

Compounding of Ethylene-Propylene Polymers for Electrical Applications

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Background

Earlier articles in this magazine have reviewed crosslinked polyethylene (XLPE) insulation and ethylene-propylene rubber (EPR). This article is intended as a sequel to the latter

Because of its combination of superior electrical properties, its flexibility over a wide temperature range and its resistance to moisture and weather, EPR is used in a diverse range of electrical applications:

- Power cables
- Flexible cords
- Control and instrument wire
- Automotive ignition wire
- Appliance wire
- Motor lead wire
- Mining cable
- Molded electrical accessories

Compounding is the technology of converting the raw rubber resin into useful materials through the addition of fillers, reinforcers, stabilizers, process aids, curatives, flame retardants, pigments, etc.

The resulting composition is called a compound. In this paper, additive levels will be expressed in parts per hundred of resin (or rubber), commonly called by compounders "phr." This is a more convenient form than weight percentages, some of which are optional, depending on the intended application and on processing requirements. A typical compound usually has the following ingredients:

- EP rubber
- Filler/Reinforcing Agent(s)
- Plasticizer
- Antioxidants/Stabilizers
- Flame Retardants
- Process Aids
- Ion Scavenger
- Coupling Agent
- Curing Coagent
- Curative

Frequently, the considerations given to the selection of compound ingredients may require compromises in one characteristic in order to improve another.

Proper selection of the ingredient(s) in each category requires that consideration be given to the desired physical, electrical and environmental properties, as well as cost, ease of mixing, chemical stability and ease of processing. Frequently these considerations may require compromises in one characteristic in order to improve another. In the following discussion, factors to be considered in the proper choice of each ingredient will be explored.

Curatives and Curing Coagents

Although these components comprise only a small percentage of the total compound, the development of a practical compound and the selection of the ingredients cannot be carried out on a rational basis without considering the crosslinking chemistry to be employed.

A sulfur cure, possible with an EPDM but not with an EPM, is used only in low voltage insulations, cable jackets, or in some molded electrical components. Its principal deficiencies in electrical applications are its electrical stability under wet and high temperature conditions and its high temperature ageing characteristics.

For most electrical applications, peroxide cures, possible with either an EPM or EPDM, are the most common curing method. Compared with sulfur cures they are more expensive both in terms of the selection of other compatible compounding ingredients and in the temperatures and pressures required to achieve void-free vulcanizates.

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Table I
Characteristics of Peroxide and Sulfur-Cured EPR

Type of Cure	Benefits	Disadvantages
Peroxide	Electrical stability	Requires higher temperatures and pressure vs. sulfur
	Heat stability	
Sulfur	High tensile and tear strength	Requires coagent if used with EP copolymer
	Can be carried out at lower pressures and temperatures in air	Inhibited by oxygen or acidic compounding ingredients
		Wet electrical stability
		Heat stability
		Odors

Peroxide cures are inhibited by oxygen, a potent scavenger of free radicals, and by acidic components in the mixture, which cause the peroxide to decompose by an ionic (no free radicals are generated) mechanism. A comparison of sulfur and peroxide cures is summarized in Table I.

The most commonly-used peroxide is dicumyl peroxide, available in several substantially pure grades (90% and above) or as 40% dispersions on inert particulate carriers such as treated calcined clay, diatomaceous earth or calcium carbonate. It can be used in all cures above 150°C, and produces a characteristic odor in the product due to a byproduct of its thermal decomposition, acetophenone.

Another commonly-used peroxide is bis-(tertiary butylperoxy)-diisopropyl benzene. Like dicumyl peroxide it is available in a concentrated or a dispersed form and has two advantages: It is free of odorous byproducts and, because of its multifunctionality (higher peroxide content), it is used at levels much lower than those used with dicumyl peroxide. It is slightly less reactive than dicumyl peroxide and is frequently used in thin-walled low voltage applications or in semiconductive insulation shields. In both cases the lower reactivity is acceptable because these products reach their cure temperatures more rapidly in a continuous vulcanization tube than does a heavy-walled medium voltage insulation, for example.

