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## TEXTILES: SOME TECHNICAL INFORMATION AND DATA III: LOW FLAMMABLE AND OTHER HIGH PERFORMANCE FIBRES

by L. HUNTER

#### INTRODUCTION

Day<sup>1 a</sup> has given the following resumé concerning various concepts of textile flammability:

What is meant by the flammability of a textile material? What exactly are the meaning of such terms as "non-burning", "flame retarded", "fire resistant", "self-extinguishing", "non-combustible", "flameproof", etc? Unfortunately the flammability properties to which the labels are attached are not absolute values. For example, whilst material such as carbon or metals may be classified as non-flammable under normal ambient conditions, if the conditions are altered sufficiently (i.e. a 100% oxygen environment and/or elevated temperatures) even these materials can be made to burn. 1 a

Thus the flammability properties of a material usually refer to the value measured by some appropriate test designed to cover typical exposure conditions. This means that when a material is classified as self-extinguishing it means that it is self-extinguishing only in some standard test and under certain standard conditions. <sup>1</sup> a-

Many factors play an important part in the characterization of the flammability of material. The environment of the test for example plays an important role in a materials performance. Factors such as temperature, oxygen concentration and relative humidity are three which immediately come to mind. Usually materials are more flammable at higher temperatures than they are at lower temperatures. The fact that oxygen influences the flammability of a material was made clear by the tragic Apollo fire not many years ago when many "fire-proof" materials were rapidly consumed. Textile people all know the importance of relative humidity on the performance of textiles, especially in the case of the natural fibres which have high moisture regain values. Its importance in the flammability performance is well illustrated in the case of certain wool shag carpets. Legislation for carpets sold in Canada requires that all carpets be tested under bone dry conditions. Consequently the natural moisture content of the wool fibres (10-14%) is not available to prevent the spread of flame. If, however, these carpets were tested under higher relative humidity conditions the burning methenamine tablet employed as ignition source would not be sufficient to cause flame spread.

In addition to these environmental effects other factors are also important. For example, the orientation of the sample surface being tested can

influence the value given to the flammability property being measured. It is possible for a textile to be classified as self-extinguishing when tested in a candle-like manner since in this case the hot combustion gases are being removed in a direction opposite to that of flame propagation. However, when the material is tested in the vertical upward burning mode it is possible that the flame will be self-propagating since we now have a situation where the hot combustion gases are assisting the flame propagation. Similarly, if one were to test the flammability characteristics of a carpet on the floor and compare the results with those obtained on a ceiling, different results should be anticipated. In the former case the hot combustion gases are directed away from the surface helping to keep the surface cool, whilst in the latter case the hot gases are fed back into the surface causing an increased flammability of the subsurface.

The nature and duration of exposure to the ignition source can also influence the measured flammability of a material. A material may be non-ignitable by a match or a bunsen burner flame but may be readily ignited if exposed to the flame from a blow torch or crib fire.

Thus, it can be clearly seen that the use of such terms as self-extinguishing, fireproof, etc., are very misleading and should be avoided at all cost, since the flammability properties of a material are dependant on many external parameters over and above those of the actual material itself<sup>1a</sup>.

This publication, the third in the series, mainly deals with special fibres which have certain high performance properties, such as low flammability and high mechanical performance, for instance. Once again most of the information has been reproduced in the words or form used in the original source.

# FLAMMABILITIES AND FIRE RESISTANT FIBRE PROPERTIES IN GENERAL

## When Do Textiles Ignite?

Ignition occurs in polymeric systems when the concentration of combustible gaseous decomposition products in an oxidative environment near the sample reaches a characteristic explosive level. If a polymeric material is heated slowly enough, it decomposes without igniting because this critical concentration is never reached. The material will ignite only when (1) it has reached its thermal decomposition temperature, and (2) it is absorbing heat at a rate high enough to produce a combustible concentration of volatile fuel.

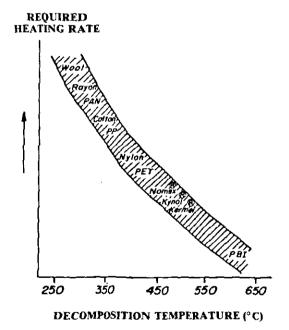
Using an experimental arrangement which they had developed, scientists at Textile Research Institute determined the minimum surface heating rate that produces ignition for a group of widely differing fabrics. For single-component fabrics an individual relationship is found between thermal decomposition temperature and this critical heating rate. In other words, the higher a material's

decomposition temperature, the less rapidly it needs to be heated to ignite.

This finding is consistent with the hypothesis that the rate of accumulation of ignitable volatiles is controlled by both the rate of polymer decomposition and the rate of molecular diffusion out of the decomposing solid.

As yet no available additive, whether it be another fibre, as in a blend, or a flame retardant, has been found to hinder ignition by increasing the heating rate requirement. On the contrary, a number of common flame retardants actually facilitate ignition of the fabric to a considerable degree.

In searching for ignition inhibitors, it should be remembered that an additive will be effective if it slows decomposition and/or interferes with diffusion out of the solid.



Relation between the critical factors for ignition for several fibres: minimum heating rate required during thermal decomposition vs. decomposition temperature!

	Burning Characteristics of Textile Fibres <sup>2</sup>
Fibre	Characteristics
Cotton	Supports combustion, burns rapidly, afterglows
Rayon	Supports combustion, burns very rapidly, no afterglow
Acetate/Triacetate	Supports combustion, melts ahead of flame
Nylon	Supports combustion with difficulty, melts and carries flame away in falling droplets
Nylon 6.6	Does not readily support combustion, melts and carries flame away in falling droplets
Acrylic	Burns readily with sputtering
Modacrylic	Melts, shrinks away from flame and sometimes burns very slowly
Polyester	Supports combustion with difficulty, melts and carries flame away in falling droplets
Polyolefin	Melts, burns slowly
Wool	Supports combustion with difficulty
Vinyon	Does not readily support combustion

## LIMITING OXYGEN INDICES AND SMOKE DENSITIES OF FABRICS3

Fabric	LOI <sup>a</sup> (% O <sub>2</sub> )	Specific O.D. <sup>b</sup>
Untreated Cotton	16-17	8
FR Treated Cotton	13-32	30
Modacrylic	30	72
Nylon	20	16
Natural Nomex	27-28	8 (0)*
Dyed Nomex	25-27	8 (5)*
Kynol	30-34	
Kermel	32	1
Durette	35-38	1
PBI	38-43	
Enkatherm	52	1

<sup>\*</sup> Non flaming

 <sup>(</sup>a) Candle-like burning mode, 153-220 g/m²:
 (b) Specific optical density as measured with the N.B.S. Smoke Chamber

## Burning behaviour of some textile fibres4

The following table illustrates the ignition temperatures and burning properties of some textile raw materials:

Fibre	Ignition temperature (°C)	Melting temperature (°C)	Burning behaviour	Heat resistance
Cotton	400		endothermic + 1)	good
Wool	600		endothermic - 2)	poor
Viscose	420		endothermic + 1)	good
Polyester	480	250	exothermic	poor
Nylon	530	220/260	exothermic	poor

<sup>1)</sup> burns quickly 2) burns slowly

## COMBUSTION PROPERTIES OF STAPLE<sup>5</sup>

Classification	Relative Flammability	Ignition Temperature °C	COMMENTS
Acetate	Moderate	438	Will not usually ignite with a match. A more sustained ignition source needed. Staple burns with melting, leaving a hard, brittle enamel-like residue.
Acrylic	Moderate	516	Ignites with match. Solid staple decomposing to a liquid, leaving hard brittle black beads.
Cotton	High	438	Ignites easily with match. A flash surface fire is followed by smoldering beneath the surface until cotton is consumed.
Modacrylic	Low	571	Supports combustion with staple melting and dripping under heat from ignition source. Flames die out after removal of ignition source.
Nylon 6.6	Low	485	Same as Modacrylic,
Olefin	Moderate	449	Ignites with match. Solid staple decomposing to liquid while burning, leaving hard black residue.
Polyester	Low	521	Supports combustion with staple melting and dripping under heat from ignition source. Flames die out after removal of ignition source.
Rayon Viscose)	High	460	Ignites easily with match. A flash surface fire is followed by complete combustion usually without leaving residue.
Triacetate	Moderate	485	Ignites easily with match. Solid staple decomposing to a liquid while burning leaving hard black residue.

## **COMBUSTIBILITY OF DIFFERENT FIBRES**

Kinds of Fibre	Value of LOI
Incombustible fibres	
Glass Fibre	
Asbestos	<del>-</del>
Metallic fibres	_
Heat resistant fibres	
Fluoric fibre	95%
Aromatic polyamide	33 — 34
"Kynol"	32 — 33
Flame retardant fibres	
Vinylidene chloride	45 — 48
Vinyl Chloride (PVC)	35 - 37
Polychlar	30 - 31
Modacrylic	27 - 31
Wool	24 — 26
Inflammable fibres	•
Polyamide	20 — 22
Polyester	20 — 22
Acrylic fibre	18 — 20
Polypropylene	19 - 20
Vinylon	18 — 20
Rayon	17 — 19
Cotton	17 — 19

# THERMAL PROPERTIES OF WOOL, POLYESTER FIBRE, AND NYLON\*6

Fibre	Ignition Tempe- rature (°C)	Melting Point (°C)	Flame Tempe- rature (°C)	Heat of Combus- tion (kJ/g)	LOI*
Wool	590		680	20,5	25,2
Polyester fibre	508	250	697	23,9	22
Nylon	510	255	875	33,1	20

<sup>\*</sup> As cited by Einsele

<sup>★</sup> LOI: Limiting-oxygen index

#### MINIMUM LOI VALUES<sup>7</sup>

Apparel	Children's wear Old men's wear Wigs Sleep wear	28 28 28 28
Interior goods	Curtain Carpet Wall cloth	29-30 24-26 25
Bedding	Bedding Blanket Sheets	24 24 24 24

Amount of combustion gas of various fibre <sup>8</sup>							
Temperature	Name of fibre	Amount of gas generated (p				)	
	, in the second	СО	CO2	NH <sub>3</sub>	HCN	H <sub>2</sub> S	
400°C	CONFX	536	370	0	0	0	
	Polyester	1166	510	0	0	0	
	Nylon	1030	135	12	0	0	
	Acrylic	1630	0	499	250	0	
	Vinylchloride	1967	615	0	) 0	) 0	
	Cotton	4000	3300	0	0	D	
	Wool	1000	1500	600	130	480	
			ı	1	6	1.	

