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<b>(54) Title:</b> ENCAPSULATING COMPOUND AND ARTICLES COMPRISING SAME		
<b>(57) Abstract</b> <p>A polyurethane-based cable splice encapsulant further comprises about 25-70% b.w. ester plasticizer, and, optionally, no more than 35% b.w. diluents, preferably less than 15% b.w. diluents, and has a cure time of at least 50 minutes at 25%. The relatively long cure time, together with other properties of the material, including a viscosity typically between 0.15 and 1.5 Pa.s (150 and 1500 centipoise), and relatively good 'bonding' to plastic-insulated conductors, results in improved water-intrusion resistance of splices enveloped in the novel encapsulant.</p>		

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## ENCAPSULATING COMPOUND AND ARTICLES COMPRISING SAME

Field of the Invention

This invention relates in general to compounds  
5 for protecting matter from hostile ambients by  
encapsulating the matter, and to matter so encapsulated.  
In an important specific application, this invention  
relates to encapsulating compounds for cable splices.  
Still more specifically, it relates to encapsulating  
10 compounds for splices in multiconductor telecommunications  
cables, e.g., telephone cables.

Background of the Invention

The need to protect an article, or a part  
thereof, from a hostile environment exists in many areas of  
15 technology and commerce. For instance, it is often  
necessary to protect electrical equipment or components  
against contact with water or moisture. A particular  
example of this is the need to so protect joints or splices  
between electrical conductors, e.g., splice points between  
20 multiconductor communications cables.

A frequently used technique for protecting such  
splice points comprises providing an enclosure around the  
completed splice, and introducing a curable or gelable  
liquid into the enclosure. The liquid should ideally fill  
25 all the voids between the conductors and other splice  
components and form closely adhering interfaces with all  
the surfaces it contacts within the enclosure, thereby  
reducing the possibility of water migration along such  
interfaces, which can result in water contact with  
30 conductors or other sensitive splice components.

An encapsulating compound for cable splices and  
the like should ideally also have other desirable  
characteristics, such as compatibility with all the  
materials it contacts, chemical stability over a wide range  
35 of operating temperatures, good electrical properties, be  
reenterable, nonsagging at high temperatures, retain  
acceptable mechanical properties at low temperatures, be

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nontoxic to personnel in both the liquid and the cured state, and be acceptable under applicable environmental laws.

Although no prior art encapsulating compound  
5 fully possesses all these desirable characteristics, splice encapsulants are in widespread use, in particular, in the telephone industry.

Among the currently most frequently used  
encapsulant are polyurethane-based material compositions,  
10 especially mineral oil extended compositions. For instance, U. S. Patent 4,168,258, issued September 18, 1979 to M. Brauer et al, discloses a cured, cross-linked grease-compatible polyurethane-based material comprising mineral oil and a coupling agent, useful for sealing an insulated  
15 electrical device. See also U. S. Patent Re. 30,321.

Similarly, U. S. Patent 4,329,442, issued May 11, 1982 to R. J. Pokorny, discloses a polyurethane prepared from an aliphatic or cycloaliphatic isocyanate, a polydiol, a  
20 a tri- or tetra-functional aliphatic polyol, a monofunctional aliphatic alcohol, and a catalyst, the composition being useful as an encapsulating compound, said to have good adhesion to greasy insulation material.

Prior art encapsulants, especially cable splice encapsulants, generally are formulated to have relatively  
25 short cure (gel) times. For instance, an encapsulant that has recently become available commercially is said to have a gel time of 20 minutes at 23°C, and a gel time at -18°C of only 35 minutes. The art considers short gel time to be advantageous, since it reduces the time required to  
30 complete a splice, and allows speedy job completion.

Prior art encapsulants, which often have initial mixed viscosity in the order of 0.3-1.5 Pa.s (300-1500 cps) (centipoise) at room temperature, tend to have rapidly  
35 increasing viscosities, due to their short cure time. This sometimes results in poor penetration of encapsulant into the splice bundle.

Prior art known compounds, useful in

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rehabilitation of water-logged cable, have very low initial mixed viscosity (e.g., about 0.1 Pa·s (100 cps) at 35°F, about 10 cps at room temperature) and long cure time. See, for instance, U. S. Patent 3,733,426. Such compounds  
5 typically are not useful as splice encapsulants, due to their tendency to stress crack polycarbonate.

Although widely used, prior art cable splice encapsulation systems do not provide fully satisfactory protection against water intrusion into the splice work,  
10 and the resulting damage and deterioration of service. As a consequence, there exists a need for a splice system that retains the important advantages of prior art systems while, at the same time, being capable of giving improved protection against water intrusion.

15 Glossary of Terms

A "cable" herein is an article comprising a multiplicity of elongated bodies, typically metallic bodies (conductors) but including also dielectric bodies of the type used as optical fibers, and further comprising a  
20 dielectric sheath surrounding the elongated bodies. It is to be understood that cables typically also comprise other constituents, such as coatings or insulation around the individual fibers or conductors, strength members, metallic shielding, and filling material. Examples of cables  
25 according to the definition are multiconductor communication cables, e.g., telephone cables, and so-called distribution wire and service wire used in telephone systems. Further examples are coaxial cables of the type used in CATV systems, power cables, including cable  
30 containing power distribution wire or service wire, and optical fiber transmission or distribution cable. Cable containing service wire typically is used to connect a customer to a distribution system, and cable containing distribution wire is typically used to branch a higher-  
35 capacity cable. Cable according to the definition can be adapted to be below ground by direct burial or in buried conduit, or to be maintained above ground.