Cure rate and cure state, the latter often called crosslink density, are terms that are frequently misinterpreted or misunderstood as applied to peroxide cures. Between EPM and EPDM elastomers in general, there is essentially no difference in the cure rate with a given peroxide, but there may be a large difference in the cure state at a given peroxide level. The primary factor affecting the cure rate for a given peroxide is temperature, although the filler type and amount may also have an effect. The polymer type, plasticizer type and level, antioxidant type and level, and peroxide level have

Table II
Effect of Ingredients on Peroxide-Cured EPM/EPDM

Ingredient	Cure Rate	Cure State
EPM/EPDM	No difference	EPDM higher
Peroxide type	May increase or decrease	May increase or decrease according to type
Amount	No change	May increase or decrease according to amount
Plasticizer	No change	Small to significant decrease
Antioxidant	No change	Small to significant decrease
Filler	Slight decrease	Small to significant decrease
Coupling agent	No change	Significant increase
Curing coagent type:		
Type I	Significant increase	Significant increase
Type II	No change	Significant increase
Sulfur	No change	Decrease

little or no effect on the rate but will have significant effects on the cure state.

Coagents are polyfunctional, unsaturated organic compounds used in conjunction with peroxides to achieve a more versatile and efficient cure system. They are used to increase both the rate and the state of cure.

Coagents may be grouped into two classes. Class I includes acrylates, methacrylates, vinyl esters and bis-maleimides, which function both by addition to a free radical and by hydrogen abstraction to increase both the rate and state of cure. Class II includes allylic compounds and low molecular weight polymers with a high vinyl content and affects only the cure state, not the cure rate. A commonly used example of Class I is trimethylolpropane trimethacrylate, and an example of Class II is triallyl cyanurate (TAC). Another more specialized coagent is elemental sulfur. Used at very low levels, it imparts higher tensile and tear strengths to the vulcanizate. It is most frequently used in molded cable accessories in which the improved hot tear strength facilitates demolding. Unfortunately, it also reduces dielectric breakdown strength because of the presence of sulfur crosslinks and also creates persistent disagreeable odors in the vulcanizate.

The effects of these various compounding ingredients on the cure rate or the cure state are indicated in Table II. Discussions of the proper selection of each of these classes of ingredients will be considered in more detail below.

EPM and EPDM Selection

As indicated above, both polymer types can be crosslinked with a peroxide and, if properly compounded, will have equivalent physical, electrical and rheological properties. But the selection of an

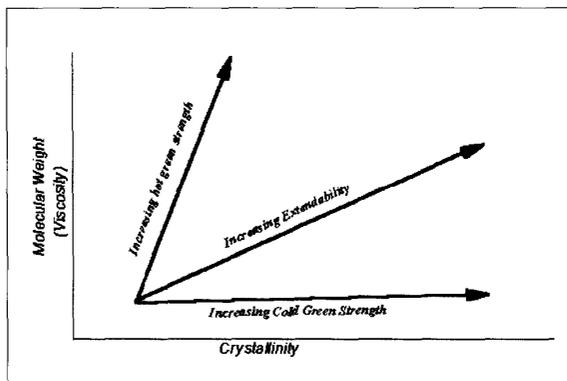


Fig. 1. Effect of molecular weight and crystallinity on polymer and compound properties.

Table III
Effect of EPM/EPDM Polymer Variables on Processing and Properties of Electrical Compounds

Property	Benefits	At the Expense of:
Increasing molecular weight	Tensile and tear strength increase Filler and plasticizer can be increased for lower cost Distortion or "sag resistance" during curing improves	Difficult mixing and dispersion More difficult to extrude Higher filler/plasticizer reduces electrical properties
Increasing ethylene content	Tensile strength increases Accepts higher filler/plasticizer levels Easier to pelletize polymer and compounds More efficient peroxide cures	Higher hardness Poorer permanent set Poorer elastic recovery Poorer low temperature flexibility
Increasing diene content	More efficient peroxide cures Improved cross link density ("cure state")	Lower heat resistance Elongation decreases Compound cost increases
Narrow MWD	Lower die swell Improved cure rate and cure state Better physical properties Accepts higher filler/plasticizer levels	Generally more difficult to mix Lower critical shear rate makes thin wall insulation difficult to smooth
Broad MWD	Easier to process in all equipment vs. narrow MWD Best for injection molding	Higher die swell Slower cure rate and cure state due to low MW components

EPM/EPDM to satisfy an individual electrical application is more complicated. A good starting point is a figure from [2], reprinted here as Fig. 1 for the convenience of the reader. This figure graphically represents the relationship of molecular weight or viscosity vs. crystallinity (only loosely correlated with ethylene content) and their effects on the ability to be extended with

fillers and/or plasticizers as well as "green" (uncured) strength. Since increasing levels of filler and plasticizer increase electrical losses and reduce breakdown strength (see Table I of [2]), it is immediately apparent that compounds suitable for low voltages will be based on polymers found in the upper right quadrant of Fig. 1. Polymers suitable for higher voltages, hence lower filler levels, will be found in the lower left quadrant. Pelletizable compounds will be based on polymers of increased crystallinity. Polymers found in the lower left quadrant will be most suited for highly flexible, low viscosity compounds with low filler levels—a molded accessory perhaps.

