OxyChem®

Dechlorane Plus®

Manual

Ver: 7-27-07

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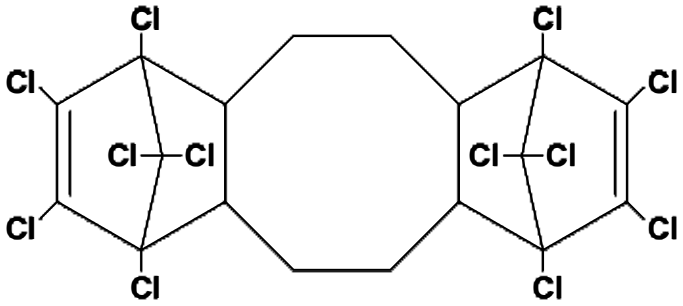


Dechlorane Plus®

($C_{18}H_{12}Cl_{12}$)

CAS Registry Number 13560-89-9

Dechlorane Plus® 515, 25, and 35 flame retardant additives are highly effective, chlorine-containing, crystalline organic compounds, which have been ground to free-flowing, white powders. They are the same chemical compound, differing only in particle size. The Dechlorane Plus® additives are used as *non-plasticizing* flame retardants in polymeric systems.

Chemical Name:	Molecular Weight
1,2,3,4,7,8,9,10,13,13,14,14- dodecachloro-1,4,4a,5,6,6a,7,10,10a,11,12,12a -dodecahydro-1,4:7,10 - dimethanodibenzo (a,e) cyclooctene	654

Specifications:

	Dechlorane Plus® - 515	Dechlorane Plus® - 25	Dechlorane Plus® - 35
Average Particle Diameter (Microns[1])	15 Maximum	6 Maximum	3.5 Maximum
pH (Methanol-Water Extract)	6.0 - 8.0	6.0 - 8.0	6.0 - 8.0
Volatility, % (4 Hours at 100°C at 5 mm Hg)	0.12 Maximum	0.12 Maximum	0.12 Maximum
Color - Rd	92 Minimum	97 Minimum	97 Minimum

(1) Micron Values list the average of particle sizes as per the Coulter Counter Method.

TYPICAL DATA:

Appearance	White, crystalline, free-flowing solid
Chlorine	65.1%
Melting Point	350°C (with decomposition)
Density	1.8 g/cc
Vapor Pressure @200°C	0.006mm of Hg
Bulk Density	
- Dechlorane Plus® 515 and 25	38-42 pounds/cubic foot
- Dechlorane Plus® 35	25-30 pounds/cubic foot
Recommended Operating Temperature	285°C (545°F) maximum

SOLUBILITY:

Solvent	Grams of Dechlorane Plus® per 100 Grams of Solvent at 25°C
Benzene	2.0
Xylene	1.0
Styrene	1.8
Trichloroethylene	1.4
Methyl Ethyl Ketone	0.7
n-Butyl Acetate	0.7
Hexane	0.1
Methyl Alcohol	0.1

PRODUCT BENEFITS:

• Colorability	Unlike many other flame retardant additives, Dechlorane Plus® is a fine white powder which easily allows color coding and matching.
• Excellent Flame Ratings	Formulations may be tailored to meet the most demanding flammability specifications.
• Thermal Stability	Operating temperatures up to 285°C allow greater ease of processing in a wide variety of polymers.
• Excellent Electrical Properties	The absence of ionic impurities provides electrical performance that is unsurpassed by other flame retardant additives.
• Low Smoke	Unlike other halogenated flame retardants, Dechlorane Plus® enhances the formation of an insulative char. This not only inhibits flaming drips and reduces flame propagation, but has the additional benefit of lower smoke generation.
• Synergist Options	Due to the unique properties of Dechlorane Plus®, cost effective synergist alternatives to antimony trioxide are available.
• Inert Filler	Dechlorane Plus® has low solubility; it is non-reactive, non-plasticizing, and hydrophobic.
• High CTI Performance	Comparative tracking index values in excess of 400 volts may be achieved through the use of OxyChem technology.
• Cost Effectiveness	With a 1.8 specific gravity, Dechlorane Plus® has the cost advantage of comparable brominated flame retardants that range between 2.3 and 3.2.
• Technical Expertise	Full service technical support to not only back our product and provide recommended starting formulations, but also to assist in your flame retardant formulation success.

POLYMER SYSTEMS

ABS
Chloroprene
DAP
EEA
EPDM
Epoxy
EPR
EVA
Hypalon®
Hytrel®
Kraton
Natural Rubber
High Impact Polystyrene
Neoprene
Nylon (6, 6/6, 12)
PBT
Phenolics
Polyester
Polyethylene
Polypropylene
Polyurethanes
SBR Block Copolymer
Silicon Rubber
TPE
TPU

DECHLORANE PLUS®
Toxicological Summary

	Toxicity Summary Study	Test Species	Result
A.	Acute Toxicity		
	1. Acute Oral	Albino Rat	LD_{50} 25 g/kg
	2. Acute Dermal	Albino Rabbit	LD_{50} 8 g/kg
	3. Acute Inhalation	Rat	LC_{50} 2.25 mg/liter
B.	Eye Irritation	Albino Rabbit	No effect as an ocular irritant was observed.
C.	Skin Sensitization	Guinea Pig	The material is not considered a sensitizer.
D.	Subchronic Toxicity		
	1. Oral 90-day	Albino Rat	The test compound was administered in the diet at 1, 3, and 10% levels. There were no study-related mortalities or untoward behavioral reactions. Growth and food intake were normal. No treatment-related differences between test and control animals were observed in clinical blood chemistry, hematology, urinalysis, gross pathology and histopathology at all feed levels. Slightly increased liver weight was observed at the 10% dietary level but not at the lower levels.
	2. Dermal 28-day Exposure	Albino Rabbit	At levels of 500 and 2000 mg/kg there was practically no irritation with repeated exposure. There were no significant adverse effects noted in the following areas: body weight, hematology, blood chemistry, urinalysis, and pathology. Statistically significant lower weight of gonads was observed in females of both test groups and lower liver weight in females at the 2000 mg/kg level. The differences were within the general range for young rabbits of this strain and age.

Toxicity Summary Study		Test Species	Result
	3. Dust Inhalation 28-day Exposure	Albino Rat	Exposure levels were 640 and 1540 mg/cubic meter. The following parameters were normal: body weight, blood chemistry, clinical chemistry, and urinalysis, and gross pathological examination. Absolute liver and lung weights and lung to body weight ratios were higher than normal in all or some of the test groups. Some histopathological alterations of the lung tissues were observed. These types of changes are frequently observed with exposure to organochlorine compounds and are normally reversible when exposure is terminated.
E.	Biodegradation	Sewage Sludge	Dechlorane Plus® is degraded under aerobic conditions but not under anaerobic conditions.
F.	Aquatic Toxicity	Bluegill Sunfish	No deaths were observed in 96-hour static and flow through tests at applied concentrations of up to 100 ppm.
G.	Mutagenicity		Dechlorane Plus® did not show a mutagenic response in the following tests: Standard Ames Assay (with and without activation), Ames Assay on Urine from rats treated with Dechlorane Plus®, and Mouse Lymphoma Forward Mutation Assay (with and without activation).

DECHLORANE PLUS®
Combustion Products

"In experiments conducted to date on the combustion of polypropylene, nylon, and high impact polystyrene containing Dechlorane Plus®, no regulated chlorodibenzodioxins or furans were detected in combustion products (20 ppb detection limit) when the flame retardant compound was heated from room temperature to 900°C. Additional experiments are in progress.