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By "curing" or "gelling" of the encapsulant is meant herein a linking process, including crosslinking, resulting in a substantial increase of the viscosity of the encapsulant.

5           The "cure time" or "gel time" at a given temperature herein is the time required for the viscosity of encapsulant at the temperature to increase from the initial value (typically the value immediately after mixing together all the components) to about  $10^5$  cps. All cure  
10 times herein are understood to be  $\pm 15$  minutes, unless stated differently.

A "prepolymer" is an adduct or reaction intermediate of a polyol and a monomeric isocyanate, in which either component can be in considerable excess of the  
15 other.

Unless defined differently herein, all terms herein are intended to have their customary meaning, as defined, for instance, in The Condensed Chemical Dictionary, 10th edition, Van Nostrand Reinhold Company,  
20 New York.

#### Summary of the Invention

This application discloses encapsulants having novel properties that result in improved water intrusion resistance. As discussed above, the prior art teaches that  
25 encapsulants of the type used to protect cable splices desirably have a short curing time. We, on the other hand, have found that increased protection of splices against water damage can be obtained with encapsulants having relatively long cure time. Although the inventive  
30 encapsulant can be used in prior art splicing systems, it is advantageously used in a forced encapsulation system.

The increased cure time results in more thorough filling of voids and interstices within the splice enclosure (e.g., the elastomer bag of the forced  
35 encapsulation system) and the cable ends, leading to improved resistance of the splice system to water intrusion and the thereby caused electrical problems.

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The correctness of this novel approach is verified by means of a water intrusion test, to be described later. In this test, splices encapsulated with a prior art encapsulant that is widely used in the telephone industry, having a cure time at 26.7°C (70°F) of 30 minutes, typically failed after about 1-3 days with a 1.524m (5 ft.) water head (cable filled with an oil-extended thermoplastic rubber). In the same test, similar splices encapsulated with the same, but uncatalyzed, encapsulant [cure time at 26.7°C (70°F) about 20-24 hours] typically failed after about 17 days with a 1.524m (5 ft.) water head. The improvement in this case clearly is dramatic. Other advantages of the inventive encapsulant will be discussed below.

In a broad sense the inventive composition of matter is a curable material comprising at least one isocyanate, at least one polyol, at least one ester plasticizer, and, optionally, one or more diluents, the material having a cure time of at least about 50 minutes at 25°C. The invention also comprises the above composition of matter, in combination with matter comprising a splice between at least two cables, the splice enveloped in the encapsulant, and the composition of matter, in combination with matter comprising at least one cable, such as in end caps and cable stubs.

The encapsulant is advantageously used by mixing, at the splice site, the previously prepared components of a two- (or multi-) component preparation, pouring the encapsulant into a splice enclosure, where it cures in situ.

As is well known, polyurethane is produced by the condensation reaction of an isocyanate and a hydroxyl-containing material such as a polyol. The total amounts of isocyanate and of polyol in the uncured encapsulant embodying the invention advantageously are such that the cured encapsulant comprises between about 20% and about 60% polyurethane, preferably between about 30% and 50%, by

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weight of the cured encapsulant, the balance comprising diluent(s) and/or plasticizers. All compositional percentages herein are intended to be weight percent of the cured encapsulant, unless stated otherwise. The weight fraction of polyurethane is typically chosen to result in a cured encapsulant having appropriate characteristics, including relatively low tear strength, relatively high pull-out strength, good resistance to water intrusion, and compatibility with cable filling compounds and splice components.

Both aliphatic and aromatic isocyanates are useful in the practice of the invention. The average number of -NCO radicals advantageously is from about 1.2 to about 1.0 per hydroxyl radical. Diluents, of the type to be discussed below, can be added to the isocyanate component as well as to the polyol component, to adjust the mix ratio, and possibly for other reasons.

A variety of hydroxyl-terminated materials can be used to practice the invention, including Castor Oil, polyether and polyester polyols, and hydroxyl-terminated polybutadiene (HTPBD). Criteria for selection include low water absorption, hydrolytic stability, and reactivity. A currently preferred polyol is a HTPBD.

The inventive encapsulant can also comprise one or more substantially unreactive components (diluents), as well as, typically, an ester (or combination of esters) plasticizer. A variety of esters can be employed in the practice of the invention, including esters based on phthalic acid and trimellitic acid, as well as esters of adipic acid and similar diacids.

As is well known, esters can cause stress cracking of polycarbonates, a material frequently present in cable splices (e.g., in connectors). The likelihood of stress cracking decreases with increasing molecular weight, and therefore viscosity, of the ester. On the other hand, the viscosity of the ester should be relatively low, in order to achieve a relatively low-viscosity encapsulant.



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Esters having viscosity between 0.05 and 0.5 Pa.s (50 and 500 cps) at 25°C can be advantageously used in the inventive encapsulant.