The analysis based on Fig. 1 ignores a very important factor in polymer selection, namely the question of molecular weight distribution (MWD). Variations in MWD usually are manifested in processing characteristics, but at equal filler loading, narrow MWD polymers are likely to have higher tensile strengths and moduli than broad MWD types. Expressed another way, narrow MWD polymers can accept higher levels of filler and plasticizer than the wide MWD types for equivalent properties—a possible factor in lower cost. Indeed, narrow MWD types may require higher levels of filler and plasticizer to attain the extrusion rates and degrees of smoothness characteristic of the broad MWD types. These considerations of MWD are summarized in Table III.

Fillers

Although the compounding of EP rubber for general industrial applications employs a wide variety of fillers, these choices are substantially narrowed by various requirements of the electrical industry, including physical properties, electrical losses, electrical stability, breakdown strength and moisture absorption. Furthermore, as has been stated previously, peroxide cross-linking is incompatible with acidic fillers. And finally, as the voltage rating increases, both the type, level and purity of the filler are substantially restricted.

Low voltage applications can use several filler possibilities, some of which are restricted to specialty constructions for specific effects—e.g., flame retardant fillers such alumina trihydrate (ATH) and magnesium hydroxide—which will be discussed in the section below entitled "Flame Retardants."

The more conventional fillers in low voltage applications are water-washed hard clay, calcium carbonate or whiting, barium sulfate or barytes and various types of synthetic silica. The clay is water-washed to remove acidic components that interfere with the free radical peroxide decomposition. The terms "hard" and "soft" as applied to clay refer to its reinforcing effect in rubber and not to the intrinsic hardness of the mineral.

Experience in industry has shown that as the voltage requirements increase, choices of mineral fillers are sub-

Table IV
Comparison of Treated and
Untreated Calcined Clay in EPR (110 phr)

Physical Properties	I	II
Original		
100% Modulus, MPa (psi)	3.9 (560)	5.4 (780)
200% Modulus, MPa (psi)	4.4 (640)	8.3 (1210)
Tensile strength, MPa (psi)	4.8 (700)	8.3 (1210)
Elongation	370	200
Oven Aged, 7d, 150°C		
Tensile strength, % of orig.	143	98
Elongation, % of orig.	54	95
Oven Aged, 14d, 150°C		
Tensile strength, % of orig.	141	94
Elongation, % of orig.	46	90
Moisture absorption, 90°C water (mg/sq inch)		
1 wk.	4.0	0.9
2½ wks.	4.7	1.2
Dielectric constant, 1 kHz in 90°C water		
Original	3.45	3.41
1 wk.	4.30	3.48
2½ wks.	4.09	3.52
% Power factor, 90°C water at 1 kHz		
Original	0.52	0.50
1 wk.	8.98	0.44
2½ wks.	6.12	0.37

stantially restricted to untreated and surface-treated calcined clays and to platy structures such as talc. Calcined clay and talc have largely been replaced in any but the lower voltages by surface-treated calcined clays (see discussion of coupling agents), which are much superior in electrical losses, electrical stability and moisture absorption.

Table IV compares some critical values obtained with an untreated and a treated calcined clay at the same level in the polymer. The original and aged properties of the EPR compound based on the surface-treated clay (Table IV, column II) are markedly improved over those of the untreated clay (Table IV, column I). Furthermore the former exhibits much lower moisture absorption at 90°C and better electrical stability.

Talc, even with a surface treatment, is markedly inferior to a treated clay at an equal level in EPR in 75°C water as shown below. Treated talc meets the requirements only for Type II, O-2kV, in ICEA-S-68-516/NEMA WC-8, while the clay-filled compound can be used at much higher voltages.

