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LIGHT EXTENDING PRODUCT AND PROCESS

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ABSTRACT OF THE DISCLOSURE

A light source which consists essentially of a plastic envelope filled with the chemiluminescent compound, tetrakis(dimethylamino)ethylene. At least one side of the envelope must be of an oxygen or water vapor permeable plastic so that activation of the chemiluminescent compound will take place when the envelope is exposed to air.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

The present invention relates to a light source based on chemiluminescent materials.

Various chemiluminescent compounds are well known such as the peraminoethylenes, but because most of them are liquid their utility as light sources has been very limited. Little work has been done on the control of intensity and duration of the luminescence of a chemiluminescent composition by its package or container. Most efforts have been directed toward the use of inert diluents or carriers. The present invention is for a new and useful extended light source which is regulated or controlled by the material from which the package or casing is made.

It is an object of this invention to provide a light source useful in night signaling.

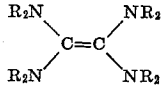
Another object is to provide an extended light source which has prolonged luminescence and durability.

Yet another object is to provide a light source which can be used as a search and rescue aid on land or sea.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same becomes better understood by reference to the following detailed description.

The invention essentially consists of a chemiluminescent material enclosed in an envelope having at least one side which has high oxygen permeability, some water permeability, and is resistant to the chemiluminescent material and its oxidation products.

The chemiluminescent composition used in this invention is a tetrakis(disubstituted)ethylene of the formula



whereinin the R's are the same or different and are monovalent alkyl or cycloalkyl groups of up to 10 carbon atoms, divalent alkylene joined to the other R attached to the same nitrogen to form a 3–5 membered monoaza heterocycle, and divalent alkylene joined to an R attached to a second nitrogen to form a 3–7 membered diaza heterocycle. The preferred member of the series of peraminoethylenes; namely, tetrakis(dimethylamino) ethylene (TMAE) was first prepared by the reaction of dimethylamine and chlorotrifluoroethylene as reported by Pruett et al., in the Journal of the American Chemical Society, 72, 3646 (1950). Other compounds of this series may be successfully used and include tetrakis(N-pyrrolidinyl)ethylene, 1,1',3,3' - tetramethyl - $\Delta^{2,2'}$ - bi(imidazolidine), and tetrakis(dimethylamino-methyleneamino) ethylene.

The envelope or casing must consist of a plastic material having at least one side of a predetermined thickness with a controlled permeability to the activating agents in air (water and oxygen). Those materials which were successfully used include vinylidene fluoride, cast vinyl chloride, polypropylene and a copolymer of polyfluoroethylene and polyfluoropropylene.

The following table gives the emission of light by peraminoethylene (chemiluminescent material) in accordance with the oxygen permeable plastics used. The intensity units shown below are recordations of a photocell response which were used to evaluate these materials.

TABLE I

Material	Thickness (mil)	Time (days)	Units
Vinylidene fluoride	3	17	4.5
Cast vinyl chloride	3.5	4	68.6
Do	3.5	9	78
Do	3.5	16	18
Cellulose	0.5	4–39	0
Polypropylene	2	4	94
Do	2	9	133
Do	2	16	317
Copolymer of polyfluoroethylene and polyfluoropropylene (Teflon FEP)	1	1	2,000
Do	1	24	230

A study was made of the oxygen permeability of a number of plastics to determine whether or not the above list (Table I) could be implemented. Tables II, III, and IV below show these results.

TABLE II.—PERMEABILITY OF PLASTIC FILMS

[from Plastics World, April 1961]

Material (Plastic Film)	Oxygen [1]	Water Vapor [2]
Cellulose Acetate	1,700–2,500	1.4–8.8
Polyamide	25	0.3–1.8
Polyester	<0.5	0.5–0.15
Polyethylene:		
Low density	1,900	1.2–1.4
High density	800–450	0.3–0.4
Polypropylene	[3]	0.06–0.7
Polytetrafluoroethylene	[3]	0.0
Polystyrene (oriented	310	4.4
Vinyl chloride (non-rigid)	150	8.0
Vinylidene chloride-vinyl chloride copolymer	2.4	0.01–0.03
Regenerated cellulose	<0.5	1.4–2.7

[1] Cc./100 sq. in.-24 hrs. 1 atm./mil.
[2] Gms./100 sq. in./24 hrs.
[3] No data.

TABLE III.—TYPICAL PERMEABILITY OF PLASTIC FILM

[Du Pont Data]

Material (du Pont plastic)	Oxygen [1]	Water Vapor [2]
Mylar (polyethylene terephthalate resin)	3	1.8
Tedlar (polyvinyl fluoride)	3.2	180
Cellulose acetate	117	219
Regenerated cellulose	[3] 0.3	134
Polyethylene	535	1.8
Teflon FEP (copolymer of polyfluoroethylene and polyfluoropropylene)	350	0.04
Nylon (long-chain polymeric amide)	1.0	8.1

[1] Cc./100 sq. in./24 hrs. atm./mil.
[2] Gms./100 sq. in./24 hrs.
[3] 100% R.H.