# IGNITION AND SELF-IGNITION TEMPEATURES AND LOI VALUES FOR VARIOUS FIBRES<sup>9</sup>

Fibre	Ignition Temperature (°C)	Self-Ignition Temperature (°C)	LOI
Cotton	350	400	19
Triacetate	325	490	18.4
Polypropylene	375	495	18.6
Polyester	390	508	22
Nylon	390	510	20
Acrylic	250	515	18
Wool	325	590	25,2
Kynol	500	614	35
Enkatherm	- 560	637	52
Nomex	490	675	28,2
PVC-Fibre	-	_	37,1

Flame temperature of wool is 680°C. It yellows at about 100°C, heating for over 200 hours at 125°C causes a loss of approximately 10% in dry strength and 25% in extension. Heating at 180°C for over 24 hours causes a brown colouration and a loss of elasticity.

## FLAMMABILITY AND RELATED PROPERTIES OF FIBRES

(Compiled from references 8 and 10-16)

		[			
Durette	35-38				
H.E.I.M. (F.R. Polyester)	27,5		480	~~	
Vinylon	19,5				
Rhovyl	37				

LOI - Limiting oxygen index - Volume concentration of oxygen - Volume concentration of oxygen + Volume concentration of nitrogen x 100

- \* Woul flame resist treated with Titanium or Zirconium complexes. (The Zirconium-Tungsten treatment of wool is fast for at least 50 washes at 40°C, although it will not meet 50 washings at 60°C).
- \*\* Teklan a synthetic fibre produced by emulsion spinning a copolymer of acrylnitrite, vinyl chloride and vinylidene chloride.
- + Polychal a synthetic blend of PVA with a copolymer of PVA-PVC.

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## FLAMMABILITY AND RELATED PROPERTIES OF FIBRES

(Compiled from references 8 and 10-16)

Fibre	LOI (Limiting Oxygen Index)	Heat of Combustion (k cal/g.)	Ignition Temperature (°C)	Melting Point (°C)	Decomposition Temperature (°C)
Acrilan (Polyacrylnitrite)	18-18,2	7,6	465-530; 560	230-320	_
Cotton: Untreated	18-21	3,9	255; 400	Does not melt	_
Cotton: F.R. treated	26-32		_	Does not melt	
Triacetate	18-18,4		450-520; 540	293	_
Diacetate	18,6	_	450-540	255	
Acetate	18-19		480		
Polypropolene	18-18,6	11,1	450-540; 576	164-170	387
Rayon (Viscose)	18-20	3,9	570; 420	Does not melt	_
Polyvinyl Alcohol	19,7				·—
Nylon (Polyamide)	20-24	7,9	485-575; 530	160-260	
Nylon 6	24,5		530	223	
Polyester	20-24	5,7	485-560;	252-292	385
Wool	24,5-25,2	4,95	570-600;	Does not melt	
PFR-Rayon (Viscose)	26,26,4		_	Does not melt	
Modacrylic	26-32			160-190	
Nomex Nylon (P.A.) T450	27-32		440; 800	316	_
Wool Ti or Zr treated*	31,8			Does not melt	<u> </u>
Polyvinylchloride	37	5,1	_	100-160	<u> </u>
Fibrolane			625		
Teklan**			690	-	
Polychal +	28-32		500-600	<u> </u>	
Kermol	32	-			_
Conex	32-35		_		
Acrylic	18-19,5		560		
Dynel	27			<del></del>	_
Verel	30			_	
P.B.I. (Polybenzinidazde)	38-43				_
Kynol	29-34				
Durette	35-38		_	_	_
H.E.I.M. (F.R. Polyester)	27,5	-	480		
Vinylon	19,5	-		_	_
Rhovyl	37	_		_	

LOI — Limiting oxygen index — Volume concentration of oxygen Volume concentration of oxygen + Volume concentration of nitrogen x 100

<sup>\*</sup> Wool flame resist treated with Titanium or Zirconium complexes. (The Zirconium-Tungsten treatment of wool is fast for at least 50 washes at 40°C, although it will not meet 50 washings at 60°C).

<sup>\*\*</sup> Teklan - a synthetic fibre produced by emulsion spinning a copolymer of acrylnitrite, vinyl chloride and vinylidene chloride.

<sup>+</sup> Polychal — a synthetic blend of PVA with a copolymer of PVA-PVC.

## RESULTS OF THERMOGRAVIMETRIC ANALYSIS OF FIBROUS MATERIALS<sup>11</sup>

Materials	Ho (cal/g)	mp, (°C)	Range of pyro- lysis at the lst stage (°C)	Self- ignition point (°C)	Flash point (°C)	Residual carbona- ceous solids (%)	Rate of mass loss (%/°C)	Char residue at 600°C, (%)	Temp. of half mass (°C)
Polypropylene	11027	176	356-453	470	448	6,0	0,97	5,0	414
PS	9812	240	344-412	496	360	3,5	1,42	1,5	386
Nylon 6	7484	225	402-453	424	421	4,5	1,87	1,5	429
PMMA	6301	160	252-379	456	l —	0	0,78	o o	331
Polyacrylic	7448	317	306-340	_	331	74,4	0,75	57,7	741
Polyester	5462	267	388-433	485	349	13,0	1,93	1,0	423
Cotton	4180	l —	316-348	254	254	23,0	2,41	1,0	335
Triacetate	4524	306	327-366	540		18,0	2,10	3,0	350

Ho = Heat of Combustion mp = melting point

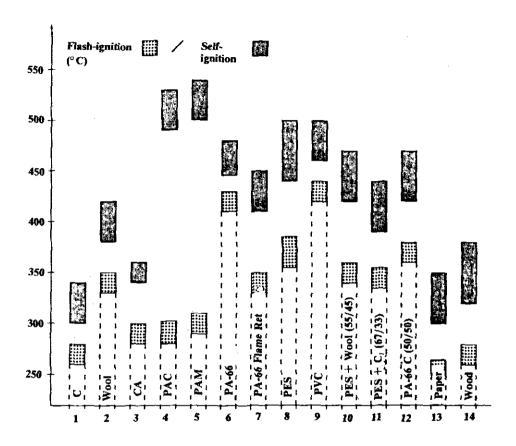
## SELF-IGNITION TEMPERATURES OF FABRICS<sup>17</sup>

	°C	°F
Cotton cloth	237	460
Wrapping Paper	252	485
Tissue Paper	221	430
Tobacco (cellulosic)	399	750
Polyester Fabric	302	575
Nylon Fabric	329	625
Acrylic Fabric	288	550

## TEMPERATURES OF IGNITION SOURCES<sup>17</sup>

°C	°F
704	1300
871	1600
816	1500
982	1800 max.
982	1800 max.
1427	2600 max.
1204	2200 max.
871	1600 max.
649	1200
1093	2000
816	1500
899	1650
	704 871 816 982 982 1427 1204 871 649 1093

# FLASH-IGNITION AND SELF-IGNITION TEMPERATURES OF VARIOUS MATERIALS<sup>33</sup>



C = Cotton; CA = Acetate; PA = Polyamide (nylon) PES = Polyester; PVC = Polyvinylchloride

# HCN FORMATION AS A FUNCTION OF DECOMPOSITION TEMPERATURES (PYROLYSIS IN AIR)<sup>3</sup>

Material	HCN (mg %)							
···	250°	350°	450°	550°	650°	750°	850°	
Cotton	35	10	15	15	15	30	50	
Wool	230	500	150	500	1500	1600	1300	
Paper	4	6	4	4	3	10	50	
Wood	20	20	15	15	7	80	70	
Acetate (CA)	20	5	10	10	4	10	5	
Polyester (PES)	3	30	10	5	5	5	5	
Acrylic (PAC)	1500	1500	600	3000	3000	4200	3000	
Nylon 6.6 (PA)	20	300	300	500	1500	1500	800	
Polyethylene (PE)	50	10	10	10	1		l —	
Tobacco	4	8	80	120	330	50	220	

# EMISSION OF CO AND CO<sub>2</sub> FOR DIFFERENT FIBRES UNDER VARIOUS CONDITIONS OF COMBUSTION

(15 min) <sup>54</sup>							
Fibre	% CO (Range)	% CO (Average)	% CO <sub>2</sub> (Range)	% CO <sub>2</sub> (Average)			
Cotton	9-54	26,4	38-129	80,5			
Cotton (flame retardant)	2-35	13,8	33-123	72,3			
Polyester	8-38	23,5	26-184	95,6			
Nylon 6	7-51	32,7	17-210	107,2			
Wool	5-37	19,0	33-148	81,2			
Aromatic Polyamide	8-58	17,7	12-230	109,9			
Acrylic	9-44	22,4	36-221	111,3			

# MAXIMUM, MINIMUM AND MEAN VALUES OF HCN IN % ON FIBRE MASS UNDER VARIOUS EXPERIMENTAL CONDITIONS<sup>54</sup>

Fibre	% HCN (Range)	% HCN (Mean)	Nitrogen content of fibre (%)	% N in HCN (max)
Acrylic	0-16,9	4,23	24,16	8,75
Nylon 6	0-10,1	1,93	12,3	5,23
Aromatic Polyamide	0-2,7	0,40	11,7	1,40
Cotton Flame Retardant	0-1,14	0,44	3,45	0,59
Wool	0-12,4	3,24	16,8	6,42

Агот. Ројувтіче	0	0	3 0,5  -	3,3
Polyamibimayloq	0	3	0,2	4 <u>.</u> .
Cplotvinyl (PVC)	0	5	20  -   45	6 46
Modacrylic	0 1,5 0	7 1,5 5	L4L	N 4 ∞
Acrylic	0,5	7, 2,6	8 6,5  -	*2 ° 0
9.9 nolųV	0	13 1,2 —	14 5	4.0
Polyester	0	<b>S1</b>	02	14
looW	00	15 1	15 3,5 —	11 5,5 —
Viscose	0,1	21	27	19 
потьо	0,2	22	29	20 
	CO HCN HCI	CO HCN HCI	CO HCN HCI	CO HCN HCI
	000°C	J ₀009	O08	1000°C

The following data concerning the temperature at which different fibres ignite, when dropped on a suitably heated hot place show that the ignition temperature of wool (i.e. 600°C) is higher than that of cotton and many of the synthetic fibres, except the last two<sup>12</sup>:

530°C
540°C
560°C
570°C
600°C
625°C
690° C

Teklan is a flame-proof synthetic fibre (Courtaulds) made from copolymer of acrylonitrile, vinyl chloride and vinilidine chloride. A recent zirconium tungsten treatment is fast to at least 50 washings at 40°C, although it will not meet 50 washings at 60°C.