Dielectric constant, 75°C water At 80v/mil, % increase	Treated clay	Treated talc
1 - 14d.	+1.5	+4.5
7 - 14d.	-0.5	+1.5

As one moves to lower voltages and electrical requirements are reduced, filler types can cover a wider range and the levels can be substantially increased. In

such cases the EPR type is selected from the range in the upper right of Fig. 1, and it is not unusual for polymer levels to be as low as 20% in portable and flexible cord insulations, the remainder being largely filler(s) and plasticizer. At these voltage levels, high performance fillers are rarely used, and unless the insulation is to meet some special requirement, water-washed hard clay admixed with whiting are the fillers of choice.

For any given combination of EPR and filler there is an optimum range of filler level. If the filler level is too low, extrusion rates will be low and it will be difficult to achieve smooth extrudate surfaces. If the filler level is too high, elongations will be too low and electrical properties will be significantly reduced.

Small amounts of special fillers can be incorporated into insulation compounds for special purposes. Zinc oxide at levels as high as 20 phr generally aids heat ageing but is quite expensive due to its high density. Hydrated alumina is used at 120-200 phr in compounds requiring track resistance or low smoke evolution when burned. Titanium dioxide at 2 phr or below provides a neutral color base for colored insulations but at some sacrifice of dielectric constant. Thermal blacks at 2 phr or below are also used as colorants and at these levels have only a slight detrimental effect on electrical properties. Reinforcing blacks are sometimes used at levels up to 30 phr in telephone wire for cost reduction while still maintaining acceptable electrical properties for this application.

Plasticizers

As the level of filler(s) increases and as the molecular weight of the base EPR increases, extrusion or molding become more difficult, requiring higher pressures and temperatures and often accompanied by a loss of surface smoothness. Plasticizers are added to reduce the compound viscosity and improve the processing characteristics. This is achieved with an increase in elongation, a reduction of tensile properties and some loss of electrical properties. Obviously, the use of plasticizers should be carefully considered, and avoided if possible, if the insulation is to be used in high performance applications

At lower voltages, plasticizers are used primarily for cost reduction, achieved because their use permits much higher filler levels. In low-cost flexible cords, it is not uncommon for plasticizer levels to exceed 50 phr.

The proper choice of a plasticizer is critical in peroxide cures because some plasticizers are potent free-radical traps and can retard or even completely prevent crosslinking. As the plasticizer becomes more saturated and linear, it becomes less reactive toward the peroxide. Thus petrolatum, paraffin waxes, highly purified paraffinic oils and even low molecular weight polyethylenes

are acceptable. Paraffinic oils designed for this purpose are extremely pure, with very low levels of phenolics, naphthenics and aromatics. For special purposes, saturated dibasic acid esters and chlorinated paraffins are sometimes used. The former class will provide improved low temperature flexibility at some increase of electrical losses. The latter will improve flame retardancy (generally in combination with other flame retardants) with an increase in electrical losses and a decrease in low temperature flexibility.

If sulfur cures are used with terpolymers, plasticizer choices and purity requirements are far less restrictive. Naphthenic oils are commonly used for these compounds and they are considerably less expensive than the highly-purified paraffinics.

Antioxidants and Stabilizers

Antioxidants function by serving as a trap for the free radicals generated during oxidation. Unfortunately, this function also interferes with the peroxide crosslinking that proceeds through free radicals. These modes of operation that conflict with each other serve to restrict the number and types of suitable antioxidants for crosslinked EPR.

As has been stated, peroxide cures may be retarded or even halted completely when used in mineral-filler EP rubbers that are even slightly acidic. This acidity may arise from the mineral filler or from the base polymer. Many EP rubbers are themselves slightly acidic, which arises from a residual catalyst as well as from the acids used to isolate and wash the polymer during manufacture. This "inherent" polymer acidity is less common now that high-purity EP rubbers are being manufactured for the medium voltage electrical industry. In any case, it has long been known that antioxidants that are members of the class of poly-dehydroquinolines are most effective in mineral-filled EPRs. In this case, their alkaline nature permits them to act as cure activators, in addition to their antioxidant activity. This class is extremely effective in EPR, is low in cost and is easily mixed and dispersed even at its relatively high use levels of up to 2 phr. This general class of aromatic amines will develop yellow-tan colors over time, which are largely masked by the pigmentary value of the mineral filler.

For insulations intended for continuous service at 150°C or above, an antioxidant combination consisting of a polymeric condensation product of diphenylamine and acetone (1 phr) and the zinc salt of mercaptotolyimidazole (2 phr) is used. Although this combination is very effective as an antioxidant system, it is quite expensive, and the zinc salt must be carefully dispersed to avoid unacceptable losses in breakdown strength.