Experiments were conducted by the Institut Fresenius, West Germany, using the "Heraeus" apparatus described by W. Merz, et at in Fresenius Z. Anal. Chem. (1986) 325:449-460 (Springer-Verlag 1986)."

More information available upon request.

FR Nylons

Dechlorane Plus® is an excellent flame retardant for all nylons with or without glass fiber reinforcement. UL-94 V-0 ratings are possible to thicknesses of 1/64" (0.4 mm), while maintaining the excellent electrical and physical properties.

Many alternative synergists to antimony oxide are available for use when flame retarding nylon with Dechlorane Plus. The synergist selected is a very important factor in determining the physical and electrical properties of the final formulation. Zinc and iron compounds are often used in place of the standard antimony oxide because of their lower cost and the special properties that they contribute. Additionally, synergist mixtures are also useful in obtaining specific properties.

Table 2-1 to 2-7 demonstrate the wide range of FR nylon formulations possible with Dechlorane Plus®.

Table 2-1 demonstrates the various synergists that may be used with Dechlorane Plus to flame retard nylon 66. While in Table 2-2, the use of mixed synergists gives UL-94 materials with lower loading levels.

TABLE 2-1
FR-NYLON 66 USING DIFFERENT SYNERGISTS

Formulation (Weight %)			1	2	3	4	5
Nylon 66			73	70	73	73	82
Dechlorane Plus®			18	20	18	18	14
Sb_2O_3			9	10	-	-	-
Zinc Oxide			-	-	9	-	-
Zinc Borate			-	-	-	9	-
Ferric Oxide			-	-	-	-	4
Performance							
UL-94	1/8"	(3.2mm)	V-0	V-0	V-0	V-0	V-0
	1/16"	(1.6mm)	V-0	V-0	V-0	V-0	V-0
	1/32"	(0.8mm)	NA*	V-0	NC**	V-0	V-0
	1/64"	(0.4mm)	NA	V-0	NC	NC	V-0
Tensile Strength	(PSI)		NA	8406	8696	8840	10435
	(MPa)			58	60	61	72
CTI (Kc)	(volts)		NA	275	600	325	275

* Not Available
** No Class

TABLE 2-2
FR-NYLON 66 USING MIXED SYNERGISTS

Formulation (Weight %)			1	2	3	4
Nylon 66			78	85	70	85
Dechlorane Plus®			16	12	20	10
Sb_2O_3			2	-	-	-
Zinc Borate			4	1.5	5	2.5
Zinc Oxide			-	-	5	-
Ferric Oxide			-	1.5	-	-
Flamtard S			-	-	-	2.5
Performance						
UL-94	1/8"	(3.2mm)	V-0	V-0	V-0	V-0
	1/16"	(1.6mm)	V-0	V-0	V-0	V-0
	1/32"	(0.8mm)	V-0	V-0	V-0	V-1
	1/64"	(0.4mm)	V-0	V-0	V-0	NA
Tensile Strength	(PSI)		9739	10174	9464	10565
	(MPa)		67.2	70.2	65.3	72.9
CTI (Kc)	(volts)		450	350	375	575

In Table 2-3, the efficiency of antimony oxide is compared to a mixed synergist system. There is a 6% reduction in flame retardant package when used in a 25% glass filled system. The most effective single synergist is ferric oxide. With only a 12% flame retardant loading and a synergist loading of 3%, one obtains a V-0 at 1/16 inch.

TABLE 2-3
FR-NYLON 66 (25% GLASS-REINFORCED)
UL-94 V-0 AT 0.4 MM (1/16 inch)

Formulation (Weight %)			1	2	3	4
Nylon 66			49	55	60	60
Fiberglass			25	25	25	25
Dechlorane Plus®			18	16	12	12
Antimony Oxide			8	2	-	-
Zinc Borate			-	2	-	1.5
Ferric Oxide			-	-	3	1.5
Performance						
UL-94	1/8"	(3.2mm)	V-0	V-0	V-0	V-0
	1/16"	(1.6mm)	V-0	V-0	V-0	V-0
	1/32"	(0.8mm)	V-0	V-0	V-0	V-0
	1/64"	(0.4mm)	V-0	V-0	V-0	V-0
Tensile Strength		(PSI)	17800	18380	15000	15100
		(MPa)	122.6	126.6	103.3	104.0
CTI (Kc) (volts)			225	350	200	200

Table 2-4 shows the further reduction in flame retardant package that is possible using Dechlorane Plus® in combination with zinc borate and the iron oxides. It is possible to achieve V-0 materials at 1/16 inch with only 13% total flame retardant loading level in a 25% glass filled system.

TABLE 2-4
FR-NYLON 66 (25% GLASS-REINFORCED) WITH IRON OXIDE SYNERGISTS

Formulation (Weight %)		1	2	3
Nylon 66		60	60	62
Fiberglass		25	25	25
Dechlorane Plus®		12	12	10
Iron Oxide (Black)		3	1.5	1.5
Zinc Borate			1.5	1.5
Performance				
UL-94				
1/8"	(3.2 mm)	V-0	V-0	V-0
1/16"	(1.6 mm)	V-0	V-0	V-0
1/32"	(0.8 mm)	V-0	V-0	V-0
1/64"	(0.4 mm)	V-0	V-0	V-0
Tensile Strength	(PSI)	15570	16260	16620
	(MPa)	107.3	112.0	114.6
CTI Kc (volts)		225	225	225

Similar improvements in flame retardant efficiency are possible in Nylon 6 formulations Table 2-5. However, in the case of Nylon 6, zinc borate is not a good enough synergist to be used alone. Table 2-6 shows how a V-0 at 1/64 inch may be obtained by using a mixture of antimony oxide and iron oxide.

Table 2-7 shows some glass filled FR-nylon 6 formulations using single synergists which are V-0 at 1/16 inch.

TABLE 2-5
FR-NYLON 6 USING DIFFERENT SYNERGISTS

Formulation (Weight %)	1	2	3	4	5
Nylon 6	73	76	70	75	74
Dechlorane Plus®	18	16	20	20	21
Antimony Oxide	9	6	-	-	2.5
Zinc Borate	-	2	10	-	2.5
Ferric Oxide	-	-	-	5	
Performance					
UL-94					
1/8" (3.2mm)	V-0	V-0	NC*	V-0	V-0
1/16" (1.6mm)	V-0	V-0	NC	V-0	V-0
Tensile Strength (PSI)	6969	7348	7609	7019	7350
(MPa)	48.1	50.7	52.5	48.4	50.7
CTI Kc (Volts)	325	375	NA**	275	425

* No class

** Not available

TABLE 2-6
FR-NYLON 6 USING IRON OXIDE SYNERGISTS

Formulation (Weight %)		1	2
Nylon 6		75	70
Dechlorane Plus®		20	20
Antimony Trioxide		-	5
Ferric Oxide (Red)		5	5
Performance			
UL-94			
	1/8" (3.2mm)	V-0	V-0
	1/16" (1.6mm)	V-0	V-0
	1/34" (0.8mm)	NC*	V-0
	1/64" (0.4mm)	NC*	V-0
Tensile Strength	(PSI)	7026	7182
	(MPa)	48.4	49.5
CTI Kc (Volts)		275	275

TABLE 2-7
FR-NYLON 6 (25% GLASS-REINFORCED USING DIFFERENT SYNERGISTS)

Formulation (Weight %)			1	2	3
Nylon 6			47	50	45
Fiberglass			25	25	25
Dechlorane Plus®			22	20	20
Zinc Borate			-	-	7
Antimony Trioxide			6	-	3
Ferric Oxide (Red)			-	5	-
Performance					
UL-94					
	1/8"	(3.2mm)	V-0	V-0	V-0
	1/16"	(1.6mm)	V-0	V-0	V-0
Tensile Strength	(PSI)		14500	12300	16407
	(MPa)		99.9	84.7	113
CTI Kc (Volts)			250	125	350

TABLE 2-8
FR-NYLON 6 HIGH CTI (25% GLASS REINFORCED)

Formulation (Weight %)		1
Nylon 6		40
Fiberglass		25
Dechlorane Plus®		11.2
Zinc Borate		7.8
Melamine Cyanurate		14
Antimony Trioxide		2
Performance		
UL-94		
	1/8" (3.2mm)	V-0
	1/16" (1.6mm)	V-0
Tensile Strength	(PSI)	14000
	(MPa)	96.5
CTI Kc (Volts)		375

Polybutylene Terephthalate (PBT)

PBT has been successfully used in conjunction with Dechlorane Plus® to produce flame retardant connectors. Table 3-1 summarizes some typical properties of FR-PBT compositions. The high CTI values attainable with such formulations are especially noteworthy.