TABLE IV.—TYPICAL PERMEABILITY OF PLASTIC FILMS

[Dow Chemical data]

Material (Dow Chemical plastics)	Oxygen [1]	Water Vapor [2]
Saran Wrap (vinylidene chloride-vinyl chloride copolymer)	0.8–6.9	0.02
Polyethylene:		
High density	140	0.55
Medium density	210	0.75
Low density	255	0.95
Trycite (polystyrene)	210	7.0

[1] Cc./100 sq. in./24 hrs. atm./mil.
[2] Gms./100 sq. in-24 hrs./mil.

Polypropylene, a Union Carbide biaxially oriented film, has an oxygen permeability of 150 cc./100 sq. in. and water vapor permeability of 0.4 gram per 100 sq. in.

Kynar, a Pennsalt Chemical Company solution cast vinylidene fluoride, has an oxygen permeability of 24×10^{-12} cm.^{3} and water vapor permeability of 1 gm./24 hrs. m.^{2} atm.

The flexible plastic envelopes used to enclose the liquid peraminoethylenes were made of a variety of polymeric films. For example at least one side or portion of the envelope was selected from one of the high oxygen permeable plastic films disclosed in the tables hereinbefore. The copolymer of polyfluoroethylene and polyfluoropropylene (Teflon FEP, 1 mil thick), polypropylene and solution cast vinylidene fluoride (Pennsalt Chemical Company, Kynar) were most successful for the high oxygen permeable portion. The other side of the envelope (and the airtight storage container for these envelopes) was selected from one of the plastics which had very low or no oxygen and water vapor permeability. These plastics included regenerated cellulose, long-chain polymeric amide (nylon), polyethylene terephthalate resin (Mylar), and others backed by metal foil such as aluminum.

The chemiluminescent envelopes of this invention are illustrated in further detail in the following examples.

Example I

An open-end envelope or casing about 2 x 5 inches was prepared by sealing by appropriate means, such as heat or tape, three edges of two sheets of Teflon FEP. This plastic film is highly permeable to oxygen and water vapor. The envelope was flushed with nitrogen and then filled under inert atmospheric conditions by dropping from a pipette about 4 ml. of tetrakis(dimethylamino)ethylene into the envelope. The open end was then sealed with tape. Several of these were prepared and packed in an airtight glass test tube and sealed. The next day when the envelopes were removed, they glowed on both sides for several hours. They were replaced in the test tube out of the presence of air and resealed. Light emission diminished. Several weeks later when removed from the test tube the envelopes glowed with a yellow-blue light.

Example II

Another quantity of envelopes about 1½ x 4 inches were prepared by the same method described in Example I, except that one side of the envelope consisted of oxygen permeable cast vinylidene fluoride film and the other side was regenerated cellulose which has low oxygen and water vapor permeability. Three edges were sealed. The envelopes were flushed with nitrogen and filled under inert atmospheric conditions with about 3 ml. of tetrakis(dimethylamino)ethylene. This was done by dropping the liquid from a pipette into the open end of the envelope. The end was then sealed with tape and each sealed envelope was placed in an airtight container and capped. Several days later when several of the envelopes were withdrawn a yellow-blue light emitted from the vinylidene fluoride side of the envelope.

Example III

Another quantity of chemiluminescent envelopes about 2" x 3" were prepared in the same manner as described in Example I, except one sheet of polyethylene film (a Du Pont product about 1 mil thick and with an oxygen permeability of 350 cc./100 sq. in.) was sealed to vinylidene chloride-vinyl chloride copolymer (Saran wrap) which has low oxygen and water vapor permeability. These envelopes or casings were filled dropwise with about 2 ml. of tetrakis(dimethylamino)ethylene and packed into an airtight test tube and stoppered. When one of these envelopes was removed from the tube light soon emitted. When replaced in the tube the glow eventually ceased, but when exposed to air again it would emit a light.

The oxygen and water vapor permeability study indicates that plastic films having a thickness of from 1 to 3 mil may be used as a casing which controls the function and duration of luminescence of the chemiluminescent material enclosed therein.

In another embodiment of this invention the oxygen impermeable side of the envelope may be backed by a metal foil, such as aluminum, which increases the light emission from the other side.

Chemiluminescent materials in the dry form may be successfully used. The dry materials overcome some of the packaging difficulties encountered in filling the envelopes with the liquid peraminoethylenes.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed is:

1. An extended light source comprising tetrakis(dimethylamino)ethylene hermetically sealed in a plastic envelope at least one portion of which is oxygen and water vapor permeable polyethylene.

2. The light source of claim **1** in which one portion of the enclosing envelope is said polyethylene and the other portion is backed with metal foil.

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