The inherent flame-resistant fibres which possessed their flame-resistant properties because of their chemical composition are 1) poly (vinylchloride) and 2) aromatic polymers<sup>18</sup>. PVC fibres might be difficult to process because of their low initial modulus, although Leavil had a higher initial modulus produced by the use of a special catalyst. Yarn tenacities of PVC yarns were in the range of 8,4 to 13,1 cN/tex and breaking extensions in the range 25 to 30%. PVC fibres produced fabrics with good flammability properties but fabrics made from 100% PVC tended to be "dead". Work was done at LIRA, using blends of PVC fibres to improve fabric appearance, and this showed that in order to maintain good flammability properties, the blend should not contain more than 25% cellulose fibre my mass<sup>18</sup>.

Two kinds of flame retardant aromatic fibres are the aromatic polyamides and the aromatic polyamidimides, but the high cost of these fibres tended to restrict their use to protective clothing. Typical yarn tenacities were in the range 13,7 to 15,7 g/tex with breaking extensions from 8 to 12%<sup>18</sup>.

Fibres can be made flame-resistant by modification either via the use of additives in the pre-extrusion mix, or by copolymerisation with a flame-resistant additive. The flame retardant viscose fibre Darelle had been withdrawn from the market and this left a gap which hopefully would soon be filled. Whereas with other materials it was a case of the higher the mass per unit area the better the flame resistance, this was not so with modacrylic fabrics, which required careful control because they inconsistent in this respect<sup>18</sup>.

Tenacities of the modacrylics were in the range 7,5 to 13,2 cN/tex and breaking extensions from 10 to 30%. Other flame-retardant fibres, include asbestos, carbon, phenolic and poly terephthylol oxamidrazone<sup>18</sup>.

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## HIGH PERFORMANCE FIBRES<sup>3</sup>

Trade Name	Producer	Chemical Structure	Class
1 Nomex	Du Pont	-NH- O-NHC- O-CNH-	polyamide (aramid)
2 Fibre B	Du Pont	NH	polyamide
3 Durette	Monsanto -NH-	O CNH-O NHC-O NHC-O-C-	polyamide
4 Cornex	Teijin	Unknown	poly(amideimine)
5 PBU	USAF/Celanese		polybenzimidazole
6 Kermel	Rhodiaceta	-N < C \ C \ C \ N - \ O \ O \ O \ O \ O \ O \ O \ O \ O \	polyamideimíde
7 Enkatherm	Akzo	~ () - c, NH <sub>2</sub> NH <sub>2</sub> O ~ () ~	polyamidrazone
8 Kynol	Carborundum	Novolak Phenol-formaldehyde	cross linked phenolic

The modern durable flame retardant began in the early fifties with the patenting of THPC, and the main members of this family of FR products and processes were THPC-amide (heat cure); THPOH-NH<sub>3</sub> (NH<sub>3</sub> and heat cure); and THPOH-amide (heat cure)<sup>19</sup>.

These treatments may, however, produce too stiff a fabric and reduce tear and tensile strength, and additions were used to obviate this effect<sup>19</sup>.

In the early 1960's work led to Pyrovatex CP, which was based on the Arbuson reaction, and a series of dialkylphosphorocarboxylic acid methylol amides were prepared and assessed as flameproofing agents. It was concluded that the N-methylol compounds of dimethyl and diethyl phosphoropropionamide and the corresponding 1-methyl propionamides were the most suitable. Reaction with the fibre did take place but reaction via N-methylol compound was more practical. From this, the process based on Pyrovatex CP had been developed to give the following properties: good flame retardancy, no afterglow, good wash and dry clean fastness, acceptable handle and fabric mechanical properties.

The application of Pyrovatex CP should be to a thoroughly prepared neutral to slightly acid pH fabric, and consideration had to be given to the optimum add-on and the choice of resin, as the resin was required not only to take part in the Pyrovatex CP-Cellulose reaction but also to act as a nitrogen donor. Melamine resins and diacetylene urea have proved the most suitable for effect and fastness<sup>19</sup>.

The conclusions from one study<sup>20</sup> were that fibre thermal stability proved to be the most important parameter for designing a thermally protective fabric—the fabric must not melt, burn or shrink to provide maximum protection. There are two fibres able to meet this "all-important" objective, namely stabilised PBI and HT-4. The chemical stability at high temperatures was critical to the retention of structural stability in the fabric. Assuming equilibrium reactions between pyrolysis gases, the fire and air environments, it was concluded that these two fibres showed no indication of combustion, while the presently used Nomex fibre (for Air Force flight suits) was found to ignite around 550°C<sup>20</sup>.

In fabric design, three parameters exhibited significant potential for ensuring adequate thermal protection. These were (i) fabric mass had to be sufficient to provide a thermally protective barrier (for example woven fabrics made from either stabilised PBI, HT-4 or Nomex/Kevlar blends should be a minimum of 153 g/m<sup>2</sup>; (ii) fabric optical properties is an area where considerable improvement might be accomplished to achieve improved thermal protection; and (iii) fabric breakthrough time must be increased<sup>20</sup>.

#### Cordelan<sup>21</sup>

A fast growing flame resistant fibre of foreign manufacture is Cordelan. It is manufactured by Kohjin Company, Ltd. (Japan). It is a bicomponent fibre containing 50% Vinal and 50% Vinion. It may be dyed with selected cationic, disperse, or direct dyes. Dyes have a tendency, however, to exhaust fast on this fibre, i.e. they have a "high strike". Special retarders are required to slow down this strike, and to produce level dyeings when dyeing below the boil. Cordelan cannot be dyed at the boil, because it is sensitive to high temperatures. Dyeing temperatures should not exceed 90-93° C<sup>21</sup>.

A popular blend of Cordelan and cotton for knitted fabrics with good flame retardance is 80% Cordelan/20% cotton<sup>21</sup>.

Although disperse dyes have better lightfastness on Cordelan, select cationic dyes seem to be preferred, apparently for reasons of brightness and colour-yield<sup>21</sup>.

Kohjin Co has perfected a new fibre called Cordelan II. It is not a minor improvement over Cordelan I fibre, but a new formulation which is the subject of current patent applications<sup>22</sup>.

Fabric blend <sup>22</sup>		Limiting o	kygen index <sup>22</sup>			
Cordelan	Polyester	Cordelan				
%	%		II			
100	<u> </u>	32,3	35,2			
75	25	25,8	29,5			
65	35	23,6	27,3			
60	40	23,0	26,4			
50	50	22,0	24,6			
_	100		0,3			

When conventional apparel fabrics are ignited on the lower edge as in NFPA 702, the mass-to-burning time ratio is a straight line which varies very little with fibre type (except for wool)<sup>23</sup>.

The curve obtained when the mass of the fabric is plotted against burning time in the "forced" CS 191 test is parabolic because of the finite burnthrough time. This is most obvious in cotton samples which have a relatively long burnthrough time<sup>23</sup>.

Heavy cotton fabrics tend to burn on the upper surface only if they are ignited on the surface<sup>23</sup>.

Prolonged surface ignition of any conventional apparel fabric produces a longer burning time than that resulting from minimum ignition<sup>23</sup>.

With the exception of wool, most conventional apparel fabrics burn at

rates very much like those of cotton and all fabrics exhibit mass-to-burning retardency time ratios which are similar<sup>23</sup>.

## CELLULOSICS

Most man-made fibres producers now include some sort of flame retardant fibre in their programmes, particularly in staple form<sup>24</sup>. One problem with most of these is that they incorporate as the protective agent a halogen compound which, when exposed to flame or heat, can emit highly toxic hydrogen halide fumes. Now a completely new viscose rayon staple has been introduced by Chemiefaser Lenzing A.G., Austria. This does not incorporate this type of aid and so does not suffer from such a disadvantage. Initially the fibre is only available in dull, raw white<sup>24</sup>.

The production programme can be summarised as follows:24

dtex	Staple lengths
1,7	38 and 40 mm
3,3	60 and 80 mm
5,5	60 and 100 mm
8,9	80, 100 and 120 mm

Physical properties of the new staple show that it has a conditioned maximum tensile strength of  $22-24\,\text{cN/tex}$  and  $13-15\,\text{cN/tex}$  when wet, while the loop strength is about  $7\,\text{cN/tex}^{24}$ .

Basically the new fibre behaves much the same as conventional viscose and will run on three-cylinder, woollen and worsted ranges and spin and weave in much the same way as standard Viscolen and Viscolan from Lenzing. However, it is noted that in subsequent dyeing there is a tendency for the staple to have a somewhat higher uptake<sup>24</sup>.

#### PHYSICAL PROPERTIES OF FLAME RETARDANT VISCOSE STAPLE FIBRES 25

dtex	HL 22 1,7	HL 20 3,3	NL 18 5,5	NL 18 8,9
Max. stress per tex cond. (cN/tex)	22-24	23-25	15-17	15-17
dry (cN/tex)	12-14	13-15	7-9	7-9
Elongation at max. stress cond. (%)	13-15	14-16	22-25	22-25
Max. stress per tex in loop strength determination (cN/tex)	6-7	7-8	5-6	5-6
Wet modulus (cN/tex)	70	70	20	20
Brightness (% *1)	арргох. 60	approx.	60	60
Moisture absorption (%*2) LOI (%)	10-11 17-29	10-11 27-29	10-11 17-29	10-11 27-29

<sup>\* 1:</sup> brightness according to A. Berger

## FLAME RETARDANT AGENTS USED IN VISCOSE MANUFACTURE<sup>26</sup>

Flame retardants used are generally phosphorus, nitrogen or chlorine compounds and often the presence of two elements shows synergestic effects. The compounds used are mainly of three types;

- 1. organic phosphates or phosphonates and their halogen derivatives,
- 2. phosphoronitrile chloride derivatives,
- 3. other products.