TABLE 3-1
FR-PBT USING DECHLORANE PLUS®

Formulation (Weight %)		1	2
PBT		78.9	80.9
Dechlorane Plus® 25		15.75	15.2
Antimony Trioxide		5.25	3.8
Teflon 6C		0.1	0.1
Performance			
UL-94	1/8" 3.2 mm	V-0	V-0
	1/16" 1.6 mm	V-0	V-0
	1/32" 0.8 mm	V-0	NC*
Tensile Strength	(PSI)	6848	6844
	(MPa)	47.2	47.2
CTI Kc (Volts)		325	575

* No class

Flame retardant glass reinforced PBT compositions with excellent properties can also be produced using Dechlorane Plus®. Tables 3-2 and 3-3 summarize obtainable property values with these formulations. As can be seen, the tensile strengths are consistently increased with increasing reinforcement as desired with UL-94, V-0 values at specimen thicknesses as low as 0.8 mm being possible.

TABLE 3-2
FR-PBT WITH DIFFERENT FIBERGLASS LEVELS

Formulation (Weight %)		1	2	3	4	5
PBT		73	69	64	56	54
Fiberglass		-	7	15	30	30
Dechlorane Plus®		18	18	14	11	12
Antimony Trioxide		9	6	7	3	4
Performance						
UL-94	1/8" (3.2mm)	V-0	V-0	V-0	V-0	V-0
	1/16"(1.6mm)	V-0	V-0	V-0	V-0	V-0
	1/32"(0.8mm)	V-2	V-2	V-2	V-1	V-0
Tensile Strength	(PSI)	6296	8562	11180	15469	14520
	(MPa)	43.4	59.0	77.0	106.5	100.0
CTI Kc (Volts)		275	NA*	NA*	225	225

* Not available

TABLE 3-3
GLASS-FILLED FR-PBT USING DECHLORANE PLUS®

Formulation (Weight %)		1	2	3	4	5
PBT		56	55.9	56	54	56
Fiberglass		30	30	30	30	30
Dechlorane Plus®		8	8	10.5	12	11
Antimony Trioxide		6	6	3.5	4	3
Teflon 6C		-	0.1	-	-	-
Performance						
UL-94	1/8" (3.2mm)	V-0	V-0	V-0	V-0	V-0
	1/16"(1.6mm)	V-0/V-2	V-0	V-0	V-0	V-0
	1/32"(0.8mm)	V-2	V-0	NA	V-0	V-1
Tensile Strength	(PSI)	14,340	14,300	14,960	14,520	15,460
	(MPa)	98.9	98.7	103.2	100.2	106.7
CTI Kc (Volts)		250	250	-	225	225

TABLE 3-4
FR-PBT FORMULATIONS WITH NO Sb_2O_3

Formulation (Weight %)	1
PBT	40%
Fiberglass	25%
Dechlorane Plus®	12%
Zinc Borate	8%
Melamine Cyanurate	15%
Performance	
UL-94 1/8" (3.2mm) 1/16"(1.6mm)	V-0 V-0
Tensile Strength (PSI) (MPa)	10290 71
CTI Kc (Volts)	300

POLYPROPYLENE (PP)

- These formulations are well suited for use in molded or extruded electrical/electronic applications.

- These formulations provide:

  - UL-94 V-0 down to 1/16 inch (1.6 mm) and V-1 or V-2 beyond that.
  - Synergist combinations of antimony oxide and zinc borate; the UL-94 rating desired determines the loading levels required.
  - Colorability - compounds are inherently white.
  - Non-dripping formulations.

TABLE 4-1

FORMULATION - WEIGHT %		1	2
Polypropylene		55	55
Dechlorane Plus®		30	35
Antimony Oxide		10	4
Zinc Borate		5	6
PERFORMANCE			
Oxygen Index		23	29
UL-94	1/8 inch (3.2 mm)	V-0	V-0
	1/16 inch (1.6 mm)	V-0	V-0
	1/32 inch (0.8 mm)	V-2	V-1
Tensile Yield	(PSI)	4030	3200
	(MPa)	27.8	22.0
Tensile Break	(PSI)	3028	2600
	(MPa)	20.8	17.9

POLYPROPYLENE - 20% TALC REINFORCED

- These formulations are well suited for use in molded or extruded electrical/electronic applications where mineral reinforcement is desired.

- These formulations provide:

  - UL-94 V-0 down to 1/16 inch (1.6 mm).
  - Synergist options including traditional (1) and reduced (2) loading levels of antimony oxide.
  - Lower costs when the synergist combination is used (2).
  - Colorability - compounds are inherently white.
  - Non-dripping characteristics.

TABLE 4-2

Formulation - Weight %	1	2
Polypropylene	50	50
Dechlorane Plus®	20	20
Antimony Oxide	10	5
Zinc Borate	-	5
Talc	20	20
Performance		
Oxygen Index	25	27.5
UL-94 1/8 inch (3.2 mm) 1/16 inch (1.6 mm)	V-0 V-0	V-0 V-0
Tensile Yield (PSI) (MPa)	4230 29.1	4054 27.9
Tensile Break (PSI) (MPa)	3660 25.2	3562 24.5

TABLE 4-3
FR-TALC-FILLED POLYPROPYLENE

Formulation (Weight %)	1	2	3	4	5
Polypropylene (Homopolymer)	45	45	-	-	-
Polypropylene (Copolymer)	-	-	45	45	45
Dechlorane Plus®	20	25	25	25	20
Antimony Trioxide	10	5	10	5	10
Zinc Borate	5	5	-	5	5
Talc	20	20	20	20	20
Performance					
UL-94 1/8" (3.2 mm) 1/16" (1.6 mm)	V-0 V-0	V-0 V-0	V-0 V-0	V-0 V-0	V-0 V-0
Tensile Yield (PSI) (MPa)	4082 28.1	3840 26.4	3372 23.2	3260 22.4	3468 23.9
Tensile Break (PSI) (MPa)	2712 18.7	3408 23.5	2294 15.8	2332 16.1	2446 16.9
Elongation (%)	2	2	31	32	25

TABLE 4-4
FR-POLYPROPYLENE

Formulation (Weight %)	1	2	3	4
Polypropylene (Homopolymer)	55	61	62	62
Dechlorane Plus	35	30	28	26
Sb_2O_3	4	6	6	4
Zinc Borate	6	2	2	-
Zinc Borate/500XPI	-	-	-	6
SFR-100	-	1	2	2
Performance				
UL-94 1/8" (3.2 mm) 1/16" (1/6"mm)	V-0 V-0	V-0 V-0	V-0 V-0	V-0 V-0
Tensile Yield (PSI) (MPa)	3352 23.1	3706 25.8	3508 24.2	2921 20.1
Tensile Break (PSI) (MPa)	2480 17.1	2822 19.5	2528 17.4	2326 16.0
Tensile Strength % Elongation	25	23	40	60

ABS

There are three major advantages of using Dechlorane Plus® in ABS: excellent UV stability, an increase in heat distortion temperature, and no blooming.