The weight fraction of esters typically is  
5 between about 25% and about 70%, preferably between about 35% and 60%. The amount advantageously present depends, inter alia, on the stress cracking potential, the viscosity and the hydrolytic stability of the ester, and the desired dry heat aging characteristics of the encapsulant.

10 Encapsulant according to the invention may also comprise hydrocarbon oil, typically less than 35%, preferably less than 15%. Some preferred compositions are substantially free of hydrocarbon oils. The hydrocarbon  
15 saturated synthetic oils, unsaturated oils, as well as mineral oils.

Mineral oils herein are those aliphatic, cycloaliphatic and branched aliphatic saturated hydrocarbons that contain about 15-20 carbon atoms and are  
20 distilled from petroleum. Included are naphthenic as well as paraffinic oils, with naphthenic oils being preferred. Also included are the above oils having a substantial proportion (typically about 5-25% b.w. of the oil) of  
25 aromatic molecules. Aromatic oils generally are compatible with polyol and are typically inexpensive diluents, but tend to be volatile and toxic, and can result in stress cracking of polycarbonate.

Saturated synthetic oils, e.g., olefins of the type disclosed in U. S. Patent 4,355,130, and  
30 U. S. Patent 3,338,861, can be used in the inventive encapsulant. These oils typically are inexpensive diluents that can reduce the likelihood of stress cracking of polycarbonate. Although use of the above mineral and  
35 synthetic oils may be advantageous for cost reasons, the amount added typically should be minimized, in order to get improved encapsulation performance.

Another type of synthetic oils, the unsaturated

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oils, also referred to as liquid rubbers, e.g., unsubstituted polybutadiene or polyisoprene, or depolymerized natural rubber, are also contemplated as diluents.

5           An important aspect of the inventive material being its relatively long cure time, the amount of catalyst present is of significance. A widely used catalyst, dibutyl tin dilaurate, has been found to be useful in the practice of the invention. Other known initiators, such as  
10 organophosphorous and organoarsenic compounds, are also contemplated. Use of a catalyst is optional, depending, inter alia, on the desired cure time. Catalyst-free encapsulants according to the invention, which may, dependent on the amount and type of polyol present, require  
15 up to 20 hours and more to cure at room temperature, can form highly water-resistant seals, and such compositions are among the currently preferred ones.

          Since long gel times are desirable in material according to the invention, it is in principle not  
20 necessary to prepare a prepolymer of the isocyanate component. However, for health and safety considerations, some prereaction may be desirable.

          A further aspect of the invention is the typically relatively low mixed initial viscosity of the  
25 encapsulant, typically between 0.15 and 1.5 Pa·s (150 and 1500 cps) at 25°C. In some preferred compositions this viscosity is about 0.4-0.9 Pa·s (400-900 cps). This relatively low viscosity contributes to thorough penetration of the splice work, and often to some  
30 penetration into the cable ends, resulting in good water-intrusion-resistance of the encapsulation.

#### Detailed Description

          A significant aspect of the invention is the discovery that the water-intrusion resistance of  
35 encapsulated splices typically can be improved by using a liquid encapsulant having relatively long (longer than about 50 minutes at 25°C) curing time, while maintaining

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(or improving upon) the other relevant material properties of the encapsulant. These other properties include initial mixed viscosity, low tear strength and compatibility with all materials present in the splice, including, possibly, 5 preexisting encapsulant in reentered splices. Some relevant aspects of compatibility are: absence of stress cracking, absence of gross materials changes at interfaces (e.g., formation of "oily" interfaces, tackifying of polymeric surfaces), and relatively high pull-out strength 10 of encapsulated (plastic insulated) conductors precoated with cable filling compound.

Compositions embodying the invention are advantageously prepared on site by mixing of two separate previously prepared materials, although all the individual 15 components could be mixed together on site, if so desired.

Typically, although not necessarily, one of the previously prepared materials comprises an isocyanate-terminated prepolymer. The prepolymer can be formed by known methods (see, for instance, U. S. Patent Re. 30,321,) 20 from aliphatic or aromatic isocyanates, or from appropriate derivatives. Aliphatic isocyanates useful in the practice of the invention include hexamethylene diisocyanate (HMDI), isophorone diisocyanate (IPDI), 3-isocyanatemethyl, and 3, 5, 5 trimethylcyclohexyl isocyanate. Such aromatic 25 isocyanates include 4, 4'-diphenylmethane diisocyanate (MDI), and 2, 4-tolylene diisocyanate (TDI). Appropriate derivatives of isocyanates include a polymethylene phenyl isocyanate available from Upjohn Company, LaPorte, Texas, under the trade name PAPI 94, and a polymethylene 30 polyphenyl isocyanate available from Mobay Chemical Corporation, Pittsburgh, Pennsylvania, under the trade name MONDUR MRS-10. Other desirable polyols are hydroxyl-terminated polyisoprene, and combinations of butadiene and/or isoprene with other monomers, such as styrene. In 35 general, the derivatives comprise aromatic and aliphatic isocyanates substituted with other organic and inorganic groups that do not adversely affect the course of the

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chain-extending and/or cross-linking reaction. Isocyanates useful in the practice of the invention typically have relatively low average "functionality", between about 2 and about 2.5.