In USA the firm FMC began production of flame retardant viscose rayon under the name. Avisco PFR. During the manufacture a liquid, water insoluble organic phosphorus containing agent is incorporated and 2-3% phosphorus content is quite adequate. The properties as given by manufacturers are:

Density	$1,45 (g/cm^3)$	
Tensile strength, dry	2,6-3 (cN/tex) $7$	gf/denier
Tensile strength, wet	1,4-1,8 (cN/tex)	<i>G</i> -1
Elongation	12-15%	
Decomposition temp.	270-280°C	
LOI	23	

<sup>\* 2:</sup> at a relative humidity of 55%

The table below<sup>27</sup> gives the minimum requirements for phosphorous and nitrogen to impart flame resistance.

Fibres	Mass of the fabric (g/m²)	Minimum phosphorous (% P)	Minimum nitrogen (% N)	Ignition time (s)	Burning time (s)	Char length (cm)
Ramie	215	1,37	0,62	8	<1	8,5
	175	1,37	0,62	6	<b>!</b> </td <td>11,5</td>	11,5
Cotton	250	1,65	0,74	} 8	3	5
-	150	1,65	0,74	6	2	11
Polynosic	78	2,45	1,10	4	1	12
Rayon	82	2,54	1,15	4	i	10

#### COMPARISON OF PHYSICAL PROPERTIES OF VARIOUS FIBRES<sup>28</sup>

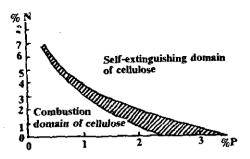
Property	DFG*	Cotton	Ordinary Rayon
dtex	1,89	1,67	1,67
Dry strength (cN/tex)	30	29	24
Wet strength (cN/tex)	24	30	15
Knot strength (cN/tex)	12,3	18,5	14,1
Dry elongation (%)	9,5	10.0	20,0
Wet elongation (%)	11,0	11,0	22,0
Relative Density	1,50	1,54	1,54
Wet modulus at 5% elongation (cN/tex)	8,8	8,8	2,6

DFG Daiwabo Flame Guard (a polynosic flame resistant fibre; LOI = 29-32 and is self-extinguishing.

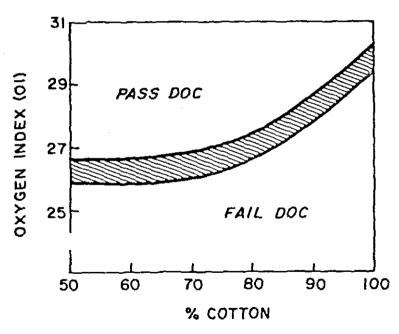
The antiflaming effect of phosphoric acid upon cellulose is due to the action of polyphosphoric acid<sup>29</sup>.

The rate of degradation of cotton by heat is accelerated by moisture<sup>30</sup>, e.g. a dry loomstate cotton fabric will withstand a temperature of 130°C for about two weeks before losing half its strength whereas in steam at the same temperature this loss in strength would occur in less than one day. The melting point of nylon is lowered by some 50°C in the presence of moisture<sup>30</sup>.

The following figure shows the effect of nitrogen on the minimum level of phosphorous required to flameproof cotton<sup>14</sup>.



The LOI values after 25X and 50X of sixteen fabrics treated at 3 or 4 levels of add-on, were plotted against the per cent cotton in the sample as shown below<sup>31</sup>:



LOI-DOC FF3-71) CORRELATION vs COTTON TREATED WITH THPOH-NH

Emission levels of CO, HCN and H $\tilde{C}l$  in parts per million are shown in the table below and the two figures illustrate the relationship between gas emission levels and fibre blend composition<sup>32</sup>.

# MAXIMUM SMOKE DENSITY AND TOXIC GAS EMISSION (in ppm) FOR "NBS" COMBUSTION OF VISCOSE BLEND FABRICS 32

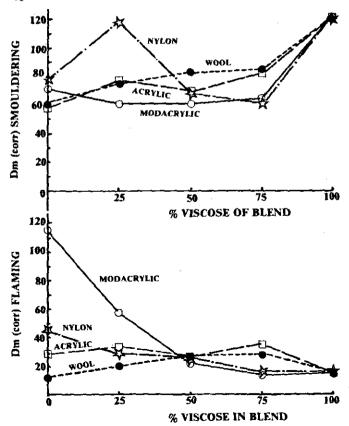
(V = viscose, M = modacrylic, A = acrylic, N = nylon, W = wool)

	Smouldering Mode				Flaming Mode			
Fabric*	Max Gas Emitted (in ppm)				Max	Gas Emitted (in ppm)		
	Smoke Density Dm	co	HCN	нсу	Smoke Density Dm	CO	HCN	нсі
100M	71	65	49	132	115	385	78	63
25V/75M	61	130	53	87	57	365	32	62
50V/50M	61	150	28	57	22	320	23	36
75V/25M	65	260	20	25	14	298	10	15
100V	122	380	0	0	15	150	0	0
100A z	58	6	22	_	28	37	5	} _
25V/75A		36	41		33	22	12	J _
50V/50A	, ,	105	34		27	70	11	
75V/25A	82	165	12		35	47	6	} –
100N	77	30	3		45	85	4	
25V/75N		45	3		28	95	3	<b>)</b>
50V/50N		43	ł ź		26	95	) 3	}
75V/25N	62	140	5	~	16	75	3	<b>)</b> —
100W ,, W001	62	39	6		12	132	2	{ _
24V/75W		91	6		20	177	5	<b>-</b>
50V/50W		181	l ž		27	140	11	l –
75V/25W		220	6		28	151	12	<u> </u>

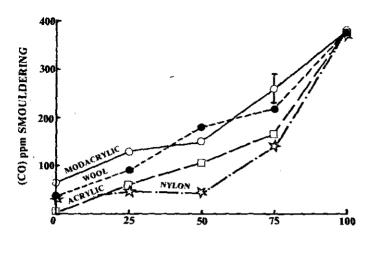
<sup>\*</sup>The smoke and gas data have been normalized to correspond to a 339 g/m² (conditioned mass) fabric³².

Some results for smoke density measurements obtained in another study are plotted against the blend composition below<sup>32</sup>. To render the measurements more self-consistent both smoke and gas data were normalized to that of a 339  $g/m^2$  (conditioned mass) fabric<sup>32</sup>.

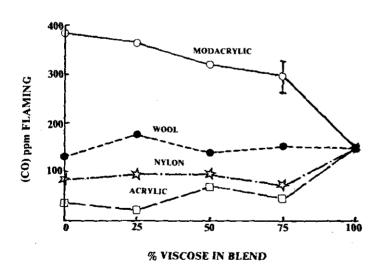
Regarding the smoke density data, with one exception (the 100% modacrylic fabric), smouldering conditions are seen to afford higher DM values than flaming conditions<sup>32</sup>. This is not surprising since smouldering is usually found to be the more smoky condition. Under flaming conditions the curves for nylon, acrylic and wool remain relatively flat through 0 to 100% viscose while the modacrylic, starting at a relatively high level, trends sharply downward with increasing cellulosic content. Under smouldering conditions the wool, acrylic and modacrylic curves remain relatively flat through 0 to 75% cellulosic content then trend sharply upwards reflecting the relatively smoky character of smouldering 100% cellulosic. The curve for nylon shows a peak at 75% nylon/25% viscose.



Maximum smoke density as a function of fibre blend normalized for a 339 g/m<sup>2</sup> fabric<sup>32</sup>



% VISCOSE IN BLEND



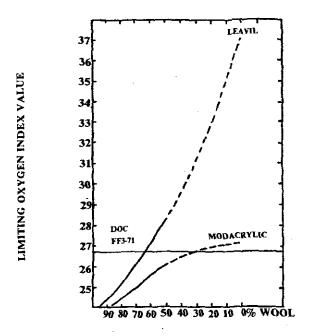
Carbon monoxide evolution as a function of fibre blend normalized for a 339 g/m² fabric³²²

Thermogravimetric data for polyester/cotton, polyester/acetylated cotton, tris treated polyester/acetylated cotton, and NaOH treated tris finished polyester/acetylated cotton are given below:

Fabric Detail Thermo- gravimetric Data	Polyester/ Cotton	Polyester/ Acetylated Cotton	Tris Treated Polyester/ Acetylated Cotton	NaOH Hydro- lyzed Tris Treated Polyester/ Acetylated Cotton
Onset Decomposition Temperature °C	360	363	263	275
Residue %	96	94,5	95,0	92,5
Inflection Point: Temperature °C	425	400	330	350
Residue %	54	65	28	48,5
Decomposition Completed Temperature °C	501	513	437	545
Residue %	16,1	16,0	12,3	17

#### WOOL AND WOOL BLENDS

LOI values obtained in one study for wool blended with either polyvinyl chloride or modacrylic are plotted against blend level in the figure below:33



Influence of wool content on the limiting oxygen index (LOI) of blends with Leavil (polyvinyl chloride) and Teklan (modacrylic) fibres<sup>33</sup>

#### **POLYAMIDES**

Since the decision banning Tris there has been increased interest in fabrics that are inherently flame resistant, without the application of a chemical flame retardant<sup>21</sup>. While there is a variety of flame resistant fibres on the market, the most popular for apparel are Du Pont's Nomex, Monsanto's SEF and Eastman's Verel. Kohjin's Cordelan is also finding acceptance, and this article includes a dyeing procedure for this material.

Materials such as Kynol, because of their deep natural colour, are not generally dyed, except in blends. PBI, developed for the Air Force, is a superior flame resistant fabric that can be dyed, but because of its expense must, for the time being, remain of limited commercial use. Rhovyl and Leavil, like Cordelan, are derived from vinyl chloride and may be dyed easily using proper methods.