Formulations "A" and "B" are shown below. With the new additives, "A" and "B" reflects improved properties compared with the old formulation.

TABLE 5-1
FR ABS FORMULATION WITH DECHLORANE PLUS®

Weight %	A	B
ABS	77.0	72.1
Dechlorane Plus®	16.9	16.9
Antimony Oxide	6.1	6.1
Additive "A"	--	1.5
Additive "B"	--	3.4
UL-94 1/8" (3.2mm)	V-0	V-0
Notches Izod Ft. Lb/Inch J/M	 1.5 80	 3.9 208

The additives used in the Dechlorane Plus® ABS formulation are listed below.

"A" STRUKTOL TR 060
(Process aid/mixture of aliphatic resins with a molecular weight <2000)

"B" GOODYEAR P715-C1
ACRYLONITRILE (BUTADIENE) COPOLYMER (27% Acrylonitrile) (Impact Modifier)

Table 5-2 demonstrates that Dechlorane Plus® is more efficient flame retardant in ABS than some popular brominated additives. The compositions are also more light and heat resistant. Figure 5-1 graphically illustrates the magnitude of these advantages for UV stability.

TABLE 5-2
COMPARISON OF FR-ABS

Formulation (Weight %)	Dechlorane Plus®	OBDPO	Bis(tribromo-phenoxy)ethane
% FR + 6.1% Sb_2O_3	16.9	13.8	15.7
% Halogen	11	11	11
Performance			
UL-94 1/8"	V-0	V-2	NC
Notched Izod (J/m)	208	283	309
(Ft. Lb/in)	3.90	5.30	5.79
HDT, °C	77	71	71
UV Aging, ΔYI (1 week)	0.2	58.6	15.8

Thermosetting Resins

Dechlorane Plus® can be conveniently used as a flame retardant in most thermosetting resins and generally behaves as a conventional filler. It is effective in all resin systems, although its primary use includes epoxy resin, unsaturated polyester resins, and phenolic resins. A flame retardant synergist is generally required in order to obtain adequate flame retardance.

Epoxy Resins

In Tables 6-1 and 6-2, the results of Dechlorane Plus as a flame retardant in epoxy resins are shown. All the synergists give a UL rating of V-1 or better with the chlorinated flame retardant except zinc oxide. In this case, the results parallel those of Nylon 66, where the efficiency of the three additives are in the order - ferric oxide > zinc borate > antimony oxide at the loading levels used.

* The epoxy used was Epon 828 from Shell Chemical. The curing agent was triethylene-tetramine (TETA) at a 13 PHR level.

TABLE 6-1
FR-EPOXY USING DIFFERENT SYNERGISTS AND DECHLORANE PLUS

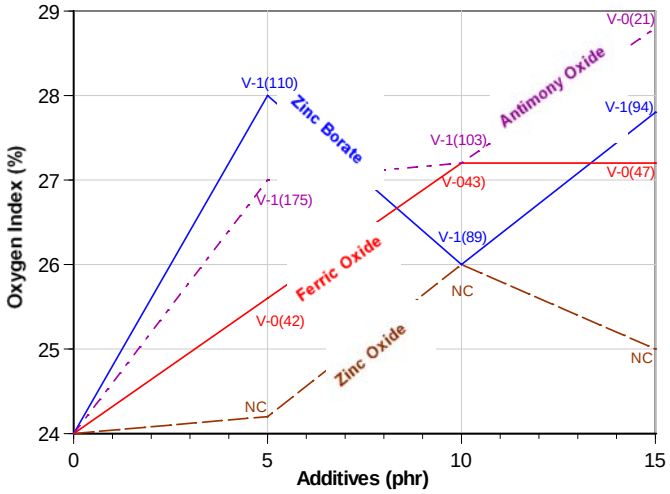
Formulation, PHR	1	2	3	4
Dechlorane Plus®	25.5	25.5	25.5	25.5
Antimony Oxide	5			
Zinc Borate		5		
Zinc Oxide			5	
Ferric Oxide				5
Oxygen Index	27	28	24.5	26
UL-94 1/8" (3.2 mm)	V-1	V-1	NC	V-0

TABLE 6-2
FR-EPOXY USING MIXED SYNERGISTS

Formulation, PHR	1	2	3	4	5
Dechlorane Plus®	25.5	25.5	25.5	25.5	25.5
Antimony Oxide	2.5	5		2.5	7.5
Zinc Borate	5	2.5	2.5	7.5	2.5
Ferric Oxide			2.5		
Oxygen Index	28.5	28.5	28	27.5	29
UL-94 1/8" (3.2 mm)	V-0	V-0	V-0	V-0	V-0

Figure 6-1 gives the oxygen index and UL-94 results of Dechlorane Plus® with different levels of synergists: 5, 10, and 15 PHR. The oxygen indexes are plotted against additive loading levels. The numbers in parentheses are the total burn time in seconds for the first and second ignitions. The oxygen indexes are only a guide when predicting UL-94 results. Ferric oxide at 5 PHR has an OI of 26 and a UL-94 V-0, while zinc borate has an OI of 28 but only a UL-94 V-1. It should be pointed out that Dechlorane Plus® always gives non-dripping formulations.

FIGURE 6-1
OXYGEN INDEX & UL-94 AT 1/8"/SYNERGIST STUDY
Dechlorane Plus® (25.5 phr) in Epon 828



The results of the mixed synergists study using antimony oxide are shown in Figure 6-2. The synergistic effect of using a mixture of zinc borate and antimony oxide can be seen. Using 2 1/2 PHR or each, we get a UL-94 V-0 and an OI of 28, which is better performance than using 5 PHR of the separate synergists.

FIGURE 6-2
OXYGEN INDEX & UL-94 AT 1/8"/SYNERGIST STUDY
Dechlorane Plus® (25.5 phr) Mixed 1/1 Synergists

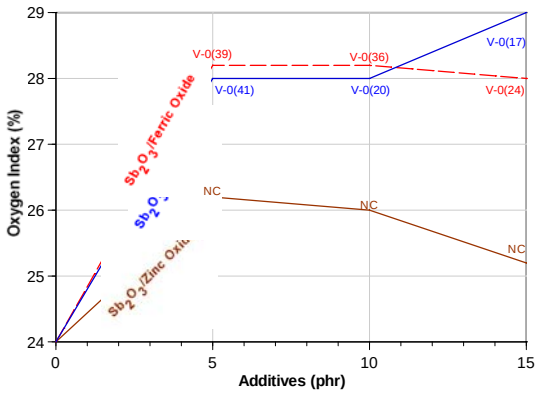
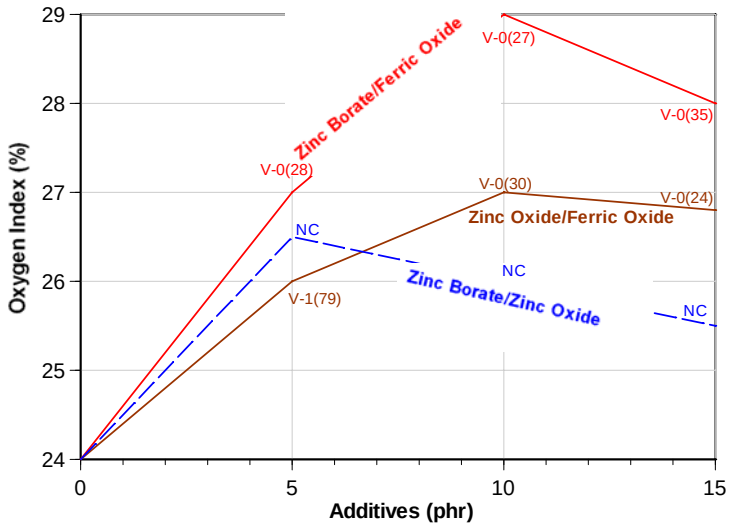


Figure 6-3 gives the results of Dechlorane Plus with the mixed synergists, without using antimony oxide. In all cases, the samples using zinc oxide in combination with zinc borate are NC. Because of the efficiency of ferric oxide, the formulations using this synergist in combination with zinc oxide or zinc borate are V-0.