5           An isocyanate-terminated prepolymer can be prepared by reacting an excess of an appropriate isocyanate with one or more polyols, typically polyol of molecular weight between about 1000 and about 6000. Among the polyols useful in the practice of the invention are those  
10 selected from compounds based essentially on HTPBD, hydroxyl-bearing ethers and/or esters, Castor Oil, or combinations thereof. Polyols of the same type can also be used in the second part of a two-part preparation, also referred to herein as the polyol part.

15           Criteria for selection of polyols comprise low water absorption and appropriate reactivity of the polyol, as well as good hydrolytic stability and flexibility of the encapsulant formed therewith. The currently preferred polyols belong to a family of liquid, hydroxyl terminated  
20 polymers of butadiene with a number average molecular weight of about 3000, and degree of polymerization typically in the range of 50, available from ARCO Chemical Company, Philadelphia, Pennsylvania, under the trade name POLY BD.

25           Polyether and/or polyester polyols are used to reduce the viscosity and to reduce the reactivity. For instance, use of polyols having secondary or tertiary hydroxyls, e.g., polyether diols and triols, reduces the rate of reaction, and delays onset of gelation, relative to  
30 a similar system using primary hydroxyl polyols. On the other hand, use of higher molecular weight polyol typically results in higher viscosity of the mixed encapsulant.

          Exemplary polyether polyols are propylene oxide adducts of diol starters, propylene oxide adducts of triol  
35 starters, ethylene oxide adducts of polyoxypropylene diols, and ethylene oxide adducts of polyoxypropylene triols, all available from Union Carbide Corporation,

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Danbury, Connecticut, under the trade name NIAX POLYOLS. Other exemplary polyether polyols are available from DuPont, Wilmington, Delaware, under the trade name TERACOL. These latter substances are polytetramethylene  
5 ether glycols.

Castor Oil is one of the polyols known to the prior art. Although it is advantageously used in prepolymer formation, its exclusive use in the polyol part is currently not preferred. An exemplary Castor Oil is a  
10 substance consisting substantially of triglyceride of ricinoleic acid, available from CasChem Corporation, Bayonne, New Jersey under the trade name DB OIL.

It will be understood that, in general, the liquid encapsulant, or its precursor materials, has to  
15 comprise enough isocyanate(s) and polyol(s) to result in a 20-60% b.w. polyurethane content of the cured encapsulant, with the balance being diluents, plasticizers, catalyst, fungicides, antioxidants, or other property-modifying additives. Various ways of achieving this end are possible  
20 and known to those skilled in the art, and all these are intended to be within the scope of the invention.

The polyol part of a two-part encapsulant preparation according to the invention typically comprises, in addition to polyol or polyols, one or more nonreacting  
25 esters (a plasticizer), and, optionally, saturated synthetic oils, unsaturated oils, and/or mineral oils, in addition to such optional additives as catalysts.

Exemplary esters are listed, for instance, in Technology of Plasticizers, J. K. Sears and J. R. Darby,  
30 J. Wiley and Sons, 1982, and include phthalates, trimellitates, high molecular weight adipates, azelates, and succinates, and similar esters which meet the criterion of low stress-cracking activity on polycarbonates. Specific examples are 6-10 trimellitates, available from  
35 USS Chemical, Pittsburgh, Pennsylvania, under the trade name PX-336, and ditridecyl phthalates, available from Exxon Corporation, Houston, Texas. Esters typically are

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about 25-70%, preferably 30-60%, of the encapsulant.

In material according to the invention, the proportion of hydrocarbon oils is preferably relatively low, typically less than 35%. Reducing the amount of hydrocarbon from the relatively high amounts present in prior art encapsulants results typically in increased water intrusion resistance by the encapsulant. Mineral oils useful in the practice of the invention are naphthenic as well as paraffinic oils, with the former generally preferred, since they are generally more compatible with HTPBD, and generally have lower pour point than the latter. The naphthenic oil content of the mineral oil is desirably greater than 25%, as determined by carbon type analysis, and desirably have an aromatic content less than 15% by weight of the oil, as determined by clay gel analysis. Desirably, the pour point is less than about  $-9.4^{\circ}\text{C}$  ( $+15^{\circ}\text{F}$ ), and the SUS viscosity is between about 200 and about 400. Such mineral oils are available, for instance, from Sun Oil Corporation, Marcus Hook, Pennsylvania (e.g., under the trade name SUNPAR LW 120), and from Penreco, Butler, Pennsylvania (e.g., under the trade name DRAKEOL 35). Synthetic oils typically are added at about the 5% b.w. level because of their generally relatively high viscosity.

Oils such as polyisoprenes, depolymerized material rubbers, or polybutadienes are typically compatible with HTPBD and thus may be used to replace in part nonreactive esters. This substitution was found to result in improved water resistance of at least some encapsulants. A preferred synthetic oil is polyisoprene, available from Hardman, Incorporated, Belleville, New Jersey, under the trade name ISOLENE XL, having a viscosity of 20 Pa.s (20,000 cps) at  $22.2^{\circ}\text{C}$  ( $72^{\circ}\text{F}$ ). An exemplary butadiene is available from Colorado Chemical Specialties, Inc., Golden, Colorado, under the trade name RICON 157, having viscosity of 7 Pa.s (7000 cps) at  $25^{\circ}\text{C}$ .