#### Nomex

The most successful high temperature fibre so far developed is Du Pont's Nomex. Introduced in 1961 and commercialized in 1967, it was slow to catch the public's attention until garments made of it saved the life of a driver in the 1969 Indianapolis 500<sup>21</sup>.

Conventional polyamide fibres such as nylon 6.6 are linear aliphatic polyamides. Nomex belongs to a new family of polyamides that are completely aromatic and have the FTC generic designation of Aramids: a manufactured fibre in which the fibre forming substance is a long chain synthetic polyamide in which at least 85% of the amide linkages are attached directly to two aromatic rings. The mechanism by which these aromatic fibres resist high temperatures is a development of a dark crust on exposure to flame, which insulates and reflects heat. Char yield is high, shrinkage low and no flammable or poisonous volatiles are produced. The most important Nomex fibres offered to the public are now Nomex Type 450 for apparel. The latter may be the strongest contender for the sleepwear industry. It has the advantage that it may be dyed at atmospheric pressure<sup>21</sup>.

Unfortunately, the aromatic molecular structure that makes Nomex flame resistant also makes it extremely difficult to dye, primarily due to the absence of the dye receptors found in conventional nylon<sup>21</sup>.

Late in 1969, Arkansas Company developed a carrier called Dymex that made it possible to dye an almost unlimited range of colours on Nomex fibre and fabrics. The development, which permitted the dyeing of Nomex with cationic dyes, is patented, and still represents the only practical method of dyeing these fibres<sup>21</sup>.

Two problems that have not been completely solved in dyeing Nomex are light fastness and odour. Arkansas has developed products that reduce odour in the plant and residual odour in the fabric, but their cost is somewhat higher than that of the standard Dymex line. Other less costly formulations are now in the research and development stages<sup>21</sup>.

The most significant recent development is that Nomex 455 (Nomex E-10) can be dyed at the boil; at atmospheric pressure, using Dymex. This should substantially reduce finishing costs and may open up new apparel markets to the fibre<sup>21</sup>. The Du Pont fibre Nomex, was the first of the flame resistant fibres apart from the modacrylic fibres which are not strictly non-burning even under ordinary conditions; others to follow from the USA have been, not necessarily in chronological order, Durette from Monsanto, PBI (polybenzimidazole) from Celanese, and Kynol from Carborundum<sup>34</sup>.

Nomex was hailed as the answer to a prayer, but it was not the complete answer by any means. Wearers complained that it was uncomfortable because of its low moisture absorption and it certainly did not provide protection against radiant heat<sup>34</sup>. It could be dyed — green and black are the only two common

colours; but Nomex yellows badly and rapidly on exposure to sunlight and loses strength about twice as fast as nylons. So the search for a suitable flame-resistant fibre continued<sup>34</sup>.

PBI of all the synthetic fibres has good moisture absorption with a regain, under ordinary conditions, of about 13%. The manufacturers also claim the fibre, normally the typical golden-brown colour of most fibres of this group, can be dyed. But can it be called dyed fibre if it reverts from its green 'dyed' colour to its original gold after exposure outside for  $2^{1}/_{2}$  days?<sup>34</sup>

Durette comes in gold or black and from its chemical composition it probably behaves rather much like Nomex<sup>34</sup>.

Kynol is the weakest of these four fibres and although cloths have been made from pure Kynol it is generally blended with others, Nomex being a common choice. Kynol is available in several colours, all of them rather transient<sup>34</sup>. Another thing that these fibres have in common is poor resistance to solar degradation. PBI is about half as bad again as Nomex<sup>34</sup>.

Innovation in this area is still incomplete. Work is continuing to try to improve the properties of the fibres so that they may be more acceptable.

Work on wool is fairly recent. Wool has usually been looked upon as a safe fibre, but of course it will burn given the right conditions. And so we have PFZ (potassium fluorozirconate) treated wool which seems to be a very good prospect as a flame-resistant fibre with all the normal good textile properties. It is apparent however that this process still has to be perfected<sup>34</sup>.

To return to the high performance fibres, Kevlar, which is a very close relative of Nomex, has caused quite a bit of excitement. It was originally developed by Du Pont as a very high strength fibre for use in motor car tyres. At something in excess of 176 cN/tex its tenacity is twice as great as any textile fibre previously produced and it has the excellent heat-resistant properties so vital to good performance in heavy duty tyres<sup>34</sup>.

Kevlar has even poorer resistance to degradation by sunlight than PBI has, but it starts off so much stronger and it was not originally intended for outdoor use<sup>34</sup>.

On the other hand fibres such as polyethylene and polypropylene were developed with outdoor use uppermost in the minds of the manufacturers. And with them, especially polypropylene, came another innovation in the form of split film fibre which in its way is one of the most unusual developments in fibre production in recent years. It certainly simplified production greatly but it did nothing to help with the problem of solar degradation of polyolefin fibres. The introduction of ropes made from the polyolefin fibres has changed particularly the approach to fibres for marine rope use.

Various antistatic finishes have been applied to yarns and fabrics but it is doubtful, at least on the basis of laboratory tests, if these are of much value. A different approach has been to incorporate about 2% of stainless steel in a blended yarn. This certainly produces conducting yarns and cloths; perhaps

greatest success has been obtained in the synthetic pile of carpets where static at times can reach quite surprising voltages especially when relative humidity is low. Attempts to use the stainless steel blend in washable or dry-cleanable garments have not been as successful because the steel tends to powder under the vigorous action of cleaning and washing machines with loss of conducting power<sup>34</sup>.

The relationship between melting point and flameproofing in the case of nylon is illustrated below<sup>14</sup>:

Nylon cloth	m.p.(°C)	Flameproofing effect (vertical method)		
Nylon 6.6 original cloth	245-250			
thiourea (10,4% mass) ammonium thiocyanate	200-210	better		
,8% mass) nmonium sulphaminate	200-210	medium		
(11,3% mass)	235-240	good		
Nylon 6 original cloth	223			
thiourea (10% mass) ammonium sulphaminate	205	better		
(10% mass)	216	good		

## COMPARATIVE FLAMMABILITY PERFORMANCE OF NYLON 6.6 AND NYLON 6 35

Property	Nylon 6	Nylon 6.6
Fibre Melting Point (°C)	215-220	250-260
LRHI(W/cm <sup>2</sup> )	0,40	0,50
Burn Rate (in/min)	0,93	0,94
Smoke Evolution (arb. units)	97	117
Heat Output (arb units)	63	67

Aramid is an aromatic polyamide<sup>36</sup>. It is a man-made fibre composed of a long chained synthetic polymer whose ratio of amide bond directly combined with the two benzene nuclei is less than 85%.

Teijin manufactures this kind of fibre under the brand name "Conex"36. Polychal is a synthetic fibre manufactured by emulsion spinning after

blending PVA with copolymer of PVA-PVC. According to the definition of the quality indication law, polychal is a fibre composed of the long chained high molecular compound whose contents are more than 35% and less than 65% of PVC (-CH<sub>2</sub>.CHCl-) in terms of mass ratio and mainly vinyl alcohol (-CH<sub>2</sub>CHOH-). This fibre is produced by Kohjin Co. Ltd. under the brand name "Cordelan"<sup>36</sup>.

#### Phenol fibre:

This fibre is a synthetic fibre made of phenol resin and has excellent flame proofing and heat resistant properties<sup>36</sup>. In Japan, the definition for this fibre in the quality indication law has not yet been established. In the U.S. phenol fibre is called "Novoloid" whose definition is given as follows:

A man-made fibre whose cross linking novolak is more than 85% of the mass. Kanebo manufactures this kind of fibre called "Kynol"<sup>36</sup>.

TENSILE PROPERTIES OF 16.7 dtex NYLON 6.6 MONOFILAMENT<sup>37</sup>

Test conditions	20° C	65% RH	in liquid nitrogen
Extension rate (%. sec -1)	30	150	8,3
Breaking load, av. (cN)	101	107	157
Tenacity (cN/tex)	61	65	94
Breaking extension (%)	41	29	6

#### TENSILE PROPERTIES OF VARIOUS HIGH PERFORMANCE FIBRES<sup>3</sup>

Fibre	Initial Modulus (cN/tex)	Tenacity (cN/tex)	Elongation at break (%)
Nylon 6.6	~ 360	44-79	- 20
Kermel	~ 500	44	~ 12
PBI	~ 990	44	24
Durette	~1200	44	15
Nomex	~1350	44	~20
Glass	1800-3150	44-123	3
Steel	~2300	29-44	2
Fibre B	3600-9900	~ 194	~ 5
Carbon/graphite	18000-27000	44-53	0,5

## HIGH TEMPERATURE BEHAVIOUR OF HIGH PERFORMANCE FIRRES<sup>3</sup>

Fibre	% Tenacity retained a at 250° C	% Shrinkage at 450° C
Nylon 6.6	0	
Nomex	45	13
PBI	75	8
Durette (M3P)	50	3

(a) From the ratio of tenacity measured immediately on heating to  $250^{\circ}$  C to that at room temperature,

## COMPARISON OF THE PROPERTIES OF HIGH-PERFORMANCE FIBRES<sup>3</sup>

Fibre	Ultimate tensile strength* (cN/tex)	Breaking extension (%)	Relative Density	Stiffness (Young's Modulus) (cN/tex)*
Carbon	92-123	0,5	1,95	18 135-21 026
Glass (E type)	67	3,0	2,54	2 756
Steel	36-51	2,0	7,80	2 654
Nylon	79	14,0	1,14	614
Polyester	79	9,0	1,38	1 087
Kevlar (aromatic polyamide)	219	6,5	1,44	3 958

<sup>\*</sup>To convert to kN/mm<sup>2</sup> multiply by 0,01 x density

Note: The strength units (kN/mm<sup>2</sup>, are kilonewtons per square millimetre: One Newton is close to 102 grammes force.

Carbon fibres are very resistant to heat in inert conditions, but they begin to oxidize in air at 410-450°C.

The melting point of Kevlar aramid fibre (610°C), and high T<sub>g</sub> (375°C), the high thermal stability and low combustibility of this fibre, its low density and good dielectric properties, make this fibre most suitable for protective garments<sup>38</sup>. Its high strength (26,5 to 53 cN/tex) is 5 times higher than that of steel.