FIGURE 6-3
OXYGEN INDEX & UL-94 AT 1/8"/SYNERGIST STUDY
Dechlorane Plus® (25.5 phr) Mixed 1/1 Synergists



Some other very efficient combinations of synergists are shown in Table 6-2. All these formulations gave a UL-94 V-0 at 1/8" thickness. Formulation #5 was one of the best performers, giving a non-dripping UL-94, V-0 at 1/8" with a very low total flameout time and a high oxygen index. When tested at 1/16", this material was also a V-0 with no dripping.

Dechlorane Plus® in Unsaturated Polyester Resins

Dechlorane Plus® can be used to confer a degree of flame retardance to standard polyester as indicated in Table 6-3. Thirty PHR of a 2:1 mixture of Dechlorane Plus® and antimony oxide is sufficient to yield a self-extinguishing classification by ASTM D-757.

TABLE 6-3
POLYESTER RESIN CASTINGS

Ingredients, Parts	1	2
Polyester	100	100
Dechlorane Plus®515	20	-
Antimony Oxide	10	-
Benzoyl Peroxide	1	1
Styrene	10	-
Wetting Agent	0.4	-
Thixotropic Agent	2.0	-
Cure 50°C	16	16
120°C	24	24
Flame Out Time, Seconds ASTM D-757	143	Not Self-Ext
Burning Rate, in Minutes	0.20	0.85

Dechlorane Plus in Flame Retardant Phenolic Laminates

As indicated in Table 6-4, a combination of less than 20% by weight of a 2:1 mixture of Dechlorane Plus and antimony oxide is sufficient to confer a self-extinguishing classification to phenolic resin paper laminates without appreciably degrading other physical properties.

TABLE 6-4
FIRE RETARDED PHENOLIC-DECHLORANE PLUS® 515 PAPER LAMINATES

Ingredients, %	1	2	3	4
Durez Resin = 12704	19.5	10.23	9.60	8.34
Durez Resin = 14139	31.6	38.30	56.40	37.95
Dechlorane Plus®	0.0	12.98	0.0	12.86
Antimony Oxide	0.0	6.48	0.0	6.43
Paper	48.9	32.0	34.0	34.40
Thickness, Inches	0.057	0.085	0.091	0.079
Barcol Hardness	60	59	58	59
Flammability Flame Out Time, Seconds	Burns	<1	Burns	3
Inches Burned		0		0
Arc Resistance, Seconds			22	28
Plies Of Paper	12	12	13	13
Impact Strength, Notched Izod Ft. Lbs/In Notch			0.218	0.301
Rockwell Hardness E			82.6	80.2
Flexural Strength, PSI			17,800	15,350
Flexural Modulus x 10^5, PSI			9.86	10.8
Compressive Strength, PSI			25,560	30,930

Importance of the Synergist of Synergist Mixture in Determining Flame Retardant Behavior in Epoxies

The choice of synergist or synergist mixture used in combination with Dechlorane Plus® is not only important in determining the halogen level necessary for obtaining a given degree of flame retardance but also can effectively reduce the amount of smoke produced during flame exposure. See Figures 6-4 and 6-5.

FIGURE 6-4
NBS SMOKE DATA EPOXIES
Flaming Mode

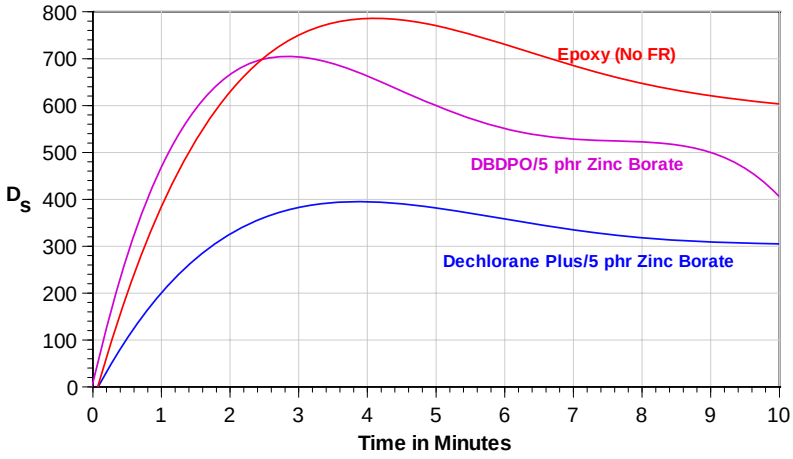
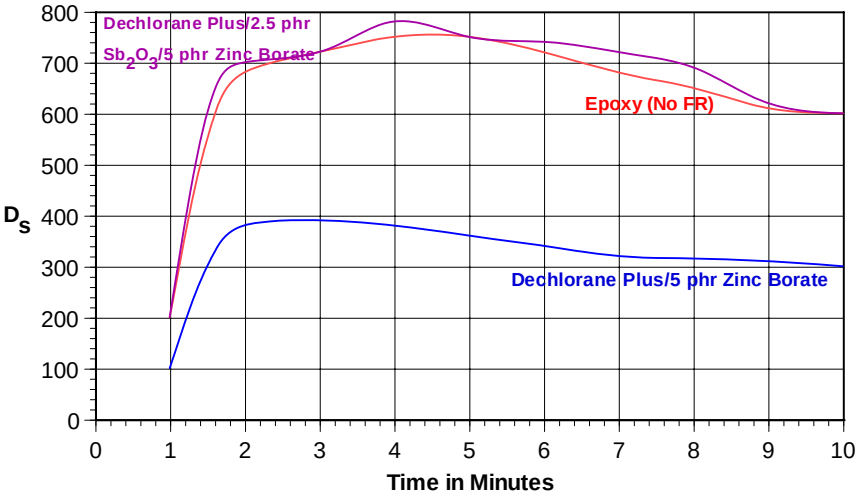


FIGURE 6-5
NBS SMOKE DATA EPOXIES
Flaming Mode



DECHLORANE PLUS® IN WIRE AND CABLE

OxyChem's Dechlorane Plus flame retardants have been used with great success in wire and cable applications. The following are some typical formulations that have proved especially successful for flame-retarded ethylene vinyl acetate (EVA) wire and cable.

These formulations in Table 7-1 have excellent electrical properties, are thermally stable, and are colorable. They also have char-forming and non-dripping characteristics. In addition to providing UL 44 VW-1 performance, the formulations produce lower smoke generation than equivalent brominated flame retardant formulations.

OxyChem has also developed low smoke flame retardant technology, capable of reducing smoke levels in FR wire and cable formulations by 95%.