In general, hydrocarbon oils of the type

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discussed here are mixtures of various molecular weight oils, including, generally, aromatic as well as naphthenic and paraffinic oils. Aromatic oils are available from Kendrick Oil of Bayonne, New Jersey (e.g., under the trade name KEMPLAST G). Such oils typically cannot be used undiluted because of their tendency to stress crack polycarbonate, as well as their toxicity. For similar reasons, the aromatic content of naphthenic and paraffinic oils should be relatively low, typically no more than about 15% b.w. of the oil (by clay gel analysis). DRAKEOL 35 is substantially aromatic-free.

Catalysts for curing polyurethanes are well known and include dibutyl tin dilaurate, available under the designation T-12 from M&T Chemicals, Inc., of Rahway, New Jersey. The amount of catalyst is to be adjusted to result in the desired gel time, no less than about 50 minutes at 25°C. The exact amount typically depends on the details of the composition and the desired objective, and its determination may require a minor amount of experimentation. A typical level is 0.06% b.w. of polyol. In an encapsulant comprising about 40% polyurethane, this amount of tin catalyst would result in a cure time of about 60 minutes. It is, of course, possible to practice the invention without use of catalyst. The cure time can be adjusted not only by choice of the amount (or type) of catalyst used, but also by proper selection of the proportion of primary or higher polyols with the presence of higher (secondary or tertiary) polyols resulting in longer cure time. However, it is not only the cure time at 25°C that is of concern in materials according to the invention, but also the cure times at higher temperatures.

Currently preferred encapsulant compositions comprise less than 10% hydrocarbon oil, more preferably still, are substantially free of such oils. Furthermore, such compositions comprise between about 50% and about 70% of trimellitate. Some of the trimellitate may be present in the prepolymer part. That part further comprises

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sufficient MDI isocyanate and Castor Oil such that, together with the HTPBD-based polyol in the polyol part, between about 30% and about 50% of the encapsulant formed from the parts, is polyurethane. Furthermore, preferred  
5 compositions have gel times greater than 75 minutes at 25°C, with some preferred compositions being substantially catalyst-free.

The ability of various encapsulants to resist water intrusion into a splice was evaluated by means of a  
10 test in which a simulated splice, comprising typically 50 pairs of standard 0.0646cm (22 AWG) plastic insulated conductor, connected by means of a commercially available connector, inserted into a test cell which was then filled with encapsulant, was subjected to water at a controlled  
15 pressure, indicated in terms of head of water. The electrical resistance between various combinations of wires (corresponding to ring-ground, tip-ground, and ring-tip in a telephone cable) was monitored, with a drop below  $10^9 \Omega$  considered a failure. Increase in time to failure  
20 indicates improved ability to resist water inversion. Table I shows exemplary results of some of these tests and Table II shows compositions of encapsulants used in Table I.



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Table I

## Water Intrusion Test

	line	encapsulant	conductors	water head		days to failure
				(m)	(feet)	
	1	D, catalyzed	b	1.52	(5)	134+
10	2	D, catalyzed	a	1.52	(5)	1-3
	3	D, catalyzed	c	1.52	(5)	<1
	4	D, uncatalyzed	a	1.52	(5)	17
	5	F, catalyzed	a	1.52	(5)	43
	6	F, catalyzed,	a	1.52	(5)	3
15		partially reacted				
	7	F-1, catalyzed	a	1.52	(5)	121
	8	F-1, Catalyzed	a	1.52	(5)	1
		partially reacted				
	9	N-6XL, catalyzed	a	1.52	(5)	59
20	10	C-1, 1/2 catalyzed	c	1.52	(5)	2
	11	C-1, uncatalyzed	c	1.52	(5)	35+
	12	D, catalyzed	b	2.44	(8)	<1
	13	D, uncatalyzed	a	2.44	(8)	<3
25	14	F, catalyzed	a	2.44	(8)	<5
	15	F, uncatalyzed	a	2.44	(8)	42+
	16	F-1, catalyzed	a	2.44	(8)	22
	17	F-1, uncatalyzed	a	2.44	(8)	69+
	18	N-6XL, Catalyzed	a	2.44	(8)	14
30	19	F-17B, catalyzed	a	2.44	(8)	1
	20	F-17B, uncatalyzed	a	2.44	(8)	88+
	21	F-1, uncatalyzed	a	3.05	(10)	69+

Table II

Composition of Encapsulants of Table I

Encapsulant	Polyurethane (%)	Ratio prepolymer/polyol	HTPBD (%)	Polyether polyol (%)	Diluents & Plasticizers (%)			
					(1)	(2)	(3)	(4)
D(a)	34.9	17/83	32.5	-	25.2	42.3	-	-
D(b)	34.9	17/83	32.5	-	25.2	42.3	-	-
F	49.2	21.4/78.6	51.1	-	17.8	31.1	-	-
F-1	49.0	21.2/78.8	49.4	-	50.6	-	-	-
N-6XL	45.7	19.7/80.3	46.4	-	45.5	-	8.1	-
C-1	40.0	17.7/82.3	40.2	-	-	-	-	59.8
F-17B	50.0	82.9/77.1	31.2	20.6	48.2	-	-	-