Kevlar was compared to nylon and to polyethylene in its creep-failure time determinations, and it was found that even with a large safety factor, the "life time" of Kevlar is much higher than that of the other fibre types. The durability of the tensile properties is also maintained in sea water, so that Kevlar is applicable in long cables. It has been pointed out that filament extensibility was 4-4,5%, and in bundle form 3,5-4%. Some medical applications of Kevlar could also be envisaged<sup>38</sup>.

Fibre <sup>39</sup>	Boron	Thornel	Kevlar
Relative density	2,45	1,78	1,45
Strength x 10 <sup>3</sup> PSI	515	425	400
cN/tex	145	165	190
Modulus x 106 PSI	56	34	20
cN/tex	15771	13180	9517

Tensile strength (lb/in<sup>2</sup>) =

12 800 x density of fibre (gf/den)

so tenacity (gf/den) = 
$$\frac{\text{Tensile strength (lb/in}^2)}{12\ 800\ x\ \text{density}}$$

The density of Kevlar 49 is  $1,45 \, \text{g/cm}^2$  and its tensile strength can be taken as  $400 \, 000 \, \text{lb/in}^2$ , so that

Tenacity (gf/den) = 
$$\frac{40\ 000}{12\ 800\ x\ 1,45}$$

$$= 21,6 = 191 \text{ cN/tex}$$

This is an extraordinarily high strength; more than twice as high as that of the strongest nylon. Coupled with it is the very low extension at break of just under 2%. The fibre is very stiff; it is 20 times stiffer than nylon, and twice as stiff as glass fibre. Kevlar 49 fibres have excellent toughness leading to a high impact strength and also to good textile processing. Under high compression loads the fibre has an almost perfect plastic behaviour and this gives its properties some resemblance to the ductility of metals.

#### KEVLAR YARN PROPERTIES<sup>40</sup>

	Keviar	Industrial Nylon	Industrial Polyester
Tenacity (cN/tex) Modulus (cN/tex) at 20°C Modulus (cN/tex) at 200°C	185	87	81
	3974	486	971
	2649	177	177

Kevlar was initially called Fibre B

Teijin, Japan, is now making international marketing efforts with its Teijin-Conex fibre, a wholly aromatic polyamide item which must compete directly with such fibres as Du Pont's Nomex<sup>41</sup>.

Teijin-Conex is resistant to temperatures of 100°C to 250°C, depending upon environmental conditions; it is self-extinguishable and 'may be referred to as fire-retardant'. Initially, the fibre was available in 2,2 5,6 and 11,1 dtex but now a 1,7 dtex version has been added to the range. Staple length can vary from 50 mm to 150 mm with 76 mm being the most widely used for needled fabrics such as papermakers felts.

Teijin Conex:	Fibre prope	rties	2,2 dtex	5.6 dtex	II,I dtex
	- Tote prope		51 mm	76 mm	76 mm
Tensile streng	th (cN/tex):	Standard	44-49	44-49	40-44
		Wet	35-42	35-42	32-35
Knot strength	Knot strength (cN/tex)		34-37	34-38	34-37
Loop strength (cN/tex)			35-39	35-39	35-39
Elongation (%) Standard			35-45	35-45	40-50
		Wet	40-50	40-50	45-55
Initial modulus (kg/mm²)			800-1000	700-900	700-800
3% Stretch elasticity (%)			75-80	75-80	70-75
	Numbe	r of Crimps			
	(/25 mm)		(/25 mm) 12-14	10-12	10-12
Crimp Proper	ties Degree	of Crimp (%)	15-20	15-20	17-22
	Residu	al Crimps (%)	12-16	12-16	14-18
Whiteness	Whiten	ess (W)	80-82	78-80	76-78
	Yellow	ness (b)	9-11	9-11	
Dry heat shrin	rinkage at 300°C (15 min) (%)		4-6	4-6	4-6
Specific resista	ince of electi	ricity(Ω)	x 10 -7	x 10 -7	х 10-7
Frictional	Against	0 cm/min	0,31	0,33	0,34
Coefficient	fibre	90 cm/min	0,26	0,28	0,26
	Against	0 cm/min	0,16	0,17	0,22
·	metal	90 cm/min	0,18	91,0	0,19

A range of 20 spun-dyed colours is available, minimum quantity in any one colour being 2 000 kg. Some variant versions of the standard fibre have been developed to offer even greater heat resistance, or better resistance to sunlight degradation<sup>41</sup>.

## **POLYESTERS**

## MECHANISM OF FLAME RETARDANCE OF POLYESTER FIBRE<sup>42</sup>

- A Increasing self-extinguishing properties by flame retardants, such as halogen or phosphorous compounds (Higher LOI is required)
- B Increasing melting properties to drop out melt fibres (Higher LOI isn't always required).

# FIBRE PROPERTIES OF EXTAR\* AND REGULAR POLYESTER<sup>42</sup>

	Tetoron Extar	Reg. polyester
Linear density (dtex/f)	83/24	83/36
Tenacity (cN/tex)	29-36	42-49
Elongation (%)	23,5-38,5	22,0-36,0
Loop Tenacity (cN/tex)	23-28	33-38
Loop Elongation (%)	7-13	6-12
Initial modulus (Kg/mm²)	1000-1200	1200-1400
Elastic recovery (3%)	99-100	99-100
Elastic recovery (5%)	95-98	95-98
Elastic recovery (10%)	68-71	68-71
Shrinkage in boiling water (%)	6,5-8,5	8-10,5
Shrinkage in an oven at 150°C	8-1 ť	9-12

<sup>\*</sup>A flame retardant polyester fibre

# FLAMMABILITY OF LACE CURTAINS (TETORON EXTAR)<sup>22</sup>

	45°	C micro burne	Coil test		
	Max. char area (cm²)	Max. after flame (sec)	Max. after glow (sec)	Min. number of impinge- ment	Judgement
Reg. polyester before dyeing	5,2	0	0	1	fail
Tetoron Extar: before dyeing	5,0	0	0	4	pass
Fluorescent dyed	5,3 5,0 5,7 5,4	0	0	4	pass
Light dyed	5,0	0	0	4	pass
Medium dyed	5,7	0	0	4	pass
Deep Dyed After 5 cycle	5,4	0	0	4	pass
laundering After 5 cycle	5,1	0	0	4	pass
dry-cleaning	6,1	0	0	4	pass
Acceptance criteria	≤30	≤3	≲5	≥3	_

# EFFECT OF WATER HARDNESS ON FLAMMABILITY<sup>42</sup> (TETORON EXTAR)

Water	45° micro	burner test	Coil test		
Hardness (ppm) Char area (cm²)		After flame (sec)	No. of impingement	Judgement	
0	7,9 9,0 6,2 6,3 5,0	000	7;5;5;5;5	pass	
50	6,8 6,7 4,3 5,4	0 0 0	5;4;5;5;5	pass	
70	6,7 6,2 6,0 6,3	000	5;5;5;4;5	pass	
90	eo 18,3 eo	0 0 21,5 34,5		fail	
120	ou 6,3 ou	0 0 60 47	<del>-</del>	fail	

# FIBRE PROPERTIES - DACRON TYPE 900F43

	Type 900F	T-54	T-64
Tenacity (cN/tex)	35	41,5	30
Elongation (%)	40	35	34
Tenacity at 7% elongation	0,8	1,1	1,1
Modulus (cN/tex)	291	371	397
Shrinkage (%)			
Boiling water	1,0	1,0	1,0
Dry Heat (196°C)	9,5	6,5	5,0
Melting Point (°C)	235	255	252
Relative Density	1,41	1,38	1,38
Dyeability	Disperse	Disperse	Basic and Disperse
Surface	Transverse Striations	Smooth	Smooth

#### **ACRYLICS AND MODACRYLICS**

## Modacrylics21

Most successful of the flame resistant modacrylics has been Monsanto's SEF (Self Extinguishing Fibre). One of the reasons for the fibres immediate success was its ease of dyeing. It can be dyed using exactly the same dyes, auxiliaries and methods applied to non-flame retardant acrylic fabrics. No strong or odouriferous dye carriers are necessary, and the fibre has a good, off-white colour of its own.

SEF, in fact, is so successful that not enough of the 100% fibre can be produced. Thus it is prevalent in flame resistant blends. One of these is 65% SEF and 35% Spectran (Monsanto) polyester. This blend is much less expensive than Nomex or 100% SEF yet provides substantial flame retardance. In dyeing this blend, it is very important to use the right dyeing assistants and to watch the process carefully to avoid uneven dyeing with high strike dyes.

## Verel Modacrylic Fibre

Verel is one of the oldest modacrylic fibres. It has been used considerably as a carpet fibre, but also has apparel applications. It has good flame resistant characteristics. The fibre was initially dyed at the boil, but it had to be relustered after dyeing, using a high amount of salt, dry steam or a combination of both. To avoid relustering, low temperature dyeing methods (71°C) were developed using dye accelerants or carriers.

Arkansas Company has two carriers for dyeing Verel — Anthrapole CM and Dyacryl MA. They have practically no odour or only a slight one. The former is somewhat more efficient, the latter more economical.

Verel can be dyed easily with selected cationic, disperse and neutral premetalized dyes, using 1%-6% (omf) of Anthrapole CM or Dyacryl MA; lower amounts for light shades, higher amounts for darker shades. Lately, however, the trend has been increasingly toward the use of cationic dyes, since they produce bright shades with good light and washfastness.

Note: It is important to dry Verel immediately after dyeing at 104° - 138°C. If it dries in the air, it becomes delustered and acquires a harsh handle.

# Lower Flammability Acrylics

This is a highly emotive item and is obviously towards the top of every fibre maker's research programme<sup>45</sup>. There are currently a number of successful candidates in this area:- Teklan, Kanecaron and Monsanto's modacrylic fibre, to name but three.

Monsanto's modacrylic fibre as related to the present and the next few years will now be discussed. When producing this modacrylic fibre, now available in staple form as fine as 17 decitex, excellent flame retardant properties are obtained but this is a compromise with deterioration in some physical properties — such as base colour, light fastness and reduced spin limit. Development and subsequent commercial trials are already advanced on a modacrylic with an improved base colour and possibly improved light fastness. The problem of reduced spin limit is a more difficult one to solve and this has been approached from several angles and currently remains unsolved, although it is considered not to be an extremely serious drawback.