TABLE 7-1
FLAME RETARDED ETHYLENE VINLY ACETATE

Formulation (Weight %)	Normal	Low Smoke	
EVA	47.9	47.8	* Key ingredients for low smoke technology **Talc** Mistran ZSC Luzanac America **Iron Compound** a) Ferric Oxide Akron Chemical b) Ferrous Gluconate Pfizer Chemical Division
Dechlorane Plus®*	25.0	25.0	
Antimony Trioxide	5.0	5.0	
Talc (Zinc Stearate Coated)*	20.0	20.0	
Iron Compound*	--	0.1	
Agerite Resin D (Anti-Oxidant)	0.7	0.7	
Luperox 500R (Peroxide)	1.4	1.4	
Oxygen Index	27	28	
UL-44 VW-1 Flame Test	Pass	Pass	

NOTE: Polyethylene may be used in a similar formulation to give a flame retarded material with an oxygen index of 27%, which also passes the UL-44 VW-1 flame test.

FR-EPDM FORMULATION

This typical formulation for FR-EPDM outlined in Table 7-2 may be used in a nuclear power plant control cable. It also provides excellent electrical properties and is colorable. Char-forming and non-dripping characteristics are present. The formulation provides UL 44 VW-1 performance, and produces lower smoke generation than equivalent brominated flame retardant formulations.

TABLE 7-2

FORMULATION - PARTS	
EPDM (Nordel 2722)*	90
Low Density Polyethylene	20
Dechlorane Plus	33
Antimony Oxide	12
Calcinated Clay	60
OTHER INGREDIENTS	
Zinc Oxide	5
Paraffin Wax	5
ERD-90 (Lead Oxide)	5
Agerite Resin D (Anti-oxidant)	2
Vinyl Silane A-172	1
SRF (Carbon Black)	2
Luperox 500-R (Peroxide	3
PERFORMANCE	
Oxygen Index	26
UL 44 VW-1 Flame Test	PASS
Tensile Strength (PSI)	1600
(MPa)	11.0
Elongation (%)	290

* Product of E.I. duPont de Nemours & Co.

Low Smoke Technology

As indicated in Figure 7-1, Dechlorane Plus® has a significant advantage over Decabromodiphenyl oxide in polyolefin compositions. A typical low smoke polyethylene composition is also summarized in Table 7-3. Low smoke properties are the result of the addition of an iron compound and talc filler to the standard Dechlorane Plus®/antimony oxide flame retardant. The importance of the additional iron and talc additives is emphasized in Figure 2, where the novel low smoke formulation is compared directly to the standard Dechlorane Plus® formulation.

FIGURE 7-1
NBS SMOKE GENERATION
FLAME RETARDANT POLYOLEFIN COMPOSITIONS SUMMARY

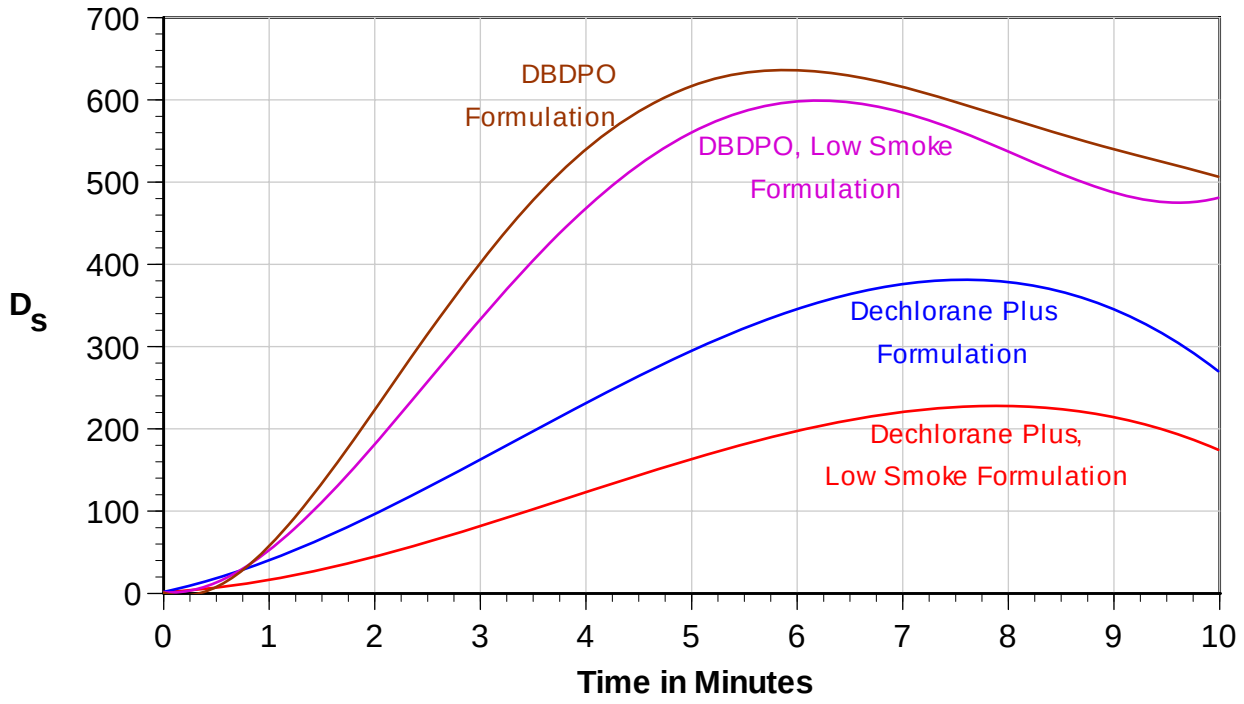


FIGURE 7-2
STANDARD AND LOW SMOKE
XLPE NBS SMOKE RESULTS

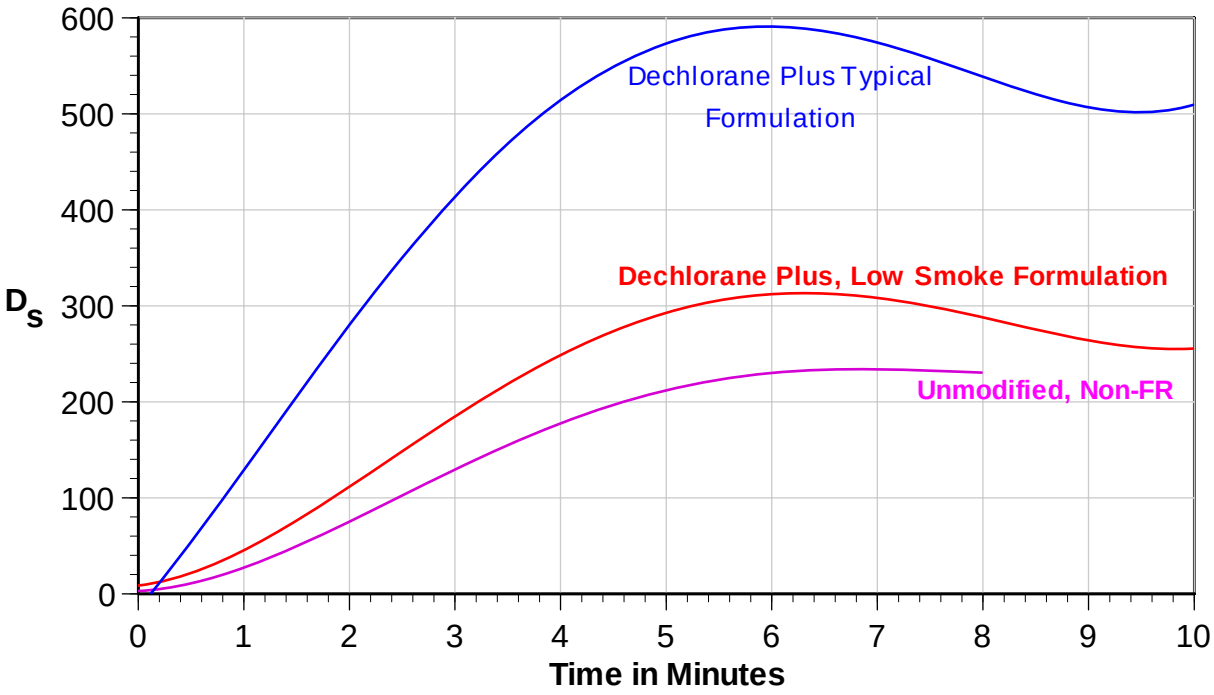


TABLE 7-3
FLAME RATARDED
CROSS-LINKED POLYETHYLENE LOW SMOKE FORMULATIONS

- This formulation is well suited for wire and cable applications.