Non-staining antioxidant types, such as the alkylated bisphenols combined with thiodipropionase synergists commonly used in XLPE, are largely ineffective in providing adequate oxidative protection in EPR.

Zinc oxide, previously mentioned as a filler, is known to have a stabilization effect at high levels (for instance, 20+ phr) in a peroxide-cured EPR. It is believed to function as an acid acceptor, reacting with acidic materials arising from residual catalyst in the polymer and those generated by the mineral filler. Due to its high density it is quite expensive, and its value as a stabilizer is less clear in modern EPRs, which now contain much lower levels of residual catalyst. Its stabilization effect is not evident at lower levels in peroxide-cured EPR. In a sulfur-cured EPR zinc oxide is required as an accelerator/activator for the cure. Used at the 5 phr level it is believed to function by solubilizing some of the cross-linking chemicals in the EPR matrix.

Process Aids

Broadly defined, this category may also include the plasticizers discussed above, but it also includes additives, which are generally used at much lower levels and are intended to provide some of the following benefits:

- Easier release from the hot metal surfaces of mixing equipment.
- Easier incorporation of fillers during mixing, with shorter mixing cycles, better dispersion and lower energy consumption.
- Better extrusion performance, including smoother surfaces and lower energy consumption.

Because of the restriction of electrical losses for medium voltage insulation in industry specifications, these process aids are limited to those based on hydrocarbon building blocks such as petrolatum, paraffin waxes and low molecular weight polyethylenes. But at lower voltages other types of process aids are used, including long chain fatty acids, their esters, amides and their salts. These latter types all tend to increase electrical losses and, in some instances, interfere with a peroxide cure. Another deleterious effect of these process aids involves the over-lubrication of the compound. Over-lubrication results in slippage of the compound in the extruder barrel, resulting in reduced extruder output and/or difficulties in dimensional control. For this reason it is important to use the minimum level of process aid to produce the desired effect.

Ion Scavenger

It has long been known that various lead compounds serve as stabilizers for mineral-filled EPR insulations used in wet applications. It is believed that these lead compounds function as efficient anion scavengers,

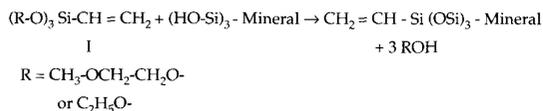
which would otherwise cause large increases in dielectric constant and power factor. These anions arise largely from the mineral filler, being extracted from the mineral matrix by the mild acidity of rain or ground water (due to dissolved carbon dioxide). Silicate, sulfide, sulfate and chloride salts of lead are extremely insoluble with solubility products among the lowest of all inorganics.

The lead oxide most frequently used for this purpose is red lead oxide, Pb₃O₄, which imparts the salmon-pink-to-red color often considered characteristic of EP insulations. Generally used at up to 5 phr, it is the least costly of the lead compounds known to serve as ion scavengers and is available in a pigmentary form. Other suitable lead compounds are litharge, (PbO light yellow) and dibasic lead phthalate (white). These two are considerably more expensive than red lead and are usually used only in insulation compounds that are to be colored other than the characteristic salmon pink. Table V summarizes data showing the effect of red lead.

Because of the high toxicity of lead compounds and the particularly severe hazards presented by their dusts, they are all available to the rubber compounder as pre-dispersed, dust-free concentrates in various carriers such as paraffin wax, petrolatum, mineral oil and EP rubber.

Coupling Agent

Organofunctional silanes, commonly referred to as "coupling agents," have the ability to react with silanol groups at the surface of silicate mineral fillers such as talc and clay. This reaction is known as "silanol condensation." These silanes contain an organofunction at the other end of the molecule, generally a vinyl group which can undergo crosslinking with the EP polymer matrix. The net result is that the polymer and the filler are held together by a chemical bond rather than by simple wetting of the filler surface. In peroxide-cured EPR for wire and cable insulation, the most commonly-used organofunctional silane is I, a tris-alkoxy-vinylsilane. This silane reacts with surface silanol groups thusly:



The surface-modified mineral now contains vinyl functions which, in the presence of peroxide-generated free radicals, will crosslink with the EP matrix and create a continuous organic phase. This bonding increases modulus and improves electrical stability in hot water (75°C and up).