PHYSICAL PROPERTIES OF 1,7 dtex, 400 m MODACRYLIC YARNS

	1/20's cc	1/34's ce	1/40's cc
Count (tex)	29,8	17,3	15,3
(cc)	1/19,8	1/34,1	1/38,6
Twist (turns/m)	6031	889	1003
Strength (cN)	397	238,1	195,2
Extension (%)	31,9	30,1	28,5
Tenacity (cN/tex)	13,3	13,8	12,8
Evenness (U%) 8 m/min	12,5	11,9	15,5
50 m/min	12,8	13,0	14,8
Yarn boil shrinkage (%)	1,1	1,3	3,2

A fibre (P Series) was engineered initially for carpet printing, and obviously the increased rate of dyeing enables the fibre to be used on continuous printing at similar speeds to nylon. When this fibre type was initially evaluated to ensure that the performance of the end product, the carpet, had not changed adversely a very pleasant surprise was obtained — there had been a significant improvement in abrasion resistance.

The technology of producing the P Series fibres is currently being translated down to apparel/home furnishing fibre description and these fibre types should be available shortly as high durability fibres, subject of course to final commercial evaluations<sup>45</sup>.

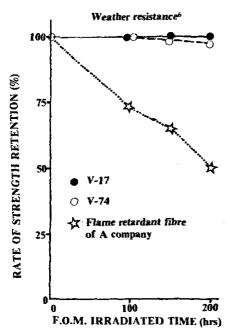
Abrasion revs	Standard fibre	High durability
Jersey 600 g headload	20 500	47 750
HF 800g headload	30 000	42 500

Lufnen is a flame retardant acrylic fibre containing more than 50% acrylonitrile<sup>44</sup>.

Lufnen does not shrink at 120°C wet heat and shrinks only 4% at 160°C dry heat. (Comparable flame-resistant acrylic fibres shrink by 30-50% at 120°C wet heat and by 25-45% at 160°C dry heat). It is self-extinguishing and has an LOI of 29. Tetoron Unfla-3 is a flame retardant polyester filament<sup>44</sup>. "Cordelan" is the brand name of polychal synthetic fibre produced with an emulsion spinning method by Kohjin Co. Ltd. in Japan<sup>52</sup>. It is composed of polyvinylalcohol and polyvinylchloride with a small amount of vinylchloride grafted polyvinylalcohol<sup>52</sup>. Cordelan ignites at 500 to 600°C.

PHYSICAL PROPERTIES OF "V-74" AND "V-17"6

Item	V-74 (Fire retarding type)	V-17 (Regular type)
Fineness (dtex)	15 (5,10)	15 (1,5-30)
Dry tenacity (cN/tex)	18,5-20,3	19,4-22
Elongation-dry (%)	45-55	45-51
Knot strength - dry (cN/tex)	17,7-18,5	18,5-19,4
Relative Density	1,20	1,17
Shrinkage in boiling water (%)	0 - 1	0 - 1
Flame retarding property (Value LOI)	27,5	18.5
Degree of whiteness (Value of PI)	92	94
Degree — fastness (Grade)		
Light Resistance	5-6	5-6
Discolouration during cleaning	5	5
Rubbing (dry)	5	5
Rubbing (wet)	4	4



Vonnel V-74 is an acrylic flame retardant fibre6

Uses of Products made mainly with acrylic fibre and their minimum required values of LOI (presumed)						
Product	Ways of Testing their combustibility	Minimum values of LOI required (Presumed)				
Curtains	Fine defense regulation Micro-burner of 45 degrees	29-30				
Nightclothes	D.O.C. FF-3-71	28-29				
Shaggy carpets	JIS-L-1091-B Method (Tokyo Fire Defense Method)	26-27				
Blankets	Mesenamine Method of 45 degrees (Wire net of 20 meshes)	24-25				
Sheets	Mesanamine Method of 45 degrees (Wire net of 20 meshes)	23				
Loop carpets	JIS-L-1091-B Method (Tokyo Fire Defense Method)	23				

Note: It is to be noted that the values of LOI shown above are affected by the product standard and kind of fibres.

#### FIBRES WITH HIGH MECHANICAL PERFORMANCE

Calvert<sup>46</sup> reviews some of the developments in the field of high strength fibres as follows:

One long-standing objective in polymer science is the production of fibres with an elastic modulus (ratio of stress to strain) approaching the theoretical maximum value. It seems self-evident that the structure should consist of polymer chains aligned as closely as possible to the fibre axis with few folds or disorientated regions. Various methods might produce this structure. The natural and most elegant method is exemplified in the production of plant cellulose where polymerisation occurs in situ so that fibres grow by addition of monomer units to one end. This has never really been achieved synthetically.

The drawing process used in the production of textile fibres produces chain orientation by stretching the fibre to several times its initial length. Two fibre industry alumni now in universities have worked on modifying this process to produce high draw ratios and orientation and so high moduli E.S. Clark of the University of Tennessee and co-workers at Du Pont have produced acetal fibres with a modulus of 35GPa (1 giga pascal-10° Nm ·2). For comparison, a carbon fibre has a modulus of 500 GPa and the theoretical maximum modulus of polyethylene is 220 GPa. These acetal fibres were drawn at about 130°C by a factor of 20 times in a two-stage process. The usual room temperature draw ratio is 7 times. The structure seems to consist of a single highly orientated phase.

I.M. Ward and co-workers at Leeds have studied the dependence of draw ratio on temperature, molecular weight and pre-draw microstructure in polyethylene. They found that the modulus increases roughly linearly to 70 GPa at a draw ratio of 30 times. The draw ratio was a maximum for a sample of high molecular weight and narrow molecular weight distribution crystallised by cooling to 110°C then quenching to room temperature and drawn at 75°C. This leaves the question as to whether it is the molecular weight itself or its influence on the microstructure that is important in allowing a high draw ratio.

Barham and Keller have taken up this point and studied the relationship between modulus, draw ratio and starting structure. They found that the dependence of modulus on draw ratio is the same for a range of pre-draw microstructures. What did change, with microstructure, was the maximum draw ratio which could be attained. Sheet which had been directly quenched or made from a sample with no low molecular weight polymer could not be drawn past 20-25 times as opposed to 30-35 times. This point is somewhat confused in the paper by the fact that the figure captions are exchanged.

From this Barham and Keller deduce that the low molecular weight chains, which segregate to the spherulite boundaries during slow crystallisation, govern the drawability of the sample. In support of this they say that samples which draw well had a black line around the spherulites when viewed with crossed polars. Presumably this region is liquid at the drawing temperatures and somehow allows large deformations without fracture. This is surprising in that one would expect strong rather than weak boundaries to be necessary.

Finally, mention should be made of the high melting point, high modulus fibres produced by spinning aromatic polyamides and polyhydrazides from strongly hydrogen bonding solvents such as dimethylacetamide-lithium chloride or sulphuric acid in which they form lyotropic liquid crystals. These fibres spun from anisotropic solutions have moduli several times those of fibres spun from similar isotropic solutions and reach 140 GPa. They are reviewed by Carter and Schenk, where the industrial significance is evidenced by that fact that most of the important references are to the patent literature. Two fibres, called Kevlar 47 and Kevlar 49 by Du Pont, which are based on poly (ρ-phenylene terephthalamide) look likely to be used in tyre belting and in composites for body armour and aircraft parts<sup>46</sup>.

where density is in g/cm<sup>3</sup> (or use relative density or specific gravity).

Some properties of high performance fibres are given in the following tables.

## TYPICAL PROPERTIES OF REINFORCING FIBRES

	Alumi- nium	Silicon Carbide	Boron Carbide	Graphite	iron	Nickel	Copper	Steel filament	Berylium filament	Glass
Relative Density	4	3,2	2,52	1,66	7,8	9	8,9	7,8	1,83	2,5
Modulus of Elasticity (cN/tex)	10350	26306	17798	59024	2565	2377	1396	2654	13197	3588
*Tensile Tenacity (cN/tex)	480	345	255	1247	168	43	33	51	48	193

# ENGINEERING PROPERTIES OF V-BELT FIBRE REINFORCEMENTS<sup>48</sup>

	Rayon		Nintan	Polyester	Glass	Steel	Aramid
	Dry	Wet	Nylon	1.0theater	June	Site	Aismo
Relative Density	1,52		1,14	1,38	2,54	7,83	1,44
Tenacity (cN/tex)	53	35	71-84	71-84	53-84	26,5-35,3	159-185
Strength (psi)	115 000	80 000	130 000	155 000	195 000/ 310 000	350 000	360 000
Young's Modulus (cN/tex) Young's Modulus (psi x 10°)	1 236 2.8	132 0.3	442 0.7	618 1,2	2 649 9.6	1 854 20	3 532 10

\*cN/tex = 
$$\frac{\text{GPa x 100}}{\text{relative density}}$$
$$= \frac{9.8 \times 10^{-3} \times \text{kgf/cm}^2}{\text{relative density}}$$

## PROPERTIES OF CARBON FIBRES<sup>49</sup>

Type of Fibre	[e.g. Hy Royce) (Mor Resea Develo	bon fibres fil (Rolls- Modmor ganite , urch & pment), rafil taulds)]	US carbon fibres (e.g. Thornel 50)	Boron fibres	Single- crystal alumi- nium oxide fibres	
Property	High Strength	High Modulus	_		]	
Diameter (µm)	8,0	7,5	6,6	100	250	
Relative Density	1,8	1,95	1,63	2,5	4,0	
Young's Modulus*				]		
(cN/tex)	13778	21231	21166	16560	11525	
Tensile strength*	162	100	121		_	
(cN/tex) Electrical resistivity at room temperature*	153	106	121	110	52	
(Ωcm)	1 3 x 10+	ı ³0,3 x 10 <i>~</i> ³		l	1 x 1016	
Thermal conductivity at room temperature*		about			about	
(cal/cm/°C/s)	0,05	0,1	0,03	_	0,1	