- It provides a high performance compound which has:
  - Colorability
  - Excellent electrical properties
  - Thermal stability
  - UL-44 VW-1 performance
  - Char forming and non-dripping characteristics
  - Extremely low smoke generation - when compared to equivalent brominated flame retardant or standard Dechlorane Plus® formulations

- Antimony oxide is used as a synergist with Dechlorane Plus® flame retardant

FORMULATION - WEIGHT %

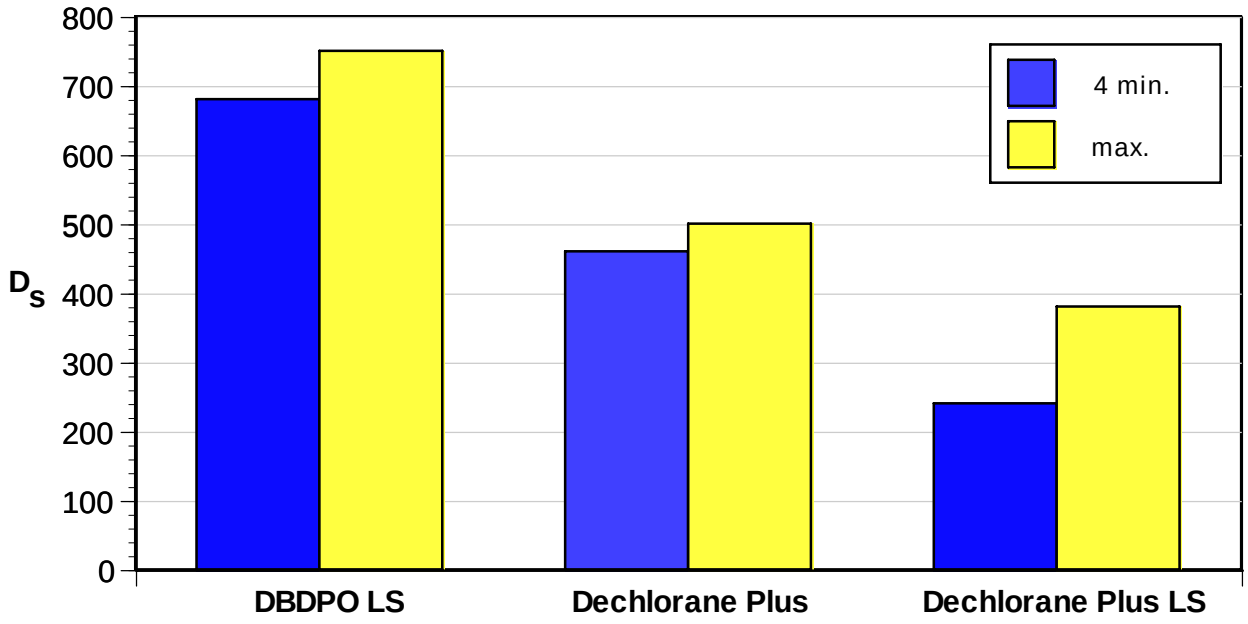
Polyethylene	47.8
Dechlorane Plus®	25.5
Antimony Oxide	5.0
Talc (Zinc Stearate Coated)*	20.0
Iron Compound*	0.1
OTHER INGREDIENTS:	
Agerite Resin D (Anti-oxidant)	0.7
Luperox 500 R (Peroxide)	1.4

* Key ingredients in Low Smoke technology

PERFORMANCE

Oxygen Index		28
UL-44 VW-1 Flame Test		PASS
Tensile Strength	(PSI)	1370
	(MPa)	9.4
Elongation (%)		200
NBS Smoke Generation - ASTM E-662-79		
D_2 @ 4 Minutes		230
D_s Maximum		310

FIGURE 7-3
NBS SMOKE DATA EVA Ds VALUES



The relative efficiency of the low smoke technology is demonstrated by the comparison of brominated EVA low smoke values to standard EVA/Dechlorane Plus® in Figure 3. Similar comparisons are made in ethylene/propylene diene monomer elastomers (EPDM) in Figure 4 and Table 13. Table 14 is a summary of the efficiency of smoke reduction possible with the new technology in the different polymer compositions.

TABLE 7-4
FLAME RETARDED ETHYLENE PROPYLENE DIENE MONOMER LOW SMOKE FORMULATION

- This formulation is well suited for wire and cable applications.
- It provides a high performance compound which has:
  - Colorability
  - Excellent electrical properties
  - Thermal stability
  - UL-44 VW-1 performance
  - Char forming and non-dripping characteristics
  - Extremely low smoke generation - when compared to equivalent brominated flame retardant or standard Dechlorane Plus® formulations.

FORMULATION - WEIGHT %

EPDM (Nordel 2722)	90
Low Density Polyethylene	20
Dechlorane Plus®	33
Antimony Oxide	12
Talc (Zinc Stearate Coated)*	60
Iron Compound*	0.1
OTHER INGREDIENTS	
Zinc Oxide	5
Paraffin Wax	5
ERD-90 (Lead Oxide)	5
Agerite Resin D (Anti-oxidant)	2
Vinyl Silane A-172	1
Luperox 500 R (Peroxide)	3

* Key ingredients for Low Smoke technology.

PERFORMANCE

Oxygen Index			26
UL-44 VW-1 Flame Test			PASS
Tensile Strength	(PSI)		1500
	(MPa)		10.3
Elongation (%)		240	
NBS Smoke Generation - ASTM E-662-79			
D_2 @ 4 Minutes			295
D_s Maximum			385