Because these silanes are liquids that are highly reactive to atmospheric moisture, they are more conven-

Table V
Effect of Red Lead Stabilizer on Long Term Wet Electrical Stability (60 phr treated clay in EP rubber)

Power factor, %, 60 Hz in 90°C water, tested at 80v/mil	With 4.5 phr Red Lead	Without Red Lead
Original	0.46	0.51
1 wk.	0.47	0.51
2 wk.	0.47	0.54
1 mo.	0.48	0.63
2 mos.	0.51	0.72
4 mos.	0.59	0.88
6 mos.	0.57	1.03

iently handled as dispersions (ca. 40 wt.%) in a low-melting paraffin wax. In the most critical medium-voltage applications in which they are used as a supplemental treatment for the surface-treated calcined clay, 1 phr of active silane is usually sufficient. For less critical applications, a treated calcined clay may be directly produced in the mixing step by treatment of an unmodified calcined clay with a larger amount of the functional silane. Generally this requires 2 phr or more of the vinyl silane, depending on the amount of clay in the insulation compound.

Flame Retardants

Since EP rubber is a hydrocarbon with a high energy content, its vulcanizates will support and sustain combustion if not properly compounded. Many wire constructions using EP insulations, such as integral singles for nuclear cable, tray cable, secondary network cable, building wire and appliance wire, must meet flame tests as required by ICEA, UL and CSA. For optimum flame resistance it is important to minimize or even eliminate combustible ingredients other than the EP rubber, such as plasticizing process oils and waxes in the composition.

Alumina trihydrate or aluminum hydroxide, Al(OH)₃, is an excellent flame retardant but non-reinforcing filler if the purpose is to pass a single application of flame. It evolves copious quantities of water at temperatures above 200°C, which function to cool the flame, dilute the flammable gases and prevent access of the flame to oxygen by blanketing the flame front with water vapor. Unfortunately, in company with other inorganic hydroxides and carbonates, its wet electrical stability is poor, and these additives may only be utilized in insulations that are not exposed to moisture during operation. These inorganics are normally used at levels of 120 phr and above to achieve an adequate level of flame retardancy. At these levels, they drastically reduce the physical toughness of the vulcanizates. Finally, these inorganics generally evolve less smoke

and acidic gases than the organohalides (see below) often used as flame retardants.

The other major class of flame retardants is the synergistic combination of a polyhalogenated organic and antimony trioxide, Sb_2O_3 . Typical polyhalogenated organics used in EP rubber are decachloro-dicyclopentadiene and decabromo-diphenylether, but many others are also available. Used alone, these polyhalo compounds provide only a limited degree of flame retardancy, functioning primarily by evolving HCl or HBr gas, which prevents access to the flame by oxygen and by interfering with free radical reactions in the flame front. Antimony trioxide serves as a synergist for this flame retardancy, permitting the use of much lower levels of polyhalide. It is believed that antimony trioxide reacts with the hydrogen halide in the flame, producing antimony trichloride or tribromide, both of which are volatile, have high vapor densities (to displace oxygen) and are potent free radical traps. Effective levels of the polyhalide range from 15 to 40 phr with a weight ratio of halogen-to-antimony in the range of 2 to 5.

These organohalide/antimony oxide combinations, when properly dispersed, provide excellent resistance to the repeated application of high energy ignition sources and show excellent wet electrical stability. Their use makes possible the manufacture of EP-insulated nuclear power and control cables that pass LOCA testing, as well as building and instrument wire. Their smoke evolution in an ignition source is heavier than with the metal hydroxides, and, of course, they evolve halogenated acidic gases.

Summary

This paper reviews the basic compounding technology of ethylene propylene rubber for electrical insulation. The factors involved in the proper selection of an EP rubber for a given end-use are discussed. It also discusses the various components of a practical insulation compound, as well as their characteristics and their effects on the physical, electrical and rheological properties of the compound.

Morton Brown received the B.A. degree in chemistry from Cornell University in 1952, the M.A. degree from Duke University in 1954 and the Ph.D. from the Massachusetts Institute of Technology in 1957. After a National Science Foundation Postdoctoral Fellowship program at M.I.T., he joined DuPont in Wilmington, Delaware, retiring after 33 years of service as a senior technical consultant. In that capacity he was responsible for polymeric materials development for solid dielectric, semi-conductive and jacket materials for cable. Since 1990 he has been a technical consultant to A. Schulman Inc. of Akron, Ohio, manufacturer of ethylene-propylene insulation compounds.

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