<sup>·</sup> Parallel to fibre axis

# PHYSICAL PROPERTIES OF HMHS AND OTHER FIBRES (JOHNSON)50

Fibre	Young's	Young's Modulus		Tensile Strength		
	(GN/m²)	(cN/tex)	(GN/m²)	(cN/tex)	to failure (%)	Relative Density
Polyester fibre (H.T.)*	18,5	1542	1,4	117	20	1,2
Nylon 6.6 (H.T.)	12,5	1042	1,0	83	17	1,2
E Glass	70	2692	2,5	96	4,0	2,6
Steel	200	2564	4,0	51	2,0	7,8
Carbon Type I	400	20513	2,0	103	0,5	1,95
Carbon Type II	260	14857	2,6	149	1,0	1,75
Carbon Type A	210	12727	1,9	115	0,9	1,65
Nomex	17,3	1236	0,7	50	22	1,40
Kevlar 29	59	4097	2,7	187	4,0	1,44
Kevlar 49	127	8759	2,9	200	2,6	1,45
X500 G	100	6849	2,2	151	4,0	1,46

<sup>\*</sup> High tenacity

## TYPICAL PROPERTIES OF EPITROPICS<sup>51</sup>

	Nylon Monofilament Epitropic	Polyester Epitopic Staple	
Linear density (dtex)	20	3	
Tenacity (cN/tex)	24,5	34	
Extension (%)	40	40	
Relative density	1,14	1,30	
Electrical Resistance (Ω/cm)	4 x 106	1 x 108	

0,3% of epitropics in carpets appears satisfactory

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#### Flame Retardant Wool Textiles

Wool is regarded as a safe fibre from the point of view of flammability.<sup>56</sup> It may be ignited if subjected to a sufficiently powerful heat source, but will not usually support flame and continues to burn or smoulder for only a short time after the heat source is removed. This is connected with the chemical and morphological structure of the wool fibre which has a high amino and amido nitrogen (16%) and moisture content (10-14%).<sup>56</sup>

The fundamental flammability properties of a number of fibres are summarised in the table. The position of wool, with a high ignition temperature, a high limiting oxygen index, a low heat of combustion and a low flame temperature, is particularly advantageous. In addition, wool does not melt and drip when ignited, but rather gives a foam-like ash which has good insulating properties and which can be handled immediately after the fire.<sup>56</sup>

In tests for flammability, the apparent flame properties of wool textiles depend on the test method specified and the fabric construction. A flammability test with the fabric in a horizontal position is much less severe than a 45 degree or a vertical test. All wool fabrics will pass a horizontal test without any special treatment, but may not pass some 45 degree tests. Very few wool fabrics will pass a vertical flame test, unless they are of a very dense construction and heavy, i.e. wool felts. The influence of fabric construction is also very important; the denser and heavier the fabric, the lower the flammability.<sup>56</sup>

Although untreated wool textiles have adequate natural flame-resistance for many common end uses such as apparel, carpets, upholstery, fabrics and blankets, sometimes a higher degree of flame-resistance is required. The application of the simple and inexpensive Zirpro treatments, involving titanium and zirconium complexes, leads to flame-resistant wool textiles without any adverse effect on the aesthetics, natural soil-resistance, colour fastness and mechanical properties of wool. The Zirpro treatments act predominantly in the solid phase with increased insulating char formation and this is being commercially exploited mainly for the contract market; public buildings and the transportation industry, with products such as carpets, upholstery fabrics, curtains, blankets.<sup>56</sup>

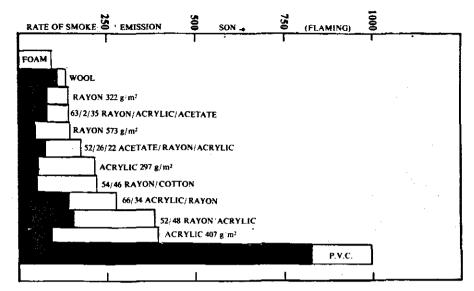
The advantages of the Zirpro treatments are also reflected in Zirpro wool protective clothing with low heat transfer from convective and radiant heat (aluminised wool) and good resistance to molten metal splashes. Multi-purpose finishes for wool protective clothing such as flame- and shrink-resistance, flame-resistance, water- and oil-repellence offer to the wearer additional comfort and protection.<sup>56</sup>

Benisek and Phillips<sup>56</sup> have consulted various sources in compiling this table, these being listed in their article.

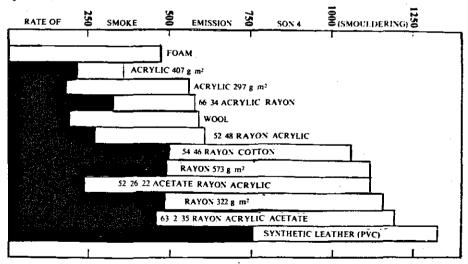
## FLAMMABILITY PROPERTIES OF VARIOUS FIBRES<sup>56</sup>

FIBRE	LOI	Heat of combustion (kJ/g)	Ignition temperature (°C)	Melting Point (° C)
Acrilan	18,2	31,8	465-530	235-320
Cotton	18,4	16,3	255	Does not melt
Triacetate	18,4	_	450-520	293
Diacetate	18,6		450-540	255
Polypropylene	18,6	46,5	570	164-170
Rayon	19,7	16,3	420	Does not melt
Polyvinyl alcohol	19,7	_		Does not melt
Nylon	20,1	33,1	485-575	160-260
Polyester	20,6	23,9	485-560	252-292
Wool	25,2	20,5	570-600	Does not melt
PFR Rayon	26,4	_		Does not melt
Modacrylic	26,8	<b>-</b>		160-190
Nomex Nylon T-450	30,0	_	800	316
Zirpro Wool	28-34	_		Does not melt
Polyvinylchloride	37,1	21,4	_	100-160

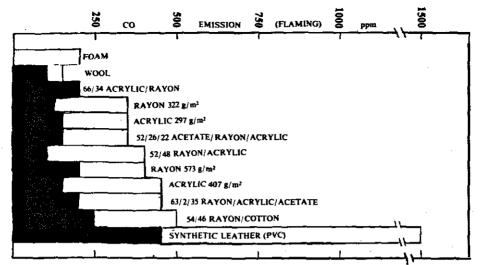
Recently Benisek and Phillips<sup>57</sup> evaluated the burning behaviour, and smoke and carbon monoxide (CO) emission of various commercial upholstery fabrics in a composite test and compared the results of the tests with those obtained on the individual components. They concluded that the burning behaviour of the composite materials (using a polyether foam) could not be predicted from the behaviour of the individual components. The wool upholstery fabrics performed best. Some of their results are illustrated in the following figures.



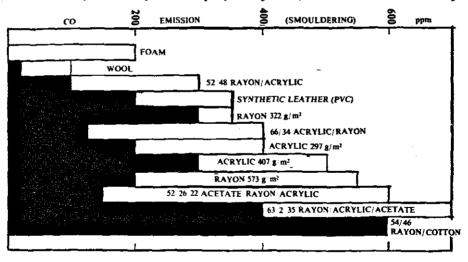
Rate of smoke emission under flaming conditions of various upholstery fabrics tested individually (shaded bars) and in a composite sample with a polyether foam (shaded and unshaded bars).



Rate of smoke emission under smouldering conditions of various upholstery fabrics tested individually (shaded bars) and in a composite sample with a polyether foam (shaded and unshaded bars).

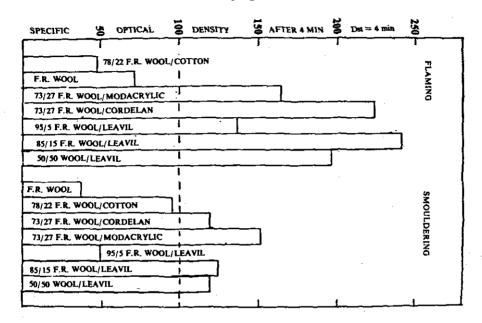


Carbon monoxide emission under flaming conditions at the maximum specific optical density of various upholstery fabrics tested individually (shaded bars) and in a composite sample with a polyether foam (shaded and unshaded bars).



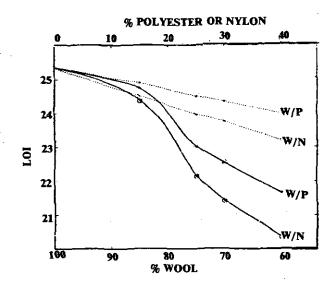
Carbon monoxide emission under smouldering conditions at the maximum specific optical density of various upholstery fabrics tested individually (shaded bars) and in a composite sample with a polyether foam (shaded and unshaded bars).

Benisek and Phillips<sup>58</sup> recently showed that flame-retardant wool-rich blends with PVC (Leavil), Cordelan or modacrylic fibres produce more particulate smoke and at a higher rate than flame-retardant 100% wool upholstery fabrics. The latter, as well as flame-retardant wool-rich blends with cotton, meet the F.A.A. proposed smoke emission standard. Some of their results are illustrated in the following figure.



Specific optical density after 4 minutes,  $Ds_t = 4 \min$  of various F.R. wool and wool containing fabrics meeting the flammability requirement of F.A.R. 25.853. The F.R. wool fabric was treated with metal complexes based on a F/Zr ratio of 2

Benisek<sup>59</sup> showed that wool-rich blends containing either polyester or nylon had significantly lower LOI values than those calculated, probably due to the higher heat of combustion and lower LOI of the synthetic fibres relative to those of wool (see Figure below). The hexafluorozirconate treatment was effective for imparting flame-resistance to blends containing at least 85% wool while zirconium-tungsten complexes were effective for blends with lower wool contents.



Limiting-oxygen index of wool/polyester/fibre (W/P) and wool/nylon (W/N) blends. Dotted lines – average calculated LOI values; solid curves – experimental LOI values

Van Rensburg<sup>60</sup> found that wool could be made flame- and shrinkresistant by treating the fabrics with chlorine and THPOH. The fabrics retained their soft handle after treatment.

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#### ERRATA

Page 8: Second sentence below the last table should read "Yellowing sets in at about 100° C" [Zahn, H. and Eckardt, K., Heimtex, 29 (2), 80 (Feb., 1977). IWS Translation (8612/77)].

Page 26: Table was reproduced from: Mehta, R.D., Sheets, D. and Loughlin, J.E., J. Fire Retardant Chemistry, 4, 209 (1977).

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