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Key to Table I: for compositions, see Table II:

"catalyzed": gel times 20-40 minutes;  
5 "1/2 catalyzed": gel time about 65 minutes;  
"uncatalyzed": gel times about 20 hours;  
(all gel times are at room temperature);  
conductors "a": had been in contact with oil extended  
thermoplastic rubber filling compound;  
10 conductors "b": had not been in contact with any filling  
compound;  
conductors "c": had been in contact with petroleum jelly  
filling compound;  
days to failure: "+" indicates that test is beyond the  
15 given number of days

Notes: i) An isocyanate-terminated Castor-Oil-based  
polyurethane prepolymer (comprising polymeric MDI, Castor  
Oil, and dioctyl adipate) available from CasChem, Inc.  
20 under the trade name VORITE 715 M-1, was used as the  
prepolymer part in all compositions listed in Table II.

ii) The percentage of polyurethane is weight percent  
of the cured encapsulant, all other percentages are weight  
25 percent of the polyol part.

Key to Table II: a) catalyzed: a HTPBD-based polyol,  
available from CasChem, Inc. under the trade name POLYCIN  
934-M3, was used. The composition of the polyol was  
30 established using IR analysis methods.

b) uncatalyzed: as in a), except that trade name is  
POLYCIN 934-M4

c) polyether triol NIAX LG56  
1) dioctyl adipate  
35 2) white mineral oil, DRAKEOL 35  
3) synthetic oil, ISOLENE XL 20097-3  
4) 6-10 trimellitate

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Table I shows, inter alia, that typically the water intrusion resistance depends on the presence or absence of a filling compound and, where present, on the type of filling compound. Compare, for instance lines 1 and 2, and lines 2 and 3, of Table I. Furthermore, the data shows that typically longer cure times, in excess of about 60 minutes, result in improved water intrusion resistance. See, for instance, lines 2 and 4, 10 and 11, 14 and 15, 16 and 17, and 19 and 20. A further point illustrated by the data is the effect of viscosity and very short gel times. See lines 5 and 6, and 7 and 8. The "partially reacted" compounds were held at room temperature for about 10 minutes after mixing, and then poured. This resulted in high initial viscosity of the encapsulant as poured, as well as in reduced subsequent gel time. The observed effect was a significant reduction in the time to failure. The table also illustrates the sensitivity of time to failure to water pressure. See lines 5 and 14, and 17 and 21. A further significant point illustrated by the data is the effect of composition, in particular, of diluent content, on water intrusion resistance. See lines 2 and 5, and 11 and 13. The beneficial effect of mineral oil reduction or elimination is also apparent. See lines 5 and 7, and 14 and 16.

Bonding of encapsulant to insulated conductors is measured by means of a pull-out test. A predetermined length of conductor [11.4cm (4.5 inches)] is embedded in encapsulant and pulled out (at a rate of 50 mm/minute) on a standard tensile test machine (Instron). Increase in pull-out force indicates improved adhesion of the encapsulant to the insulation. In order to get results representative of actual conditions, wires removed from actual cables (air core, petroleum jelly filled, and oil-extended thermoplastic-filled) were used. Exemplary results are shown in Table III.

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Table III

## Pull-Out Test

	line	encapsulant	conductors	pull-out load	
				N	(lb.)
	1	D, catalyzed	a	10.68	(2.4)
	2	D, catalyzed	b	32.92	(7.4)
	3	F, uncatalyzed	a	16.46	(3.7)
10	4	F-1, uncatalyzed	a	19.57	(4.4)
	5	C-1, uncatalyzed	a	45.37	(10.2)
	6	C-1, uncatalyzed	b	58.27	(13.1)
	7	C-1, uncatalyzed	c	11.12	(2.5)
	8	C-1, 1/2 catalyzed	a	56.05	(12.6)
15	9	C-1, 1/2 catalyzed	c	13.34	(3.0)

Note: see keys to Tables I and II for explanation.

The data of Table III shows, inter alia, the effect of insulator contamination on pull-out strength, with clean (unfilled cable) conductors tending to have higher strength. See lines 1 and 2, and 6 and 7. Furthermore, the type of filling compound is shown to be important. See lines 9 and 10. The results also indicate that long cure time does not have a significant effect on the pull-out strength. This suggests that the improved water intrusion resistance of encapsulants according to the invention is due to their low viscosity and long cure time. Desirably, encapsulants according to the invention have pull-out strength (as measured by the above-described test) of at least about 17.79N (4 lb.) for 0.0646cm (22 AWG) plastic insulated wires whose insulation is treated with oil-extended thermoplastic, and of at least about 8.90N (2 lb.) for such wires whose insulation is treated with petroleum jelly.

Although the preferred application of compositions of matter according to the invention is as cable splice encapsulant, these compositions can typically

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also be employed for making pressure plugs in cables, preferably in filled telecommunications cables. In this application the inventive composition is typically injected under pressure into the cable and allowed to cure in situ.

5 This procedure does typically require no further preparatory steps, and results in pressure plugs that permit splicing of unpressurized cable to pressurized cable.

