
28.	373.	2069.	1.29	2642.	9.6	19.31
29.	365.	2107.	1.33	2631.	9.8	19.36
30.	359.	2131.	1.37	2622.	10.1	19.24

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Experimental neoprene rubber compositions were prepared to investigate the feasibility of reducing smoke formation and improving other fire performance characteristics with incorporated additives. Specimens were tested in an NBS smoke density chamber under flame and nonflame exposure conditions. The smoke optical density and the concentrations of CO, CO <sub>2</sub> , NO <sub>x</sub> , hydrocarbons, hydrogen chloride and hydrogen cyanide were monitored during these tests. Ferric acetylacetonate by itself and in combination with poly(ammonium phosphate) reduced smoke optical density generated from neoprene foam under flame exposure.		

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Item 20 Abstract (cont'd)

conditions by approximately 10%. Cupric acetylacetonate, another candidate char formation catalyst evaluated in this work, catalyzed thermal degradation of the neoprene foam.

Ferric acetylacetonate catalyzed the formation of CO under nonflame exposure conditions. This catalytic effect was destroyed by incorporating poly(ammonium phosphate) with ferric acetylacetonate.

Crosslinking catalysts more effective than those used in the presently reported work are needed to reduce smoke formation from neoprene foam significantly.

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