FIGURE 7-4
NBS SMOKE DATA EPDM Ds VALUES

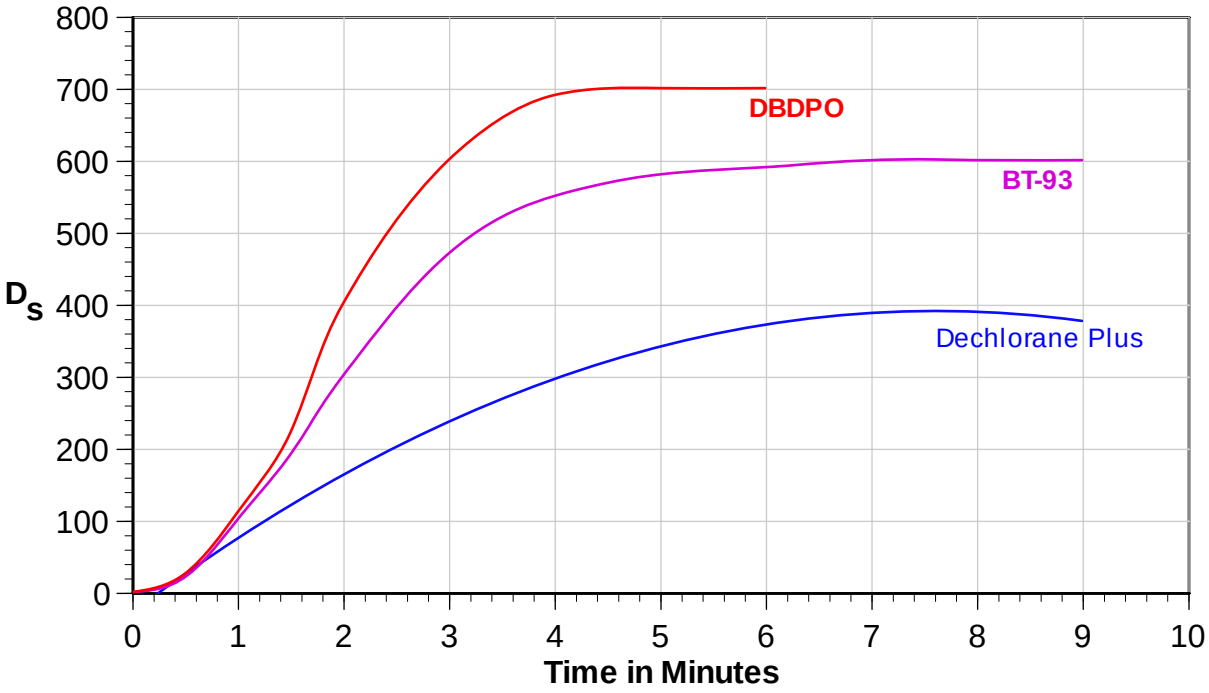


TABLE 7-5
DECHLORANE PLUS® POLYOLEFIN FORMULATIONS
SMOKE GENERATION SUMMARY
ASTM E-662-79

		Standard Formulation	Low Smoke Formulation
XLPE	D_s @ 4 Minutes	525	230
	D_s Maximum	592	310
EVA	D_s @ 4 Minutes	430	216
	D_s Maximum	484	343
DPDM	D_s @ 4 Minutes	321	295
	D_s Maximum	441	385

$D_s = 132 \text{ LOG } (100/\%T)$

TABLE 7-6
TESTING OF WIRE & CABLE SAMPLES
Hydrogen Halide Generation

Sample	FR	Standard Composition % Hydrogen Halide*		Low Smoke Composition % Hydrogen Halide	
		D_{max}	20 Minutes	D_{max}	20 Minutes
EPDM	Dechlorane Plus®	<2	0.5	0.5	<2
EPDM	DBDPO	<2	<2	<2	<2
EPDM	BT-93	1	<2	<2	1
XLPE	Dechlorane Plus®	1.5	0.5	0.5	1
XLPE	DBDPO	0.5	<4	0.5	<4
EVA	Dechlorane Plus®	0.5	1	0.5	0.5
EVA	DBDPO	<4	<4	1	4

* Measured and based on total composition while burning in the NBS smoke chamber.

Dechlorane Plus® as a Flame Retardant in Various Elastomer Substrates

Tables 8-1 to 8-9 indicate typical Dechlorane Plus® formulations developed for use in various synthetic elastomers. Physical property data is also included. Oxygen index values are included to indicate the degree of flame retardancy obtained.

TABLE 8-1
CHLOROSULFONATED POLYETHYLENE (HYPALON) - A

Formulation - PHR		
Hypalon 40		90
EPDM SC-1		10
Dechlorane Plus®		5
Antimony Oxide		5
Dibasic Lead Phthalate		30
Aluminum Trihydrate		40
Calcined Clay		20
Triallyl Cyanurate		2
Antioxidant - 1		3
Antioxidant - 2		2
Peroxide Curing Agent		3
Performance		
Oxygen Index		35
Tensile Strength	(PSI)	1930
	(MPa)	13.3
Elongation (%)		685

TABLE 8-2
CHLOROSULFONATED POLYETHYLENE (HYPALON) - B

Formulation - PHR		
Hypalon 45		70
EPDM SC-1		30
Dechlorane Plus®		6
Antimony Oxide		6
Dibasic Lead Phthalate		30
Calcined Clay		90
Triallyl Cyanurate		1
Antioxidant - 1		2
Antioxidant - 2		1.5
Vinyl Silane		1
Peroxide Curing Agent		2.5
Performance		
Oxygen Index		30
UL-44 VW-1 Flame Test		PASS
Tensile Strength	(PSI)	2452
	(MPa)	16.9
Elongation (%)		245

TABLE 8-3
FLAME RETARDED POLYCHLOROPRENE (NEOPRENE)

Formulation – PHR		
Neoprene W	100	100
Dechlorane Plus®	10	10
Antimony Oxide	5	-
Witco HAF Black (Carbon Black)	60	60
Sun Circosol 4240 (Processing Aid)	15	15
Stearic Acid	0.5	0.5
Sulfur	1	1
Zinc Oxide (New Jersey)	5	5
Magnesium Oxide	4	4
MBT (R.T. Vanderbilt) - (Accelerator)	1	1
NA-22 (Harwick) - (Accelerator)	0.5	0.5
TMTMS (Uniroyal) - (Accelerator)	0.3	0.3
Performance		
Oxygen Index	40	37
Tensile Strength (PSI)	3010	2840
(MPa)	20.7	19.6
Elongation (%)	250	240

TABLE 8-4
FLAME RETARDED SBR BLOCK COPOLYMER (KRATON)

Formulation - PHR	
Kraton 3200	100
Dechlorane Plus®	30
Antimony Oxide	10
Performance	
Oxygen Index	30
Tensile Strength (PSI)	1780
(MPa)	12.3
Elongation (%)	840

TABLE 8-5
NATURAL RUBBER

Formulation - PHR		
Natural Rubber		100
Dechlorane Plus®		18.7
Antimony Oxide		7.5
Performance		
Oxygen Index		24
Tensile Strength	(PSI)	2870
	(MPa)	19.8
Elongation (%)		650

TABLE 8-6
FLAME RETARDED SILICON RUBBER

Formulation - PHR			
Silicon		100	100
Dechlorane Plus®		18.8	40
Antimony Oxide		9.3	-
CAB-O-SIL (Fumed Silica)		0	6
Luperco CST (Peroxide)		0	1.5
Performance			
Oxygen Index		31	29
Tensile Strength	(PSI)	580	60
	(MPa)	4.0	4.1
Elongation (%)		460	380

TABLE 8-7
FLAME RETARDED POLYESTER BASED URETHANE ELASTOMER (TPU)

Formulation - PHR		
Polyester Based Urethane		80
Dechlorane Plus® 25		16
Sb_2O_3		4
Performance		
UL-94	1/8" (3.2mm)	V-0
	1/16" (1.6mm)	V-0
Tensile Strength	(PSI)	4060
	(MPa)	28
Elongation (%)		536

TABLE 8-8
FLAME RETARDED URETHANE RUBBER

Formulation - PHR		1	2
Urethane		100	100
Dechlorane Plus®		30	20
Antimony Oxide		15	10
Witco HAF Black (Carbon Black)		25	25
Stearic Acid		2	2
Dicup 40C (Peroxide)		4	4
Performance			
Oxygen Index		30	28
Tensile Strength	(PSI)	2600	3000
	(MPa)	17.9	2037
Elongation at Break (%)		880	890
Hardness, Durometer A		66	64

TABLE 8-9
FLAME RETARDED URETHANE FOAM

Formulation - Weight %	1	2
Urethane	72.47	50.25
Dechlorane Plus®	17.5	35.0
Antimony Oxide	2.5	8.75
Talc	7.5	6.0
Ferric Oxide	0.03	-
Performance		
Oxygen Index	25	29
Foam Density (Lbs./Cubic Feet)	4.8	6.6