Other possible uses of the invention composition are as encapsulant in cable end caps, as encapsulant in cable restoration, and as filling compound in cable stubs. End caps are used to protect a cable termination, e.g., the termination of a telephone distribution cable. Plastic insulation is subject to deterioration in some environments, and such cables can be restored by removing the portion containing the cracked insulation, splicing a cable stub to the virgin conductor, and encapsulating the splice. Cable stubs are short lengths of cable that may be attached to equipment such as terminals or repeater housings.

The following examples are intended to further illustrate the practice of the invention, in particular, the synthesis of encapsulants according to the invention.

Example 1: A prepolymer was prepared, substantially as described in U. S. Patent Re. 30,321, Example XIII. The reactants and their amounts were also as given in that example. A polyol mixture was prepared by mixing 40.2 parts b.w. hydroxyl-terminated polybutadiene (POLY BD) with 59.8 parts normal alkyl trimellitate (6-10 TM) until homogeneously dispersed, with a resulting viscosity of about 0.896 Pa·s (896 cps) at 23.9°C (75°F). Mixing 17.7 parts by weight prepolymer with 82.3 parts of polyol mixture (NCO/hydroxyl ratio of about 1.15, in this and all subsequent examples) resulted in encapsulant having a gel time of about 20 hours, relatively low tear strength [4.99N/cm (2.85 lb/inch) width], and good water intrusion resistance (see line 11 of Table I).

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Example 2: A prepolymer of the same composition as in Example 1 was prepared substantially as in Example 1, except that the prepolymer was prepared at room temperature, without any heating step. The polyol mixture had the same composition as the mixture of Example 1. The encapsulant resulting from mixing of the two components had properties substantial as the material of Example 1.

Example 3: A prepolymer is prepared by a procedure substantially as described in U. S. Patent Re. 30,321, Example XIII, except that polymeric MDI is replaced by IPDI, and that the following percentages of materials are used: 25.6% IPDI, 14.9% Castor Oil, and 59.5% dioctyl adipate. All percentages are by weight of prepolymer. A polyol is prepared by mixing 40.2% POLY BD and 59.8% 6-10 trimellitate. Mixing the prepolymer and the polyol results in an encapsulant having a polyurethane content of 40% b.w. and a cure time of about 20 hrs.

For the examples below VORITE 715 M-1 was used. See Notes to Table II.

Example 4: A polyol mixture was prepared as in Example 1, and encapsulant formed by mixing 17.7 parts of the commercial prepolymer with 82.3 parts of the polyol mixture. The properties of the resulting encapsulant were substantially the same as those described in Example 1.

Example 5: A polyol mixture was prepared by mixing 49.4 parts of HTPBD with 50.6 parts of dioctyl adipate. The viscosity of the resulting material was about 0.577 Pa·s (577 cps) at 23.9°C (75°F). Mixing 21.2 parts of the commercial prepolymer with 78.8 parts of the polyol mixture resulted in an encapsulant with a gel time of about 20 hours.

Example 6: A polyol mixture was prepared as in Example 5, to which 0.0165% of T-12 dibutyl tin dilaurate catalyst was added. Adding 78.8 parts of this mixture to 21.2 parts of the commercial prepolymer yielded encapsulant having a gel time of about 30 minutes at 23.9°C (75°F).

Example 7: 22.9 parts of the commercial prepolymer is

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mixed with 77.1 parts of a polyol mixture formed by blending HTPBD with a polyether polyol (NIAX LG56) in a weight ratio 1.86:1 and dioctyl adipate, for a total polyurethane content of the encapsulant of 50% b.w. The  
5 polyol mixture had a viscosity of 0.348 Pa.s (348 cps) at 23.9°C (75 °F). The encapsulant had a cure time in excess of 20 hours, and showed good water intrusion resistance. See line 20 of Table I.



Claims

1. A multipart composition of matter which yields, upon mixing together of the parts, a curable liquid adapted for forming therefrom an encapsulant, characterized  
5 in that the encapsulant comprises between about 20% and about 60% polyurethane, formed from at least one isocyanate and at least one polyol, the encapsulant further comprising at least one ester plasticizer, the total amount of ester plasticizer being between about 25% and about 70%, all  
10 percentages being by weight of the encapsulant, the liquid having a cure time of at least about 50 minutes at 25°C.
2. A composition according to claim 1, characterized in that the liquid has a viscosity at completion of mixing of the parts, this viscosity to be  
15 referred to as the "initial mixed viscosity", the initial mixed viscosity being between 0.15 and 1.5 Pa·s [150 and 1500 centipoise (cps)] at 25°C.
3. A composition according to claim 2, characterized in that the initial mixed viscosity is at  
20 least 0.4 Pa·s (400 cps) at 25°C.
4. A composition according to claim 1, characterized in that the isocyanate is selected from diphenylmethane diisocyanate (MDI), polymeric MDI, and derivatives of MDI.
- 25 5. A composition according to claim 1, characterized in that the polyol is selected from hydroxyl-terminated polybutadiene, polyether, castor oil, and combinations thereof.
6. A composition according to claim 1,  
30 characterized in that the ester plasticizer has a viscosity between 0.05 and 0.5 Pa·s (50 and about 500 cps) at 25°C.
7. A composition according to claim 1, characterized in that the cured encapsulant substantially  
35 consists of 30% to 50% b.w. of polyurethane and 50% to 70% b.w. of ester chosen from the group consisting of trimellitates, phthalates, and combinations thereof.

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8. A composition according to claim 1, characterized in that the multipart composition is a two-part composition.

5 9. A composition according to claim 1, characterized in that the encapsulant further comprises a diluent chosen from the saturated synthetic oils, the unsaturated oils, and the mineral oils, the unsaturated oils selected from polybutadienes, polyisoprenes, and depolymerized natural rubbers, the total  
10 amount of diluent in the encapsulant being no more than 35%.

10. A composition according to claim 9, characterized in that the amount of diluent is less than about 15%, and the liquid has a viscosity at completion of  
15 mixing of the parts, this viscosity to be referred to as the "initial mixed viscosity", the initial mixed viscosity being between 0.4 and 1.5 Pa·s (400 and 1500 cps.) at 25°C.

11. A composition according to claim 9,  
20 characterized in that the mineral oils are mineral oils having a naphthenic content greater than about 25% by weight of the oil, as determined by carbon type analysis, and an aromatic content less than about 15% by weight of the oil, as determined by clay gel analysis.

25 12. A composition according to claim 9, characterized in that the unsaturated oils are unsaturated oils having a viscosity less than about 20 Pa·s (20,000 cps) at 25°C.

13. A composition according to claim 9,  
30 characterized in that the saturated synthetic oils are chosen from polypropylene, polyethylene, and polybutene.

14. A composition according to claim 2,  
characterized in that the isocyanate and polyol content is  
35 such that the encapsulant comprises between 30% and 50% b.w. of polyurethane, and the encapsulant further comprises between 50% and 70% b.w. of ester chosen from the group

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consisting of trimellitates, phthalates, and combinations thereof and at most 10% b.w. of hydrocarbon oil.

15           15. A composition according to claim 1 or 9, characterized in that said composition forms an  
5 encapsulant for a cable comprising a tubular dielectric sheath enveloping a multiplicity of elongate metal bodies, at least some of the metal bodies extending longitudinally beyond an end of the tubular sheath; said encapsulant enveloping at least a part of the metal bodies extending  
10 beyond the tubular sheath.

          16. A composition according to claim 15, characterized by further comprising at least a second cable and a splice between the first and the second cable, the splice being enveloped in the encapsulant.

15           17. A composition according to claim 15, characterized in that the encapsulant forms part of a cable termination.

          18. A composition according to claim 15, characterized by further comprising a cable stub spliced to  
20 the said cable.

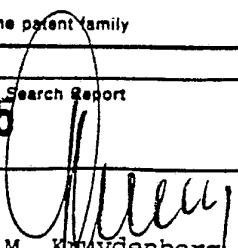
          19. A composition according to claim 1 or 9, characterized by said composition forming a pressure plug in a cable comprising a filling compound and a pressure plug, the pressure plug formed by injection of said curable  
25 liquid into the cable, the curable liquid forming, in situ, a cured material.

          20. A composition according to claim 19, characterized in that the filling compound is selected from petroleum jelly and oil-extended thermoplastic rubber.

30           21. A composition according to claim 15 or 19, characterized in that the cable is a multiconductor telecommunications cable.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US 85/00998

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC <sup>4</sup> : H 01 B 3/30; C 08 L 75/04		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System <sup>1</sup>	Classification Symbols	
IPC <sup>4</sup>	H 01 B C 08 L	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>9</sup></b>		
Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	GB, A, 2014595 (NL INDUSTRIES) 30 August 1979, & US, A, 4168258 see page 7, tabel B, page 12, example 5; claims (cited in the application)	1-21
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X	US, A, 4171998 (M. BRAUER et al.) 23 October 1979, see column 8, tabel A; claims	1-21
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X	US, A, 4176239 (M. BRAUER et al.) 27 November 1979, see column 8, tabel A; claims	1-21
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X	US, E, 30321 (M. BRAUER et al.) 1 July 1980, see column 9, tabel A; claims (cited in the application)	1-21
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X	US, A, 4008197 (M. BRAUER et al.) 15 February 1977, see column 8, tabel A; claims	1-21
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X	US, A, 4029626 (G.W. SILLEMOT et al.) 14 June 1977, see column 5, lines 45-56; claims	1-21
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X	US, A, 4355130 (R.E. HEINZE) 19 October 1982, see column 4, lines 43-54; claims (cited in the application)	1-21
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<p><sup>10</sup> Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&amp;" document member of the same parent family</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
10th September 1985	10 OCT. 1985	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	 G.L.M. Kravdenberg	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	US, A, 4375521 (J.M. ARNOLD) 1 March 1983, see column 4, lines 33-50; claims  -----	1

## ANNEX TO THE INTERNATIONAL SEARCH REPORT ON

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 INTERNATIONAL APPLICATION NO.            PCT/US 85/00998 (SA        9771)  
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This Annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 02/10/85

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For more details about this annex :  
 see Official Journal of the European Patent Office, No. 12/82

INTERNATIONAL APPLICATION NO.  
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